High Strength Particulate Aluminum Matrix Composite Design: Synergistic Strengthening Strategy

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Abstract

Aluminum-alumina (Al-Al_2O_3) composites with ultrahigh strength were designed by combining dispersion strengthening via the addition of Al_2O_3 particles and matrix strengthening via *in situ* strain hardening. Cold spraying additive manufacturing technology was applied to fabricate the composites. The yield strength ($\sigma_{0.2\%}$) of the Al-46 wt.% Al_2O_3 composite was 317.4 MPa and was among the highest value reported in the literature, to the best of our knowledge, for a pure Al-based Al-Al_2O_3 composite. Experimental evaluation of the material showed that the compressive failure mode of the composite changed from gradual plastic collapse to shear-dominated behavior as the percentage of Al_2O_3 was increased or the matrix was pre-strain strengthened in the Al-Al_2O_3 composite.

Keywords: Metal–matrix composites (MMCs); Mechanical properties; Particle-reinforcement; Synchronous strengthening.

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1. Introduction

Aluminum matrix composites (AMCs) have attracted much attention during the past decade since they exhibit lower density, high specific strength/stiffness, good wear and corrosion resistance, and show economic viability [1-3]. A wide range of reinforcement particulates such as SiC, Al_2O_3, TiC, TiB_2, and B_4C have been used to date in AMCs [1]. Among all these particle-reinforced AMCs, Al-Al_2O_3 composites have shown better thermal stability and oxidation resistance at high temperatures, since undesirable phases (e.g., Al_4C_3, Al_3Ti, AlB_2 precipitates [4-6]) are not produced in such materials [7]. However, the mechanical properties (e.g., hardness, yield and ultimate strengths) of most Al-Al_2O_3 composites remain lower compared to that of other structural materials (such as Ti alloys [8] and steels [9]) when fabricated through traditional metallurgical methods such as casting [10] and powder metallurgy [11]. It becomes even more challenging for AMCs to satisfy the stringent service criteria for functional components (e.g., hydraulic manifold, fan exit guide vane, engine blocks [1, 12]) that are used in the aerospace and automotive sectors in light of continued increase in demand for performance.

The challenges for commercial application of Al-Al_2O_3 composites as a structural material comes from both material fabrication and strengthening mechanisms. Some undesirable defects (e.g., voids because of different shrinkage and poor wettability of Al_2O_3 in the Al matrix [13, 14]) can be introduced inevitably during metallurgical processing. In addition, Al_2O_3 particulates tend to agglomerate in the Al matrix (e.g., sinking behavior of Al_2O_3 in the melt [15]) due to the different particle-to-liquid densities between the constituents, leading to a non-homogenous particle distribution and a further deterioration of the wettability [15]. In addition to these manufacturing challenges, the strengthening effects from Al_2O_3 particles may still be limited due to microscopic inhomogeneous deformation in the composite that manifests during mechanical loading, which has its origin from the large modulus difference between the matrix and

reinforcing particles [16]. To date, some efforts have been made to reduce the difference in the particle-matrix modulus in particulate AMCs by subsequent mechanical processing, including compressing [17], rolling [18], extrusion [19], and severe plastic deformation methods [20, 21], so as to improve the overall strength of the composite. However, these particulate AMCs commonly contain a relatively low level of particulate reinforcement content (commonly less than 10 vol.% [20-22]), and thus the strengthening effect is theoretically [16] and experimentally [17, 18, 20, 21] limited. In short, the competition between the matrix self-strengthening and the particle dispersion strengthening results in a trade-off for AMC design: if we pursue matrix strengthening, dispersion strengthening has to be sacrificed because of the reduced number of particles. Whereas, if we pursue dispersion strengthening, matrix strengthening will be limited due to lack of subsequent mechanical processing after the synthesis of the composite precursor.

In this Letter, we introduce an additive manufacturing method to fabricate advanced high-strength AMCs based on a synchronous strengthening design strategy, whereby matrix strengthening, dispersion strengthening, and interactional strengthening were achieved simultaneously. This approach was explored in pure Al-based Al-Al_2O_3 composites.

2. Experimental section

2.1. Materials preparation

Our processing strategy consisted of four steps: gas atomization, sieving, mixing, and cold spraying, as illustrated in Fig. 1a. Specifically, spheroidal pure Al (99.0 %) powders (CenterLine, Ltd., Windsor, ON, Canada), shown in Fig. 1c, were produced by the gas atomization method, and pure alumina (α-Al_2O_3, 99.5 %) (Oerlikon Metco Inc., Westbury, NY, USA) were fused and crushed into powders with an angular morphology, shown in Fig. 1d. The sieving processes were carried out to obtain powders with size

distributions of 40 to 60 μm for Al and 30 to 45 μm for Al_2O_3. Sieved Al and Al_2O_3 powders were then mixed at varying weight percentages (0, 60, and 90 wt.% of Al_2O_3 with the balance, pure Al powder). Powder mixing was performed in a rotated cylinder of 20 mm diameter at 20 RPM for 30 min. Shown in Fig. 1b is the schematic of the cold spray system setup. The deposition of the Al-Al_2O_3 composites was performed using a low-pressure cold spray system (SST series P, CenterLine, Ltd., Windsor, ON, Canada) equipped with a volumetric powder feeder (5MPE, Oerlikon Metco, Westbury, NY, USA). The air temperature was set to 375°C and the pressure was 90 psig, which has been experimentally proved to be a preferred setting for Al(-Al_2O_3) deposition [23, 24]. The cold-spray nozzle was attached to a robot (Motoman HP-20, Yaskawa Electric Corp., Waukegan, IL, USA) to allow for control and repeatability of the deposition. The nozzle traverse speed was set to 15 mm/s for deposition of all the powder feedstock compositions, and the deposition process was repeated 5 times (layer by layer) to fabricate the coating. It should be noted that in order to improve the reproducibility and to develop composites with definite weight fraction of reinforcements, controlling the powder size (distribution) and optimizing the spraying parameters are of great importance [24, 25].

2.2. Test and characterization

The specimens used in the compression tests were approximately 2.3 mm × 2.7 mm × 3.5 mm in size, and were cut using wire electrical discharge machining of the AMC deposit (Fig. 1e and f). Tests were performed parallel to the 3.5 mm direction. Some of the specimens were annealed at 170°C/4 h, 300°C/1 h, or 500°C/30 min in an Ar environment before conducting the compression tests. Experiments on annealed samples were performed to unravel the effects of matrix condition on strengthening and failure mechanism in the composites. Room temperature uniaxial compression tests were conducted at a constant strain rate of 1×10^{-3} s^{-1} to a maximum displacement of 1 mm using an Instron 3365 testing machine equipped with a

Promon U750 high speed camera and a VIC 900170WOF LED laser light guide for illumination. The loading platens made from M2-graded high-speed steel (HSS) with a diameter of 1 in. were used to compress the specimen. Extreme pressure grease covered the surfaces of HSS platens as lubricant to eliminate the frictional effect. The compression tests follow the ASTM C1424-15 standard [26]. Each test was performed at room temperature and was repeated for 4 times. Before testing, an airbrush with a 0.15 mm diameter nozzle was used to produce a fine speckle pattern (i.e., 5 to 10 pixels per speckle) on the specimen surface for digital image correlation (DIC) measurements. DIC analysis was performed using VIC-2D (v6 2018) software (Correlated Solutions Irmo, SC, USA). A field-emission SEM operated at 20 kV (Zeiss Sigma, Oberkochen, Baden-Württemberg, Germany) equipped with energy dispersion spectroscopy (EDS) and EBSD capabilities was used to characterize the microstructure of the composites before conducting experiments. The porosities of the samples were calculated from SEM images using a graphical analysis software (ImagePro, Media Cybernetics, Inc., Rockville, Maryland, USA). According to the EDS analysis, it was found that for the powder blends with 60 and 90 wt.% Al_2O_3, there was 34 ± 2.56 wt.% and 46 ± 2.04 wt.% Al_2O_3 in the deposited composites, respectively.

3. Results and discussion

3.1. Mechanical properties of the pure Al and Al-Al_2O_3 composites

Figure 2a shows typical compressive engineering stress-strain curves of the as-sprayed pure Al and Al-Al_2O_3 samples at room temperature. Note that each mechanical test was repeated, and the results were consistent and repeatable. From Figure 2a, the yield strength at 0.2% strain offset increased with increasing Al_2O_3 content in the samples from approximately 102.4 MPa for pure Al to 317.3 MPa for Al-46 wt.% Al_2O_3. There was a transition of the work hardening rate from positive to negative (see Fig. 2b). To the best

of our knowledge, the present particulate-based Al-Al_2O_3 composites exhibit the highest yield strength among all pure Al-based Al-Al_2O_3 composites prepared by various methods [10, 11, 15, 21, 27-29], as summarized in Fig. 2c.

It is our hypothesis that the combined strengthening from both the matrix self-strengthening and dispersion strengthening led to the ultra-high strength in the present composites, and the following annealing experiments helped to support the hypothesis (Fig. 2d-f). Specifically, the magnitude of the flow stress decreased for both the pure Al (Fig. 2d) and Al-46 wt.% Al_2O_3 (Fig. 2e) samples after annealing at high temperatures. Compared to the unreinforced annealed Al, the yield strength of the as sprayed Al-46 wt.% Al_2O_3 composite increased more than 8-fold (i.e., from 38.9 MPa to 317.3 MPa as shown in Fig. 2f).

3.2. Microstructures and strengthening mechanisms in pure Al and Al-Al_2O_3

An image analysis software program (ImagePro, Media Cybernetics, Bethesda, MD, USA), coupled with the SEM micrographs, was used to estimate the porosity in the coatings. The porosities were found to be 2.84 ± 0.31 vol.% in pure Al, 0.23 ± 0.04 vol.% in Al-34 wt.% Al_2O_3, and 0.17 ± 0.03 vol.% in Al-46 wt.% Al_2O_3. Figure 3a shows the typical microstructure features of the Al-46 wt.% Al_2O_3 composite. The porosity is low in this material because of the strong Al-Al particle bonding, resulting from the asperities on the deposited layer caused by incoming cold-sprayed Al_2O_3 particles [30]. For the Al matrix, major lattice distortion was found in the as-sprayed grains, considering the continuous change of crystal orientation as displayed in the EBSD map shown in Fig. 3b. In addition, some ultrafine grains or sub-grains with sizes of about 1 μm or less were distributed in local regions (Fig. 3d), similar to that which was observed by Dewar, *et al*. [23]. In contrast, however, the grain size was larger (approximately 7 μm on average) in the annealed Al matrix (Fig. 3c), and the lattice distortion is much lower in the grains (Fig. 3e).

According to Bourgeois [16], the composite yield stress, σ_y, and the *in situ* matrix yield stress, σ_y^m, is described as

$$\sigma_y = \frac{(1-V_p)\left[G^m + \beta_m\left(G^p - G^m\right)\right] + V_p G^p}{G^m + \beta_m\left(G^p - G^m\right)}\sigma_y{}^m = \left[1 + \frac{(1-\beta_m)\left(G^p - G^m\right)V_p}{G^m + \beta_m\left(G^p - G^m\right)}\right]\sigma_y{}^m, \quad (1)$$

where V_p denotes the volume fraction of particles, G^m/G^p represents the shear modulus of the matrix/particle, and β_m is the deviatoric component of the Eshelby tensor. According to Eq. 1 (the middle term), a higher matrix strength and a smaller difference between G^m and G^p will correspond to a higher yield stress of the composite. Since Al_2O_3 is a rigid particle with a G^p of 88 to 165 GPa [31] and Al is a light metal with a low G^m of approximately 25 GPa [32], strengthening the Al matrix can reduce the modulus difference and further improve the overall composite strength. In this work, the shear modulus of as-sprayed Al was 50.4 ± 5.2 GPa, compared to 22.1 ± 2.3 GPa for the annealed state (Table 1). The yield strength of the pure Al increased from 38.9 MPa (500°C annealed) to 102.4 MPa (as-sprayed), as shown in Fig. 2d. Beyond the modulus difference, Eq. 1 (far right-hand term) emphasizes the contribution of particle content (i.e., V_p) on the strength of the composite: increasing the number of particles will increase the strength. Our experimental results showed that the yield strength was improved from 38.9 MPa to 168.8 MPa by adding Al_2O_3 particles from 0 to 46 wt.% in an annealed Al matrix (Fig. 2d and 2e). Other experimentalists have observed similar trends for other material systems [33]. This study, however, explores combining the influence from matrix strengthening (σ_y^m), dispersion strengthening (V_p), and interactional strengthening ($G^p - G^m$) to show that the final yield strength ($\sigma_{0.2\%}$) of Al- Al_2O_3 composites increased from 38.9 MPa to 317.4 MPa (Fig. 2f), and are among the highest values reported in the literature for Al-Al_2O_3 (Fig. 2c).

3.3. Deformation and failure modes in pure Al and Al-Al_2O_3

Videos 1-3 in the supplementary data show the spatial distribution of the axial strain evolution of the

as-sprayed pure Al, Al-46 wt.% Al_2O_3, and 500°C annealed Al-46 wt.% Al_2O_3, respectively, according to the *in situ* DIC analysis. Pure Al, regardless of annealing, always exhibits a typical ductile deformation feature. Specifically, strain first concentrates in the centre of the specimen and then extends to the surrounding regions, leading to a temporary uniform deformation. Eventually, the plastic instability occurs at the edges of sample (see Video 1; left in Fig. 4a). In comparison, a deformation band forms directly after yielding occurs in the as-sprayed Al-46 wt.% Al_2O_3 (Video 2, middle image in Fig. 4a), indicating a shear-band-mediated fracture (i.e., the main fracture developed progressively by the initiation, growth, and coalescence of ductile microcracks), commonly observed in other high-strength materials [34, 35]. However, when the Al-46 wt.% Al_2O_3 composite material is annealed (Video 3, right-hand image in Fig. 4a), thereby recrystallizing the matrix (Fig 3c, e), the failure is transformed to an intermediate state: gradual plastic instability accompanied by multiple shearing.

For the pure Al, the normal stress is believed to cause its plastic collapse (or instability). Normal stress causes uniform strain in the entire sample at first, and the edges begin to deform with further loading due to loss of structural symmetry (Video 1, left-hand image in Fig. 4b). In contrast, it is the shear stress that caused failure in the deposited Al-46 wt.% Al_2O_3 composite. Shear strain appears to concentrate in a narrow band (i.e., macroscopic shear band [35, 36]) with an angle of approximately 45° to the loading direction, leading to shear failure (see Video 2, middle image in Fig. 4b). A similar phenomenon was observed in an Al-Ti composite [3]. For quasi ductile-brittle materials like the annealed Al-46 wt.% Al_2O_3, both the normal stress and the shear stress contributed to failure (i.e., normal stress caused uniform strain and hardened the entire sample at first, then shear stress caused shear banding in the hardened material). The strain concentrated at the centre and at the shear bands (Video 3, right-hand image in Fig. 4b). In short, the propensity of shear failure increased with increasing Al_2O_3 content and matrix strength in the composite

under compression. Shear stress gradually dominated the initiation and growth of damage.

4. Conclusion

In summary, the present work proposes a feasible advanced manufacturing approach to realize high strength in metal-ceramic composites during *in situ* synthesis. The microstructural strategy for developing high-strength AMCs proposed in this study was the combination of a well strain-strengthened matrix with an abundant amount of uniformly distributed reinforcing particles. A preparative route based on the cold-spraying technique was then designed according to this strategy, and particulate Al-Al_2O_3 composites with high yield strength and minimum porosity were successfully fabricated. The compressive damage and fracture of the present Al-Al_2O_3 composites was observed to be mainly controlled by the shear stress, an important observation in future materials design and simulations.

Acknowledgements

Financial and technical support for this project was received from InnoTech Alberta (Dr. Gary Fisher), the Major Innovation Fund (MIF) Program of the Province of Alberta – Ministry of Jobs, Economy, and Innovation, Imperial Oil, and the Natural Science and Engineering Research Council of Canada.

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Table caption

Table 1. Material properties of sprayed pure Al before and after annealing.

Figure captions

Figure 1. Preparation of Al-Al_2O_3 composites. (a) Schematic illustration of the fabrication process for high strength Al-Al_2O_3 composites. (b) Schematic of cold spray system experimental setup. (c,d) SEM images showing the morphology and shape of sieved Al (c) and Al_2O_3 (d) powders. (e) An example of Al-Al_2O_3 composite fabricated by cold spraying. (f) Typical specimens for compression tests.

Figure 2. Mechanical properties of Al and Al-Al_2O_3 composites. (a) Room-temperature engineering stress–strain curves under uniaxial compression. (b) Strain hardening rate versus true strain curves corresponding to curves in 'a'. (c) Comparison between the present materials and other Al-Al_2O_3 composites for the yield strength versus Al_2O_3 content. (d,e) Comparison of compressive curves for pure Al (d) and Al-46% Al_2O_3 (e) with and without annealing. (f) Correlation of yield strength versus Al_2O_3 content in present composites before and after matrix strengthening.

Figure 3. Microstructures of the composites. (a) SEM images showing typical microstructure in the Al-46% Al_2O_3 composite; (b,c) inverse pole figure (IPF) maps for as-sprayed Al before (b) and after (c) annealing at 500°C; (d,e) image quality (IQ) maps corresponding to 'b' and 'c', respectively.

Figure 4. Damage features and mechanisms under compression. (a) DIC results showing distribution of axial strain in specimens under global strain of 10%; (b) schematic illustration of deformation modes in pure Al and Al-46% Al_2O_3, and annealed Al-46% Al_2O_3.