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THE EFFECT OF SINTERING TEMPERATURE ON THE HARDNESS AND WEAR TESTS OF ALUMINUM MATRIX COMPOSITES REINFORCED WITH MWCNT NANOPARTICLES

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Abstrak

Penelitian ini bertujuan untuk mengetahui pengaruh variasi temperatur sintering terhadap sifat mekanik komposit Al-MWCNT yang dibuat dengan metode metalurgi serbuk. Matriks yang digunakan adalah aluminium seri AA1100 dengan penambahan 1% nano Multi-Walled Carbon Nanotube (MWCNT). Campuran dipadatkan dalam cetakan baja, kemudian disinter pada suhu 450°C, 500°C, 550°C, dan 600°C, serta dibandingkan dengan spesimen tanpa sintering. Hasil penelitian menegaskan bahwa sintering pada suhu 500°C memberikan hasil paling optimal, ditunjukkan dengan peningkatan signifikan pada nilai kekerasan sebesar 50,05 HV dan ketahanan aus dengan laju keausan hanya 0,4387 mm³/s, lebih baik dibandingkan spesimen yang tidak disinter. Analisis EDS membuktikan bahwa peningkatan ini terkait dengan rendahnya kandungan oksigen pada suhu 500°C, sementara analisis SEM menunjukkan ikatan antarpartikel yang lebih rapat dan merata. Sebaliknya, sintering pada suhu di atas 500°C justru menurunkan kualitas material akibat terbentuknya retakan dan rongga. Temuan ini secara jelas mengonfirmasi bahwa peningkatan kekerasan dan ketahanan aus pada material dicapai melalui kondisi sintering optimal pada suhu 500°C.

Kata kunci: AMC, MWCNT, Metalurgi Serbuk, SEM-EDX, Uji Kekerasan, Uji Keausan.

Abstract

This study aims to investigate the effect of sintering temperature variations on the mechanical properties of Al-MWCNT composites produced by the powder metallurgy method. The matrix used was AA1100 aluminum with the addition of 1% nano Multi-Walled Carbon Nanotube (MWCNT). The mixture was compacted in a steel mold and sintered at temperatures of 450°C, 500°C, 550°C, and 600°C, and the results were compared with non-sintered specimens. The findings confirm that sintering at 500°C provides the most optimal results, as shown by a significant increase in hardness of 50.05 HV and improved wear resistance with a wear rate of only 0.4387 mm³/s, which is superior to the non-sintered specimens. EDS analysis revealed that this improvement is related to the lower oxygen content at 500°C, while SEM analysis showed tighter and more uniform particle bonding at this temperature. In contrast, sintering at temperatures above 500°C reduced material quality due to the formation of cracks and surface voids. These results clearly confirm that the increase in hardness and wear resistance of the material can only be achieved at the optimal sintering condition of 500°C.
Keyword: AMC, MWCNT, Powder Metallurgy, SEM-EDX, Hardness Test, Wear Test.

INTRODUCTION

Aluminium Matrix Composites (AMCs) have garnered significant research attention due to their superior mechanical and physical properties compared to traditional aluminium alloys. Incorporating metal or ceramic reinforcements, in the form of particles or fibers, into the aluminium matrix can result in enhancements in tensile strength, hardness, wear resistance, and dimensional stability at elevated temperatures[1]. These composites are widely utilized in automotive, aerospace, biotechnology, electronics, and military industries, where high strength-to-weight ratios, excellent corrosion resistance, and reliable performance at high temperatures are critical[2]. Aluminum metal matrix composites are formed by dispersing reinforcement in the metal matrix, reinforcement is used to improve the mechanical properties of the metal matrix such as hardness, strength, temperature holding capacity, wear resistance, environmental influences, density, porosity, etc[3][4].

Widely recognized methods for manufacturing Aluminium Metal Matrix Composites (AMCs) include Powder Metallurgy, Squeeze Casting, Pressure Infiltration, and Stir Casting. Although the production costs can be high, Powder Metallurgy offers the advantage of a more uniform distribution of reinforcement within the matrix, leading to a smoother surface finish that minimizes the need for additional finishing processes. Furthermore, this manufacturing method can be performed quickly and with automated equipment[5]. The powder metallurgy method is an advanced material formation technique that is a solution for the production of structural components due to the limitations of making components by other methods, this method has high efficiency in material use, low energy consumption, and production of false shapes[6], this method also produces less waste, allows for better control over the porosity of the resulting material, and demonstrates more rapid advancements in manufacturing technology compared to traditional casting[7].

Aluminum matrix composites produced through powder metallurgy are typically reinforced with several potential materials such as Carbon Nanotubes (CNT), Graphene, Silicon Carbide (SiC), Boron Carbide (B4C), Alumina (Al_2O_3), Fly Ash, Titanium Diboride (TiB_2), and Graphite[4]. Aluminum reinforced with CNTs aims to increase its strength and stiffness while maintaining its lightweight[4]. CNTs have the stiffness of up to 1000 GPa, strength around 100 GPa, and thermal conductivity of up to 6000 W/m.K[8], so widely regarded as one of the most promising reinforcement materials for enhancing Aluminium Metal Matrix Composites (AMCs) produced via powder metallurgy techniques. This is due to their lightweight nature, high strength, and excellent corrosion resistance, making them highly effective in improving the stiffness and strength of aluminium composites, particularly in terms of tensile strength, yield strength, wear resistance, thermal conductivity, and hardness[4].

CNT as reinforcement in AMC material has been widely researched and achieves Young's modulus of 88 GPa, tensile strength of 820 MPa, and elongation to failure close to 5%. By using CNT, the strength, ductility, and electrical conductivity of copper were also found to simultaneously increase[4]. The use of CNT as a reinforcement in powder metallurgy processes has demonstrated an increase in strength by up to 50% and an improvement in Young's modulus by 23%. The duration of ball milling plays a crucial role in achieving a more uniform distribution of CNTs within the matrix, which significantly contributes to enhancing the overall strength of the material[4]. The addition of CNT material as reinforcement has a good effect on the results of hardness and wear tests[9][10]. The variation in CNT weight affects the wear mechanisms under different operational conditions. The results indicate that CNT-Al composites exhibit lower wear rates compared to pure aluminium, with a decrease in wear rate up to 1.5 wt% CNT. However, the wear rate increases at 2 wt% CNT and beyond, suggesting reduced plastic deformation on the worn surfaces[9]. The addition of CNTs in Al composites enhances hardness, wear resistance, and reduces the coefficient of friction compared to pure aluminium, particularly under varying sliding speeds and applied loads. The study found that hardness and wear resistance improved by up to 78.8% at 5 wt% CNT, with a decrease in the coefficient of friction as CNT content and sliding speed increased. CNTs played a crucial role in forming a carbon layer that acted as a solid lubricant, significantly improving wear performance[10].

Research on aluminium matrix nanocomposites reinforced with 1.00 vol.% carbon nanotubes

(CNT) using powder metallurgy manufacturing methods has shown that the addition of CNTs enhances the tensile strength and hardness of the nanocomposites, although it may potentially reduce ductility due to CNT distribution along the grain boundaries. The findings indicate that incorporating CNTs increases the yield strength by 185% compared to pure aluminium, thanks to an effective load transfer mechanism. However, the presence of MWCNTs at the grain boundaries also results in decreased ductility[11]. With a fixed CNT content of 2 wt.% and varying graphene levels (1, 2, 3, and 5 wt.%), the study examined the effect of adding graphene and carbon nanotubes (CNT) on the hardness and wear rate of aluminium hybrid composites produced through powder metallurgy. The results showed that the addition of CNT and graphene enhanced hardness, with the highest value observed at 3 wt.% graphene, and reduced the wear rate due to the solid lubricating effect of CNT, which minimizes surface contact[12].

The material produced through cold compaction and sintering is significantly affected by the content of CNTs in the composite. Adding CNTs up to 2.0 wt.% increases the hardness of the composite by 22.85% and lowers the friction coefficient and wear rate due to the self-lubricating effect of CNTs, which reduces surface contact. However, excessive CNT content may cause agglomeration, which adversely affects the material performance[13]. The distribution and interaction of CNTs within the aluminium matrix play a crucial role, with a uniform distribution of CNTs significantly enhancing the mechanical properties of the nanocomposite. CNTs act as bridges between points within the matrix, preventing crack propagation and improving the composite's resistance to mechanical loads[14]. Efforts to improve the quality of Al/CNT composites in the powder metallurgy process were made by enhancing CNT dispersion within the composite matrix using a planetary ball mill. The study results showed a uniform dispersion of CNTs in the final product structure, which subsequently enhanced the mechanical properties and hardness of the Al/CNT nanocomposites compared to aluminium processed without using a planetary ball mill[15].

Besides the distribution of CNTs in the composite achieved through the mixing process, the sintering process is also a crucial step in the fabrication of the material. High-temperature sintering enhances material density and improves homogeneity, which are crucial for mechanical properties such as strength and fatigue resistance. In this process, compacted powder is heated, leading to particle agglomeration and the integration of particulate reinforcements within the metal matrix. This results in stronger bonds between particles, ultimately enhancing the overall mechanical integrity of the material[6].

The distribution and interaction of CNTs within the aluminium matrix play a crucial role, with a uniform distribution of CNTs significantly enhancing the mechanical properties of the nanocomposite. CNTs act as bridges between points within the matrix, preventing crack propagation and improving the composite's resistance to mechanical loads. The study results indicate that the optimal sintering temperature for achieving maximum hardness is 550°C[16]. The variation in sintering temperature on the mechanical properties of aluminium matrix composites reinforced with carbon nanotubes (CNT) produced by powder metallurgy was evaluated for its impact on hardness, wear rate, and porosity. The results indicate that increasing the sintering temperature significantly enhances hardness and wear resistance while reducing porosity. Sintering at 500°C yielded the best results, with the highest hardness of 32.5 HR, the lowest wear rate of 0.049 mm^3/m, and a porosity of 4.45%. This is due to the more uniform distribution of CNTs within the matrix, which strengthens particle bonding and improves the overall mechanical properties of the composite[17].

From the results of the study on sintering temperature parameters conducted in the research mentioned above, several weaknesses were identified. In the study [17], aluminum-CNT composites were produced using approximately 0.4% of the total weight of the composite mixture. However, other studies suggest that the optimal weight fraction of CNT ranges between 1% to 1.5% of the total composite weight. This raises the question: if 1% CNT weight is used, would the optimal sintering temperature remain the same? In the previous study [16], research was conducted using CNT as a fixed variable with a composition of 1%, but due to two varying variables are 500 °C for 90 minutes, 550 °C for 60 minutes, and 600 °C for 30 minutes (where both temperature and time different), the impact of temperature in this study was confounded by the varying sintering times, leading to the question of whether it is the time or temperature that actually affects the sintering results?

From the review of the research weaknesses above, the author aims to verify both studies to refine the determination of the optimal sintering temperature, thereby establishing precise parameters for temperature, time, and CNT composition, which could serve as a reference for future research. The planned study involves creating Aluminum Matrix Composites (AMC) reinforced with Multi-Walled Carbon Nanotube (MWCNT) using powder metallurgy, with variations in temperature. The fixed variables will include a CNT composition of 1% and a sintering time of 1 hour, while other parameters will be kept constant or controlled as fixed (controlled variables).

METHODS

Design Experiment

The matrix uses 1100 series aluminium powder with a purity is 99.7%, powder size 63 µm/ 250 Mesh, CAS Number: 6429-90-5. The reinforcement used is Multi-Walled Carbon Nanotube (MWCNT) has a purity of 95%, and powder size is 8-10 µm. Technical specification data obtained from the data of the MWCNT material manufacturer can be seen in Table 1.

Table 1. Properties of Multi-Walled Carbon Nanotubes Powder

Properties	Specifies
Purity	>95 wt%
Inner Diameter	3-5 nm
Outer Diameter	8-15 nm
Length	3-12 m
Specific Surface Area	>233 m^2/g
Density	0,15 g/cm^3
Actual Density	2,1 g/cm^3
Resistivity	1412 m
Preparation Method	CVD
Appearance	Black Powder
Package	100 G

The composite was made by mixing 99% Al powder and 1% MWCNT weight fraction[11]. The materials were mixed using a horizontal milling machine for 1 hour at a speed of 7 Rpm. The powder mixture was compressed with a Krissbow brand hydraulic press with a pressure of about 460 MPa to form specimen pellets, where the specimen manufacturing process uses cold compaction[13].

The variation used in the research is specimen non-sintering treatment and sintering treatment at temperatures 450 0C, 500 0C, 550 0C, and 600 0C, with heating time in the sintering process is 1 hour. The sintering tool uses a Muffle Furnace W/Flap Door Max. temp 1100 °C, manufacturer Nabertherm (Germany), Model: L5/11/B180, Cap: 5 Lt. Schematic of Al/MWCNTs nanocomposite fabrication process by powder metallurgy method can be seen in Figure 1.

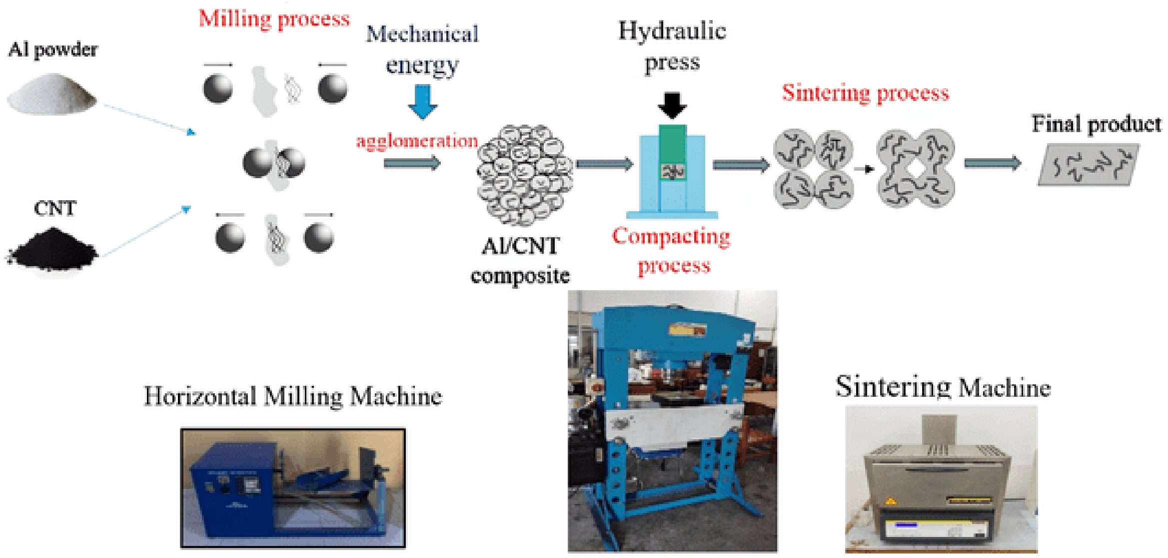


Figure 1. Schematic of Al/MWCNTs Nanocomposite Fabrication Process by Powder Metallurgy Method[15].

Testing method

The material characterization will be carried out is hardness testing, wear rate testing, and observation of material morphology and phase formation with SEM-EDS. This research uses the Vickers hardness method, because it provides results in the form of a continuous scale of hardness. The advantage of testing with this vikers hadness method is that it can analyse specimens of small size. specimen testing is carried out at 3 test points on the surface of the material. The hardness test carried out using the Vickers micro method is carried out using a load of 30 Kgf for 20 seconds. Wear testing is carried out using the Ogoshi Wear Testing machine (Type OAT-U) to determine the value of wear resistance on composite specimens. This machine serves as a tool to test the wear rate of composite specimens. The wear test used the Ogoshi method with a load of 62.3 N with a duration of 1 minute, this test can also handle small specimens. Testing procedure and calculation of VHN hardness value and wear rate value by Oghoshi s method refer to the article on carbon steel hardness and wear analysis[18].

To determine the morphological condition of the Al-MWCNT composite material, SEM (Scanning Electron Microscope) test was conducted, which is used for surface analysis and microstructure examination of materials with high resolution. Tthe material composition analysis on the composite surface was carried out by EDS (Dispersive X-ray Spectroscopy)test. which is used in conjunction with SEM to analyze the chemical composition of materials on a microscopic scale. Zeiss Type Evo 10 equipment was used to analyze the surface shape of the composites examined by SEM and the phase formation was identified by the EDS technique. preparation of the surface of the material to be tested SEM and EDS is done based on Standard Practice for Microetching Metals and Alloys, this aims to get optimal results[19]

RESULTS AND DISCUSSION

Specimens that have been pressed with a hydraulic press machine are then subjected to a sintering process aimed at improving the quality of the bond between particles in the composite structure, The specimen that has been sintered can be seen in Figure 2.

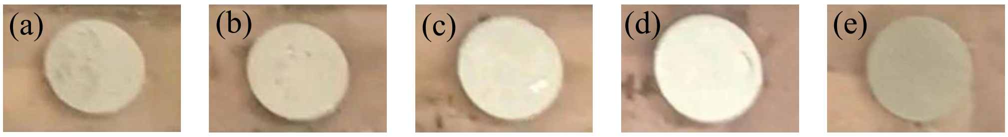


Figure 2. Sintering Result Specimens with Sintering Temperature Variations; (a) 450ºC, (b) 500ºC, (c) 550ºC, (d) 600ºC and (e) Non Sintering

Each variation consists of 3 specimens so a total of 15 specimens are ready to be tested for hardness and wear. The first test carried out is the Vickers microhardness test in each of the 3 test points on the surface of the specimen. After that on the same specimen wear testing is carried out with the oghosi method. Figure 3., is an example of a specimen after hardness and wear testing.

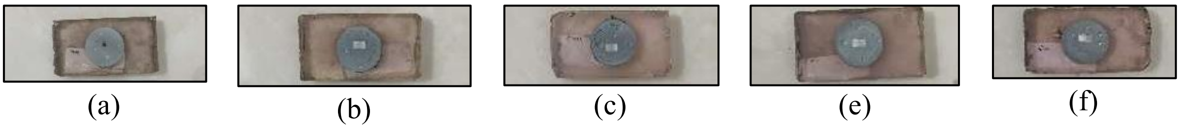

(a) (b) (c) (e) (f)

Figure 3. Specimens After Hardness And Wear Tests With Sintering Temperature Variations; (a) 450ºC, (b) 500ºC, (c) 550ºC, (d) 600ºC and (e) Non Sintering

Microhardness Test

The research results indicate that the sintering variation at a temperature of 500°C achieved the highest hardness value of 50.05 HV. This increase in hardness is likely due to the formation of intermetallic phases in the specimen. However, when the sintering temperature exceeds 550°C, the hardness of the specimen decreases significantly. This phenomenon can be attributed to the formation of voids and craters in the specimen, as well as oxidation with aluminium metal. Similarly, at a temperature of 450°C, the hardness value remains below that of 500°C, which is due to the suboptimal agglomeration process of the particles within the composite structure. Figure 4. shows the results of the Vickers microhardness test observed on the specimen.

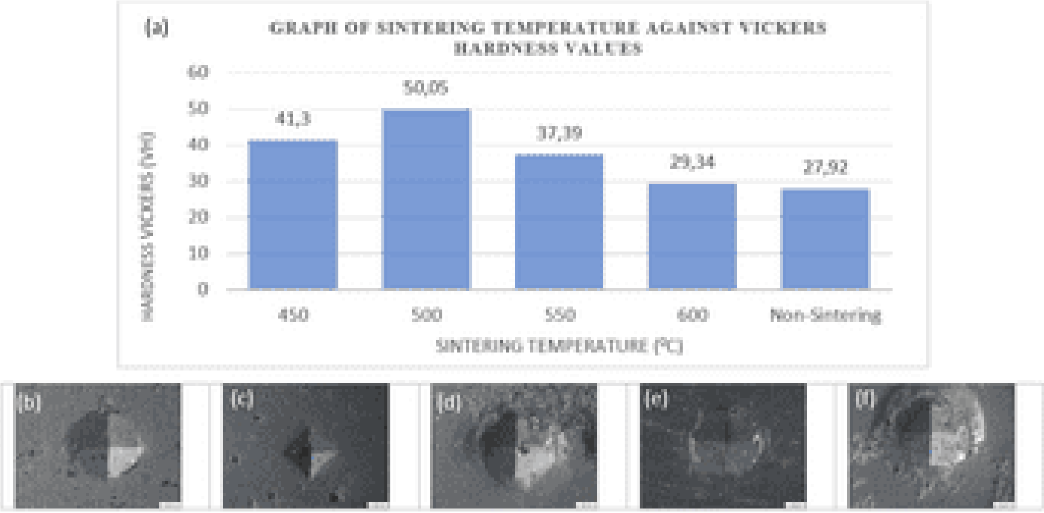


Figure 4. (a) Graph Of Vickers Microhardness Test Results, Measured On Synthesis Specimens Made With Variations Sintering; (b) 450^{0}C, (c) 500^{0}C, (d) 550^{0}C, (e) 600^{0}C, and (f) Non-Sintering

Wear Rate Test

In this test, it was found that the optimal wear resistance was achieved at a sintering temperature of 500°C with a value of 0,4387 mm^3/s. Increasing the sintering temperature above 500°C (specifically at 550°C and 600°C) led to an increase in the wear rate, indicating that the material becomes more prone to wear. The primary factor contributing to the increased wear rate of the specimens is the formation of oxide phases on the specimen surface. At a sintering temperature of 600°C, the MWCNT material is more susceptible to oxidation, forming MWCNT-O2. This is evident from the increased oxygen content observed in the EDS map sum spectrum analysis and the appearance of void craters seen in the SEM analysis. At 450°C, the wear rate is lower compared to 500°C. This is because, at this temperature, the particles within the composite structure have not yet undergone optimal agglomeration. Consequently, the material retains a denser and more uniform structure, which enhances its resistance to wear. Figure 5., shows the wear rate analysis results of the Al-MWCNT composite specimens.

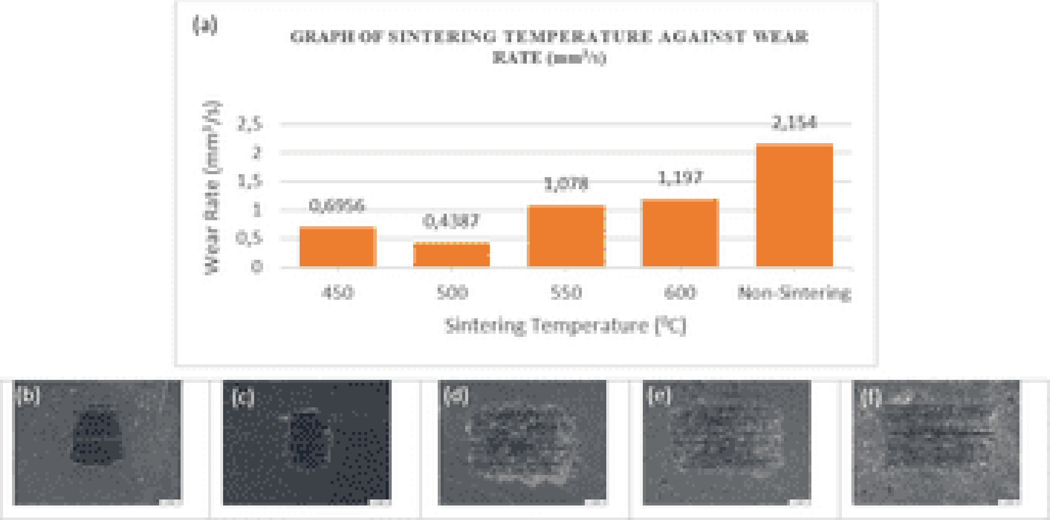
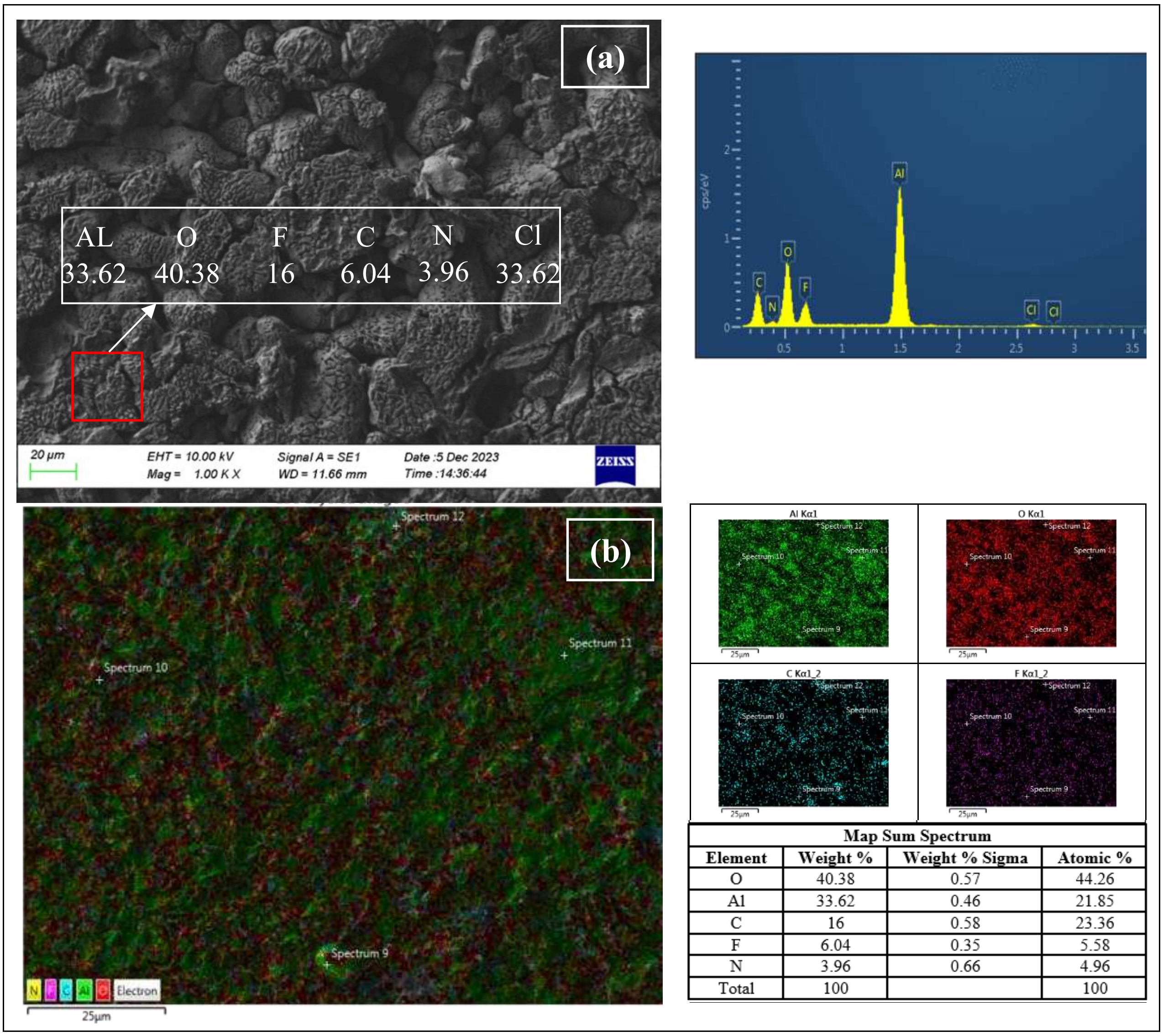


Figure 5. (a) Wear Rate Graph, Observed On Sintering Specimens With Variations Sintering; (b) 450^{0}C, (c) 500^{0}C, (d) 550^{0}C, (e) 600^{0}C, and (f) Non-Sintering

Microstructure characterization

SEM (Scanning Electron Microscopy) and EDS (Energy Dispersive X-ray Spectroscopy) tests were conducted on specimens with the best and lowest wear and hardness values, specifically at temperatures of 500°C and 600°C. Additionally, specimens without sintering were also analyzed using SEM and EDS to serve as baseline data for comparison purposes. This approach allowed for a comprehensive assessment of how sintering temperatures affect the material's microstructure and properties. The results of hardness and wear tests at sintering temperature variations of 550°C and 600°C produced values that were not far adrift so that in this study samples were taken at the 600°C sintering temperature variation to be evaluated by SEM-EDS analysis.

In the non-sintering variation, the oxygen content was the highest reach 40.38%, which is expected since the mixing and pressing processes were conducted in open conditions. As a result, the composite particles readily bonded with oxygen, and a significant amount of oxygen became trapped in the internal spaces of the compacted particles. This condition makes the wear quality and hardness of non-sintered specimens lower than the specimens with sintering at all temperature variations (450 °C, 500 °C, 550°C and 600°C) this phenomenon can be seen can be seen in Figure 6(a). On the other hand, SEM observation shows clear lines between particles so that it can be confirmed that the bond between particles is not very good. This phenomenon can be seen can be seen in Figure 6(b).



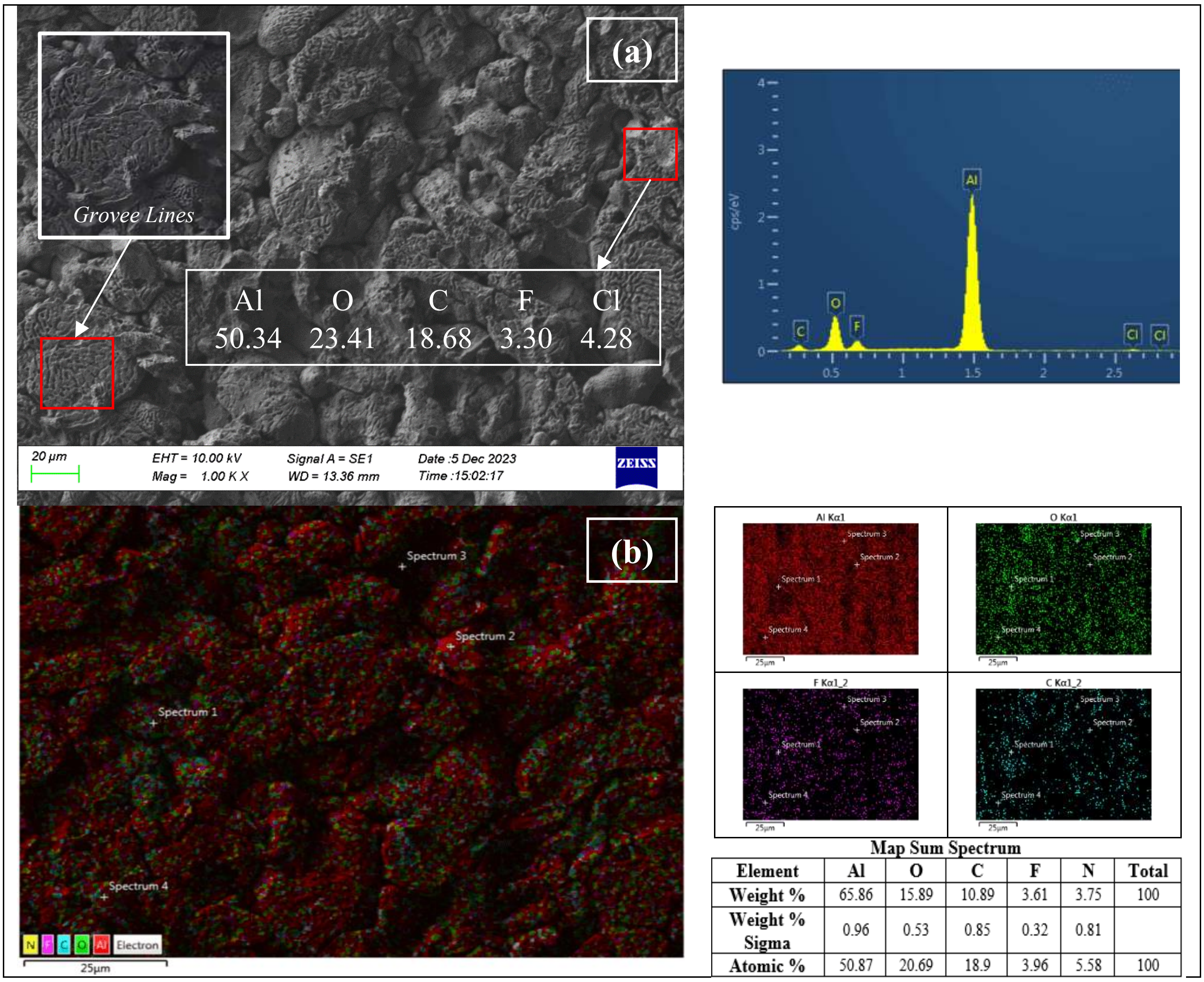
Map Sum Spectrum

Element	Weight %	Weight % Sigma	Atomic %
O	40.38	0.57	44.26
Al	33.62	0.46	21.85
C	16	0.58	23.36
F	6.04	0.35	5.58
N	3.96	0.66	4.96
Total	100		100

Figure 6. Specimen Analysis; (a) SEM Non-Sintering, (b) EDS Non-Sintering

The best Vickers hardness and wear rate were achieved at a sintering temperature of 500°C. The EDS mapping and spectrum results indicate that the oxygen content at 500°C was the lowest, at 15.89%. This is because, at the sintering temperature of 500°C, the oxygen is expelled from the spaces between the agglomerating powder particles. However, when the sintering temperature was increased above 500°C (specifically at 550°C and 600°C), the oxygen content increased, leading to a decrease in hardness and an increase in wear rate. This deterioration is due to the oxidation of the composite surface, as evident from the higher oxygen content at 600°C, which reached 23.41%.

On the surface of the specimen sintered at 500°C, there are groove patterns visible on the surface, as seen in Figure 7(a), This is also evidenced by the oxygen content on the surface of the lowest composite, see the data in Figure 7(b). This occurs due to the release of oxygen from the solidification process. The elemental composition observed by EDS shows that the dark phase can be identified as Al, and the irregularly shaped white particles can be identified as Al-O_2 and MWCNT-O_2. The formation of Al-O_2 and MWCNT-O_2 is inevitable because the mixing and pressing process is carried out in an open environment with high oxygen content so that the powder material is easier to bind with O_2 and form an oxide layer on the surface of the material. SEM observations at a sintering temperature of 500°C show fewer cracks, and the grains appear quite large because the agglomeration of powder material grains occurs well, resulting in a good interface bond between al powder and MWCNT grains.

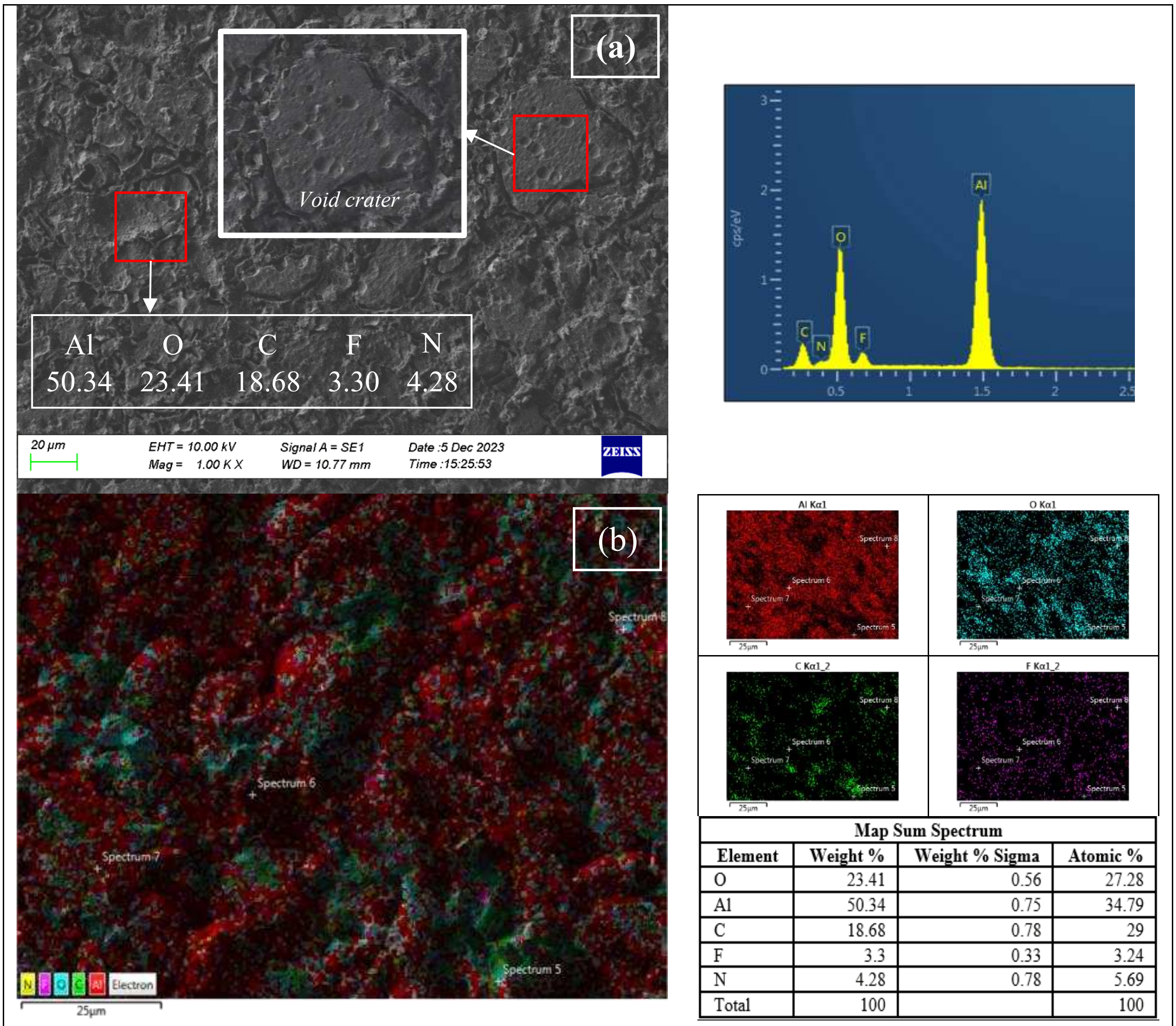


Map Sum Spectrum

Element	Al	O	C	F	N	Total
Weight %	65.86	15.89	10.89	3.61	3.75	100
Weight % Sigma	0.96	0.53	0.85	0.32	0.81	
Atomic %	50.87	20.69	18.9	3.96	5.58	100

Figure 7. Specimen Analysis; (a) SEM of Sintering Variation at 500^{0}C, (b) EDS of Sintering Variation at 500^{0}C

oxidation between the metal and oxygen, forming metal oxides. This observation is further supported by the increased oxygen content on the material's surface at 600°C, reaching 23.41%, as analyzed by EDS. This phenomenon is illustrated in Figure 8(a), where a greater number of craters are visible on the specimen's surface compared to other specimens. The surface chemical composition data, shown in Figure 8(b), indicates a significant oxygen content in the non-sintered variation. The increased oxygen content on the composite surface, indicating a higher amount of metal oxides such as Al-O_2 and MWCNT-O_2, negatively affects the material's hardness and wear resistance, as evidenced by the lower hardness values compared to specimens sintered at 500°C. The SEM and EDS results collectively demonstrate that oxidation activity in the specimens increases with rising temperatures, especially above 500°C. During the initial heating phase, volatile gases (such as oxygen and nitrogen) trapped within the specimen are expelled. However, as the temperature continues to rise, approaching the material's melting point above 500°C, surface oxidation accelerates, leading to the formation of more metal oxide phases. This phenomenon adversely affects the specimen's quality, particularly in terms of hardness and wear resistance.



Al	O	C	F	N
50.34	23.41	18.68	3.30	4.28

Map Sum Spectrum			
Element	Weight %	Weight % Sigma	Atomic %
O	23.41	0.56	27.28
Al	50.34	0.75	34.79
C	18.68	0.78	29
F	3.3	0.33	3.24
N	4.28	0.78	5.69
Total	100		100

Figure 8. Specimen Analysis; (a) SEM of Sintering Variation at 600^{0}C, (b) EDS of Sintering Variation at 600^{0}C

CONCLUSION

The sintering process is one of the crucial processes to produce Al-MWCNT composites with good quality, at temperatures above 500°C (specifically 550°C and 600°C), the surface composition of the composite tends to be dominated by Al-MWCNT, $Al\text{-}O_2$, and $MWCNT\text{-}O_2$ phases. EDS analysis at 600°C and in non-sintered variations shows a high oxygen content, which negatively affects the hardness and wear test results. At the sintering temperature of 500°C, the amount of metal oxides is reduced, as evidenced by the lowest oxygen content, which only contains 15.75% oxygen on EDS analysis. This reduction is identified as the reason for the improved hardness and wear resistance of the specimen. However, for specimens sintered at 600°C, SEM observations reveal that the bonds within the specimen exhibit many cracks and numerous void craters on the material surface. In SEM testing, it can be seen that in specimens that are not sintered, there are clear boundary lines between materials, this indicates poor particle bonding. in contrast to sintered specimens, it can be seen that the particles bind well, especially at 500°C. This study indicates that the optimal sintering temperature to achieve enhanced hardness and wear resistance in the material is 500°C, it increased the hardness and wear values compared to the non-sintered specimens, achieving 50.05 HV and 0.4387 mm^3/s respectively.

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