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Experimental investigation on mechanical properties of Multi Wall Carbon Nanotubes (MWCNT) reinforced aluminium metal matrix composites

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The aim of this research is to obtain better micrographs and to perform the mechanical characterization of Multi Wall Carbon Nanotubes (MWCNT) consolidated aluminum matrix composite (AMC). This research is focused on the mechanical behavior and microstructure characteristics in MWCNT consolidated aluminum matrix composites. The composites of MWCNT/Al were successfully fabricated using powder metallurgy (PM) mechanical alloying (MA) sintering. X-ray diffraction (XRD) and Scanning electron microscopy (SEM) micrographs were utilized in order to determine the porosity, density and hardness characteristics to observe if they are affected or not concerning the performance of composites in different reinforced % MWCNT rate conditions. Along with SEM and XRD studies, this study details the homogeneous distribution of nanotubes which showed no evidence of decomposition; this was supported by a successive increase in hardness and abrasion resistance. This increase in abrasion resistance and hardness can be accredited to the further activation of the slip system due to the presence of MWCNTs. Based on the conclusions of this study, the authors determined that the mechanical alloying sintering method provides promising results for the mechanical alloying fabrication of MWCNT/Al composites, especially due to the different % rate reinforced up capabilities.

Keywords: Powder metallurgy, Multi Wall Carbon Nanotubes, Mechanical behaviour.

Introduction

Powder Metallurgy (PM) is a significantly important but also an easy method of fabrication combining shaping of elemental and/or pre alloyed powders after mixing and pressing, then sintering in an atmosphere under control, thus rendering metallurgical bonding of compact powders possible by a cost effective way [1, 2]. Metal Matrix Composites (MMC), which are a combination of reinforcement material(s) and partly metal(s), present superior properties compared to that of a single material could offer.

Upgraded mechanical and physical properties such as wear resistance and thermal expansion coefficients at higher temperatures have attracted attention for several decades, particularly in case of the utilization of particles or whiskers that can be produced by standard metal forming processes [3-7].

Due to their thermal and electrical conductivity, wear resistance and high impact, elevated melting point, chemical corrosion resistance carbon nanotubes are used in various applications, such as sensors, nanocomposites, and transparent electrodes [8-11]. Aluminum MMC, which is part of the new advanced materials, combines high strength with low density, plus exhibits a low thermal expansion coefficient and good wear resistance [12]. Despite these improved features, the application of their use is limited to aerospace and military areas, which could be attributed to their higher processing cost [13-16].

Powder metallurgy production methods are designed to produce qualified engineering materials using aluminum material powder. In this research, CNT and Aluminum materials are chosen as reinforcement and matrix materials respectively. This study aimed to examine and improve the mechanical properties of AMC and identified different process conditions such as temperature, sintering condition, time and atmosphere, production with Al/ CNT type composite material.

Matrix powder and an additional powder wasmixed by attrition milling with the help of a mechanical alloying method. The amount of added powder depended on experimental results and studies. According to the International American Society of Tests and Materials (ASTM) standards and MPIF powder mixture shaped by using uniaxial pressing technique. After the critical temperature determined by Differential Thermal Analysis (DTA), formed wet specimens density were measured in different sintering condition. Then the sintered specimens were examined in terms of their microstructure, chemical and physical properties, size change, XRD

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and SEM analysis, density, hardness and wear resistance tests one by one.

Experimental Details

The pure aluminum and composite made in this research strengthened the MWCNT particles by 2.5% to 7.5% by weight. The materials were manufactured by using a PM technique (Alfa Aesar, Johnson Matthey GmbH & Co. KG). The material used as raw material in this research was atomized aluminum powders with values of 10 µm (99.99% purity, density 2,699 g/cm³). The material used as the matrix material was trading naive aluminum (Al) powder and MWCNT. Alfa Aesar, Johnson Matthey GmbH & Co. MWCNT reinforcing material purchased by KG (USA) has a density of 2.31 g/cm³. The mean particle dimension of MWCNTs powders were about 10-30 nanometers. The variable weights of the MWCNTs were distributed over the Al matrices using a turbula ball mill (Turbula Ch PM 400 MA, Switzerland). The as-received Al powders and the pre-determined weight percentage of MWCNTs were charged into glass vials (100 ml capacity and 40 mm inner diameter) along with stainless steel balls of the same sizes (diameter $(\emptyset) = 20$ mm, combined in a weight ratio of 2:1). The same size of grinding balls was used to ensure that sufficient powder energy was provided to the powder particles during the dispersion process. [17, 18]. All materials used in this research were bought without being purified and replaced.

In the course of the manufacturing composites, the 2.5% to 7.5% by weight MWCNT was strengthened to aluminum by wear milling. Milling time was five hours. Ball/powder weight had a ratio of 2:1. It has a grinding speed of 450 rpm. The ball diameter was 10 mm diameter stainless steel ball. After the produced material was dried at 60 C, 0.5% (wt.) of acrowax was appended to the medium as a duration check agent. Powdered materials were easily pressed with a single axis press at 250 MPa. The selected sintering atmosphere is a high



The longitudinal sections of the specimens were prepared metallographically. In the next step, the microstructures of the composites were evaluated with a LEICA optical microscope. The SEM images were obtained by the specimens surface by scanning it with a high-energy beam of electrons [20, 21]. The powder morphologies and microstructures of sintered specimens were examined using a SEM (JEOL Ltd., JSM-5910LV).

Phase detections of pure Al and MWCNT powders, also composites of different powder compositions were carried out using a Rigaku X Ray diffractometer. There is a Cu/K α radiation with a beam angle of 2°. The angle of refraction has a value between 5°-105°. The step increase is 0.02° and the count time is 2 seconds. Energy Dispersive Spectrometer (EDS, OXFORD Industries INCAx-sight 7274, (133-eV resolution) and Al and MWCNT powders was analyzed.

The hardness measurement of specimens is indeed a means of determination for mechanical properties of the specimens. In this research, Instron Universal Hardness Tester Machine, with the help of Vickers 136° diamond indenter, was used.

The testing load used for every specimen was 100 g. The hardness value of the material refers to the bulk hardness of the composite. This is because both the matrix and the reinforcing material of the recess track are coated. The results of the hardness test were evaluated with approximately 5 consecutive measurements. Display of the hardness measurement specimens as shown in Fig. 2. Vickers images produced at the specimens surface as a result of the measurement made by the hardness measurement method.

Abrasion testing is used to test the abrasive resistance of solid materials. The dimensions of the composite specimens were 16 mm in diameter and 35-45 mm of length. The abrasion testing wheel has a 60 mm diameter and 550 rev/min. It performed that abrasion resistance under dynamic load which different distance used as illustrated in Fig. 3.

Results and Discussion

The powder morphologies and microstructures of sintered specimens were examined using a SEM (JEOL Ltd., JSM-5910LV). The microstructural analysis of pure aluminum and 7,5% strengthened Al/MWCNT composite material are shown in Figs. 4(a) and 4(b).

In these studies, the alpha and beta shows the structure of Al powder which can be seen in Fig. 5(a) and (b). As can be seen by the black and gray areas of the MWCNT structures. Black and gray areas increased



Fig. 1. Sintering process for horizontal pipe furnace system.



Fig. 2. (a) images formed on the specimens surface and (b) magnified view of the specimens surface.



Fig. 3. Abrasion test results of the specimens in different temperatures and CNT ratios.

as the ratio of the MWCNT % increased [22]. EDS analysis of phases are shown in Fig. 5(a) and Fig. 5(b). XRD analysis of phases are shown in Fig. 6.

The X-ray diffraction of specimens with several MWCNT contents, CNT and Al alone is displayed in

Fig. 6. Fig. 6 demonstrates the peak sat 2θ of 36, 38.44, 44.7 and 65 belongs to the Al, and, CNT alone peaks are observed at 26. Moreover, the intensity of the peaks showed an obvious enhancement depending on the MWCNT ratio. All this suggests that increased MWCNT contents lead to an increase in the main peaks of the MWCNT. The selected specimens are MWCNT-reinforced Al powder manufactured by powder metallurgy.

The features and information of these powders are mentioned in the previous section. In addition, the mean density worth of composites including dissimilar amounts of MWCNT is given as shown in Fig. 7. As the temperature of the sintering process increased, the density of the specimens approached the densities in theory [22].

Theoretic densities of the samples were calculated with the help of the following equation [23].

$$1/\rho_c = w_f / \rho_f + w_m / \rho_m \tag{1}$$

Where subscripts c, f, and m, represent the terms



Fig. 4. The MWCNTs reinforced matrix Al SEM images of the composite material. (a): Spherical Al particulate, (b): 1. Al/MWCNT phase and 2. Al composite materials.



Fig. 5. The composite material's (7.5 % content Al/MWCNT) of (a) SEM image, (b) EDS analysis.



Fig. 6. XRD pattern of 2.5-7.5 % content MWCNTs particles in Al matrix, Aluminum and CNT.

composite, fiber (or in the more general case, reinforcement), and matrix respectively.

It is the increasing weight percentage of the MWCNT and the sintering temperature that causes the composite to increase its hardness. With the addition of MWCNT, the distribution of the rise in stiffness of the composites can be correlated to the reinforcing effect.

The results of the abrasion test are as shown in Fig. 3. It is localized as a function of MWCNT, meaning about changing sintering temperatures. Even though

the rise in MWCNT meaning and sintering temperature in the matrix causes an increase in wear resistance, there is little difference between the 2.5% and 5% reinforced MWCNT at 640 C. Fig. 3 shows the weight loss for all specimens as calculated using Eq. (2) and abrasion resistance for all specimens calculated with Eq. (3) [24].

$$Wa = \Delta G/d.M.S$$
(2)



Fig. 7. Density at different % MWCNT content sintering temperatures.

Wa: Percantage of Abrassion, ΔG : Weight Loss, M: Force, S: Distance, d: Density

Aluminum density, wear properties and hardness of the aluminum reinforced MWCNT specimens were examined by a mission of temperature and time.

The results of this research are; the density of the specimens approaches the theoretic density with the rise in sintering temperatures for all dissimilar MWCNT means. Microscopic images displayed that the MWCNT particles were particularly homogeneously dispersed in the matrix, the MWCNT did not dissociate. However, the asset of porosity at the ends of the MWCNT particles is present. Microscopic pictures of the experiments in which MWCNT reinforced aluminum particles are present showed that MWCNT particles is not distributed homogenously. Carbon may have a negative effect with aluminum matrix [25].

Increasing the weight percentage of the MWCNT

 Table 1. Hardness value of MWCNT with different sintering temparature.

% wt MWCNT —	Values of Hardness	
	620 C	640 C
% 2.5 MWCNT	27.8	29.2
% 5 MWCNT	30.8	34.7
% 7.5 MWCNT	34.8	39.1
% 10 MWCNT	37.7	41.1
% 12.5 MWCNT	40.2	43.2
% 15 MWCNT	44.4	48.1
% 17.5 MWCNT	47.6	52.2
% 20 MWCNT	51.8	54.7

and the sintering temperature leads to an increase in the hardness of the composite demonstrated in Fig. 8. Increasing the hardness with extra MWCNT may be connected to the distribution reinforcement effect. The increase in the weight percentage of MWCNT and the sintering temperature resulted in an increase in the wear resistance of the composites. Content of composite has much more effective than the sintering temperature. However, some differences were observed between specimens, 2.5% and 5% MWCNT content which had a 640 °C sintering temperature. The 7.5% MWCNT content specimens have lower abrasion resistance than the others. 2.5% and 5% MWCNT content may be more homogeneously distributed compared to 7.5% MWCNT content. All values are shown in Table 1.

Conclusions

In this study, density, hardness and abrassion properties



Fig. 8. Wear-tested MWCNT reinforced Al/MWCNT composites. (a) 620 C % 2,5 MWCNT, (b) 620 C % 5 MWCNT, (c) 620 C % 7,5 MWCNT, (d) 640 C % 2,5 MWCNT, (e) 640 C % 5 MWCNT, (f) 640 C % 7,5 MWCNT.

of aluminum specimens reinforced with different MWCNT ratios were investigated as a function of temperature and time. The results of this research may be expressed as follows; The composites of MWCNT/ Al were successfully fabricated using a PM sintering process. Approaches the theoretical density with the effect of increasing specimens density and increased sintering temperature with all different MWCNT contents. Microstructural analysis showed the homogeneous distribution of MWCNT particles in the matrix.

In addition, the presence of porosity is present at the ends of the MWCNT particles. The studies carried out in SEM and XRD detailed the homogeneous distribution of nanotubes. As a result of this research, no evidence of decomposition was found. This was further supported by a simultaneous increase in forces, stiffness and ductility. The results showed increased wear resistance in all composites having high nanotube concentration of 5% by weight due to the homogeneous MWCNT distribution in the matrix phases [26]. The simultaneous increase in hardness and wear resistance can be attributed to the activation of more slip systems due to the presence of CINs. Due to the effects on the mechanical features of carbides and composites formed on the surface of MWCNTs, MWCNTs should be evaluated further as they can improve load transfer ability and strength.

References

- R.M. German, in Powder Metallurgy and Particulate Materials Processing (Metal Powder Industry, 2005) p.331-333.
- I. Topcu, H., Gulsoy, N. Kadioglu, and A.N. Gulluoglu, J. Alloys Compd. 482[1-2] (2009) 516-521.
- 3. M.D. Huda and M.S. Hashmi. Key Eng. Mater. 104-107 (1995) 37-64.
- 4. I. Topcu, Tehnicki glasnik 14[1] (2020) 7-14.
- G.M. Dieter, in Mechanical Metallurgy, Second Edition (McGraw-Hill Education, 1988) p.139-362.
- 6. T.-W. Chou, in Microstructural Design of Fiber Composites

(Cambridge University Press, 1992) p. 589-592.

- 7. S. Iijima, Nature 354 (1991) 56-58.
- H.W. Kroto, J.R. Heath, S.C. O'Brien, R.F. Curl, and R.E. Smalley, Nature 318 (1985) 162-163.
- M.M. Schwartz, in Composites Materials Handbook (Mc Graw-Hill Book Company, 1984) p. 651.
- J.M. Berthelot and J.M. Cole, in Composite Materials, Mechanical Behavior and Structural Analysis (Springer, 1999) p.620.
- S. Park, S.W. Choi, C. Jin. J. Ceram. Process. Res. 20[5] (2019) 464-496.
- 12. F.F Komarov, A.M. Mironov, Phys. Chem. Solid. State. 5[3] (2004) 411-429.
- Y. Feng, Hai Long Yuan, Min Zhang, Mater. Charact. 55[3] (2005) 211-218.
- H. Kwon, M. Estili, K. Takagi, T. Miyazaki, A. Kawasaki, Carbon 47[3] (2009) 570-577.
- R. George, K.T. Kashyap, R. Rahul, S. Yamdagni, Scripta. Mater. 53[10] (2005) 1159-1163.
- S. Jain, in Nano -Scale Events with Macroscopic Effects in Polypropylene / Silica (University of Twente, 2005) p.2-4.
- D. Gavrilov, O. Vinogradov, and W. Shaw, in Tenth International Conference on Composite Materials. III. Processing and Manufacturing (Woodhead Publishing, 1995) p.11-17.
- X. Feng, J. Sui, W. Cai, and A. Liu, Scr. Mater. 64[9] (2011) 824-827.
- İ. Topcu, A.N. Güllüoğlu, M.K. Bilici, H.Ö. Gülsoy, J. Fac. Eng. Arch. Gazi Univ. 34[3] (2019) 1441-1449.
- 20. S. Panigrahi, S. Praharaj, S.Basu, S.K. Ghosh, S. Jaha, S. Pande, T. Vo-Dinh, H. Jiang, and T. Pal, J. Phys. Chem. B 110[27] (2006) 13436-13444.
- 21. G. Gül and F. Kurtulmuş, Mater. Sci (Meziagotyra) 24[1] (2018) 104-111.
- K. Dvorak, D. Dolak, D. Paşousek, L. Čelko, and D. Jech. Mater. Sci (Meziagotyra) 24[1] (2018) 29-34.
- 23. F. Huang and L. Shangguan, Mater. Sci (Meziagotyra). 23[2] (2017) 129-132.
- 24. I. Topcu, B., Nilgün, A.N. Güllüoglu, and H. Gülsoy, J. Chem. Soc. Pakistan 42[1] (2020) 70-80.
- 25. R. Wu, Q. Li, L. Guo, and Y.M. Taiyuan, Mater. Sci (Meziagotyra) 23[4] (2017) 317-321.
- M.K. Esawi Amal and A. El Borady Mostafa, Compos. Sci. Technol. 68 (2008) 486-492.