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# Effect of Sintering Time on Hardness and Wear Behaviours of Carbon Nanotubes Reinforced Aluminium Matrix Composites

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In this study, the effects of sintering time on hardness and wear behaviour were investigated of carbon nanotubes reinforced aluminium matrix composites. 1% multi wall carbon nanotubes (90% purity with 9.5 nm in diameter, 1.5  $\mu$ m in length) and gas atomized 7075 Al alloy powders were mechanical milled for 120 min in a planetary ball mill. Mechanical milled aluminium composite powders were cold pressed under 520 MPa. Pre-shaped samples were sintered in atmosphere controlled furnace at 580 °C for three different sintering times (1, 2, and 3 h). As a result of study, it was observed that the hardness values of composites were decreased with increasing sintering time and the weight loss was decreased. It was determined from worn surface SEM images that adhesive wear mechanisms were dominant.

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### 1. Introduction

Al matrix composites are used in a number of fields including primarily automotive and space industry due to having low density, high toughness, and high corrosion resistance [1–3]. Aluminum composites are produced with addition of SiC, Al<sub>2</sub>O<sub>3</sub>, TiB<sub>2</sub>, B<sub>4</sub>C ceramic particles into Al alloys as reinforcement element have profoundly high strengths and wear resistances [4–6]. Along with the discovery of carbon nanotubes (CNT) recently, these materials have been used as reinforcement phase in metal matrix composites (MMC) due to having high elasticity modulus (600–1100 GPa) and high tensile strength (between 35 and 110 GPa) [7, 8]. As wettability of CNTs is low and these forms weak interbond with metal, it is very hard to produce CNT-reinforced composited with conventional casting method. On examination of previously conducted studies, it is understood that CNTreinforced composites are produced with mechanical alloying/mechanical milling (MA–MM) as a powder metallurgy method [9–11]. In this method, metal powders and carbon nanotubes as the reinforcement phase is mechanically milled, thus producing composite powders. The produced composite powders are shaped in a mould, and composite piece production is performed. The greatest problem encountered in production of CNT-reinforced composites is the difficulties in distribution of reinforcement phase within structure uniformly [12–14]. Shaping and sintering procedures of composite powders produced with mechanical milling method are the most important stages determining mechanical features of the composites produced. Parameters such as sintering temperature and sintering time affect microstructure, density and strength of the composite produced [15]. Hence, in this study, an effect of different sintering times in composites produced with addition of CNT in an amount of 1% into AA7075 alloy with mechanical milling procedure on microstructure, rigidity and wear resistance of the composites are evaluated.

## 2. Material and method

The chemical composition of gas-atomized (GA) AA7075 alloy (diameter of mean particule size 124  $\mu$ m) used as the matrix material in experimental studies is given in Table I. Multi-walled carbon nanotube (MWCNT) with a purity of 90%, a diameter of 9.5 and a length of 1.5  $\mu$ m supplied from Nanografi company was used as the reinforcement phase.

#### TABLE I

Chemical composition of AA7075 alloy used as the matrix material.  $\ensuremath{\mathsf{C}}$ 

Zn	Mg	Cu	Fe	Si	$\mathbf{Cr}$	Mn	Zr	Al
5.480	2.596	1.568	0.549	0.403	0.012	0.014	0.03	bal.

CNT reinforcement element was mechanically milled for 400 rpm in 0.2 ml ethanol in stainless steel grinding cell in order to prevent agglomeration. Afterwards, GA AA7075 powders were mechanically milled with stearic acid of 1% (as process control chemical) with the aim of preventing cold welding and agglomeration in the grinding cell. In the mechanical milling procedures, 2 h of milling period, stainless balls of a dimension of 8 mm, ball/powder ratio of 10:1 and vessel fullness ratio of 50% were used. With the aim of preventing heating of the powders during the mechanical milling procedure, 10 min of time was allowed for standing after each 20 min grinding. AA7075-CNT composite powders produced with mechanical milling were cold pressed under 520 MPa load and

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then hot pressed under 2 MPa at 300 °C for 1 h, and small cylindrical samples of a diameter of 12 mm and height of 7 mm were obtained. These pre-shaped samples were sintered for three different times (1, 2, and 3 h) under  $10^{-6}$ millibar vacuum at 580 °C. After sintering procedures, the samples prepared with standard metallographic procedures were etched for 50-60 s with 2 ml of HF, 5 ml of HNO<sub>3</sub>, 3 ml of HCl solution. The etched samples were examined with scanning electron microscope (SEM). Hardness values were determined by averaging the measurements from 10 different areas of each sample in a Shimadzu micro-hardness device (0.2 N load). Wear tests were carried out in standard pin-on disc type wear devices in accordance with ASTM: G99-05 standards.  $1 \text{ ms}^{-1}$ sliding speed, 30 N loads and five different sliding distances (500–2500 m) were used in wear tests. Abrasive disc and sample surfaces were cleaned with acetone prior to each test. Worn samples were weighed in precision balance and weight losses were determined. AISI 4140 steel disc of a diameter of  $\phi 230$  mm, thickness of 20 mm and hardness of 60–64 HRC was used as counter material in wear tests. The worn sample surfaces were examined with SEM after completion of the wear tests.

## 3. Results and discussion

## 3.1. Microstructural characterization

SEM images of composites produced by adding CNT of amount of 1% to AA7075 alloy and sintered for three different periods of time at 580 °C are given in Fig. 1.

On examination of SEM images given in Fig. 1, it was seen that CNTs in the samples sintered for 1 h (a) exhibited a uniform distribution within the structure while there was agglomeration in particularly particle boundaries. It was understood that the samples sintered for 2 h and 3 h had more agglomeration in both within particles and on particle boundaries (b,c). Moreover, it was seen that Al particles decreased as sintering period increased. SEM images obtained at higher magnification for clear vision of CNTs in samples sintered for different periods of time are shown in Fig. 2.

On examination of SEM images obtained at higher magnifications, it was seen that CNTs were seen as network in samples sintered for 1 h (a) while shape of CNTs aggregated on particle boundaries with the increase of sintering period had thick, short and needle-like structure. This situation may be explained with formation of stronger bonds between CNTs with the increase in sintering process (b,c). Also, as sintering period prolonged, formation of CNTs  $\alpha$ -Al particles forming a stronger bond is prevented, and thus  $\alpha$ -Al particles in the samples sintered for 3 h may be said to be smaller.

#### 3.2. Hardness results

Hardness changes of composites produced with addition of CNT in amount of 1% and sintered for three different periods of time at 580 °C are shown graphically in Fig. 3.



Fig. 1. SEM images and EDS results of samples sintered for different periods of time. (a) 1 h sintered, (b) 2 h sintered, (c) 3 h sintered.

On examination of hardness results given in Fig. 3, the highest hardness value was measured in samples sintered for 1 h while decrease in hardness values was seen as sintering period increased. This decrease seen in hardness was also seen in microstructure of SEM images given in Figs. 1 and 2, and CNTs agglomerated on particle boundaries welded into each other along with the increase in sintering period and formed a stronger bond between each other (due to formation of thicker structures). Thus, dimension of CNTs as the reinforcement phase increased. It is believed that this increase agglomeration on particle boundaries affects hardness results negatively. This relationship can be explained with Orowan mechanism. As dislocations cut these larger particles more easily, it may be said that a decrease in hardness is seen due to the inability for full conductivity of the force applied on the matrix to the reinforcement phase. This situation is caused by the fact that the intersurface bonds formed between the CNTs and the matrix (Al) are weak although contact area between the matrix and the reinforcement phase increased as a result of increase in dimension of the reinforcement phase.

#### 3.3. Wear test results

Weight losses and wear rates of composites produced by adding CNT of an amount of 1% into the AA7075 alloy at the end of wear tests and sintered for different periods of time at 580 °C are shown in Fig. 4.



Fig. 2. SEM images at higher magnifications of samples sintered for different periods of time. (a) 1 h sintered, (b) 2 h sintered, (c) 3 h sintered.



Fig. 3. Hardness changes of aluminum composites sintered for different periods.

As can be understood from graphics given in Fig. 4, the highest weight loss was obtained with samples sintered for 1 h at the end of wear tests followed by samples sintered for 2 h and 3 h (a). Also, on examination of wear rates the results obtained are understood to be compatible with weight loss results. However, an incompatibility



Fig. 4. Weight losses and wear rates of composites sintered for different periods of time. (a) weight loss, (b) wear rate.

can be seen between hardness results and weight loss and wear rates obtained at the end of wear tests (b). At the end of wear test, the sample with the lowest hardness value was expected to have the highest weight loss while the lowest weight loss was obtained in these samples. For clear explanation of this situation, surfaces of worn samples are examined with SEM.

## 3.4. Worn surface examinations

Wear surface SEM images of composites produced with 1% CNT addition into AA7075 alloy at the end of wear tests and sintered for three different periods of time at 580 °C are given in Fig. 5.



Fig. 5. Wear surface SEM images of aluminum composites sintered for three different periods of time: (a) 1 h sintered, (b) 2 h sintered, (c) 3 h sintered.

On examination of wear surface SEM images given in Fig. 5, it was seen that particles were separated from surfaces of samples sintered for 1 h and local oxidations were formed on the surface (a). Moreover, excessive plastic deformation is also seen formed due to cutting effect during friction on wear surface. On examination of surfaces of samples sintered for 2 and 3 h (b,c), it was understood that particles separated from the samples readhered onto the surface with the effect of friction during wear so hardness of these samples was lower. Moreover, active wear mechanism can also be stated as adhesive wear mechanism. Hence, it is thought that weight loss of the composites sintered for 2 and 3 h is lower than the composites sintered for 1 h.

## 4. Conclusion

In this study examining the effect of sintering period time of composites produced with 1% CNT addition into AA7075 alloy, pre-shaped after 2 h of mechanical milling and sintered for three different periods of time at 580 °C on microstructure, hardness and wear behaviour were examined. The following results were obtained:

- Along with the increase in sintering period, CNTs condensed on particle boundaries were more agglomerated.
- The more the sintering period increased, the more hardness values decreased.
- On examination of wear surfaces, it was observed that adhesion onto the wear surfaces increased with the increase of sintering period and adhesive wear mechanism was active.

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