Research Article

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Microstructure and compressive behavior of lamellar Al₂O₃p/Al composite prepared by freeze-drying and mechanical-pressure infiltration method

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Abstract: Infiltrated molten Al matrix by mechanical-pressure infiltration method into the ceramic scaffold prepared by freeze-drying technology could prepare dense lamellar Al matrix composites without damage of the biomimetic microstructure of the scaffold. However, the investigation of lamellar Al matrix composites prepared by freeze-drying and mechanical-pressure infiltration method has not been fully understood yet. In the present work, the Al₂O₃ scaffold with pearl layer structure was prepared by freezing-dry method, and eventually the lamellar Al₂O₃p/Al composite was fabricated by mechanical-pressure infiltration method. The Al matrix was infiltrated well into the large pores of the Al₂O₃ scaffold, and the lamellar structure of the Al₂O₃ was well preserved. The hardness of the lamellar Al₂O₃p/Al composite was isotropic in transvers and perpendicular directions. However, the compressive strengths of the lamellar Al₂O₃p/Al composite were significant anisotropic while the compressive strength in transvers direction was 127.7% higher than that in the perpendicular direction, indicating the integrality of the lamellae microstructure (especially the bridging layers). Due to the mismatched deformability, weak debonding was observed between Al and Al₂O₃p/Al layers in the fracture surface of the lamellar Al₂O₃p/Al composite. It indicates that the interfacial bonding between Al and Al₂O₃p/Al layers is rather strong, which is beneficial for higher strength in transvers direction but lead to lower strength in perpendicular direction.

Keywords: Metal matrix composites; Al matrix composite; lamellar microstructure; freeze-drying; compressive behavior.

1 Introduction

Al matrix composites reinforced with homogeneous distributed particles have been widely investigated due to their high specific stiffness [1], strength [2] and tailorable thermal-physical properties [3]. However, the strength enhancement in homogeneous composites is usually achieved at the expense of plasticity [4]. Recently, it has been found that inhomogeneous composites demonstrate good compatibility in strength and plasticity compared with the homogeneous distributed reinforcements [5]. Therefore, more attention in the past decades has been paid on the design, preparation and characterization of the inhomogeneous distributed microstructure in the composites.

Lamellar Al matrix composites, whose microstructure is bio-inspired by the nacre, have been considered as candidates for light-weight armor protection materials due to their high strength and toughness. Therefore, recently, the lamellar Al matrix composites have been extensively
investigated [6]. Several advanced methods, such as mechanical rolling [7], slip casting [8], centrifugal casting [9] and flake powder metallurgy [10], have been adopted to prepare the composites with layered microstructure. Li et al. [11, 12] found that the Al matrix nanolaminated composites reinforced with carbon nanotubes or graphene demonstrated improved strength and plasticity than that of the uniform distributed composites.

Freezing-dry method has been developed recently to prepare the ceramic-based bio-inspired layered structure, which could be further infiltrated with liquid matrix to obtain the composites with pearl-like layer structure. During the freezing-dry process, the ceramic particles could be dispersed in water to build sophisticated, nacre-like architectures with the layer thickness varying from 10 to 200 μm. The microstructure of the scaffold in micron-scale could be easily modified by controlling the freezing kinetics precisely. After infiltrated with selected second phase (organic or inorganic), the biomimetic microstructure of the scaffold could also be preserved, the hard and ductile phases could be alternately arranged and both strength and toughness of the composites could be enhanced. The Deville et al. [13] firstly reported the preparation of the biometric composites based on freezing-dry method including artificial bone, ceramic-metal composites, and porous scaffolds for osseous tissue regeneration with strengths up to four times higher than those of materials currently used for implantation. Currently, most of the lamellar Al matrix composites have been prepared by the pressure-less infiltration method to avoid the breakdown of the ceramic lamellae in the scaffolds with relative low strength. The Deville et al. [13] firstly reported the preparation of the lamellar Al₂O₃p/Al-12Si composites as well by freeze-casting and vacuum infiltration method, and found that the lamellar Al₂O₃p/Al-12Si composites demonstrated good strength-toughness properties. Shen et al. [14] deeply investigated the microstructure and mechanical properties of the lamellar SiCp/Al-Si-Mg composites prepared by freeze-casting and pressure-less infiltration method and found that the composites demonstrated weak orthotropic properties with maximum 722 MPa compressive strength in longitudinal direction. Shen et al. [15] further investigated the microstructure evolution and mechanical properties of the lamellar Al₂O₃p-ZrO₂/Al-Si-Mg composites prepared by freeze-casting and pressure-less infiltration method, and revealed the reaction between Al-12Si-10Mg matrix and ZrO₂ to form (Al₁₋₇m, Si₃m)₃Zr, Al₂O₃ and ZrSi₂ phases. Hautcoeur et al. [16] fabricated the lamellar ZrO₂-Al composites by freeze-casting and pressure-less infiltration method, and reported that the thermal conductivities of the composites were significantly anisotropic, which were 80 and 13 W/(m·K) parallel and perpendicular to the freezing direction, respectively.

Generally, utilization of pressure during infiltration and solidification of liquid metals is beneficial for the densification of the composites [17]. Shen et al. [18] utilized 2 MPa gas-pressure in vacuum atmosphere to infiltrate liquid pure Al into porous Al₂O₃–ZrO₂ scaffolds. The few pores and breakage of the Al₂O₃–ZrO₂ scaffolds were also found [18], and revealed that the Al penetration and subsequent interfacial reaction with ZrO₂ would be affected significantly by the porous structure of the ceramic lamellae. However, due to the low heating and cooling rate caused by the vacuum atmosphere, the preparation process of the gas-pressure infiltration was rather time and energy consuming. Moreover, it is difficult to control the reaction between the scaffolds and Al matrix due to the long contact time [18]. Furthermore, due to the technique restriction, it is very difficult to apply higher gas-pressure (more than 5 MPa), which is unfavorable for the infiltration and densification process. Liu et al. [19] explored the preparation of the lamellar SiCp/2024Al composites by freeze-casting and pressure infiltration method with infiltration pressure of 80 MPa by mechanical method. The composites demonstrated lamellar microstructure composed of alternating ceramic and metallic lamellar and high bending strength (931 MPa) and roughness (18.8 MPa·m⁻¹²). However, the breakdown of the bridges between ceramic lamellae in the scaffolds was also found in the produced composites.

Therefore, compared with other methods, the mechanical-pressure infiltration method has the advantages in flexibility of the Al matrix, low requirement for equipment and good preservation in lamellar microstructure of the scaffold. However, the investigation of lamellar Al matrix composites prepared by freeze-drying and mechanical-pressure infiltration method has not been fully understood yet. In the present work, the Al₂O₃ scaffold with pearl layer structure was prepared by freeze-drying method, and eventually the lamellar Al₂O₃p/Al composite was fabricated by mechanical-pressure infiltration method. The microstructure and the compressive behavior of the lamellar Al₂O₃p/Al composite have also been investigated.

2 Materials and methods

Al₂O₃ particles with average size of 400 nm were provided by Dalian Luming nano-materials Co., Ltd. China. Al alloy was used as matrix alloy in the present work. The chemical composition of the Al alloy was 2.6wt.% Mg, 1.25wt.% Si,
1.1 wt.% Fe and Al balance. C. Garcia-Cordovilla et al. [20] and Yoshida et al. [21] reported that a small amount of Mg in Al alloy could decrease the surface energy significantly, while the surface energy of Al-Mg alloys was decreased from 0.99 N/m in pure Al to 0.73 N/m in Al-9.1Mg. Therefore, the Al alloy with high Mg content was chosen as the matrix to promote the infiltration of the Al matrix [22, 23]. Based on our previous work [24], the porous Al$_2$O$_3$ scaffold was initially prepared by the freezing-dry method (Figure 1a). An aqueous suspension at a solids loading of 20 vol% was prepared by mixing Al$_2$O$_3$ powder, Darvan 7-N (dispersant, 1.2 wt% of the Al$_2$O$_3$ powder, R.T. Vanderbilt Co., Connecticut, US), and glycerol (10 wt% of the solvent) into distilled water. The mixture was then milled for 24 h with zirconia balls (6 mm, the mass ratio of balls and powder is 3:1) as the ball-milled media at a rotation speed of 120 rpm. The resulting suspension was poured into a polyethylene mold (40 mm diameter, 15 mm long) and placed on refrigerated equipment (−20°C) to induce the unidirectional solidification of the slurry from the bottom to the top. As the freezing process was completed (~12 min), the frozen sample was freeze-dried at −40°C and <10 Pa (Freeze Dryer, Beijing SongYuan Huaxing Technology Development Co. Ltd., China), for 48 h to remove the solvent. The green part was sintered in air for 4 h at 1500°C at a constant rate of 5°C/min, followed by naturally cooling in furnace to room temperature to prepare the Al$_2$O$_3$ scaffold. The porosity of the prepared Al$_2$O$_3$ scaffold was about 64.3%.

Later on, the lamellar Al$_2$O$_3$/p/Al composite was prepared by mechanical-pressure infiltration method. The porous Al$_2$O$_3$ scaffold was put into a steel mold and then preheated at 500°C for 2 h, while the Al matrix alloy was melted at 900°C. Considering the densification effect and preservation of the integrity of the lamellae microstructure of the scaffolds, a pressure of 5 MPa was applied and maintained for 10 min during the infiltration process, followed by the solidification of the composites in air within 10 min (Figure 1b). Before microstructure observation and compressive tests, annealing treatment of all the samples were performed at 415°C for 1 h and eventually cooled in air.

The morphologies of the porous Al$_2$O$_3$ scaffold and the lamellar Al$_2$O$_3$/p/Al composite were observed by the Axiosvert 40 MAT optical microscope (Carl Zeiss, Germany) and FEI Sirion Quanta 200 (FEI Co. Ltd., Philips, Netherlands) scanning electron microscope (SEM). X-ray computed tomography (CT) was performed on the nanotom® (Phoenix x-ray, Wunstorf, Germany) to reveal the 3D-distribution of the Al$_2$O$_3$ phase. The phase composition of the lamellar Al$_2$O$_3$/p/Al composite were analyzed by the X-ray diffraction (XRD), which were performed on the Rigaku D/max-rB diffractometer with Cu-Kα radiation (0.15418 nm) and the scanning speed was set at 2°/min.

The density of the lamellar Al$_2$O$_3$/p/Al composite samples (10×10×2 mm) was measured using Archimedes principle according to ASTM B311-17, and four samples have been tested to improve the statistical significance of the results. The hardness tests were carried out on HBS210-3000 Brinell hardness tester according to ASTM E10-07a. The dimensions of hardness test samples were 15×15×4 mm. During the hardness test, 187.5 kgf load was applied for 30 s, and five samples have been tested to improve the statistical significance of the results. Compressive tests were performed on Instron 5569 universal electrical testing machine (Instron Co., USA) with a speed of 0.1 mm/min according to ASTM E9-89a. The dimensions of the compressive test samples were 4×4×5 mm. The compressive tests were performed on the transverse direction (parallel to the Al$_2$O$_3$ layers) and perpendicular direction (vertical to the Al$_2$O$_3$ layers) to reveal the effect of the distribution of the Al$_2$O$_3$ phase. In order to observe the fracture behavior of the Al layers and the Al$_2$O$_3$/p/Al layers, the bending fracture surface of the lamellar Al$_2$O$_3$/p/Al composite were analyzed by FEI Sirion Quanta 200 SEM.

3 Results and discussion

Representative microstructure of the porous Al$_2$O$_3$ scaffold prepared by the freezing-dry method is shown in Figure 2. The Al$_2$O$_3$ scaffold demonstrated aligned lamellar porous structure, which was parallel to the growth direction of the ice (Figure 2a). Moreover, the scaffold was constituted by the Al$_2$O$_3$ layer and interlayer (Figure 2b),
representative microstructure of the porous Al$_2$O$_3$ scaffold prepared by the freezing-dry method. (a) Low magnification image, (b) Microstructure of the layer and interlayer, (c) Microstructure of the interface and the bridges, (d) Porous microstructure in Al$_2$O$_3$ interlayer.

whose thickness was about 20 and 8 µm, respectively. The paralleled Al$_2$O$_3$ layer-interlayer and interlayer-interlayer were connected by the thin Al$_2$O$_3$ bridges (Figure 2c). Moreover, the Al$_2$O$_3$ layer and interlayer were also porous, which were formed by the individual Al$_2$O$_3$ particles (Figure 2d).

X-ray CT characterization of the lamellar Al$_2$O$_3$/Al composite is shown in Figure 3. The degree of X-ray attenuation depends on both the density and the atomic number of the material composing the samples, and higher density and higher atomic numbers result in higher attenuation of X-rays, which then show brighter colors in the processed image [25]. The samples were composed by the lamellar Al$_2$O$_3$/p/Al composite (marked by green braces) and the Al matrix (marked by yellow braces), as shown in Figure 3b and 3c. Moreover, the samples were composed of four components, which could be distinguished by four colors. The lamellar Al$_2$O$_3$/p/Al composite was mainly composed of dark gray, light grey and bright regions, while the Al matrix was mainly composed of light grey and black regions. The light grey phase was Al matrix, while the dark grey region was assigned to the Al$_2$O$_3$ layers infiltrated with Al matrix. The black regions, which were further confirmed to be graphite impurities, were mainly found in Al matrix, and were not observed in the composites. The bright phase was confirmed by EDS analysis to be the Fe-rich phase. It is clear that the lamellar Al$_2$O$_3$/p/Al composite demonstrated well lamellar structure of the Al$_2$O$_3$.

The representative microstructure of the lamellar Al$_2$O$_3$/p/Al composite has been shown in Figure 4. The bright grey and dark grey areas in Figure 3 were Al and Al$_2$O$_3$/Al, respectively, which revealed the layered struc-
Figure 3: X-ray CT characterization of the lamellar $\text{Al}_2\text{O}_3/p/\text{Al}$ composite. (a) 3D-image, (b)(c) Cross-section images observed perpendicular to the freezing direction, (d) Cross-section images observed parallel to the freezing direction.

Figure 4: Representative microstructure of the lamellar $\text{Al}_2\text{O}_3/p/\text{Al}$ composite. (a) Low magnification image, (b) High magnification image.

ture of the composites. Meanwhile, several individual bright phases with irregular sharp were also found in the composite (Figure 4a). Further EDS analysis indicated that the bright phases were rich with Fe elements, which might be introduced into the composites during infiltration process as impurities. Higher magnification observation indicated that the Al matrix has been infiltrated well into the large pores of the $\text{Al}_2\text{O}_3$ scaffold (Figure 4b). However, the $\text{Al}_2\text{O}_3$ layers, interlayers and bridges have not been fully infiltrated, which has been marked out by the red
Figure 5: XRD analysis of the lamellar Al₂O₃p/Al composite.

Figure 6: Representative compressive curves of the lamellar Al₂O₃p/Al composite in transvers and perpendicular directions and Al matrix.

circles in Figure 4b. Correspondingly, the measured relative density of the prepared lamellar Al₂O₃p/Al composite was about 92.3%. Since the porosity of the prepared Al₂O₃ scaffold was about 64.3%, the real weight percentage of the Al₂O₃ was about 48.2%. Pressure infiltration method has been widely used to infiltrate the Al matrix into the ceramic scaffolds with uniform pore structure [26]. However, high infiltration temperature and pressure, which could overcome the poor wettability and high infiltration resistance, is usually used to achieve full densification [27]. In the present work, in order to avoid the destruction of the lamellar porous structure in Al₂O₃ scaffold, relative low infiltration pressure (5 MPa), which was lower than in other system (usually higher than 10 MPa) [28, 29], was applied. It was mainly due to the relative low compressive strength of the Al₂O₃ scaffold (measured to be about 15 MPa at 500°C). However, the micro-scale pores between Al₂O₃ interlayer were fully filled with the Al while the submicron-scale pores within the Al₂O₃ interlayer were rarely infiltrated. Therefore, the infiltration parameters (such as temperature and pressure) should be further optimized for the fully densification of the composites.

XRD analysis of the lamellar Al₂O₃p/Al composite has been shown in Figure 5. Only the diffraction peaks of the Al, Al₂O₃ and very small content Mg₂Si phases were found, while the Mg₂Si phase is the common strengthening precipitates in Al-Mg-Si matrix composites [30, 31]. However, no significant diffraction peaks of the Fe-rich phases were detected, indicating the content of the Fe-rich phases was rather low. It should be noted that the Al₂O₃ could react with Mg element to form MgAl₂O₄, which has been widely reported in Al₂O₃/Al composites prepared by pressureless infiltration method [32, 33]. However, no significant presence of the MgAl₂O₄ phase has been found in the present work. It might be due to the short preparation time (about 10 min) used in the present work. Usually, the chemical reaction is strongly related to the atmosphere temperature and contact time. Since the diffusion rate of Mg element in solid Al is much lower than that in molten Al [34], the reaction to form MgAl₂O₄ phase is mainly occurred at the interface between Al₂O₃ and molten Al-Mg matrix. However, due to the short contact time in the present work, the reaction should be very weak and lower than the XRD detection accuracy, which agrees well with the interfacial microstructure observation results in Al₂O₃ particles reinforced 6061Al and 2024Al composites prepared under similar parameters [35, 36].

The hardness of the lamellar Al₂O₃p/Al composite in the transvers (parallel to the growth direction of the ice) and perpendicular (vertical to the growth direction of the ice) directions were 212.1±5.5 and 209.7±7.2 HB, while the hardness of the Al matrix was 84.4±4.7HB, respectively. The hardness of the lamellar Al₂O₃p/Al composite increased about 150% than that of the Al matrix. However, it is difficult to reveal the anisotropy characters of the lamellar Al₂O₃p/Al composite since the hardness values in different directions were very close. The representative compressive curves of the lamellar Al₂O₃p/Al composite in transvers and perpendicular directions have been shown in Figure 6. For comparison, the compressive curve of the Al matrix was also shown in Figure 6. Regardless of the test direction, it is clear that the compressive strengths of the lamellar Al₂O₃p/Al composite were higher than that of the Al matrix alloy. The compressive strengths of the lamellar Al₂O₃p/Al composite in transvers and perpendicular directions were 748.2±17.3 and 328.6±11.7 MPa, which increased 234.5% and 46.9% than that of the Al matrix.
Microstructure and compressive behavior of lamellar Al$_2$O$_3$/Al composite

Figure 7: SEM observation of the fracture surface of the lamellar Al$_2$O$_3$/Al composite. (a) Low magnification image, (b) High magnification of the selected area in (a).

alloy (223.7±5.8 MPa), respectively. Meanwhile, the fracture behavior was also changed from plastic to brittle fracture character after the introduction of the Al$_2$O$_3$ scaffold since the compressive stress increased linearly to maximum before failure. Moreover, the compressive curve of the lamellar Al$_2$O$_3$/Al composite in perpendicular direction demonstrated ladder-like character, which is an important characteristic in lamellar composites and corresponds to the crack deflection along the Al$_2$O$_3$ layer or interlayer [31]. However, the compressive curve of the lamellar Al$_2$O$_3$/Al composite in transvers direction demonstrated linear character without ladder-like deflection or deformation. It was mainly due to the holistic strengthening effect of the Al$_2$O$_3$ scaffold attributed to the connection and bridging of the Al$_2$O$_3$ layer or interlayer.

Moreover, the compressive strength of the lamellar Al$_2$O$_3$/Al composite in transvers direction (748.2 MPa) was 127.7% higher than that in the perpendicular direction (328.6 MPa). Therefore, the lamellar Al$_2$O$_3$/Al composite demonstrated significant compressive anisotropic behavior. Shen et al. [14] reported that the anisotropic compressive behavior of the lamellar SiCp/Al composites prepared by freeze casting and pressure-less infiltration. The compressive strength of the composites loaded along the longitudinal direction was about 100–200MPa higher than the perpendicular direction [18]. However, Liu et al. [19] reported that the mechanical properties of the lamellar SiC/2024Al composites prepared by freeze casting and pressure infiltration method (infiltrated at 80 MPa) were quasi-isotropy. The values of the flexural strength, fracture toughness and the hardness of the composites were very close in the longitudinal and perpendicular directions, which were mainly related to the breakdown of the bridges between ceramic lamellae in the scaffolds [19].

Gas-pressure infiltration expands the option list of the Al matrix. However, due to the low heating and cooling rate caused by the vacuum atmosphere, the preparation process of the gas-pressure infiltration was rather time and energy consuming, and it is difficult to control the reaction between the scaffolds and Al matrix due to the long contact time [18]. Furthermore, due
to the technique restriction, it is very difficult to apply higher gas-pressure (more than 5 MPa), which is unfavorable for the infiltration and densification process. The high mechanical-pressure infiltration method (more than 80 MPa) has the advantages in densification effect [19]. However, the breakdown of the bridges between ceramic lamellae in the scaffolds was also found in the produced composites due to low strength of the scaffolds. Compared with other methods, the mechanical-pressure infiltration method has the advantages in flexibility of the Al matrix, low requirement for equipment and good preservation in lamellar microstructure of the scaffold. However, the infiltration parameters should be controlled carefully.

The fracture surface of the lamellar Al\textsubscript{2}O\textsubscript{3}/p/Al composite has been shown in Figure 7. The lamellar Al\textsubscript{2}O\textsubscript{3}/p/Al composite demonstrated significant layered-like fracture behavior. The fracture of the Al area was mainly characterized by the larger tearing edges (as pointed out by blue circle in Figure 7b). Moreover, small tearing edges of Al around Al\textsubscript{2}O\textsubscript{3} particles (as pointed out by yellow circle in Figure 7b), which were the representative phenomena in particles reinforced Al matrix composites [38], were found in the fracture of the Al\textsubscript{2}O\textsubscript{3}/p/Al area. Furthermore, due to the mismatched deformability, weak debonding was observed between Al and Al\textsubscript{2}O\textsubscript{3}/p/Al layers. During the loading process, both the Al and Al\textsubscript{2}O\textsubscript{3}/p/Al layers participated in the strengthening effect. The weak debonding behavior implied that the interfacial bonding between Al and Al\textsubscript{2}O\textsubscript{3}/p/Al layers was rather strong, which is beneficial for the strengthening effect of Al\textsubscript{2}O\textsubscript{3}/p/Al composite scaffold and leads to the high compressive strength in transvers direction (about 748.2 MPa). However, the strong interfacial bonding between Al and Al\textsubscript{2}O\textsubscript{3}/p/Al layers is unfavorable for the crack deflection, which is a main strengthening and toughening mechanism in lamellar composites. The compressive strengths of the lamellar Al\textsubscript{2}O\textsubscript{3}/p/Al composite in perpendicular direction were rather low (about 328.6 MPa). Therefore, the interfacial bonding properties should be further optimized to improve the mechanical properties in different directions.

### 4 Conclusions

In the present work, lamellar Al\textsubscript{2}O\textsubscript{3}/p/Al composite has been prepared by freezing-dry and mechanical-pressure infiltration method. After the infiltration of Al matrix, the lamellar Al\textsubscript{2}O\textsubscript{3}/p/Al composite also demonstrated well lamellar structure of the Al\textsubscript{2}O\textsubscript{3}. The Al matrix has been infiltrated well into the large pores of the Al\textsubscript{2}O\textsubscript{3} scaffold, while the Al\textsubscript{2}O\textsubscript{3} layers, interlayers and bridges have not been fully infiltrated. The hardness of the lamellar Al\textsubscript{2}O\textsubscript{3}/p/Al composite was isotropic in transvers and perpendicular directions. However, the compressive strength of the lamellar Al\textsubscript{2}O\textsubscript{3}/p/Al composite in transvers direction was 748.2±17.3 MPa. It should be noted that the lamellar Al\textsubscript{2}O\textsubscript{3}/p/Al composite demonstrated significant compressive anisotropic behavior, while the compressive strength in transvers direction was 127.7% higher than that in the perpendicular direction. The anisotropic characters in the compressive properties represent the integrality of the lamellae microstructure (especially the bridging layers), which also implies that the anisotropic microstructure of the scaffold has been well preserved in the present work. The fracture surface of the lamellar Al\textsubscript{2}O\textsubscript{3}/p/Al composite demonstrated significant layered-like fracture behavior. It indicates that the interfacial bonding between Al and Al\textsubscript{2}O\textsubscript{3}/p/Al layers is rather strong, which is beneficial for higher strength in transvers direction but leads to lower strength in perpendicular direction.

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