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EXPERIMENTAL INVESTIGATIONS OF MICROSTRUCTURAL AND MECHANICAL BEHAVIOUR OF ALUMINIUM ALLOY 7075 HYBRID NANOCOMPOSITES

In this paper, the microstructural and mechanical characterisation of hybrid composites with a metal matrix was conducted. Al_2O_3 nanoparticles and CNTs were added to strengthen an aluminium alloy (AA 7075) using solid-state powder metallurgy. Utilizing XRD analysis and scanning electron microscope (SEM), microstructural characterization was carried out. Uniaxial compression and microhardness testing were performed to determine how the hybrid composites behaved mechanically. The microstructural research revealed that the nanoparticle dispersion in the matrix is uniform. The XRD plots and the Williamson-Hall equation were used to evaluate the crystallite size, lattice strain and dislocation density. In comparison to the base alloy, the composites have better strength and micro hardness.

Keywords: aluminium 7075 alloys, hybrid metal matrix composites, micro hardness, compressive strength, microstructure

INTRODUCTION

The aircraft industry makes substantial use of aluminium alloy 7075 because of its high strength, lightweight, and resistance to corrosion [1, 2]. This alloy is utilized to create control valve components, missile parts, gears and shafts, in addition to fittings for aircraft in the fields of aerospace and energy production [3]. Al_2O_3 structural ceramics are well-known as high-performance materials [4]. The greater mechanical strength and hardness of Al_2O_3, make it a suitable material for various industrial applications [5, 6]. The best nanofillers for enhancing mechanical and other physical qualities are carbon nanotubes. Adding CNTs to polymers in a very small volume can significantly increase the mechanical, electrical and thermal properties of polymers [7, 8].

Metal matrix nanocomposites (MMNCs) consist of nanoscale reinforcement, and the nanocomposite matrix should have a lower density, whereas the reinforcing material requires higher strength to withstand high temperatures [9]. Nanoparticles have a propensity to aggregate into clusters in the matrix because of their low wettability and high surface-to-volume ratio, which can cause cracks and deteriorate the mechanical properties of the composites. It is a challenging task to uniformly distribute and disperse nanoparticles in the matrix material. There are several techniques described in the literature for improving the dispersion of nano-reinforcements within the matrix. The development and improvement of processing methods include traditional ball milling, plasma milling, electrochemical deposition and molecular mixing [10].

To strengthen the bonding between the base alloy and the reinforcements, powder metallurgy is frequently utilized in a variety of ways to create metal matrix composites. The ball milling technique can change the shape of the initial powder particles, and different sintering methods, including vacuum sintering, spark plasma sintering and microwave sintering, are employed to strengthen the interfacial bonding. Kumar et al. [11] investigated the impact of the TiO_2 content on the micro hardness and wear of aluminium-based composites. The results demonstrated a notable improvement in the micro hardness and wear resistance of the composites.

Aluminium matrix composites with heterogeneous structures reinforced with copper-GNS hybrids were reported by Pu et al. [12]. The results showed that the fabricated composite exhibited excellent improvement in strength and ductility simultaneously. Microstructural heterogeneities in a composite contribute to a significant increase in stress-strengthening strain-induced hardening. Aluminium/zinc oxide nanorod reinforced

aluminium composites were successfully fabricated by Bhoi et al. [13]. In comparison to pure aluminium, the created composite demonstrated encouraging results in terms of mechanical behaviour.

Industrial waste was employed as reinforcement by Nayak et al. [14] to examine the mechanical characteristics and yield stress behaviour of composites made of aluminium. The samples with the highest percentage of grinding sludge (GS) reinforcement showed improvement in compressive strength and deformation. Sethi et al. [15] studied the effects of copper-coated carbon nanotubes (CNT) on aluminium matrix hybrid composites, specifically Al-30 wt.% $Y_2W_3O_{12}$ and AlN, and improved the mechanical properties with the inclusion of Cu-CNTs. Liu et al. [16] developed rGO and GNS-reinforced Al composites using the powder metallurgy method. Higher hardness levels were obtained for the composites compared to the pure aluminium sintered samples.

Raj Mohan et al. [17] improved the strength and hardness characteristics of an Al composite by stir-casting mica and SiC ceramic particles into the A356 alloy. Tharani Kumar et al. [18] synthesized Al-Zn-Mg alloy hybrid nanocomposites with different weight fractions of Si_3N_4-BN nanoparticles and improved tensile, compressive strength and wear resistance compared with the base alloy. Nanocomposites were created by Toozandehjani et al. [19] using a hybrid powder metallurgy and microwave sintering technique. The authors reported that the mechanical properties can be controlled by the content and dispersion of the nano reinforcements. In their work, they obtained the maximum density at 8 hours and optimized the mechanical properties at 12 hours of ball milling time which increased the dislocation density and grain refinement. The tensile strength, micro hardness, and density of aluminium composites containing 10 wt.% SiC and (0-2) wt.% copper oxide nanoparticles were assessed by Raj Mohan et al. [20]. According to the authors, the mechanical characteristics rise with the weight percentage of CuO nanoparticles.

The present study focused on the synthesis of AA 7075 alloy hybrid nanocomposites using the powder metallurgy technique. The reinforcements were selected as Al_2O_3 nanoparticles and CNTs. This study aims to explore the alterations in the microstructure and mechanical characteristics of AA 7075 composites as the weight percentage of CNTs is increased.

MATERIALS AND METHODS

The matrix material was AA 7075 powder from Paraswamani Metals in Mumbai, with a mean particle size of 50 μm and a purity of 98%. As reinforcements, 10 nm-diameter carbon nanotubes and 30 nm Al_2O_3 nanoparticles were used.

Previous research on the impact of incorporating Al_2O_3 nanoparticles and CNTs in AA 7075 composites has shown that the optimal percentage for Al_2O_3 is 5 wt.%, while the ideal range for CNTs is between 0-0.5 wt.%. In this work, Al_2O_3 and CNTs were used to create hybrid composites with the above-said proportions to maximize the mechanical characteristics. The percentage of AA 7075 was varied from 100-94.5 wt.% and CNTs from 0.1-0.5 wt.% while the content of Al_2O_3 nanoparticles was fixed at 5 wt.%. The dimensions of the samples were taken as Φ10 mm×15 mm length for the compression test and 10 mm × 10 mm × 10 mm for the microstructure, density and micro hardness investigations. A total of 21 samples and three samples per formulation were produced for the compression tests. The ball milling process was performed in a planetary ball mill with tungsten carbide balls and a vial. The ball to powder ratio was 10:1 and the ball speed was 300 rpm. The resulting powder was cold-pressed with a 3-ton load at the pressure of 60 bar. The acquired raw pressings were sintered in a tube furnace at the temperature of 450°C for two hours. The composition, proportion, and designation of the composites are shown in Table 1.

TABLE 1. **Compositions of AA7075/Al_2O_3/CNT composites**

Sample	Al7075 [wt.%]	Al_2O_3 [wt.%]	CNTs [wt.%]	Designation
1	100	-	-	A_1
2	95	5	-	A_2
3	94.9	5	0.1	A_3
4	94.8	5	0.2	A_4
5	94.7	5	0.3	A_5
6	94.6	5	0.4	A_6
7	94.5	5	0.5	A_7

MICROSTRUCTURAL AND MECHANICAL CHARACTERIZATION

SEM micrographs were used to assess the microstructural characteristics of the composite materials. By means of a Joel JCM 6000 Plus scanning electron microscope, the morphology of the composites was investigated. A Benchtop X-ray diffractometer (Rigaku) was employed to perform phase analysis and to determine the crystallite size and lattice strain. A universal testing machine was utilised for uniaxial compression tests, and a Rockwell hardness tester was used for microhardness testing. The density of the composites was measured by means of the Archimedes method.

RESULTS AND DISCUSSIONS

Microstructural behaviour

Microstructural analyses were carried out on the created composites to ascertain whether there is uniform distribution of the reinforcement in the matrix.

The microstructural characterization of hybrid aluminium composites with a distribution of reinforcement and with a bimodal grain size was presented in [21]. The authors revealed the homogeneity of the reinforcement distribution in the synthesized composites. High dislocation density and grain refinement were observed at higher ball mill grinding times in Al-CNT-Al_2O_3 composites [19]. Wang et al. [22] examined the impact of tungsten copper-coated graphite plates and aluminium nitride nanoparticles on the microstructure and mechanical properties of copper matrix composites. The researchers observed uniform dispersion of the AlN nanoparticles within the Cu matrix. On the other hand, Sethi et al. [23] investigated the effect of the milling time, sintering temperature, and duration on the microstructural changes of Al-$Y_2W_3O_{12}$-AlN hybrid composites and verified the absence of any interfacial reaction between the matrix and the reinforcements.

XRD analysis

Figure 1 shows the XRD patterns of the AA 7075 pure alloy, AA 7075+5 wt.% Al_2O_3, and AA 7075+ +5 wt.% $A1_2O_3$ + 0.1-0.3 wt. % of CNT powders after six hours of ball milling. The diffraction pattern showed aluminium peaks at various 2θ positions, indicating the absence of intermetallic. The Al_4C_3 intermetallic phase which is usually formed due to the interfacial reaction between aluminium and CNTs was not detected either, owing to the shorter grinding time in the ball mill. In the AA 7075+5% Al_2O_3 composite, the Al (111) peak broadened and a notable shift in the peak position was observed in the AA 7075+5 wt.% Al_2O_3 + 0.2 wt.% CNTs composite. The shift occurred because the reinforcement particles and minor alloying components within the matrix broke down [24, 25]. The sample showed an increase in the height and breadth of the Al (111) peak, indicating the flexibility and refinement of the microstructure. These results align with those reported by Karunanidhi et al. [26].

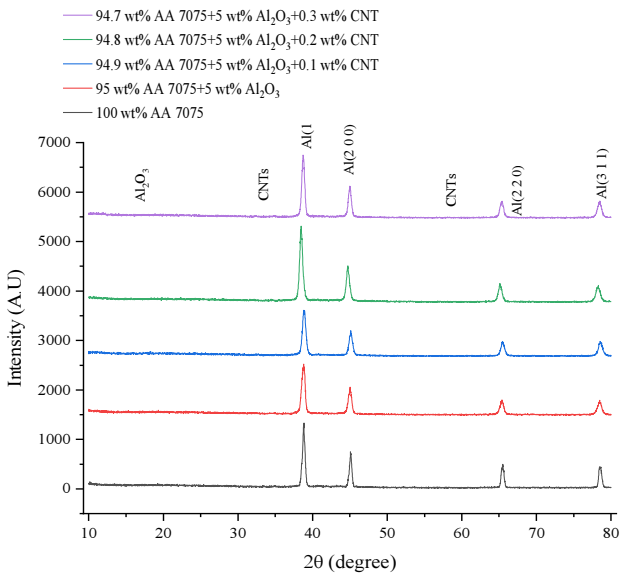

Fig. 1. XRD patterns of AA7075/Al_2O_3/CNT composites

The Williamson-Hall equation was used to evaluate the crystallite size, lattice strain, and dislocation size. Using XRD plots, Williamson Hall suggested a method for calculating the crystallite size and stress broadening. For a diffraction angle of 2θ, the diffraction peak is calculated by using the equation [24]

$$\beta_{hkl}\cos\theta_{hkl} = \frac{k\lambda}{t} + 4\sin\epsilon\sin\theta_{hkl} \quad (1)$$

where t is the effective crystallite size perpendicular to the reflection planes, k stands for the shape factor, λ is the X-ray length, θ_{hkl} is the Bragg angle, and ϵ is the lattice strain. Based on β_{hkl} and θ_{hkl}, crystallite size, lattice strain and the dislocation size were calculated for each weight fraction. The composite with AA 7075 + 5 wt.% Al_2O_3 + 0.1 wt.% was milled for 8, 10 and 12 h consecutively and the XRD analysis results revealed sizes of 17.60 nm, 17.63 nm and 20.20 nm, respectively. The results of the study revealed that the crystallite size of the synthesized composite was smaller than 100 nanometres, suggesting that it is a hybrid nanocomposite.

The variation in dislocation size as the composition changes from A1 to A7 is illustrated in Figure 2. The increase in dislocation size from A1 to A3 is because of the weak bonding between the matrix and reinforcement at the interface. As the weight percentage increases, the dislocation density in samples A4 and A5 undergoes a significant change, which is likely because of the even dispersion and distribution of reinforcements resulting from the appropriate duration of ball milling and sintering temperature. The dislocations density in samples A6 and A7 increased, owing to the higher percentages of secondary reinforcement, resulting from strain effects and grain refinement.

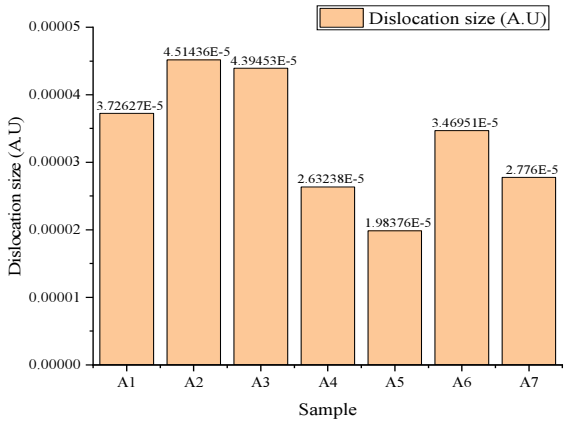

Fig. 2. Dislocation size of AA7075/Al_2O_3/CNT composites

As shown in Figure 3, the inclusion of 0-0.2 wt.% CNTs resulted in lattice deformation and a corresponding increase in lattice strain from 0.00339 to 0.00474. The increase in grain size causes a decrease in lattice strain at 0.3 wt.% CNTs. Samples A6 and A7 exhibited improved strain values due to lattice distortion and high grain refinement.

As the composition changes from A1 to A2, the crystallite sizes of the composites dwindle, as depicted in Figure 4. Equiaxed grain formation causes a reduction in the crystallite size relative to the base alloy. A slight rise in the crystallite size is observed when incrementing the CNT content from 0.1 to 0.3 weight percent. The widening of the grain boundary is the cause of the growth in crystallite size in the A5 sample. The maximum reduction in the crystallite size relative to the base alloy is 32%.

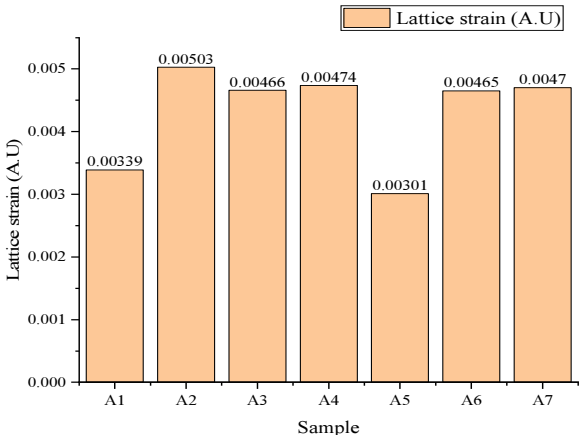

Fig. 3. Lattice strain of AA7075/Al_2O_3/CNT composites

SEM analysis

Wang et al. [27] prepared an SiCp (CNT) aluminium hybrid composite by vacuum hot pressing. The results demonstrated that CNTs can improve the dislocation density at the micro zone contact and dislocation movements pinning points. Sethi et al. [28] investigated at how the milling time, sintering temperature, and time affected the microstructural properties of aluminium metal matrix composites. In their study, they discovered that after 10 hours of ball mill grinding, the smallest crystallite size and uniform distribution of the reinforcements were seen.

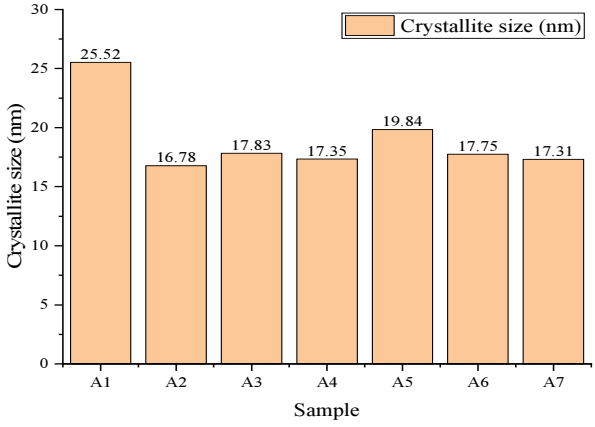

Fig. 4. Crystallite size of AA7075/Al_2O_3/CNT composites

Dinaharan et al. [29] investigated the microstructure evolution and wear of copper matrix composites produced using powder metallurgy. The microstructure revealed that the reinforcements were evenly distributed throughout the matrix and that each reinforcement caused the grains in the copper matrix to become more polished. Powder metallurgy was used by Akcamli et al. [30] to create graphene-reinforced near-eutectic Al-Si composites. Powders that had been mechanically alloyed and those that had not were combined using uniaxial pressing and pressure-less sintering. The powders that had undergone mechanical alloying were characterized by a refined, semi-equiaxial shape and lower crystallite size values. Perez et al. [31] investigated the microstructural and corrosion behaviour of A413/Al_2O_3 composites with a metal matrix. Their microscopic examinations revealed that the porosity had marginally increased and that the Al_2O_3 particles were equally dispersed. Joshua et al. [32] looked into the effects of TiO_2 nanoparticles on the micro hardness and microstructural behaviour of AA 7068 composites. The homogenous dispersion of the TiO_2 nanoparticles in the matrix and harder reinforcing particles led to a robust connection at the interface after 40 hours of ball milling. Akinwamide et al. [33] evaluated the microstructural and nanomechanical properties of TiFe-SiC-reinforced aluminium matrix composites. The sample containing 2 wt.% TiFe-2 wt.% SiC particles demonstrated superior mechanical performances and sintered composites with better microstructural characteristics.

Figure 5a shows the microstructure of the ball-milled AA 7075 alloy. The SEM micrographs reveal that the composites reinforced with 5 wt.% Al_2O_3 exhibited homogeneous particle distribution in the matrix structure. The SEM micrograph 5b shows Al/Al_2O_3 micro zones, which is consistent with the results obtained according to [27]. At 0.1 wt.% CNTs, the number of micro zones in the composite microstructure was reduced due to the pinning effect of dislocations. No cavities or micro holes were observed at 0.2 wt.% CNTs marked in Figure 5c. Grain refinement and interfacial bonding improved when the CNT wt.% was increased from 0.3 to 0.5. SEM micrograph 5d displays enhanced interfacial bonding between the matrix and reinforcements. Increasing the wt.% of CNT leads to restricted grain growth and a more uniform distribution of the reinforcements. These findings are in line with the results obtained in [28-33].

Density

The density of the composite material is heavily influenced by factors such as the compaction pressure, sintering conditions, and the compressibility of the reinforcing particles. The sintering temperature and duration can be adjusted to increase the density of the composites. [34]. Since the reinforcement density surpasses the matrix density, the density of the composite increases in relation to the base material. As the weight fraction of reinforcements grows, so does the porosity, leading to swelling and a rise in volume. The density of the composite is reduced as a result of an increment in volume [14].

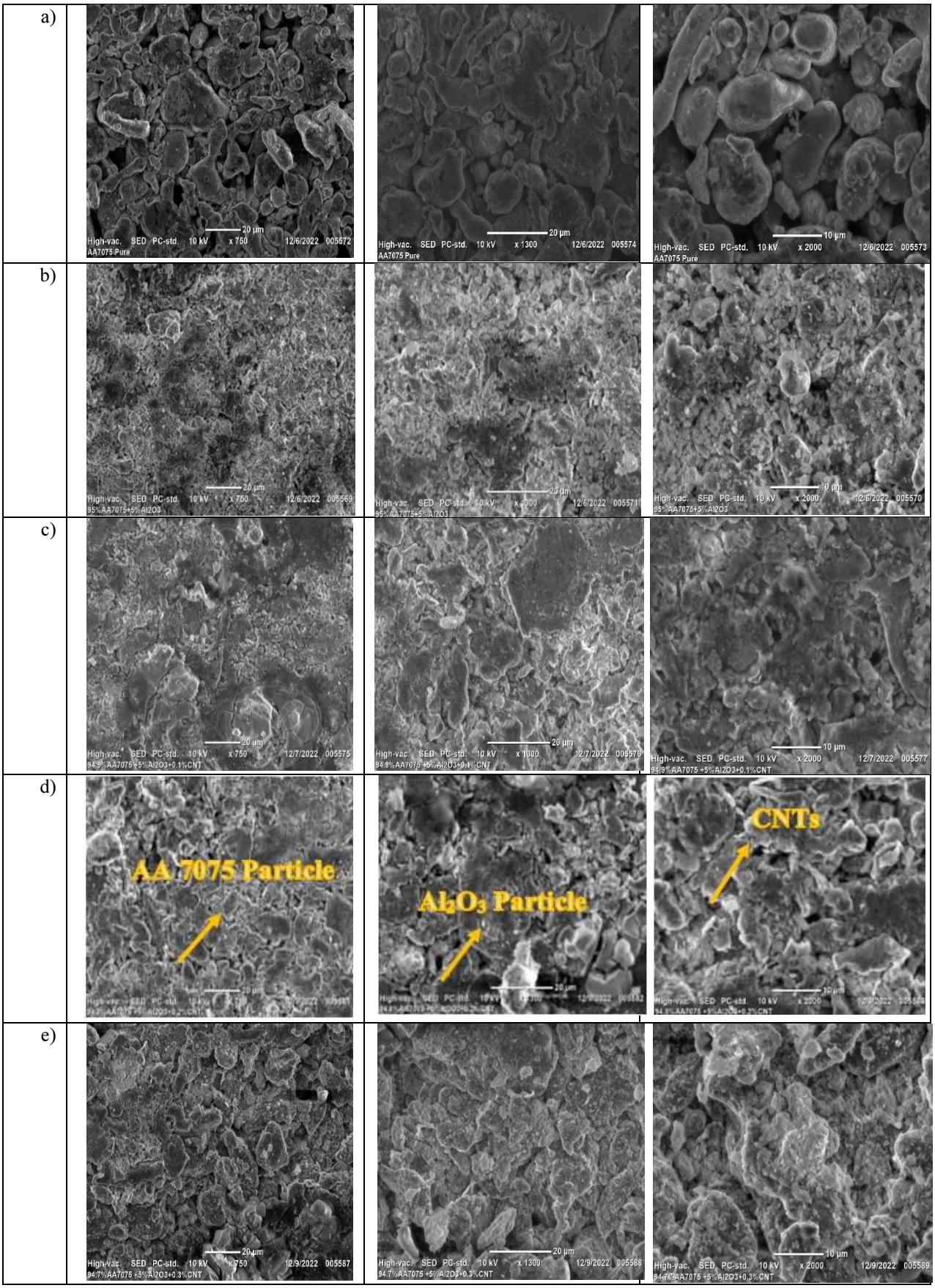

Fig. 5. SEM micrographs of: a) pure AA7075, b) AA7075+5 wt.% Al_2O_3, c) AA7075+5 wt.%Al_2O_3+0.1 wt.% CNTs, d) AA7075+5 wt.%Al_2O_3+ +0.2 wt.% CNTs, e) AA7075+5 wt.%Al_2O_3+0.3 wt.% CNTs

According to Figure 6, composite A2 has a higher density than composite A1 because of the greater density of the Al_2O_3 reinforcement compared to the AA7075 alloy. When the CNT content increased, the density dropped to 2.11 gm/cm^3 as a consequence of greater porosity. Adding 0.3 wt.% CNTs increased the density from 2.11 gm/cm^3 to 2.35 gm/cm^3, and then declined to 2.16 gm/cm^3 at 0.5 wt.% of CNTs. All the examined composites have minor density variations, with a maximum of about 7%.

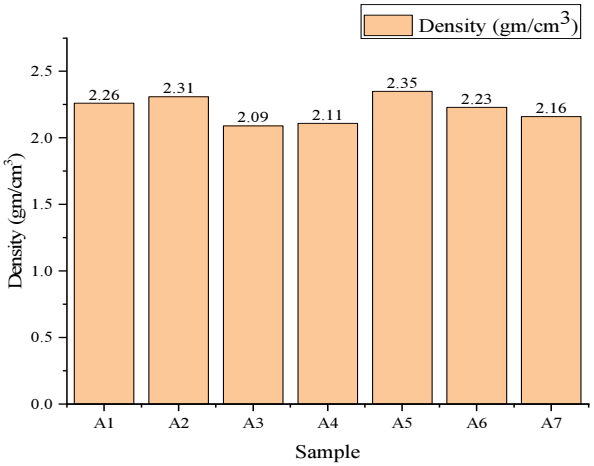

Fig. 6. Density of AA7075/Al_2O_3/CNT composites

Compressive strength

With more CNT reinforcement, the compressive strength of the composite rose as shown in Figure 7. The composite strength increased to 201.76 MPa as the weight percentage of CNTs increased from 0.1 to 0.2 resulting from even distribution of reinforcements, grain refinement, and reduced porosity. The composite containing 0.2 wt.% CNT exhibited superior compressive strength compared to both the base alloy and the alloy with 5 wt.% Al_2O_3. Evenly distributed reinforcements help to decrease the distance between particles and enhance the strength of the material. Nonetheless as the percentage of carbon nanotubes was increased, the strength of the composite material dwindled. The rise in weight percentage of CNTs from 0.2 to 0.4 in the cases of A4 to A6 led to a decrease in compressive strength from 201.76 MPa to 182.46 MPa as a consequence of the porosity and amplification of micro cracks and holes. Additionally, it was discovered that the degree of porosity and cracks significantly influences the mechanical properties of the sintered compacts. Aluminium composites with B4C micro particles and SiC nanowires were produced by Hua et al. [35]. The B4C micro particles were distributed uniformly, and it was determined that the increase in geometrically required dislocations with secondary reinforcement contributes to the hetero-deformation induced strengthening and load transmission efficiency between the matrix and reinforcement. In the present study, increasing the percentage of CNTs led to the aggregation of nanoparticles, resulting in a decrease in the ultimate compressive strength in sample A7.

Figure 8 displays the yield strength with a varying CNT wt.%. As the proportion of CNT weight increased to 0.4 wt.%, the yield strength values improved. The main factor contributing to the increased results is the strong bonding between the matrix and the reinforcement at their point of contact. The main mechanisms responsible for strengthening in materials are load transfer, Orowan looping, temperature mismatch, and the increase in strength due to the presence of dislocations [36-38]. With a rise in the weight percentage of CNTs from A6 to A7, the yield strength dropped from 159.66 to 98.66 MPa. This was attributed to inadequate load transfer and a rise in dislocation migration.

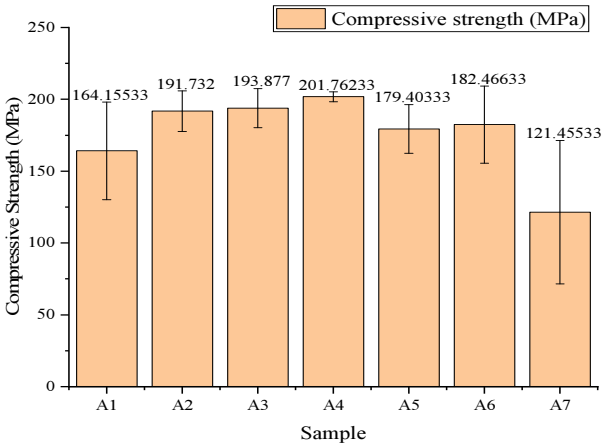

Fig. 7. Compressive strength of AA7075/Al_2O_3/CNT composites

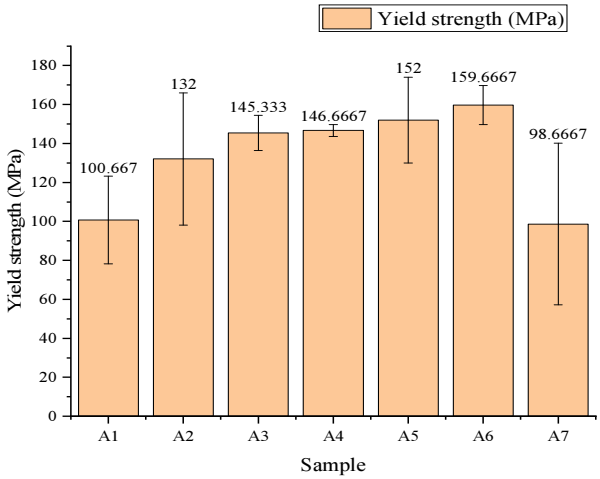

Fig. 8. Yield strength of AA7075/Al_2O_3/CNT composites

The compressive deformation values of the AA7075/Al_2O_3/CNT composites with variable wt.% CNT reinforcement are shown in Figure 9. After 6 hours of ball milling, as the percentage of CNT reinforcement increased, the compressive deformation gradually rose to Composite A4 and then declined until Composite A7.

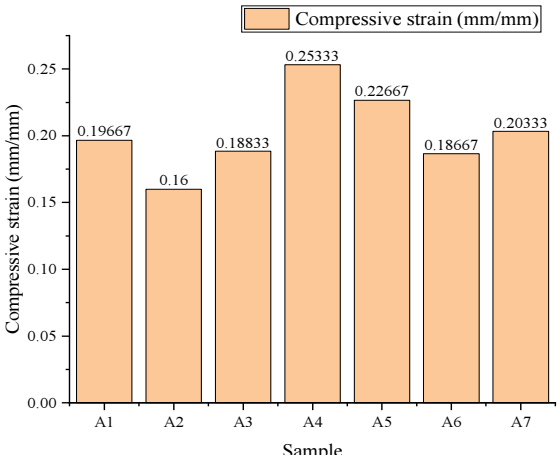

Fig. 9. Compressive strain of AA7075/Al_2O_3/CNT composites

The bonding of the nanoparticle/matrix interface improves the deformation behaviour of the composites [39, 40]. The processing variables including strain and the strain rate, as well as the particle size and weight percentage, have a significant impact on the deformation mechanism of particle-reinforced metal matrix composites [41].

Micro hardness

The literature suggests that an increase in milling duration results in alterations to both the particle size and morphology. Owing to evenly distributed hardened nanoparticles in the matrix, higher hardness values were attained during longer grinding periods in the ball mill. The employed method of mixing affects how evenly the reinforcements are dispersed throughout the matrix material, increasing the hardness of aluminium composites [42].

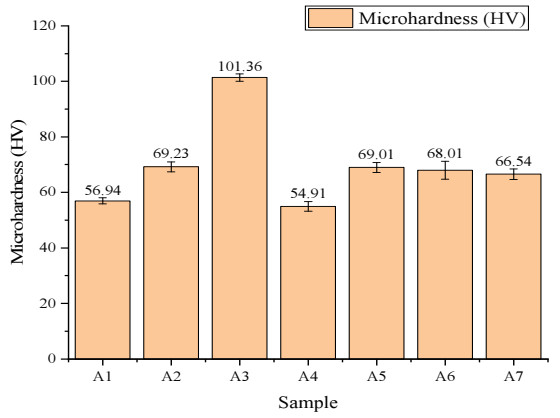

Fig. 10. Micro hardness of AA7075/Al_2O_3/CNT composites

They concluded that, in comparison to a V-Blender and a V-Blender with steel balls, the barrel mix approach enhances hardness. In this study, as the amount of CNT reinforcing material in the composite was increased, its micro hardness also grew, indicating that the amount of reinforcement directly correlates with the hardness. The composite containing 0.1 wt.% CNT exhibited a peak value of 101.36 HV, surpassing the base alloy and other composites, as illustrated in Figure 10. Salur et al. [43] reported on increased hardness values that were obtained during longer ball milling times as a result of significant plastic deformation.

CONCLUSIONS

Using the powder metallurgy procedure, AA7075 hybrid composites were successfully created. Microstructural analysis revealed homogeneous distribution of the nano-reinforcement with smaller crystallites and refined matrix grains at the lower CNT wt.%. The development of the matrix grain was constrained by both reinforcements. The differences in the dislocation size, lattice strain, and crystallite size of the composites were clearly shown by the XRD examination. The compressive strength of the composite is higher than pure AA 7075 alloy at 0.2 weight percent CNT reinforcement. The maximum micro hardness of the A3 composite is 101.36 HV (0.2) at 450°C sintering temperature, 0.1 wt.% CNT reinforcement, and a 6-hour milling time.

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