An investigation on the relationships between hardness, elastic modulus and the work of 7075 aluminium alloy by nanoindentation

Aijun Liu¹³⁴, Qichen Pan¹, Minghai Chen², Wenlei Xu⁴, Wenlin Chen¹*

¹School of Materials Science and Engineering, Hefei University of Technology, Hefei 230009, China;
²Suzhou Institute of Nano-Tech and Nano-Bionics, CAS, Suzhou 215123, China;
³Anhui Powder Metallurgy Engineering Technology Research Center, Hefei 230009, China;
⁴Suzhou aromi Technology Co., Ltd, Suzhou215134, China

*Corresponding author: chenwl@hfut.edu.cn

Abstract: Nanoindentation method is suitable for testing the mechanical properties of micron scale grains to ensure the effect of the second phase. In this experiment, the relationships between hardness, elastic modulus and the work of 7075 aluminium alloy grains with different single wall carbon nanotubes content were investigated. The results show that grain size decreases with the increase of single wall carbon nanotubes (SWCNTs) content, and MgZn2 and Al2Cu can be found. Hardness, young's modulus and the elastic reverse deformation of 7075 aluminium alloy increase firstly with increasing single wall carbon nanotubes, and then decrease obviously with the further increase of single wall carbon nanotubes, while the mechanical work and the plastic deformation work of the alloy have an opposite trend. It can be conclude that the single-walled carbon nanotubes at the grain boundary obviously changed the strain.

1. Introduction

Single wall carbon nanotubes are currently used as reinforcing phase due to their excellent combination such as high strength, specific stiffness, specific surface area, larger interface with matrix junction and closer bonding[1-4]. Single wall carbon nanotubes as reinforcing phase are mainly distributed near the grain boundary of the matrix. Aluminum(Al)-CNT composite have actively studied in accordance with transport equipment such as automobiles and aircrafts[5,6]. Grain size mainly dominates the mechanical properties of aluminium alloy. Accurate acquirements of the individual mechanical properties of grain are still challenging. Nanoindentation technique that indentation experiments can be mapped over a wide area, and the mechanical properties of grain can be obtained from the modulus and hardness contours.

In this paper, the relationships between hardness, elastic modulus and the work of 7075 aluminium alloy were investigated. The major interest lies in understanding the strengthening mechanisms of 7075 aluminium alloy.
2. Experimental procedure

2.1. Materials preparation
The SWCNTs/7075 mixed powder is pre-pressed in a cylindrical mold and sintered by vacuum hot pressing (803 K, 10 MPa for 1h) to produce a cylindrical aluminum billet with a diameter of 30 mm and a height of 4 mm. 7075 aluminum matrix composites with the mass fraction of single wall carbon nanotubes of 0.00%, 0.05%, 0.10%, 0.15%, and 0.20% were obtained. The microstructure analysis of the specimen was performed using metallurgical microscope. Load-displacement curves of the cermets were conducted using a G200 Agilent Nano-indenteter (Load 1N). Scanning electron microscope (SEM) with energy disperse spectroscopy (EDS) and electro probe micro-analysis (EPMA) were used to analysis second phase.

2.2. Nano indentation testing
The calculation formula of indentation hardness and indentation modulus is as follows:

\[ H_{IT} = \frac{F_{\text{max}}}{A_p} \]  

(1)

\[ F_{\text{max}} - \text{Maximum load, } A_p - \text{Contact projected area under corresponding load.} \]

Equation 2-1 is the general definition of indentation hardness. According to this definition, the indentation hardness here is obtained by dividing the contact load by the corresponding loaded contact area. The calculation formula of reduced modulus is shown:

\[ E_r = \frac{\sqrt{\pi} S}{2\beta \sqrt{A}} \]  

(2)

\[ S \text{ is the slope at the top of the unloading curve, also known as elastic contact stiffness or contact stiffness, and } E_r \text{ is the reduced indentation modulus, } \beta \text{ is a constant related to the indenter geometry [5]. Equation 2-2 is based on elastic contact theory [6-8], but it is also suitable for indenters of other shapes, except } \beta \text{ Different values. } E_r \text{ is used to explain the composite elastic deformation of indenter and sample, and the indentation elastic modulus } E_{IT} \text{ of the tested material can be obtained from } E_r. \]

\[ \frac{1}{E_r} = \frac{1-v_i^2}{E_i} - \frac{1-v_i^2}{E_i} \]  

(3)

\[ v \text{ is the Poisson's ratio of the tested material, } E_i \text{ and } v_i \text{ are the indentation modulus and Poisson's ratio of the indenter respectively. Fig 1 shows that the mechanical work } W_{\text{total}} \text{ indicated during the indentation procedure is only partly consumed as plastic deformation work } W_{\text{plast}}. \text{ During the removal of the test force the remaining part is set free as work of the elastic reverse deformation } W_{\text{elast}}[7]. \]
3. Results

3.1. Microstructure

The microstructure of 7075 aluminium alloy with different SWCNT content were examined by metallurgical microscopy as shown in Fig 2. The grain size of aluminum alloy decreases with the increase of the single wall carbon nanotubes content. This is because the grain growth is inhibited during the sintering of carbon nanotubes. In order to better understand the internal structure of the alloys, the mapping spectrum analysis of 7075 aluminum alloy with 0.15 shows that there may be second phases MgZn2 and Al2Cu as shown in Fig 3. In order to further understand the phase structure, the results of electro probe micro-analysis (EPMA) as shown in Fig 4 and Table 1 can also verify the above inference.
Fig 2. BSE/SEM images showing the microstructure of the 7075 aluminium alloy with different SCNT content (a) 0.0wt%, (b) 0.05wt%, (c) 0.1wt%, (d) 0.15wt%, (e) 0.2wt%.

Fig 3. The mapping of 7075 aluminium alloy with 0.15wt% SWCNTs obtained by Scanning electron microscope: (a) electronic image, (b) Cu Mapping, (c) Ta Mapping, (d) Zn Mapping, (e) Mg Mapping.
5

3.2. Mechanical properties

Basic mechanical properties of nanoindentational experiments of 7075 aluminium alloy are shown in Fig 5-Fig 8. Fig 5 shows the typical force displacement curve, with increasing single-walled carbon nanotubes, the force displacement curve make left, and the indentation morphology are shown in Fig 6. With the increase of the content of single-walled carbon nanotubes, the hardness first increased and then decreased, the indentation depth first decreased and then increased, the elastic modulus first increased and then decreased, and the optimal addition amount was 0.1% as shown in Fig 7. Fig 8 shows that the elastic work increases continuously, and the plastic deformation work decreases first and then increases.

Table 1 EPMA analysis results of point mark in Fig 4.

| Point | Mg    | Zn    | Al    | Cu    |
|-------|-------|-------|-------|-------|
| 2-1   | 4.5354| 6.4827| 87.9180| 1.0640|
| 2-2   | 2.8040| 4.3298| 92.0948| 0.7714|

Fig 5. Typical nanoindentation force-displacement curve A 0.0wt%, B 0.05wt%, C 0.1wt%, D 0.15wt%, E 0.2wt%
Fig 6. Optical micrographs showing the indentation of the 7075 aluminium alloy with different SCNT content (a)0.0wt%, (b)0.05wt%, (c) 0.1wt%, (d)0.15wt%, (e)0.2wt%
Fig 7. effect of SCNT content on the mechanical properties of 7075 aluminium alloy (a) hardness, (b) depth, (c) Young's modulus

Fig 8. Effect of SWCNT content on the energy of 7075 aluminium alloy

Johnson's ECM assumes the process of indentation as spherical shell expansion by a hydrostatic pressure $F$ in a hemispherical “core” of radius $a$ Fig 9\(^{[8]}\). The elastic-plastic boundary lies at a radius $c$. The stress and displacement outside the core are radial symmetry. When the grain deforms under load, the SWCNTs, MgZn2 and Al2Cu near the grain boundary will hinder the direct mutual movement of the grain boundary. Moreover, the dislocations in the crystal move near the grain boundary, which cause by stress concentration. The dislocation encounters the second phase (SWCNTs, MgZn2 and Al2Cu) near the grain boundary, which can obtain the high elastic modulus and strength, more energy is required to balance the deformation energy of the second phase \([9-12]\)
Fig 9. A schematic illustration of stress field with hydrostatic core radius \(a\) and plastic region outer radius \(c\) for conical indentation[8]

4. Conclusions
The relationships between hardness, elastic modulus and the work of 7075 aluminum alloy grains with different single wall carbon nanotubes content were investigated. Grain size of the alloy decreases with the increase of single wall carbon nanotubes (SWCNTs) content, and MgZn2 and Al2Cu can be found. Hardness, young's modulus and the elastic reverse deformation of 7075 aluminum alloy increase firstly with increasing single wall carbon nanotubes, and then decrease obviously with the further increase of single wall carbon nanotubes, while the mechanical work and the plastic deformation work of the alloy have an opposite trend. 7075 aluminum alloy with 0.1wt% single wall carbon nanotubes addition have excellent comprehensive performance. It can be conclude that the SWCNTs, MgZn2 and Al2Cu at the grain boundary obviously changed the strain.

Acknowledgement
The authors would like to acknowledge the support of Jiangsu Province Key R&D Program (Industry Prospects and Common Key Technologies BE2018073) and Fundamental scientific research business fees of central universities (JZ2018HCBZ0130, PA2019GDPK0090)

References
[1] Iijima S, Brabec C, Maiti A, et al. Structural flexibility of carbon nanotubes[J]. The Journal of chemical physics, 1996, 104(5): 2089-2092.
[2] Thostenson E T, Ren Z, Chou T W. Advances in the science and technology of carbon nanotubes and their composites: a review[J]. Composites science and technology, 2001, 61(13): 1899-1912.
[3] Lu J P. Elastic properties of carbon nanotubes and nanoropes[J]. Physical Review Letters, 1997, 79(7): 1297.
[4] Berber S, Kwon Y K, Tománek D. Unusually high thermal conductivity of carbon nanotubes[J]. Physical review letters, 2000, 84(20): 4613.
[5] Amke A , Km B , As A , etal. Fabrication and properties of dispersed carbon nanotube-aluminum composites [J]. Materials Science and Engineering: A, 2009, 508(1-2):167-173.
[6] Zhou W, Yamaguchi T, Kikuchi K, etal. Effectively enhanced load transfer by interfacial reactions in multi-walled carbon nanotube reinforced Al matrix composites[J]. Acta Materialia, 2017, 125: 369-376. [7]ISO14577-1:2015Metallic materials - Instrumented indentation test for hardness and materials parameters - Part 1: Test method
[8]Rong Y, Zhang T, Peng J, et al. Experimental verification and theoretical analysis of the relationships between hardness, elastic modulus, and the work of indentation[J]. Applied Physics Letters, 2008, 92(23):1564.
[9] Chen B, Kondoh K, Imai H, et al. Simultaneously enhancing strength and ductility of carbon nanotube/aluminum composites by improving bonding conditions[J]. Scripta Materialia, 2016, 113: 158-162.
[10] Kim K T, Cha S I, Hong S H, et al. Microstructures and tensile behavior of carbon nanotube reinforced Cu matrix nanocomposites[J]. Materials Science and Engineering: A, 2006, 430(1-2): 27-33.

[11] Shah S P, Mc Garry F J. Griffith fracture criterion and concrete[J]. Journal of the Engineering Mechanics Division, 1971, 97(6): 1663-1676.

[12] Han G Q, Shen J H, Ye X X, et al. The influence of CNTs on the microstructure and ductility of CNT/Mg composites[J]. Materials Letters, 2016, 181: 300-304.