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DETERMINATION OF THE MECHANICAL PROPERTIES OF Al/MWCNT COMPOSITES OBTAINED WITH THE REINFORCEMENT OF CU-COATED MULTIWALL CARBON NANOTUBES (MWCNTs)

DOLOČANJE MEHANSKIH LASTNOSTI Al/MWCNT KOMPOZITOV, OJAČANIH Z BAKROM OPLAŠČENIMI VEČSTENSKIMI OGLJIKOVIMI NANOCEVČICAMI (MWCNT)

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In this study, Al/MWCNT composites were produced by milling copper-coated MWCNTs in small amounts (2.5–7.5%) with aluminum powders using the mechanical alloying method. Then, sintering was applied to the composite material. A phase analysis and microstructure investigations were performed on the as-sintered samples. A hardness test was conducted and tribological performances of the unreinforced and MWCNT-reinforced composites were examined at room temperature. Results showed that the hardness of the base-aluminum alloy was improved. The phase belonging to the MWCNTs was detected with an X-ray diffraction analysis. The wear rate generally decreased with the addition of MWCNTs in all test conditions. Sintering had a positive effect, enhancing the wear and hardness behaviour. Abrasive, adhesive, oxidative and thermal wear mechanisms were observed with a scanning electron microscope (SEM).

Keywords: powder metallurgy, MWCNT, mechanical behaviour, Cu coating

Avtor v članku opisuje študijo, v kateri so izdelali vzorce kompozitov tipa Al/MWCNT, ojačane z majhno vsebnostjo (od 2,5 *w*/% do 7,5 *w*/%) ojačitvene faze in določili njihove mehanske lastnosti. Za ojačitev kovinske osnove iz 99,9 % Al so uporabili delce z bakrom oplaščenih večstenskih ogljikovih nanocevk (MWCNT; angl.: multi-wall carbon nano-tubes). Kompozitne prahove so izdelali s postopkom mehanskega legiranja ter nato njihovega zgoščevanja z enoosnim stiskanjem in sintranjem. Na sintranih vzorcih kompozitov so nato izvedli mikrostrukturne in fazne analize. Pri sobni temperaturi so izmerili še njihovo trdoto in izvedli tribološke raziskave. Rezultati testov mikrotrdote so pokazali, da se je le-ta izboljšala z dodatkom ojačitvene faze. Z rentgensko difrakcijsko spektroskopijo (XRD) so zaznali prisotnost MWCNT v kovinski osnovi. Pri vseh pogojih preizkušanja se je v splošnem hitrost obrabe zmanjšala z dodatkom MWCNT delcev. Zgoščevanje kompozita s sintranjem je imelo pozitiven učinek na zvišanje njegove trdote in izboljšanje odpornosti proti obrabi. S pomočjo vrstičnega elektronskega mikroskopa (SEM) so opazovali abrazivne, oksidativne in termične učinke mehanizmov obrabe.

Ključne besede: metalurgija prahov, MWCNT, mehanske lastnosti, oplaščenje z bakrom

1 INTRODUCTION

Aluminum and aluminum alloys receive great attention in the aerospace and automobile sectors due to their density. Aluminum alloys are known as heat-treatable materials, in addition to exhibiting high stiffness and strength properties.[1] Researchers generally try to use them as a matrix when developing high-strength composite materials in order to meet the demands of the industry.

Multiwall carbon nanotubes have become a popular reinforcement for metal-matrix composites because of their superior thermal, electrical and mechanical performance. They have an elastic modulus of 1 TPa and strength of 30–100 GPa.[2,3] MWCNTs are lightweight and have a large aspect ratio. When MWCNTs are incorporated into a matrix material, they significantly increase the mechanical and thermal properties. The main challenge is to obtain a homogenous MWCNT distribution in a matrix composite due to the strong Van der Waals attraction energy between carbon atoms.[4] For this purpose, different production techniques have been used so far. In general, powder metallurgy is used as it allows us to attain high-density materials. It was reported that high-energy ball milling can be effective for homogenous distribution, but after a certain milling time, it might cause serious damage to the MWCNTs.[5] Although an agglomeration of the reinforcement particles is prevented, this technique can cause extra heat, interfacial reactions and damage to the integrity of carbon.

Nowadays, chemical methods, surface modification or ultrasonication of MWCNTs in aqueous solutions, known as semi-powder techniques are tried to fabricate nanocomposite materials. A study indicates that semi-powder metallurgy can be practical for obtaining

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homogenously embedded carbon-based nano-reinforced composites with no defects. On the other hand, some small agglomerations were observed for MWCNTs compared to graphene and fullerene because of the structural properties of one-dimensional MWCNTs.[6] In recent years, friction-stir processing, spark-plasma sintering and stir-casting methods have been used for the fabrication of nanocomposites. A melted aluminum ingot is mixed with nano-reinforcement with a stirring bar in a stir-casting process, then the melted mixture is poured into a die and it is solidified. It is possible to produce large and complex-shape products, but the reinforcement phase in the matrix cannot be uniform.[7]

Al-based MWCNT-reinforced composites are fabricated with the above techniques and their mechanical properties are examined in the literature. Generally, the effect of MWCNTs or their amount during a room-temperature mechanical performance on aluminum were investigated. But few studies can be found about the high-temperature mechanical or tribological behaviour of such composites.[8] A multi-walled carbon nanotube-reinforced aluminum-alloy-matrix composite was produced with a newly developed technique, which combines semi-powder metallurgy and stir casting with an induction furnace for the first time. Furthermore, to the best of our knowledge, there is no study about the effect of carbon nanotubes on the wear properties of aluminum at elevated temperatures. Both the room and high temperature wear performances of the produced composite are investigated in detail in this report.

Powder-metallurgy production methods were designed to produce qualified engineering materials using aluminum powder. In this study, Cu-coated MWCNTs and aluminum materials chosen as the reinforcement and matrix were used. The aims of the work were to examine and improve the mechanical properties and determine different process conditions such as the temperature, sintering condition, time, atmosphere and use of the Al/CNT type composite material.[9]

Matrix powder and additional powder were mixed using attrition milling with the help of the mechanical-alloying method. The amount of added powder depended on the experimental results and studies. In accordance with ASTM standards, the MPIF powder mixture was shaped using the uniaxial pressing technique. After the critical temperature, determined with a DTA analysis, the wet-sample density was measured in different sintering conditions. Then we examined the microstructure, chemical and physical properties and size change of the sintered samples, using XRD and SEM analyses as well as density, hardness and wear-resistance tests.

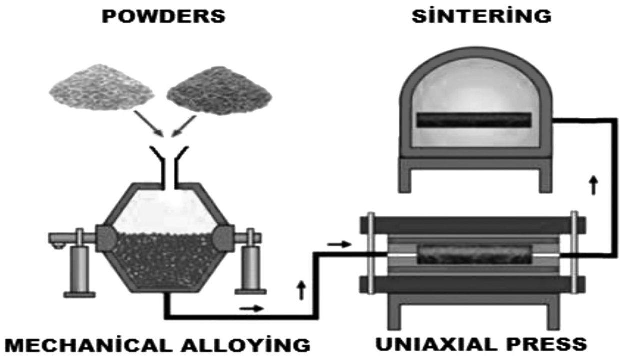

Figure 1: Fabrication stages for the MWCNT-reinforced Al-matrix composite

2 EXPERIMENTAL PART

2.1 Raw materials and fabrication

Aluminum powder (99.9 % purity, 2.690 g/cm^3 density, a powder size of 10 μm) and MWCNT powders (a density of 2.31 g/cm^3, size of 10–30 nm, from Nanografi Co. Ltd.) were used to produce a composite in this investigation. **Figure 1** illustrates the fabrication of the 2.5 *w*/% MWCNT-reinforced Al-matrix composite.

Firstly, the experiment aimed to obtain Cu-coated MWCNTs, made from $CuSO_4$. 2290 mg of $CuSO_4$, 200 mL of ethanol, 200 mL of diethylene alcohol and 400 mg of carbon nanotubes, batched and mixed with magnetic stirring at 200 °C. The Cu-coated multi-walled carbon nanotubes (MWCNTs) were prepared with electrodeposition using MWCNTs in an acid-copper plating bath under optimised bath conditions and process conditions. The structural and mechanical properties of the Cu–MWCNT thin film were investigated.[10]

Secondly, different amounts (2.5–7.5 %) of Cu-coated MWCNTs and aluminum particles were mechanically alloyed in a Turbula mixer at 450 min^{-1} for four hours.[11,12] The agitation of a powder (especially powders with different bulk densities) may result in a migration of smaller particles downwards and larger ones upwards. Another problem is the segregation whose main cause is the difference in the particle size, density shape and resilience. The Turbula device used is a Kenics-type static mixer. Static mixers save energy, prevent segregation and influence particle migration.[13]

Powders were pressed at 200 MPa, then sintered in a high-purity argon atmosphere at 600 °C and 650 °C for one hour. The horizontal-tube furnace arrangement of the sintering process is shown in **Figure 2**.

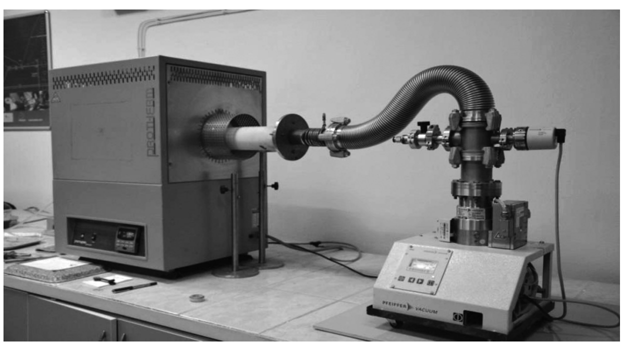

Figure 2: Sintering process of a horizontal-tube furnace system

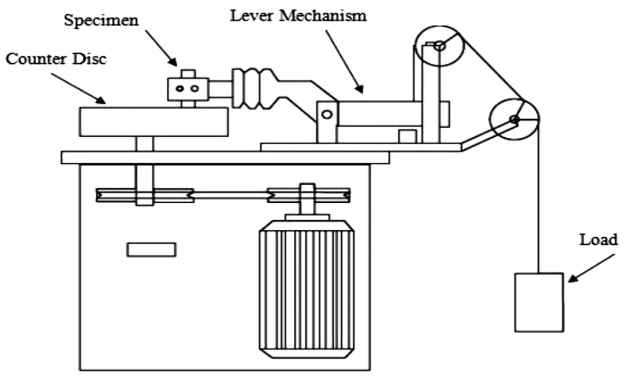

Figure 3: Schematic of the pin-on-disc wear device

2.2 Characterization of the fabricated composites

The sintered samples were prepared for microstructure and phase analyses. For this purpose, grinding (180–1200 SiC grit paper) and polishing (3- and 1-micron diamond solutions, respectively) were applied. An X-ray diffraction machine (Rigaku Ultimate) was operated to observe the present phases in the material. Then the samples were etched with a Keller solution for microstructural investigations. Scanning electron microscopy using energy dispersive X-ray spectroscopy (EDX) was used to observe the microstructure of both the as-cast and extruded samples.

Dry-sliding wear tests were carried out using a pin-on-disk device (a UTS tribometer, ball-on-disc test apparatus, as shown in **Figure 3**).

The 52100 steel was chosen as the counter-face material. The wear tests were conducted under a normal load of 10 N at room temperature. To determine the wear rate, the wear volume of the samples was calculated and this value was multiplied by the stroke distance. Then, the calculated values were divided by the sliding distance in order to obtain the units in mm^3/m. Scanning electron microscopy was used to display worn surfaces and understand the wear mechanisms of the samples versus the applied load and test temperature.

The hardness measurements of the Al/MWCNT samples were measured on a Future Tech-700 microhardness testing machine using a Vickers 136° diamond tip and 50-g test load. The hardness value indicates the hardness of the composite. The hardness-test results are the average of 10 consecutive measurements.[2,12]

Figure 4 shows the Vickers images produced at the sample surface as a result of the measurement made with the hardness-measurement method.

Abrasion testing is used to test the abrasive resistance of solid materials. The dimensions of the composite samples included a diameter of 13 mm of and length of 30–40 mm. The abrasion testing wheel had a diameter of 75 mm and rotation rate of 520 min^{-1}. It showed that the abrasion resistance under a dynamic load changes with distance.

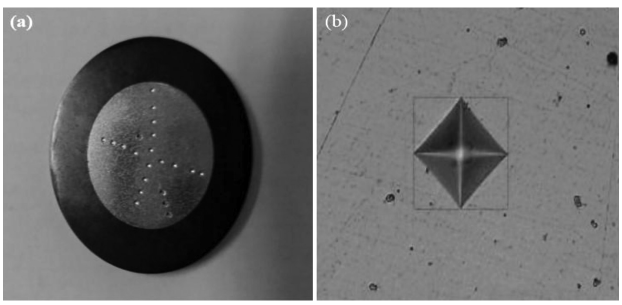

Figure 4: a) image of the sample surface and b) a magnified view of the sample surface

3 RESULTS

3.1 X-ray diffraction

X-ray diffraction results from **Figure 5** show the X-ray diffraction patterns of the sintered MWCNT-reinforced composite. An X-ray diffraction machine with a fixed monochromatic filter was operated in a range of 10–90° at 40 kV and 40 mA. Four obvious peaks corresponding to the Al reflections can be seen on the graph. It may be concluded that the MWCNT addition can react with the matrix material and new phases, such as Al_4C_3, which may weaken the bonding between the aluminium alloy and reinforcement, may occur.[11,12] The XRD analysis of the phases is shown in **Figure 6**.

However, no intermetallic phases were detected with the measurement. Because the reinforcement based on a low amount of carbon was added to the matrix, Al4C3 could not be observed. During continuous scanning in the range of defined angles, the MWCNT peak could not be seen. This can be attributed to the fact that MWCNTs might get amorphized during production stages. It can be claimed that carbon peaks may overlap with strong intensity peaks of the Al solid solution phase.[13] Furthermore, previous studies showed that a low amount of carbonaceous reinforcement may be below the detection limit.[6,14] In this study, the fixed-time method, i.e., very slow scanning in defined ranges was used to reveal the presence of MWCNTs in the composite material. It is known that commercial MWCNTs generally have a

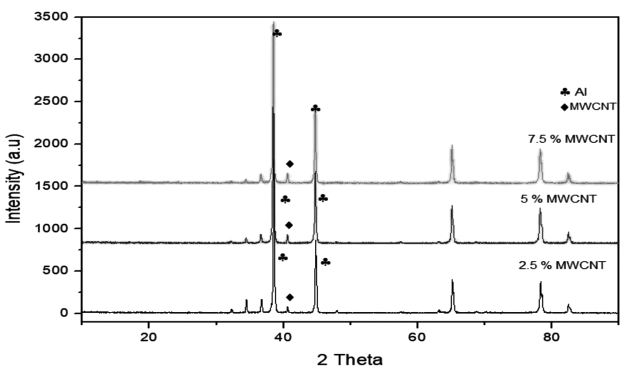

Figure 5: XRD patterns of 2.5–7.5 % MWCNT particles in the Al matrix

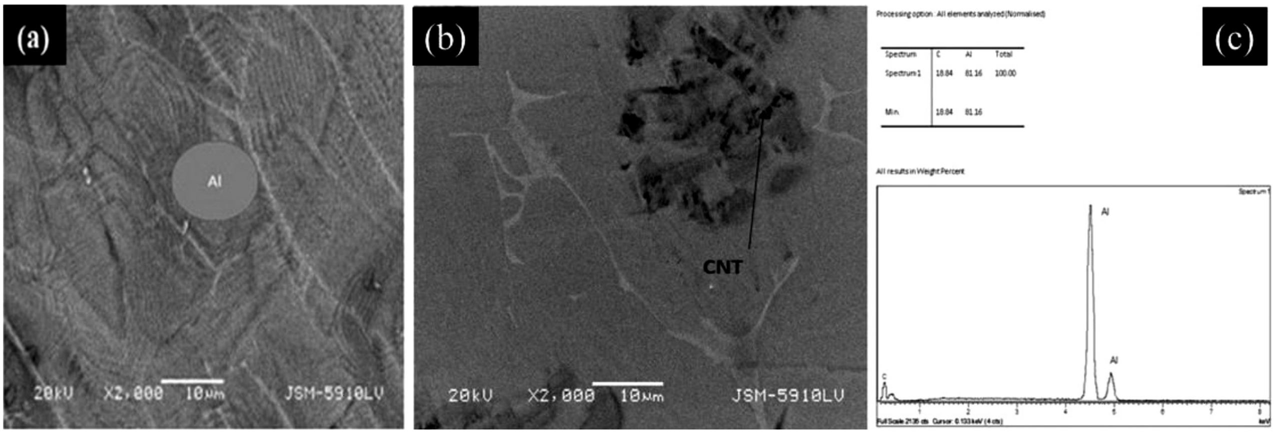

Figure 6: MWCNT (7.5 %) reinforced Al matrix: a) pure Al, b) SEM image, c) EDS analysis

strong peak at 2θ equal to 40–50°.[15] The counting time and step width were determined as 120 s and 0.05°, respectively, for 2θ of 42.6°. The highest peak obtained belonged to the MWCNTs at around 2θ of 42.6°. As shown in **Figure 5**, this peak indicates the existence of MWCNTs in the material. **Figure 6** shows the XRD analysis results for the samples.

3.2 Microstructure analysis

Sintering changes the grain structure and orientation. Grain refinement can be obtained with sintering, which provides the enhancement of the mechanical behaviour of a composite. MWCNTs cannot be recognized from the figure due to their high surface area and low amount. No cracks or macroporosity can be seen on the composites.

The powder morphologies and microstructures of the sintered samples were examined using a SEM (JEOL Ltd., JSM-5910LV). Microstructural analyses of pure aluminium and the composite of 7.5 % MWCNT/Al are shown in **Figures 6a** to **6c**. The structure of the composite material (7.5 % of MWCNT) is given in **Figure 6b** as a representative image. It consists of black and grey areas where the former represents the MWCNT structure and the latter is the Al matrix. The black area became larger as the MWCNT content increased. Additionally, the EDS analysis of the phases shown in **Figure 6c** demonstrates the presence of C and Al.

The microscopic examination showed that the MWCNT powders were distributed homogenously in the matrix and that there was no segregation in the copper-coated MWCNTs.[16]

3.3 Density and hardness results

The density of the composites reinforced with different amounts of MWCNTs is shown in **Figure 7**. The sample density approached the theoretical density with the increasing reinforcement amount and sintering temperature.[17]

Theoretical densities of the composite specimens were calculated with the following equation:[12,18]

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

where subscripts *m*, *f* and *c* refer to the matrix, reinforcement fibres and composite, respectively.

This reduction in the grain size and a uniform dispersion of Cu-MWCNTs in the Al matrix obtained

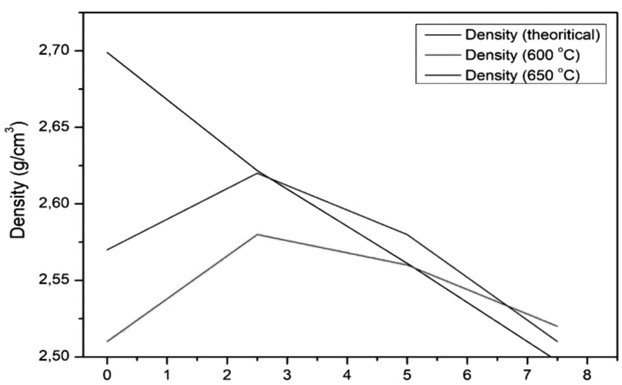

Figure 7: Density of the Cu-coated samples at different sintering temperatures

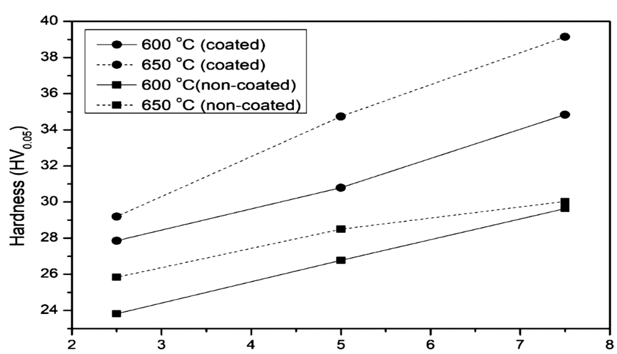

Figure 8: Hardness vs. CNT amount for the coated and non-coated samples

with the electrodeposition method was also confirmed by the XRD and EDS studies. The Vickers-hardness tests were carried out on different composites and at two different sintering temperatures. Ten hardness measurements were taken in different regions of the samples. The hardness values are shown in **Figure 8**.

Increasing the weight percentage of MWCNTs and the sintering temperature increased the hardness of the composites. The increase in the hardness of the composites due to the addition of MWCNTs can be attributed to the dispersion of the strengthening effect. The hardness-test results revealed that the MWCNT addition increases the hardness of the aluminium alloy. The hardness increased as a result of sintering the aluminium-matrix composites produced with 2.5–7.5 % of MWCNTs.

This can be based on the presence of harder reinforcements in the aluminium alloy. MWCNTs probably affect the dislocation motion, especially in the grain boundaries and an improvement in the material strength might be obtained.[19] Therefore, as a result of the restriction in the dislocation with the MWCNT reinforcements, the hardness begins to increase. This is due to the grain-size reduction in the Cu–MWCNT/Al composite. Such a very small crystalline size of pure Al and Cu–MWCNT/Al composite causes an increase in the microhardness of the composite.[10]

3.4 Abrasion tests of the produced samples

In **Figure 9**, abrasion-test results are plotted as a function of the MWCNT amount for different sintering temperatures. The weight loss for all the samples was calculated with Equation (2) and the abrasion resistance for all the samples was calculated with Equation (3).[20]

$$W_{\mathrm{a}} = \frac{\Delta G}{d \cdot m \cdot S} \tag{2}$$

$$W_{\mathrm{r}} = \frac{1}{W_{\mathrm{a}}} \tag{3}$$

W_{a}: percantage of abrassion, ΔG: weight loss, M: force, S: distance, d: density

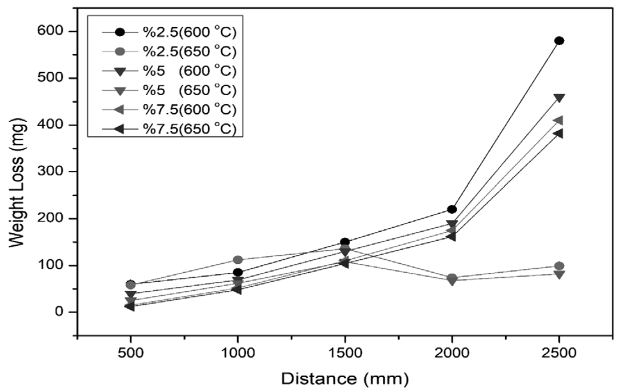

Figure 9: Display of the wear rates of composites with different reinforcements

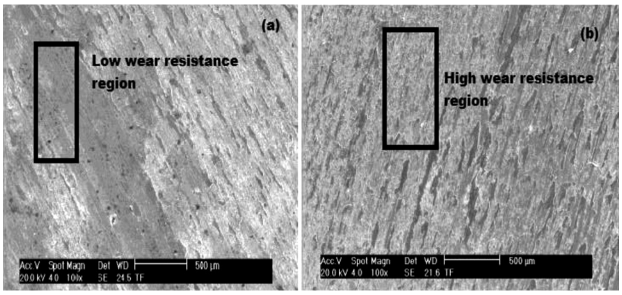

Figure 10: Worn surfaces of the produced samples: a) 2.5 % MWCNT/Al wear resistance, b) 7.5 % MWCNT/Al wear resistance

Increasing the weight percentage of the MWCNTs and the sintering temperature increased the abrasion resistance of the composite. The composite amount is much more effective than the sintering temperature. However, there are some differences between the samples: those with 2.5 % and 5 % of the copper-coated MWCNTs were sintered at 650 °C; the samples with 7.5 % of MWCNTs exhibited a lower abrasion resistance than the others.

Low-reinforcement samples exhibited the lowest wear rate under the load of 10 N. When the time increased, the wear began to increase. After the applied load, there was a dramatic increase in the wear rate, especially for the low-reinforcement alloy. Among the samples, the one exposed to a high sintering temperature and with a high reinforcement showed the best wear performance. The wear behaviour improved as the Cu-MWCNT/Al composite obtained a higher relative texture than pure aluminum.[21]

Once the as-sintered and reinforced composites are evaluated, the grain-size refinement and internal porosities might help us to determine the wear behaviour. Furthermore, the compressive residual stress, formed during the fabrication process affects the wear performance.[22,23] When the compressive residual stress increases, the mechanical and wear behaviour of the composite begin to develop, preventing the micro-crack growth. When a sample is sintered at a high vacuum, a compressive residual stress begins to occur.[24,25]

The SEM was used to understand the wear phenomenon at different temperatures of the sintered MWCNT-reinforced composite. As shown in **Figure 10**, the effects of different reinforcement rates (2.5–7.5 % of MWCNTs) and test temperatures on the wear mechanism were investigated. Obvious marks can be seen on the figure and they are parallel to the sliding direction. This is a result of the abrasive-wear mechanism. These marks are more clearly recognized when the applied load increases due to the higher pressure of the load on the contact surface of the sample.

4 DISCUSSION

As mentioned above, the sample with copper-coated MWCNTs has a higher hardness than the low-reinforcement sample. It is noted that an enhancement of the hardness improves the load-bearing capacity of the matrix material so that the wear resistance of the alloy increases.[26,27]

The wear behaviour of the sintered MWCNT-reinforced composites with different amounts of reinforcement (2.5–7.5 %) was studied. Low-reinforcement samples exhibit the lowest wear rate under the load of 10 N. When the applied distance and time increase, the wear rate begins to increase. There is a dramatic increase in the wear resistance, especially for the 7.5 % MWCNT-reinforced alloy. Among the samples, the highly reinforced composite sintered at a high temperature shows the best wear performance. Once the as-sintered composites are evaluated, the grain-size refinement and internal porosities might help determine the wear behavior.[28]

5 CONCLUSIONS

A Cu-MWCNT reinforced Al-matrix composite was fabricated and sintering was performed successfully. Based on the experimental tests, the following findings were obtained:

- No intermetallic phases such as Al_2Cu and Al_4C_3 were found according to the XRD analysis.
- The Al/MWCNT composite has a higher hardness versus wear resistance compared to the unreinforced aluminum.
- The wear rate significantly decreases with an addition of MWCNTs.
- The weight loss increases directly with the applied time and distance.
- The abrasive, adhesive and oxidative wear are the main mechanisms at room temperature.
- A homogeneous distribution of the MWCNTs in the matrix increases the load-transfer ability due to the formation of carbide on the material surface.[29]
- The Cu–MWCNT/Al composite show higher microhardness and wear properties than pure aluminum.

Acknowledgments

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