Fabrication of Fullerene-Reinforced Aluminum Matrix Nanocomposites

Hamed Asgharzadeh¹ · Hamid Faraghi¹ · Hyoung Seop Kim²

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Abstract Fullerene-reinforced Al matrix nanocomposites were fabricated by high-energy mechanical milling followed by consolidation through hot extrusion or high-pressure torsion (HPT). The results indicate that a relatively homogeneous microstructure consisting of elongated, micrometer-sized Al grains is formed in the hot-extruded specimens. However, the microstructure is not uniform along the radius of the HPT disks, which includes coarse grains near the center of the disk and ultrafine grains in the middle and along the edge of the specimen. Microstructural evaluations of the HPT disks indicate that Al grain refinement occurs due to the addition of fullerene, as grain size is reduced to 60 nm from 118 nm. The formation of the harmful aluminum carbide phase is not detected during the fabrication of Al/C₆₀ nanocomposites. The hardness, yield stress, and ultimate tensile strength of the Al-2 vol.% C₆₀ nanocomposites are about 27–160% higher than those of the monolithic Al samples, revealing the effective strengthening of fullerene particles in Al matrix. Moreover, mechanical properties of the Al/fullerene nanocomposites are significantly enhanced (59–272%) by utilizing HPT in comparison to hot-extruded specimens due to their much finer Al grain structure. The reduction in the number and the size of the dimples, as well as the formation of smooth regions on the tensile fracture surface of Al/C₆₀, results in their overall lower ductility compared to monolithic Al.

KEY WORDS: Aluminum; Fullerene; High-pressure torsion; Hot extrusion; Microstructure; Mechanical properties

1 Introduction

Fullerenes are hollow carbon molecules in the form of a sphere (buckyballs) or tubes (carbon nanotubes: CNTs) [1]. Buckyballs can be essentially regarded as graphite layers, i.e., graphene sheets, wrapped into a spherical shape

instead of a cylindrical shape as in CNTs. Considering the staggering cost and limited availability of purified single-walled carbon nanotubes, it is obvious that fullerenes with a spherical structure offer an interesting alternative to CNTs since they are available in abundance and at a much feasible cost. Furthermore, these fullerenes offer a high degree of purity and maintain their quality even during the subsequent reproduction cycles. [2–4]. Thus, fullerenes are currently widely investigated and have the potential for various technical applications [5].

A significant potential application of fullerenes would be as reinforcement in metallic alloys used in structural parts. It should be noted that buckyballs, such as C₆₀ fullerenes, may be considered as fascinating reinforcements in comparison to CNT or graphene, due to their zero-dimensional geometry. They can be easily dispersed

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✉ Hamed Asgharzadeh
asgharzadeh@tabrizu.ac.ir

¹ Department of Materials Engineering, Faculty of Mechanical Engineering, University of Tabriz, Tabriz 51666-16471, Iran

² Department of Materials Science and Engineering, Pohang University of Science and Technology (POSTECH), Pohang 790-784, South Korea

and are scarcely damaged during severe mechanical dispersion processes carried out in metal matrix composites (MMCs) [2–4, 6]. C₆₀ fullerene-reinforced MMCs have been typically prepared using high-temperature processing methods, such as squeeze casting [7], liquid metal infiltration [8], co-deposition [9, 10], and powder metallurgy (PM) [11]. However, the interaction between C₆₀ fullerene and metal has been one of the main issues in the research related to fullerene. Although the C₆₀ fullerene might be considered as a chemically nonreactive molecule, it actually displays a significant reactivity with other atoms and molecules due to its icosahedral architecture that possesses both sp^2 and sp^3 bondings [12, 13]. Furthermore, a harmful chemical reaction between fullerenes and metal matrix might be facilitated by the large surface area of fullerenes.

Among all MMCs, aluminum is the most commonly used metal as matrix owing to its high specific strength and stiffness, wear resistance, and electrical and thermal conductivities [8, 14]. To improve the mechanical properties of Al matrix MMCs, various reinforcements such as Al₂O₃ [15], SiC [16], SiC/Al₂O₃ [17], B₄C [18], AlN [19], ZrO₂ [20], CNT [21], and graphene [22] have been used. Pioneering studies on Al/C₆₀ composites manufactured by casting and PM have been reported in Refs. [2–4, 11]. These reports have hinted at the formation of the brittle aluminum carbide (Al₄C₃) in the interface between C₆₀ and Al matrix resulting in the premature fracture of the composite. Furthermore, the homogeneous dispersion has not occurred due to the inadequate diffusion in the solid state or the limited solubility of fullerene in Al in the liquid state [3]. Severe plastic deformation (SPD) techniques, e.g., equal channel angular pressing (ECAP), high-pressure torsion (HPT), accumulative roll bonding (ARB), and friction stir processing (FSP) provide an interesting alternative to casting and PM methods to overcome these setbacks. The manufacturing of fullerene-reinforced MMCs at low temperatures by utilizing SPD techniques hinders the formation of detrimental phases, homogeneously disperses the fullerene within the matrix, and significantly refines the matrix microstructure to the ultrafine size range [23, 24]. Morisada *et al.* [25] have utilized FSP to fabricate AA5083/(C₆₀ + C₇₀) composites and have reported that FSP increased the microhardness of the substrate by the promotion of the grain refinement phenomenon to ~200 nm due to the pinning effect of the fullerene and its exceptionally high hardness. Tokunaga *et al.* [26] have consolidated a mixture of C₆₀ and C₇₀ fullerene with pure Al powder using HPT and have shown that the addition of fullerenes raised the tensile strength from about 150 to 250 MPa, whereas no meaningful effect in the elastic modulus was discovered.

The purpose of this work is to fabricate fullerene-reinforced Al matrix composites through the consolidation of a

mixture of Al and C₆₀ fullerene powders and to examine the influence of the fullerene addition on the microstructural evolution and tensile properties of Al. Although some researchers have investigated the microstructure and mechanical properties of fullerene-reinforced Al matrix composites, the effects of processing method on the grain structure of the matrix and the distribution of fullerene within the matrix of Al/fullerene composites have not been examined in detail. To investigate the roles of processing temperature and strain on the microstructure and mechanical properties, two methods of consolidation were employed: (a) hot powder extrusion and (b) room temperature HPT. To achieve a homogeneous dispersion of fullerene particles throughout the Al matrix, high-energy mechanical milling has also been used prior to the consolidation.

2 Materials and Methods

2.1 Al/C₆₀ Powder Preparation

The commercial purity Al powder (99%, Norlin) with an average particle size of 63 μm was used as the base metal, and fullerene C₆₀ powder (99.9%, Cheaptubes) with an outer diameter of ~1 nm was used as the reinforcing phase of the composite. Fullerene was dispersed in ethanol via sonication for 1 h, and then Al powder was added into the mixture and sonicated for 0.5 h to achieve Al-2 vol.% fullerene powder mixture. The powder mixture was then dried at 60 °C for 4 h. 1 wt% stearic acid (CH₃(CH₂)₁₆-COOH, Merck) was added to the Al/C₆₀ powder as the process control agent in a 125-mL volume vial made of hardened steel and mixed for 1 h in a low-energy ball mill. Hardened steel balls with diameters of 11 and 19 mm were put into the vial to achieve the ball-to-powder weight ratio of 10:1. High-energy mechanical milling of the composite powder was performed in a planetary ball mill with a rotational speed of 350 rpm for 4 h in Ar atmosphere. For powder characterization, a small amount of the milled powder was taken out of the vial and stored in an argon-filled glove box.

2.2 Al/C₆₀ Powder Consolidation

The powder was degassed at 450 °C for 1 h in Ar atmosphere. The consolidation of the composite powder was performed via either HPT at room temperature or powder extrusion at an elevated temperature. For HPT consolidation, first the powder was pre-compacted into the disk-shaped sample with a diameter and thickness of 10 and 1.5 mm, respectively. Then, the disk was placed between HPT anvils with a circular shallow hole (diameter of

10 mm and depth of 0.25 mm) and a pressure of 6 GPa was applied. The HPT processing was conducted under a quasi-constrained condition by imposing severe plastic strains through ten revolutions at a rotating speed of 1 rpm. The processed disk had a thickness of ~ 0.8 mm with some limited outward material flow between the upper and lower anvils.

To consolidate the powder by hot extrusion, the composite powder was filled in a 30-mm-diameter Al can and pre-compacted to a height of ~ 70 mm. After preheating at 450 °C for 30 min, the compacted powder was extruded with an extrusion ratio of 9:1 into a 10-mm-diameter cylindrical billet. As a reference, the monolithic Al powder was mechanically milled and consolidated with the same procedure as described above.

2.3 Microstructural Characterization

The morphology of mechanically milled powders was examined by a scanning electron microscope (SEM, CX-200, COXEM). X-ray diffraction (XRD) analysis was performed using a Siemens D500 diffractometer with Cu K_α radiation source working at the voltage of 40 kV and the current of 30 mA. The density of the consolidated powders was measured with a Mettler Toledo XP205 densimeter. For metallographic observations, the polished samples were etched using a solution of 1% HF in distilled water, and the microstructure was observed by an Olympus BX61 optical microscope. Transmission electron microscopy (TEM) observations were performed on a cross-sectional cut milled out from the HPT disk at a distance of 2.5 mm from the center using a focused ion beam (FIB) milling (3D total analysis, Helios Nanolab Dual Beam, FEI, Hillsboro), as shown in Fig. 1. TEM characterization was carried out using a JEOL JEM 2100F STEM operating

at a voltage of 200 kV. The Al grain size was measured by the line intercept method.

2.4 Mechanical Properties

The microhardness of HPT disks was measured across the diameter of each disk with an applied load of 2.94 N using a Vickers indenter (Future-Tech FM-700), as illustrated in Fig. 1. The Vickers hardness of the powder extrudates was measured at a load of 0.49 N. The hardness and microhardness measurements were performed at least three times each.

To investigate the tensile properties of the HPT disks, flat and dog bone-shaped microtensile specimens with a width of 1 mm and a gauge length of 1.5 mm were cut from the disks at 2.5 mm distance from the center using electro-discharge machining (EDM), as shown in Fig. 1. The round cross-sectional tensile specimens with a diameter of 4 mm and a gauge length of 16 mm were machined from the extrudates as well. The tensile test was conducted at room temperature at an initial strain rate of $\sim 10^{-3} \text{ s}^{-1}$. Fracture surfaces resulted in the aftermath of the tensile test were examined on a JEOL JSM-6330F field emission scanning electron microscope (FE-SEM).

3 Results and Discussion

Figures 2 and 3 show the morphology and microstructure of Al and Al/C₆₀ powder particles. The gas-atomized Al powder exhibits mostly irregular morphology (Fig. 2a). The Al particles were flattened and had a flake-like morphology which was formed due to the impacts of the balls during mechanical milling for 4 h (Fig. 2b). Furthermore, Al flakes started to weld together forming large particles with an average size of $\sim 100 \mu\text{m}$, as shown in Fig. 3a. It can be seen from Fig. 2c that for Al/C₆₀ powder, microwelding between the particles is more pronounced. On the other hand, a slight fragmentation of the flattened particles occurred so that the average size of particles was decreased to $\sim 80 \mu\text{m}$ (Fig. 3b).

The XRD patterns of the Al and Al/C₆₀ powders are presented in Fig. 4. The diffraction patterns of the Al and Al/C₆₀ powders are similar and exhibit the typical reflections for Al. Neither fullerene nor second phases were detected owing to their very low amount, well below the detection limit of the XRD method.

The densities of Al and Al/C₆₀ specimens after consolidation can be seen in Table 1. It is clear that a high degree of densification with a relative density (the ratio of density to theoretical density) of more than 98% was achieved through both HPT and powder extrusion methods. Figure 5a, b presents optical micrographs of the longitudinal

Fig. 1 Schematic illustration of HPT disk and locations for tensile samples, microhardness measurements, and OM and TEM observations

Fig. 2 SEM images of powders: **a** gas-atomized Al; **b** mechanically milled Al; **c** mechanically milled Al/C₆₀

Fig. 3 Optical micrographs of mechanically milled powders: **a** Al, **b** Al/C₆₀

cross sections of the Al and Al/C₆₀ extrudates. The microstructure of the hot-extruded specimens was extended parallel to the direction of extrusion so that elongated micrometer-sized Al grains were formed. No agglomerations of fullerenes were observed after hot extrusion. The microstructure of the top surface of ten revolutions HPT disks at different areas is shown in Fig. 5c–h. It is well known that for ideal rigid-body torsion, the center of the disk should not be subjected to any shear strain, and thus, the microstructure of the powder should be preserved in

Fig. 4 XRD pattern of: **a** Al powder; **b** Al/C₆₀ powder; **c** Al/C₆₀ extrudate

this area. However, a moderate shear deformation is seen in the central area of the disk (Fig. 5c, d), especially in the case of monolithic Al, revealing the incidence of deviations from the ideal rigid-body torsion. Similar observation has been reported by Cao *et al.* [27]. Furthermore, a few small pores were detected in the central area of the disks. On the other hand, a circular macroscopic shear deformation pattern was observed in the other areas of the HPT disks. It is well established that the shear strain (γ) increases in direct proportions with the distance from the torsion axis (r), based on the following relationship [28]:

$$\gamma = \frac{2\pi N r}{h}, \quad (1)$$

where N is the number of revolutions and h is the thickness of the disk. Thus, a fully dense microstructure consisting of severely deformed particles would be observed at the middle ($r = 2.5$ mm; $\gamma = 196$) and near the periphery ($r = 4.5$ mm; $\gamma = 353$) areas (Fig. 5e–h). Also, the extent of deformation increases, as the distance from the center increases in agreement with Eq. (1). The equivalent von

Table 1 Density, hardness, and tensile properties of Al and Al-2 vol.% fullerene fabricated by high-pressure torsion and hot extrusion

Material type	Consolidation method	Density (g cm ⁻³)	Relative density	Hardness (HV)	Offset yield stress (MPa)	Ultimate tensile strength (MPa)	Strain to fracture (%)
Al	HPT	2.659 ± 0.004	0.985	92 ± 1	305	321	25.9
Al	Hot extrusion	2.661 ± 0.002	0.986	30 ± 3	63	124	36
Al-2 vol.% fullerene	HPT	2.647 ± 0.002	0.984	139 ± 2	409	409	0.6
Al-2 vol.% fullerene	Hot extrusion	2.655 ± 0.003	0.983	78 ± 9	110	258	14.1

Mises strain (ε) for the HPT and extrusion processes can be calculated using Eqs. (2) and (3), respectively [29]:

$$\varepsilon = \frac{\gamma}{\sqrt{3}}, \quad (2)$$

$$\varepsilon = \ln R, \quad (3)$$

where R is the extrusion ratio. Here, since the equivalent von Mises strain varied from 0 to 245 for the HPT process, it had a constant value of 2.2 for extrusion. Thus, the microstructural changes are mostly related to the amount and uniformity of the imposed strain during the consolidation.

The detailed microstructure analysis for the HPT-processed Al and Al/C₆₀ specimens was conducted using TEM. Bright-field TEM micrographs and corresponding selected area diffraction (SAED) patterns are shown in Fig. 6. A significant grain refinement occurred after HPT processing for ten revolutions so that ultrafine grain structure was attained in both monolithic Al and Al/C₆₀ composites. A relatively equiaxed grain structure with an average grain size of ~118 nm was formed in monolithic Al (Fig. 6a). Microstructural refinement is more pronounced in Al/C₆₀ composite, and the average grain size was measured as ~60 nm. Moreover, as shown in Fig. 6b, most of the grains are flattened in a direction perpendicular to the compression axis having dimensions of 33 nm × 86 nm. The greater grain refinement of Al₆₀₆₁/SiC [30] and Al/(CNT, graphene) [31] in comparison to the monolithic metal during the HPT processing has been previously stated.

The finer grain size of the Al matrix in Al/C₆₀ composite is also reflected in the SAED patterns. The SAED pattern of monolithic Al contains separate spots indicating the presence of low-angle grain boundaries (Fig. 6c). Conversely, the SAED pattern in Fig. 6d consists of more diffraction spots than that in Fig. 6c. These spots situated around circles forming continuous diffraction rings represent the existence of a large amount of high-angle grain boundaries [29]. In both cases, the azimuthal spreading of some spots in the SAED patterns discloses the existence of high internal stresses induced by severe straining through HPT [32]. Figure 7a illustrates the high-angle angular-

dark-field (HAADF) TEM micrograph of Al/C₆₀ nanocomposite. It can be seen that fullerene uniformly dispersed within the Al matrix. The high-resolution TEM (HRTEM) image as shown in Fig. 7b indicates that no detrimental phases, e.g., Al₄C₃, were formed in the microstructure of the Al/C₆₀ composite.

Figure 8 shows the variation of the Vickers microhardness with position on the Al and Al/C₆₀ disks subjected to HPT for ten revolutions under the pressure of 6 GPa. It is clear that Hv values tend to grow in proportion to the distance from the center of the disk, especially for Al/C₆₀ composite. However, the rate at which microhardness increases is greater near the center of the disk so there is a tendency toward saturation in Hv near the periphery of the disk. This is a typical behavior in HPT processing for a material with slow recovery rate, where the hardness is initially lower in the center, but the microstructure gradually evolves into a homogeneous condition [33]. Thus, for both monolithic Al and Al/C₆₀ composites, the microstructure was not completely evolved after ten revolutions, which is in agreement with Fig. 5c–h. Furthermore, higher imposed strains are necessary for microstructural homogeneity and hardness saturation.

The hardness level of the Al/C₆₀ composite is invariably higher than that of the monolithic Al, so that the average hardness value throughout the disk is 51% higher in the composite sample. This situation is much more significant in the hot-extruded samples, since reinforcing of Al with fullerene increased the average hardness about 2.6 times (Table 1). It should also be noted that the hardness values for extrudates are much lower than HPT samples, mostly due to their coarse grain structure.

Tensile tests of the samples have demonstrated a strong effect of the fullerene addition and processing method on the strength and ductility of Al. The measured yield stress (YS), ultimate tensile strength (UTS), and strain to fracture (SF) values are listed in Table 1. It is obvious that YS and UTS values are in accordance with the hardness values. The highest YS and UTS values were observed for the Al/C₆₀ composite processed via HPT (409 MPa), being 34 and 27% higher than those of monolithic Al, respectively. Such

Fig. 5 OM micrographs of hot-extruded and HPT-processed specimens: **a** Al, hot extrusion; **b** Al/C₆₀, hot extrusion; **c** Al, HPT-center; **d** Al/C₆₀, HPT-center; **e** Al, HPT-middle; **f** Al/C₆₀, HPT-middle; **g** Al, HPT-edge; **h** Al/C₆₀, HPT-edge

Fig. 6 TEM images and SAD patterns from the microstructure of the samples subjected to HPT for ten turns: **a, c** Al, **b, d** Al/C₆₀

high levels of strength improvement in the Al/C₆₀ composite can be attributed to the ultra-high strength of fullerene, the uniform dispersion of fullerenes within the Al matrix, the strong interfacial bonding between fullerenes and Al matrix, and the significant grain refinement of the Al matrix. It is pertinent to point out that the strength of the HPT-processed monolithic Al is much higher than those of the hot-extruded Al and even the Al/C₆₀ composite, revealing the significant role of nano-sized grain structure on the strengthening of Al. It should be noted that in the case of HPT specimens, the tensile samples were cut at a position 2.5 mm from the center of disks. Thus, the tensile properties of the HPT specimens are mostly the representative of the mechanical behavior of the middle regions of the disks. The inhomogeneous microstructure of the HPT disks which resulted from the heterogeneous nature of flow in HPT processing is likely accompanied by local variations in the tensile behavior of the samples similar to the hardness variations. Nevertheless, even though the hardness heterogeneity in HPT disks was extensively studied, the heterogeneity in their tensile properties has not been

reported owing to the difficulties in the sample preparation and testing. In the case of hot-extruded samples, YS and UTS values for the C₆₀-reinforced Al are 75 and 108% higher than those for Al without reinforcement, respectively, indicating the significant effect of fullerene on the strength enhancement. To better realize the influence of fullerenes, the strengthening efficiency (η) was calculated according to the following equation [34]:

$$\eta = \frac{\sigma_c - \sigma_m}{V_f \sigma_m}, \quad (4)$$

where σ_c and σ_m are the UTS values of composite and monolithic material, respectively, and V_f is the volume fraction of the reinforcement. The strengthening efficiency values of fullerene in Al matrix in the HPT and hot-extruded samples are 13.7 and 54, respectively. Note that these values are larger than those obtained previously for press-sintered Al-8 vol.% C₆₀ + C₇₀ (9.1) [2], press-sintered Al-1.3 vol.% fullerene (48.4) [3], spark plasma-sintered Al-20 vol.% C₆₀ + C₇₀ (24.2) [4], and HPT-processed Al-5 wt% C₆₀ + C₇₀ (8.9) [26].

Fig. 7 **a** HAADF TEM, **b** HRTEM images of HPT-processed Al/C₆₀ nanocomposite. The arrows in **a** show fullerene within the Al matrix

In contrast to the effective influence of the addition of fullerene into the Al matrix as reinforcing agent, it had a detrimental effect on the strain to fracture ratio. The fracture surfaces of Al and Al/C₆₀ samples resulting from tensile testing were studied using SEM (Fig. 9). The hot-extruded monolithic Al specimen displayed a dimpled surface typical for intragranular ductile fracture mode (Fig. 9a, b). The dimples are mostly equiaxed with a mean size of $\sim 3 \mu\text{m}$. A sharp decrease in the size of the ductile dimples to $\sim 100\text{--}300 \text{ nm}$ was observed on the fracture surface of the monolithic Al processed by HPT, as clearly seen in Fig. 9d. The size of dimples is in agreement with the grain size of the sample (Fig. 6a). Furthermore, several smooth regions were detected on the fracture surface

Fig. 8 Variation of Vickers microhardness with distance from the center of the HPT disks

(Fig. 9c). These fracture surface features indicate that the ductility of the HPT Al sample should be considerably diminished compared with the hot-extruded sample. This result is well consistent with the tensile testing results given in Table 1. By comparing the hot-extruded monolithic Al (Fig. 9a, b) and Al/C₆₀ composite (Fig. 9e, f) samples, a remarkable change in the morphology of the fracture surface was observed. The amount of dimples was significantly reduced, and a layered morphology consisting of weakly bonded powder particles was monitored on the fracture surface of Al/C₆₀. This observation may be attributed to the employment of improper processing conditions during hot extrusion of this sample, e.g., low imposed strain and temperature, resulting in the deterioration of ductility. The fracture surface morphology of the HPT-processed Al/C₆₀ composite (Fig. 9g, h) is similar to that of the monolithic Al (Fig. 9c, d) except that the size of dimples is reduced and the amount of smooth regions is increased. Additionally, some cracks were observed on the fracture surface which may be formed due to the poor interface between the fullerene cluster and the Al matrix, which acted as the crack nucleation sites. Under the tensile loading, these cracks propagated, accounting for the inferior ductility of the sample ($<1\%$). Similar results have been reported in the case of Al/fullerene composite fabricated by HPT [26] and AA6061/graphene composite prepared by semisolid sintering [35]. Thus, the ductility decrease in the Al/C₆₀ composite was most likely due to the fullerene addition and its dispersion rather than solely from the hardening effect caused by high-energy mechanical milling or consolidation, especially HPT processing.

Fig. 9 Tensile fracture surfaces of Al and Al/C₆₀ samples at two different magnifications: **a, b** Al, hot extrusion; **c, d** Al, HPT; **e, f** Al/C₆₀, hot extrusion; **g, h** Al/C₆₀, HPT

4 Conclusions

Near full density Al-2 vol.% C₆₀ nanocomposite samples were prepared from mechanically milled Al and fullerene powder mixtures by either hot extrusion or HPT. The main conclusions can be drawn as follows:

1. An inhomogeneous microstructure consisting of moderately to severely deformed Al grains was formed after HPT due to the variation of the imposed shear strain along the radius of the consolidated disk. Nevertheless, the microstructure of the hot-extruded sample was entirely composed of elongated Al grains.
2. A micrometer-sized grain structure with a thickness of $\sim 3 \mu\text{m}$ was formed after hot extrusion. However, a significant grain refinement to nanoscale range (average grain thickness of $\sim 33 \text{ nm}$) occurred in the HPT of Al/C₆₀ nanocomposite due to the application of severe plastic deformation.
3. The hardness and strength of Al/C₆₀ nanocomposite were much higher than those of the monolithic Al. The strengthening efficiency of fullerene in the Al matrix for the hot-extruded sample was greater than that for the HPT sample.
4. The fracture surface morphology of the HPT-processed Al/C₆₀ nanocomposite consisted of equiaxed ultrafine dimples and some cracks. Nonetheless, a layered morphology comprised of weakly bonded particles with some coarse dimples was observed on the fracture surface of hot-extruded sample. Thus, the addition of fullerene had a detrimental effect on the ductility of the monolithic Al.

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