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MICROSTRUCTURE AND MECHANICAL PROPERTIES OF GRAPHENE-REINFORCED ALUMINUM-MATRIX COMPOSITES

MIKROSTRUKTURA IN MEHANSKE LASTNOSTI Z GRAFENOM OJAČANIH KOMPOZITOV Z ALUMINIJEVO MATRICO

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Graphene has a high fracture strength, good ductility and low coefficient of thermal expansion, making it an ideal reinforcement for composite materials. This paper reports on an aluminum-matrix composite reinforced with graphene (graphene/Al) using ball milling and hot-press sintering. The effect of the graphene content on the microstructures, mechanical properties and fracture mechanisms of the composites were investigated. It is shown that 0.25 *w*/% and 0.5 *w*/% graphene contents modified with copper are homogeneously dispersed into the aluminum matrix. The composite with the 0.5 *w*/% graphene content exhibits the highest tensile strength and elongation; its improvement over the unreinforced aluminum matrix is 31.0 % and 30.6 %, respectively. With the increasing graphene content up to 1.0 *w*/%, the aggregation of graphene in the composite is apparent, seriously reducing the tensile properties. The fracture mechanism of the composites changes from ductile to brittle.

Keywords: aluminum-matrix composites, graphene, microstructure, mechanical properties

Grafen ima visoko lomno trdnost, dobro duktilnost in nizek koeficient termičnega raztezka, zato je idealen za ojačitev kompozitnih materialov. V članku avtorji poročajo o kompozitu na osnovi aluminija, ojačanem z grafenom (grafen/Al), izdelanem s krogličnim mletjem in sintranjem pod tlakom. Raziskovali so vpliv dodatka grafena na mikrostrukturo, mehanske lastnosti in mehanizem loma izdelanih kompozitov. V prispevku so pokazali, da je dodatek 0,25 *w*/% in 0,5 *w*/% grafena, modificiranega z bakrom, homogeno porazdeljen v matrici Al kompozita. Kompozit z 0,5 *w*/% grafena ima najvišjo natezno trdnost in raztezek, kar je 31,0 % oz. 30,6 % bolje v primerjavi z neojačanim kompozitom. Z naraščajočo vsebnostjo vsebnosti grafena do 1,0 *w*/% se pojavlja kopičenje grafena v matrici kompozita, kar močno zmanjša njegove natezne lastnosti. Mehanizem loma kompozita se tako spreminja iz duktilnega v krhkega.

Ključne besede: kompoziti na osnovi aluminija, grafen, mikrostruktura, mehanske lastnosti

1 INTRODUCTION

Aluminium-matrix composites have a high strength-to-weight ratio, high modulus-to-weight ratio, good damage tolerance, good wear resistance, excellent thermal conductivity and corrosion resistance, which make them highly competitive against the conventional aluminium alloys or even titanium alloys and steels for many structural applications, such as the construction, aerospace, transportation, automotive and military industries.[1–3] At present, particulate (such as graphite, Si_3N_4, SiC, TiN, BN, MgO and Al_2O_3 , etc.) reinforced aluminum-matrix composites are widely studied because of their advantages such as low cost, isotropic properties and the possibility of secondary processing, facilitating the fabrication of secondary components.[4–7]

The main research trend of aluminum-matrix composites is to continue to improve their comprehensive properties (such as tensile and fracture strength, thermal conductivity, friction and wear properties); meanwhile, they have good ductility and fatigue resistance. In order to get high-ductility, high-strength aluminum-matrix composites, high-performance reinforcement materials are necessary. Among them, carbon materials are outstanding, such as graphite whiskers, carbon fibers, carbon nanotubes (CNTs) and graphene (GNP).

In recent years, carbonaceous nanomaterials including graphene and carbon nanotubes (CNTs) have emerged as an important class of new materials for structural engineering and functional device applications due to their extraordinary high elastic modulus and mechanical strength as well as excellent electrical and thermal conductivities. In addition, most studies show that graphene is considered a most effective reinforcing filler for fabricating composite materials.[8–11] It is because graphene has some exceptional properties: excellent electric properties,[12] high thermal conductivity,[13] high Young's modulus[14] and high tensile stress.[15] Additionally, graphene is extremely light. The density of graphene was estimated to be as low as 1.06 g·cm.[3,16] These exceptional properties make it a perfect reinforcement for metal-matrix composites. A lot of research work has been carried out for the fabrication of aluminium composites reinforced with graphene nanosheets,[17–19] graphene nanoflakes,[18–21] few-layered graphene,[22] or its derivatives such as graphene oxide and reduced graphene oxide.[23,24] Cold compaction followed by hot isostatic pressing,[18] as well as hot extrusion,[17,24,20,21] vacuum hot press and/or hot rolling[22] are commonly used, according to the literature, to manufacture solid pure aluminium and graphene-reinforced aluminium-matrix composite specimens.

Wang et al. reported that the tensile strength of Al composites reinforced with only 0.3 *w*/% graphene nanosheets using powder metallurgy was 249 MPa.[24] It was a 62-% enhancement over the unreinforced Al matrix. Li et al. reported that an Al composite reinforced with only 0.3 *w*/% graphene oxide showed an 18-% and 17-% increase in the elastic modulus and hardness, respectively, over the unreinforced Al.[23] Yan Shao-jiu et al. reported that the tested tensile strength and yield strength of 0.3 *w*/% graphene-reinforced Al-matrix composites fabricated with powder metallurgy, showed an increase of 25 % and 58 %, respectively, compared to the pristine Al alloy.[18] The ductility of the composite was almost as good as or even a little better than the pristine alloy. Although the above studies showed that the mechanical properties of aluminum alloys can be improved by adding graphene, it is still a great challenge to produce aluminum-matrix composites reinforced by well-dispersed graphene via a conventional metallurgical process due to a high-temperature environment and a huge density difference between graphene and aluminum matrix. Great difficulties are encountered during the synthesis and unknown interfacial chemical reactions between graphene and metal matrices.

In this work, Al composites reinforced with graphene were fabricated by ball milling and hot-press sintering. The distribution and morphology of graphene in the Al matrix were analyzed. The mechanical properties of Al composites reinforced with graphene were tested and the relevant strengthening mechanisms of graphene were discussed based on the primary experimental evidences.

2 EXPERIMENTAL PART

2.1 Materials

The graphene sheets used in our experiments were prepared with modified Hummers method as reported previously.[25] The experimental material was natural graphite with a particle size of about 45 um. Natural graphite was reduced at 95 °C for 24 h using hydrazine hydrate. The preparation method was similar to that from Reference[25].

To improve the interface wetting between graphene and aluminum matrix, graphene was coated with copper using the chemical plating method. Graphene was added into a plating solution including 10 mL/L $CuSO_4$, 1 mL/L $NiSO_4$, 30 mL/L NaH_2PO_2, 30 mL/L H_3BO_3 and 15 mL/L $Na_3C_6H_5O_7$. The solution was stirred with a magnetic stirrer, then heated to 65 °C in a water bath for 2 h in order to keep a homogeneous growth of copper on the surface of graphene.

Atomized pure-aluminum powder (Anshan Steel Industrial Fine Aluminum, Inc., China) with an average size of 20 um and chemical composition of (Fe 0.071 *w*/%, Si 0.067 *w*/%, Cu 0.002 *w*/%) was used as the matrix material.

2.2 Composite preparation

A planetary machine was used for ball milling without an interruption under high-purity (99.999 %) argon gas. The stainless-steel vial was sealed with an elastomeric O ring. The stainless-steel balls to powder weight ratio was 7:1, and the rotation rate of the vial was 40 min^{-1} under the total milling time of 72 h.

A variety of ball-milled powders were fabricated by varying the mass fraction of graphene sheets (i.e., 0.25, 0.5, and 1.0 *w*/%). The ball-milled powder was containerized in a heat-resisting steel die (50 mm in diameter, 200 mm in height, and 10 mm in thickness) and was compacted. The compacted composite billets were sintered at 610 °C for 1.5 h under a pressure of 25 MPa with hot-press sintering. For comparison, a pure Al sample was also fabricated under the same conditions.

2.3 Characterization

An X-ray diffraction analysis of polished sintered sample bars was carried out with an X-ray diffractometer (Rigaku Ultima IV), using Cu K_α radiation for 20–90 degrees. Raw XRD data were refined and analyzed via the MDI Jade 6.0 program (Materials Data Incorporated: Livermore, CA, USA). The surface morphology was observed with a scanning electron microscope (SEM) S-3400N equipped with an energy-dispersive spectrometer (EDS).

The tensile properties of the sintered samples with the standard specimen sizes of (20 × 4 × 2) mm were determined with a testing machine (UTM4304, Shenzhen Suns Technology Stock Co., Ltd, China) at ambient temperature using a displacement rate of 0.5 mm/min.

3 RESULTS AND DISCUSSION

3.1. Microstructure

Figure 1 shows the representative morphology of graphene and graphene-coated Cu. Graphene has a two-dimensional high-aspect-ratio sheet geometry, and the wrinkles and folds are also shown on the exfoliated graphene sheet, as depicted in **Figure 1a**. The graphene consists of platelets with a morphology of irregularly shaped flakes, whose mean diameters are in a range of 0.5–15 um. Agglomerated powders have an opaque structure. The thickness of the graphene sheets is several nanometers (3–20 nm), corresponding to approximately 10–50 sheets of graphene (assuming that the thickness of monolayer graphene is 0.35 nm[26]). In **Figure 1b**, the graphene is covered with fine crystal particles. These particles are distributed uniformly on the surface of the graphene slice. It is concluded that these particles contain a large amount of copper particles.

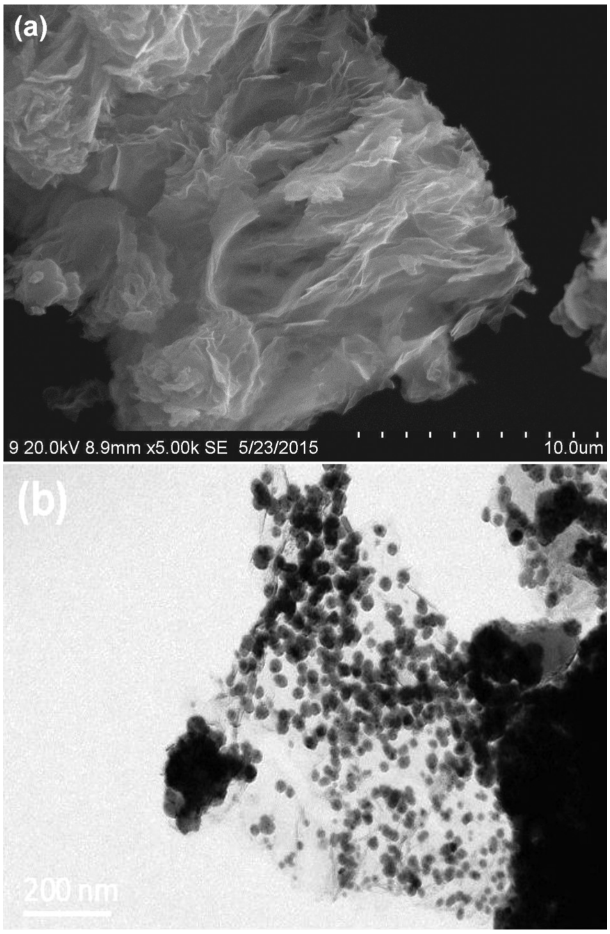

Figure 1: SEM and TEM images depicting the morphology of graphene, a) SEM morphology of graphene, b) TEM image of the copper-coated graphene

Figure 2 shows the distribution of graphene on the aluminum surfaces of the samples with different graphene contents. As shown in **Figure 2**, there are many fine platelets on the surfaces of the graphene/Al composite powders (**Figures 2b** to **2 d**), while the surface of the pure Al flakes is very clear. As the graphene contents increase, the fine platelets show a dense distribution. It is indicated that the fine platelets are signs of a homogeneous dispersion of the graphene nanosheets. It is believed that, in this study, the shape compatibility of the Al flakes and graphene nanosheets is crucial to achieve a uniform dispersion.

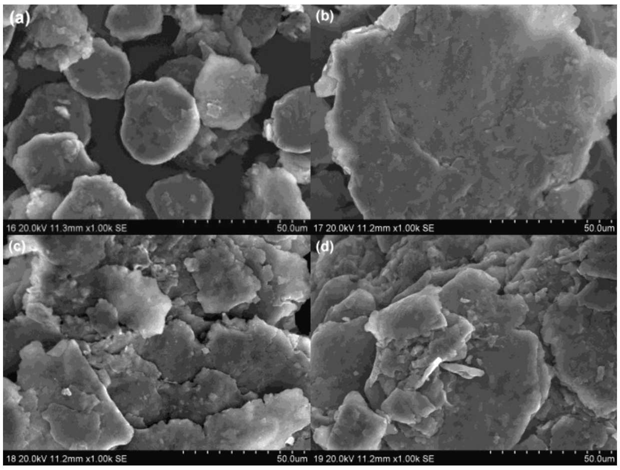

Figure 2: SEM micrographs of the mixture of aluminum powder particles and different graphene amounts after 72 h, a) pure Al, b) 0.25 *w*/%, c) 0.5 *w*/%, d) 1.0 *w*/%

Figure 3 shows X-ray diffraction patterns from pure Al and its composites in the sintered condition. As shown in **Figure 3**, a small amount of magnesium (2θ = 35.2°) is included in pure Al. No diffraction peaks of the carbon element are present in the graphene/Al composites. This is attributed to the nanometric size and the low content of the reinforcement phase, which cannot be detected due to the detection limit presented by XRD for the second phases.[27] However, no aluminium carbide (Al_4C_3) peak is recorded for any of the samples. It is believed that there is no chemical reaction between graphene and the Al matrix under this condition. Bartolucci et al. reported a formation of aluminum carbide in graphene/aluminum composites processed with hot extrusion.[17] Bustamante et al. thought that the formation of aluminum carbide is strongly dependent on the processing temperature of the production of the composites.[28] Our results calculated in the previous work showed that the interaction between C and Al atoms is in the Van Der Waals range, indicating that there is no chemical bonding between graphene and Al.[29] As also shown in **Figure 3**, an amount of the Al_2CuMg (S) phase is also detected in the graphene/Al composites, which is the main strengthening phase of Al-Cu-Mg alloys. However, in the current study, Al_2CuMg phases are generated from the copper-coated graphene and Al matrix during the sintering process.

3.2 Mechanical properties

The tensile properties of the graphene/Al composites with different graphene contents are shown in **Figure 4**.

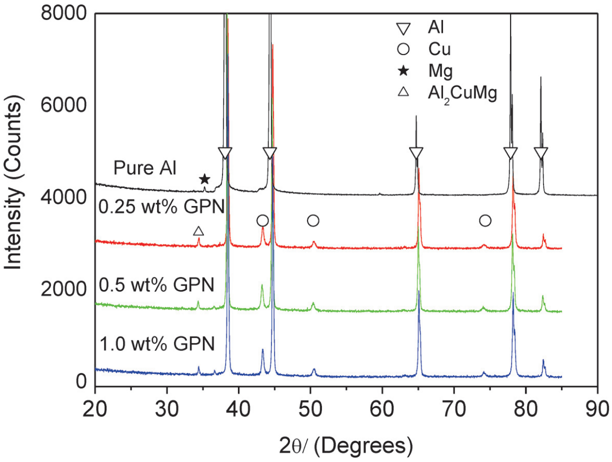

Figure 3: XRD patterns of pure Al and various graphene/Al composites

Figure 4a shows the representative engineering stress-strain curves of the graphene/Al composites with different graphene contents. **Figure 4b** shows the relationship of the ultimate tensile strength (UTS) and elongation to fracture (δ) of the graphene/Al composites. The strengthening effects are first enhanced and then they deteriorate with the increasing graphene contents. When the graphene content is 0.5 *w*/%, the UTS and δ of the composites reach the maximum value. As shown in **Figure 4b**, the composites with additions of 0.25 or 0.5 *w*/% graphene exhibit UTS values of 174.4 and 178.3 MPa, which indicate 28.1-% and 31-% enhancements over the aluminum-matrix sample (136.1 MPa). The same strong improvement of the tensile strength is seen in the aluminum-graphene composite.[24] Generally, the strengthening mechanisms include: a) Orowan strengthening, b) grain refinement, c) dislocation generation due to a different coefficient of thermal expansion (CTE) of reinforcements and matrix, and d) load transfer of graphene.[30] In the present work, the contribution of Orowan strengthening can be ignored considering the size and volume fraction of the graphene.[31]

The strengthening mechanism can be explained as follows: Firstly, the graphene with a super-high strength, a nanometric size and a homogeneous dispersion in the aluminum matrix (**Figures 2a** and **2b**) acts as the reinforcement contributing to the mechanical strengthening of bulk nanostructured graphene/Al composites. Secondly, the Cu on the graphene surfaces improves the interfacial bonding between the aluminum matrix and graphene (**Figures 2d** and **3**), effectively loading the transfer from the aluminum matrix to graphene, reported as the strongest material ever measured (ultimate strength of 130 GPa)[14]. Thirdly, graphene is uniformly dispersed at the grain boundaries of the aluminum matrix, as shown in **Figures 2a** and **2b**, thus hampering the migration of the grain boundaries and effectively hindering the grain growth during the hot-press sintering at elevated temperatures. Finally, the thermal-expansion-coefficient mismatch between aluminum and graphene (-0.8×10^{-6}/K for graphene[32] and 23.6×10^{-6}/K for Al[30]) may result in a multidirectional thermal stress at the graphene-Al interface and an improvement in the dislocation densities, which will increase the strength of the composites.

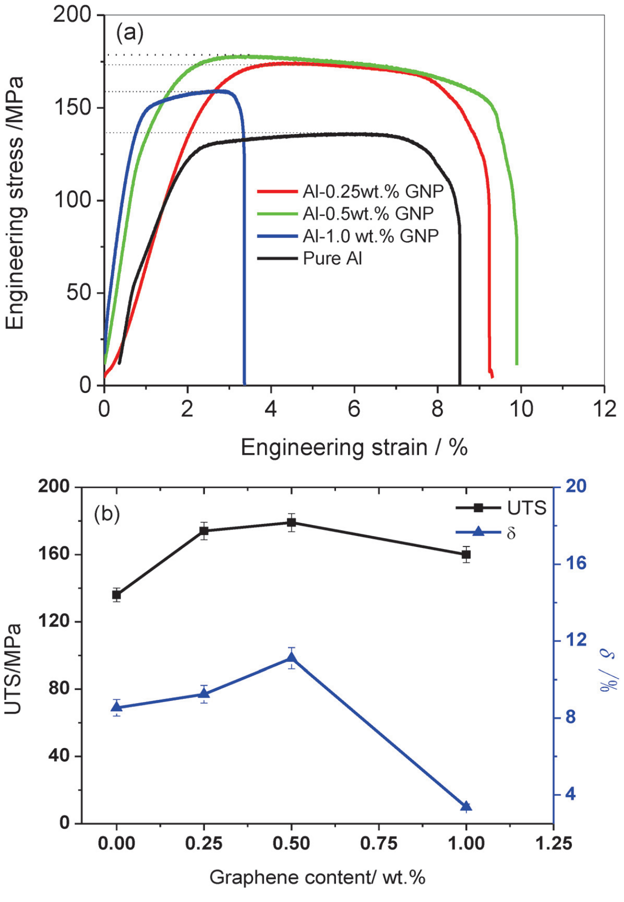

Figure 4: Tensile properties of graphene/Al composites with different graphene contents, a) stress-strain curves, b) relationship of UTS and δ with the graphene content

Meanwhile, a significant ductility is accompanied by elongation of the samples with the additions of 0.25 *w*/% and 0.5 *w*/% graphene, which increases with a greater addition of graphene and reaches the values of 9.2 % and 11.1 % for the 0.25 *w*/% and 0.5 *w*/% graphene contents, compared with 8.5 % for pure aluminum. This means that the strength and the ductility of the graphene/Al composites simultaneously increase. A possible reason for such unique mechanical properties of graphene/Al composites is the strong interface interaction between the graphene and aluminum matrix, leading to an effective transfer of the load from aluminum to graphene.

In contrast, the effect of a larger content (1.0 *w*/%) of graphene on the tensile properties is adverse. Both the tensile strength and the elongation decrease with an increase in the graphene additions. In particular, the elongation of the graphene/Al composite dropped sharply from 8.5 % of pure aluminum to 3.2 % of the graphene/Al composite with a 1.0 *w*/% graphene content. This is attributed to the fact that it would be more and more difficult for nanostructured graphene with a large aspect ratio and great specific surface area to be dispersed homogeneously into the aluminum matrix. Thus, a poor dispersion of graphene (>1.0 *w*/%) resulted in increased amounts of agglomeration and clusters of graphene, which in turn degraded both the strength and ductility of the graphene/Al composite.[20]

The tensile-fracture behavior of pure Al and graphene/Al composites with various contents of graphene is presented in **Figure 5**. It can be seen that dimples and tear ridges are tiny and uniform with distinguishing features of an obvious trend along the loading direction of the tensile stress, indicating a ductile fracture of pure Al. Compared with the pure Al (**Figure 5a**), the fracture surfaces of the graphene/Al composites are flat. The number of the dimples and tear ridges decreases. A few

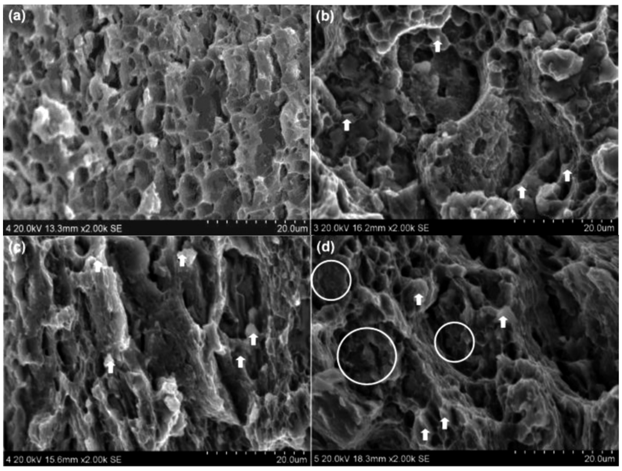

Figure 5: Tensile-fracture images of a) pure Al, b) 0.25 *w*/%, c) 0.5 *w*/%, d) 1.0 *w*/% composites

graphene sheets are observed on the fracture surface. For the 0.25 *w*/% and 0.5 *w*/% graphene/Al composites (shown in **Figures 5b** and **5c**), the fracture surfaces show graininess-distribution characteristics, indicating a brittle fracture. This is because graphene is mainly distributed around the grain boundaries in composite materials. When subjected to an external stress, the cracks will preferentially nucleate at the grain boundaries and expand continuously, which will eventually lead to a fracture of the composite. However, a plastic deformation of composite materials during a tension process is depressed by graphene. With an increase in the external force, the stress concentration occurs in the aluminum matrix sandwiched in graphene, in which holes appear and gradually grow up. These would cause a formation and continuous expansion of the cracks, indicating an obviously ductile fracture. Thus, there is a combination of fracture mechanisms, including ductile fracture and brittle fracture, affecting the graphene/Al composites with a lower graphene content.

With the increasing graphene contents up to 1.0 *w*/%, it is found that some graphene sheets are pulled out at the edges of the tear ridges, and the graphene/Al composite exhibits many flat fracture surfaces of brittle fracture, characterized by flat dimples of different sizes or almost no dimples. Many graphene sheets are observed on the fracture surface (**Figure 5d**). The aggregation of the graphene is apparent as it is seriously separated from the matrix, so the cracks preferentially form in the graphene and extend to the aluminum matrix during the tensile process, decreasing the tensile strength and elongation of the graphene/Al composite. The fracture mechanism of the graphene/Al composite changes from ductile fracture to brittle fracture with the increasing graphene content.

4 CONCLUSIONS

Graphene-reinforced aluminum-matrix composites were successfully fabricated with ball milling and hot-press sintering. Ball milling can cause a uniform dispersion of graphene into the aluminum matrix. A modification of the graphene surface with a copper coating can improve the interfacial bonding between the graphene and aluminum matrix. When the graphene content was small, the interfacial bonding between the graphene and aluminum matrix was good. Both the UTS and δ of the graphene/Al composites increased with the increasing graphene content. The composite with a 0.5 *w*/% graphene content exhibited a tensile strength and elongation improvement of 31.0 % and 30.6 %, respectively, over the unreinforced aluminum matrix. This improvement of the strength and ductility can be attributed to the homogeneous distribution of graphene in the aluminum matrix. However, as the content of graphene increased (especially up to 1.0 *w*/%), the extent of the agglomeration of graphene within the aluminum matrix increased, lowering both the UTS and δ of the graphene/Al composites. The fracture mechanism changed from ductile to brittle with the increasing graphene content.

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