Arch. Metall. Mater. **62** (2017), 2B, 1109-1112

DOI: 10.1515/amm-2017-0161

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MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Al-3 VOL% CNT NANOCOMPOSITES PROCESSED BY HIGH-PRESSURE TORSION

Al-3 vol% CNT nanocomposites were processed by high-pressure torsion (HPT) at room temperature under the pressure in the range of 2.5-10 GPa for up to 10 turns. Optical microscopy, scanning electron microscopy, and transmission electron microscopy (TEM) were used to investigate the microstructural evolutions upon HPT. Mechanical properties of the HPT-processed disks were studied using tensile tests and microhardness measurements. The results show gradual evolutions in the density, microstructure, and hardness with increasing the number of turns and applied presure. Nanostructured and elongated Al grains with an average grain thickness of ~40 nm perpendicular to the compression axis of HPT and an aspect ratio of ~3 are formed after 10 turns under 6 GPa. Evaluating the mechanical properties of the 10-turn processed Al/CNT nanocomposites indicates a tensile strength of 321 MPa and a hardness of 122 Hv. The tensile fracture surface of the Al/CNT nanocomposite mostly demonstrates a smooth fracture manner with fine dimples resulting in a low tensile ductility of ~1.5%.

Keywords: High-pressure torsion; Aluminum; CNT; Microstructure; Mechanical properties

1. Introduction

Nanostructured (NS) and ultrafine-grained (UFG) materials are in the focus of materials science due to their unique physical and mechanical properties. Several methods of severe plastic deformation (SPD), i.e., large plastic straining under high hydrostatic pressure at low temperature, such as equal channel angular pressing (ECAP), high-pressure torsion straining (HPT), accumulative roll bonding (ARB), etc., have been developed to fabricate bulk NS/UFG materials with a grain size from 20 to 200 nm [1]. Among the various SPD methods, the HPT process has been widely noticed due to its ability to impose extremely high strain and hydrostatic pressure to develop homogeneous nanostructures with high angle grain boundaries (HAGBs) and high intrinsic stresses [2]. In this technique, a disk sample is located between two anvils rotating under a high pressure of ~2-8 GPa. Applying the high hydrostatic pressure during straining can hinder vacancy migration during deformation thereby retarding the annihilation mechanisms of dislocations [3]. Furthermore, it enables HPT method for consolidating metallic powders to produce densely-packed bulk materials.

Besides single phase metals, HPT has been also used to fabricate metal matrix composites (MMCs) reinforced with carbon nanostructures [3-6]. Carbon nanotubes (CNTs) are promising reinforcement phase in MMCs because of their high aspect ratio, high elastic modulus (>1 TPa) and high tensile strength (>30 GPa) [7]. However, improvement of properties in MMCs is restricted due to the lack of uniform dispersion of CNTs in the metallic matrix and low interfacial bonding strength between the matrix and the CNT reinforcements. In this paper, the influence of CNTs on the grain structure and mechanical properties in composites consolidated from Al/CNT powders by HPT was investigated.

2. Experimental

Commercial purity Al powder and 3 vol% multi-walled CNTs (MWCNTs) were mixed by high-energy mechanical milling. The diameter and the length of MWCNTs were 5-20 nm and 1-10 μm, respectively. The powders were pre-compacted in a die with a diameter of 10 mm and then consolidated by HPT at room temperature. The disks were compressed and deformed under various applied pressures of 2.5, 6 and 10 GPa. HPT staining was performed for different numbers of revolutions in the range of 1 to 10. The HPT-processed disks were 10 mm in diameter and ~0.8 mm in thickness.

The microhardness was measured across the diameter of each disk using a Vickers indenter. Dog-bone shaped microtensile samples with a 1.5 mm gage length and 1 mm width were cut from the disks using electro-discharge machining at a position 2.5 mm from the centre. During the tensile test, precise strains were measured using the digital image correlation (DIC) method. The fracture surface of the tensile samples was examined using a field emission scanning electron microscope (FE-SEM).

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The mass density of the consolidated samples was determined using Archimedes' principle. The polished samples were etched using a solution of 1% HF in distilled water and the microstructure was observed by optical microscope. The internal microstructure of the disks was examined by scanning transmission electron microscopy (STEM) operating at 200 kV. The STEM specimens were prepared by dual beam focused ion beam (FIB) milling cut from the HPT disks at a distance of 2.5 mm from the centre.

3. Results and discussion

Figures 1 and 2 show the polished surface of Al-3 vol% CNT composites at different areas of HPT disks processed at various numbers of revolutions and applied pressures. It is clear from Fig. 1 that the structure of HPT disk is not homogeneous after one turn so that there are numerous pores at the centre of the disk while a few small pores appeared at the edge of the HPT sample. By increasing the number of turns, the densification

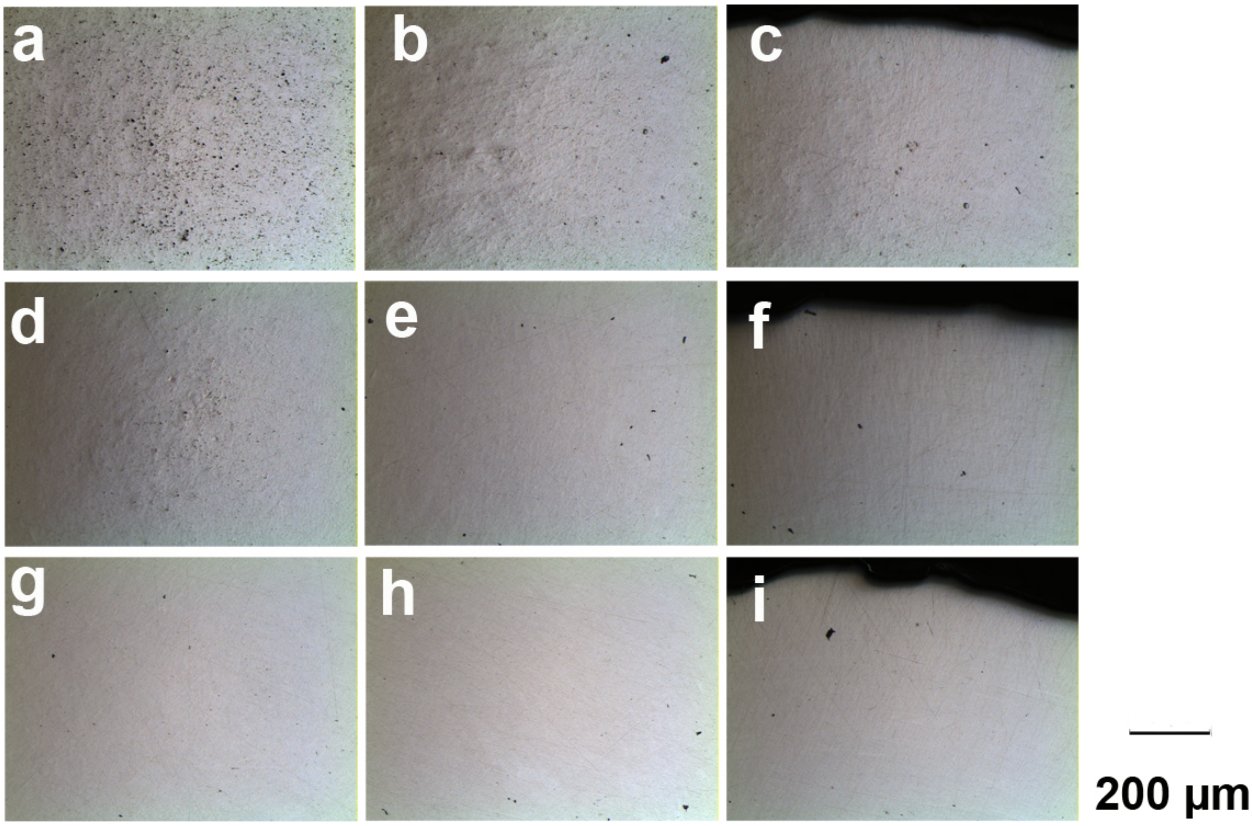

Fig. 1. Optical micrographs from the polished surfaces of the HPT-processed Al/CNT disks at number of turns of: (a-c) 1, (d-f) 5 and (g-i) 10 at the centre (left-hand), middle (middle) and edge (right-hand) of the disks (P = 6 GPa)

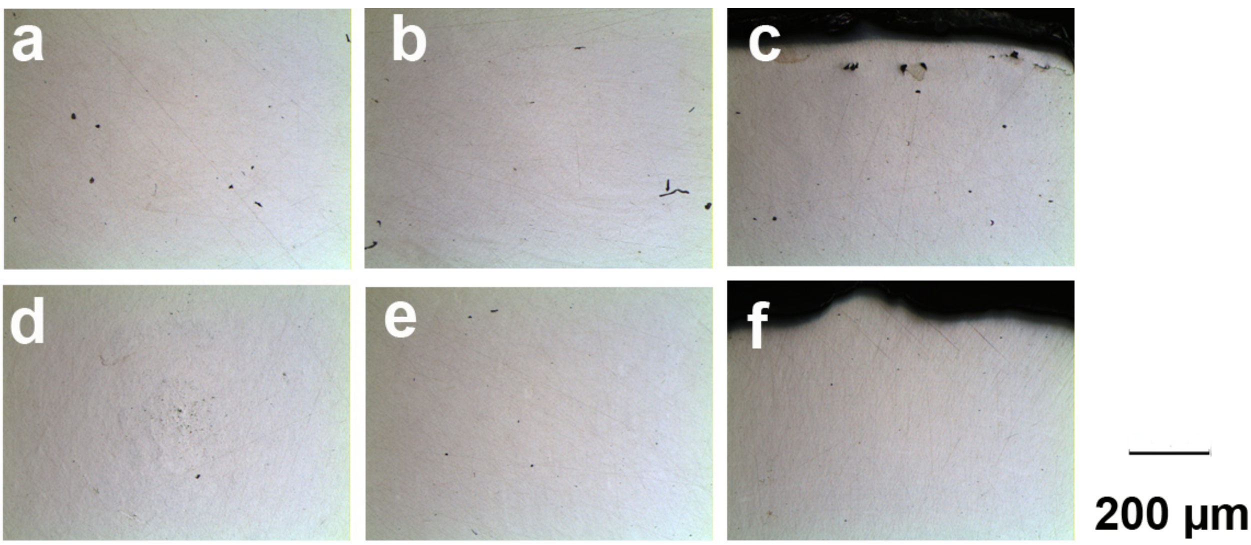

Fig. 2. Optical micrographs from the polished surfaces of the HPT-processed Al/CNT disks at pressures of: (a-c) 2.5 GPa and (d-f) 10 GPa at the centre (left-hand), middle (middle) and edge (right-hand) of the disks (n = 10)

was improved and almost full densification was achieved after 10 turns. The structural observations of the HPT disks consolidated at various pressures (Fig. 2) revealed that as the applied pressure increased, the densification was improved.

The variations of relative density of Al-CNT against the number of turns and the applied pressure are depicted in Fig. 3. It is clear from Fig. 3a that a high degree of densification to the relative density of 98.5% was achieved after only one turn. The consecutive straining to 10 turns was increased the density of HPT disks. On the other hand, as the applied pressure increased, the relative density of disks was improved. The production of near full dense disks through HPT consolidation is attributed to the severe straining concurrent with the large pressure imposed by the HPT process.

The microstructure of the HPT processed Al-3 vol% CNT disk is illustrated in Fig. 4. It is clear from Fig. 4a that after consolidation for 10 turns, the powders were well bonded together and the prior particle boundaries were not visible due to the severe shear straining upon HPT. The inspection of the microstructure at a higher magnification by means of TEM (Fig. 4b) shows that HPT deformation gives rise to a significant grain refinement. It is noticed that most of the grains are flattened in a direction perpendicular to the compression axis. The size range of the grains is 10 to 400 nm. The average length and thickness of grains were measured from 100 grains and determined to be ~120 nm and ~40 nm, respectively. Furthermore, the TEM image discloses the accumulation of a high density of dislocations within the grains.

The Vickers microhardness values of the HPT disks as a function of the number of turns and the applied pressure are shown in Fig. 5. The Hv significantly increases at the central region of the disks by increasing the number of turns (Fig. 5a). Nevertheless, consolidation at the higher number of turns resulted in a slight degradation of the microhardness at regions near the periphery. The decrease in the hardness level at higher number of turns is mainly related to an increase in the fraction of damaged CNTs that were bent or broken through HPT [8]. Moreover, microhardness increases with increasing the applied pressure upon HPT, as shown in Fig. 5b. It is apparent that the error bars are larger in the central regions and decrease with an increase in the number of turns and applied pressures, revealing a gradual transition to microstructural uniformity after the large number of turns and applying high pressures.

Figure 6a shows tensile stress-strain curve of Al-3 vol% CNT composite processed via HPT for 10 turns under an applied pressure of 6 GPa. A rapid hardening occurred during deformation and a high tensile strength of 321 MPa with a low ductility of 1.5% was achieved. The tensile fracture surface of the sample exhibited a smooth fracture manner with many fine and shallow dimples (Fig. 6b). On the other hand, no pull-out of CNTs was observed on the fracture surface of the composite. Thus, since the load transfer from the Al matrix to the CNTs has

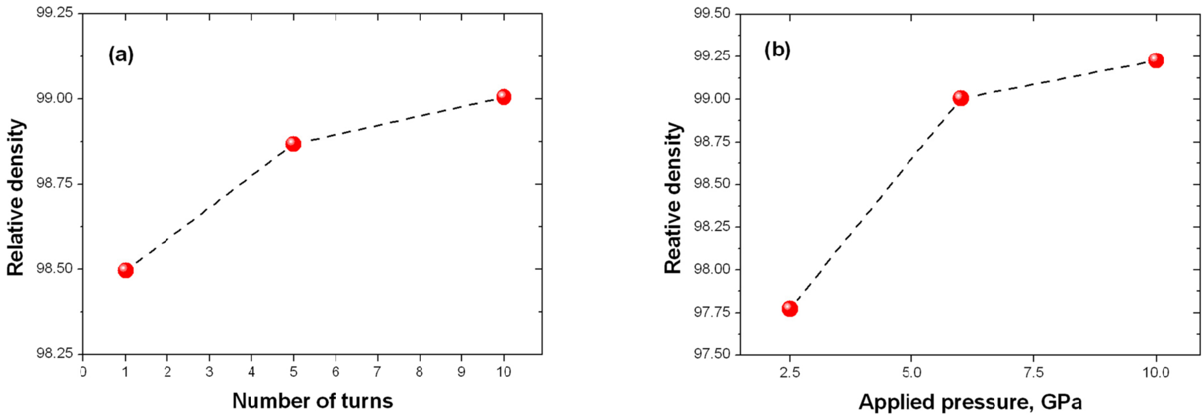

Fig. 3. Variation of relative density of Al-3 vol% CNT samples pro-cessed by HPT as a function of: (a) number of turns (P = 6 GPa) and (b) applied pressure (n = 10)

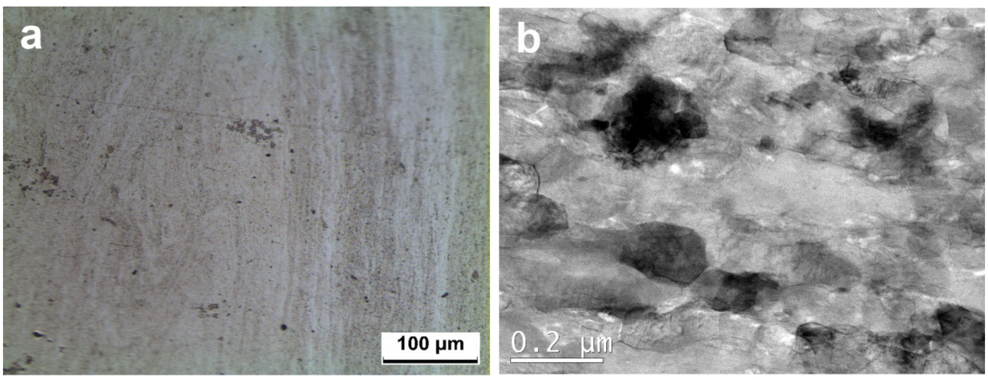

Fig. 4. Microstructure of HPT Al-3 vol% CNT disk processed for 10 turns under the applied pressure of 6 GPa in the middle region: (a) OM image and (b) TEM image

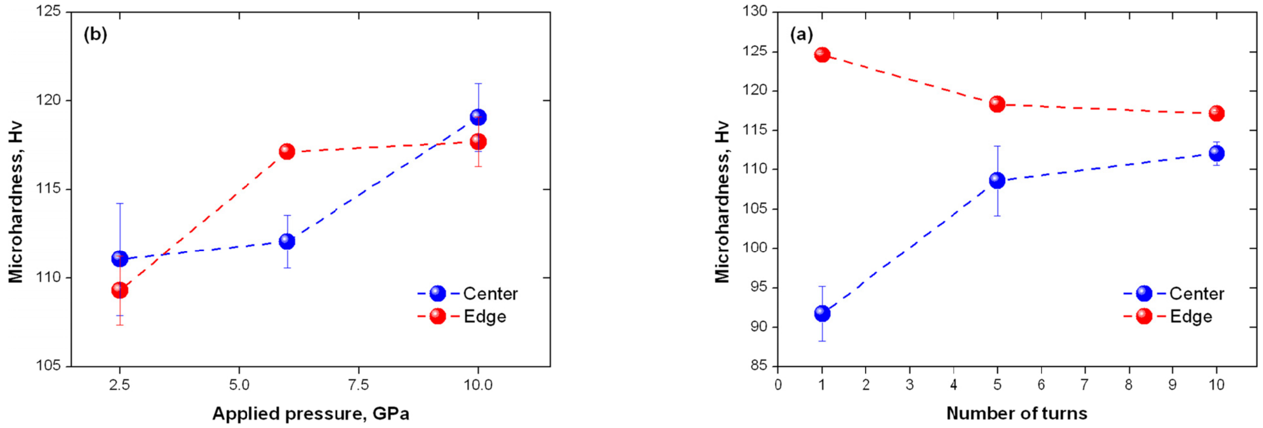

Fig. 5. Microhardness of Al-3 vol.% CNT samples processed by HPT at the center and near the edge of the disks as a function of: (a) number of turns (P = 6 GPa) and (b) applied pressure (n = 10)

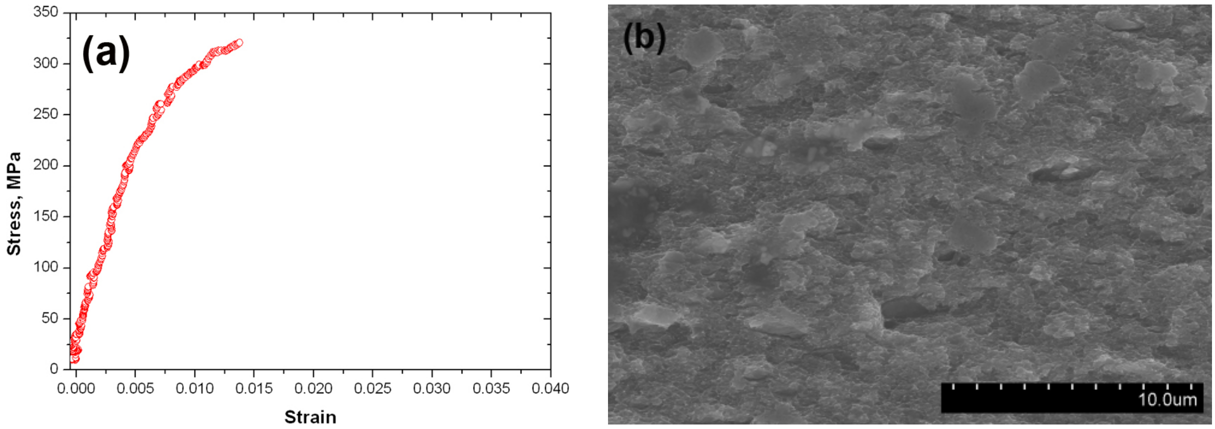

Fig. 6. (a) Tensile stress-strain curve and (b) tensile fracture surface of Al-3 vol% CNT disk processed by HPT for 10 turns under the applied pressure of 6 GPa

a slight contribution to the tensile strength, it can be considered that Al-3 vol% CNT composite is mainly strengthened by the Al matrix grain refinement and the Orowan looping of the matrix. Furthermore, an intense strain localization near CNTs caused matrix cracking due to the severe modulus mismatch between Al and CNT and hence brittle fracture behavior was prevailed and a low tensile ductility was achieved [9].

4. Conclusion

The microstructure and mechanical properties of Al-3 vol% CNT composite fabricated by HPT were investigated. Almost full densification with an average grain size of ~78 nm was achieved in the Al/CNT composite due to the large applied pressure and intense shear strain introduced by HPT. The inspection of hardness evolution during HPT revealed that the microhardness of disks increased and homogenized by increasing the applied pressure and number of turns. A high strength together with a low ductility was achieved after HPT processing due to the synergistic effect of grain refinement strengthening of the Al matrix, the Orowan looping strengthening and strain localization near CNTs.

Acknowledgments

This work has been supported by the Center for International Scientific Studies & Collaboration (CISSC).

REFERENCES

[1] R.Z. Valiev, R.K. Islamgaliev, I.V. Alexandrov, Prog. Mater. Sci. **45**, 103-189 (2000).

[2] A.P. Zhilyaev, T.G. Langdon, Prog. Mater. Sci. **53**, 893-979 (2008).

[3] P. Jenei, E.Y. Yoon, J. Gubicza, H.S. Kim, J.L. Lábár, T. Ungár, Mater. Sci. Eng. A **528**, 4690-4695 (2011).

[4] T. Tokunaga, K. Kaneko, Z. Horita, Mater. Sci. Eng. A **490**, 300-304 (2008).

[5] S.H. Joo, S.C. Yoon, C.S. Lee, D.H. Nam, S.H. Hong, H.S. Kim, J. Mater. Sci. **45**, 4652-4658 (2010).

[6] T. Tokunaga, K. Kaneko, K. Sato, Z. Horita, Scripta Mater. **58**, 735-738 (2008).

[7] V.N. Popov, Mater. Sci. Eng. R **43**, 61-102 (2004).

[8] H. Asgharzadeh, S.H. Joo, H.S. Kim, Metall. Mater. Trans. A **45**, 4129-4137 (2014).

[9] H.J. Choi, J.H. Shin, D.H. Bae, Compos. Sci. Technol. **71**, 1699-1705 (2011).