

Synthesis and characterization of graphene nanoplatelets reinforced AA7068 matrix nanocomposites produced by liquid metallurgy route

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Abstract

In this paper, we report the synthesis of graphene nanoplatelets (GNPs) reinforced AA7068 metal matrix nanocomposites by a combination of ultrasonication, powder and liquid metallurgy techniques. The effect of GNPs with a varying weight fraction (0.1, 0.3, 0.5, 0.7 and 1 wt.%) on microstructure, mechanical and tribological properties of AA7068 metal matrix is studied. In addition to this the developed nanocomposites were subjected to the standard T6 heat treatment conditions and its effect on aforementioned properties was also studied. Mechanical properties of as cast and T6 heat treated nanocomposites showed increase in tensile strength and elongation values up to 0.5 wt.% GNPs reinforced AA7068 nanocomposites, thereafter drop in these properties was observed. A similar behavior was observed in case of grain size, where grain refinement of AA7068 matrix by GNPs reached saturation point for 0.5 wt.%. The average ultimate tensile strength (UTS) of the specimens before and after extrusion processes and T6 heat treatment increases from 263 MPa to 372 MPa and 495-615 MPa after adding 0.5 wt.% GNPs, respectively. Finally, the wear resistance of the AA7068 alloy and its nanocomposite with 0.5 wt.% GNPs in T6 conditions was significantly higher than that of as cast conditions. The obtained results indicate that GNPs

(up to 0.5 wt.%) are effective in improving the mechanical properties and wear resistance of AA7068 metal matrix in both as cast and T6 conditions.

Keywords: Graphene, Nanocomposites, Mechanical properties, Wear

1- Introduction

The demand for cost reduction in the area of transportation like automotive or aerospace applications calls for decrease in fuel consumption which in turn can be achieved by weight reduction. In this regards aluminium and its alloys have been the primary candidate for the structural parts owing to their low density, high strength and ductility. In particular the newly designed and developed AA 7000 alloy series are mainly used for high strength applications due to their important properties like age treatable, high formability, good welding properties and energy absorption. Examples of this series include bumper beams, chassis parts and crash boxes for light weight cars while extrusion of fuselage and upper wing structure for commercial aircrafts [1,2]. The properties of these alloys can be further improved by adopting new processing techniques [3], usage of grain refiners [4], proper heat treatment [5] and inclusion of micron or nano-sized reinforcements [6]. Super high strength aluminum alloys have been extensively studied after mechanical deformation for several decades [5–7], but little attention has been made on the alloy in as-cast condition and semi-solid state. As-cast structures of these alloys have a significant influence on their mechanical properties and the quality of finished products [6]. The structure of such materials can be controlled by some important factors such as: changing the composition, adding grain refining agents, minimizing inclusions and applying thermomechanical treatments [7]. The use of high concentrations of alloying elements results inhomogeneity in the microstructure and severe

segregation of second phases. In casting products, the mechanical properties may vary from location to location due to the variations of grain size, the amount of eutectic phases and the amount of precipitates. Much attention has been made to reduce the segregation of the alloying elements during solidification period of high-alloyed Al alloys [5-9].

After the discovery of carbon nanotubes in the year 1991 by Japanese scientist Iijima [10], much work has been done in the development of new carbon allotropes like diamond nanorods, carbon nanofoam and GNPs. Out of all these the carbon nanotubes had dominated last decade as a potential reinforcement for polymer, metal and ceramic matrices. In particular carbon nanotubes have been used to reinforce most of the metallic matrix materials like, aluminium, copper, magnesium and nickel. Most of these studies were focused on improving the mechanical and tribological properties of these metallic matrices [11-14]. But past few years, single and multilayer GNPs is been used as a reinforcement owing to its excellent mechanical and thermal properties. The mechanical properties like elastic modulus and intrinsic strength of defect free monolayer GNPs measured by Lee et al. [15] using nanoindentation in an atomic force microscope were 1.0 TPa and 130 GPa respectively. The thermal conductivity of a suspended single layer GNPs measured at room temperature was close to that of 4000-5300 W/mK [16]. Such unique properties of GNPs make them attractive for making nanocomposites as a reinforcement material.

Most of the works reported on the metal matrices using GNPs were either on aluminium or copper based. Out of various processing routes used for development of GNPs composites most of the studies adopted powder metallurgy as primary processing and hot extrusion or hot rolling as secondary processing route [17,18]. Kumar et al. [17] reported mechanical properties of aluminium-GNPs composite synthesized by powder metallurgy route and hot

extrusion. In order to obtain uniform dispersion of GNPs, both aluminium and GNPs were dispersed in ethanol and then subjected to magnetic stirring. The cold compaction was carried out under a pressure of 350 MPa and sintered at 560 °C for 4 h. By addition of GNPs a significant amount of grain refinement in the aluminium matrix was observed. The yield and tensile strength was increased from ~150 and ~180 MPa for pure aluminium to 260 and 270 MPa for composite respectively. The increase in mechanical properties of composites was attributed to the strengthening mechanisms like, dislocation strengthening and grain refinement. Rashed et al. [19] reported the synthesis of pure aluminium and GNPs (0.3 wt.%) composite by semi powder technique in which the powders were mixed using mechanical agitator, then cold compacted, sintered and finally hot extruded at 470 °C. Mechanical properties such as yield and ultimate tensile strength of composite compared to that of monolithic aluminium were increased by 14.7 and 11.1 wt.%. Both the above mentioned studies opted for suspension of GNPs in organic media and then addition of metallic powder to it so that there is uniform dispersion of GNPs in metallic powder. The dispersion of GNPs is very important step to obtain good mechanical and tribological properties. But however with the increase in percentage of GNPs in the metallic matrix, irrespective of processing technique used it is very hard to obtain good dispersion. These GNPs will form some agglomerates and act as a porosity leading to poor mechanical and thermal properties [20,21]. So it is very necessary to obtain a saturation point of GNPs inclusion in the metallic matrix so as to obtain good properties.

In this paper we report the development of GNPs reinforced AA7068 matrix nanocomposites. Here the AA7068 alloy is chosen as a matrix material because of its high hardness and ultimate tensile strength. In addition this, it is a heat treatable alloy and light

weight compared to that of brass, copper and steel. The AA7068-Gr nanocomposites were synthesized by a combination of powder metallurgy and stir casting with ultrasonic waves. The AA7068 with and without GNPs were subjected to mechanical and wear testing to evaluate the strength and wear resistance.

2- Experimental procedures

The materials used in the present investigation were 99 wt.% pure aluminum powder with an average particle size of 45 μm , GNPs with an average thickness of approximately 5-10 layer and an average diameter of 3-25 μm and AA7068 alloy. Fig. 1 shows the scanning electron microscopy (SEM) micrographs of GNPs. To produce nanocomposites, the GNPs were dispersed in 99.5 wt.% ethanol by ultrasonication. The composite slurry was subjected to ball milling for 2 h at 250 rpm using a ball to powder ratio of 10:1.

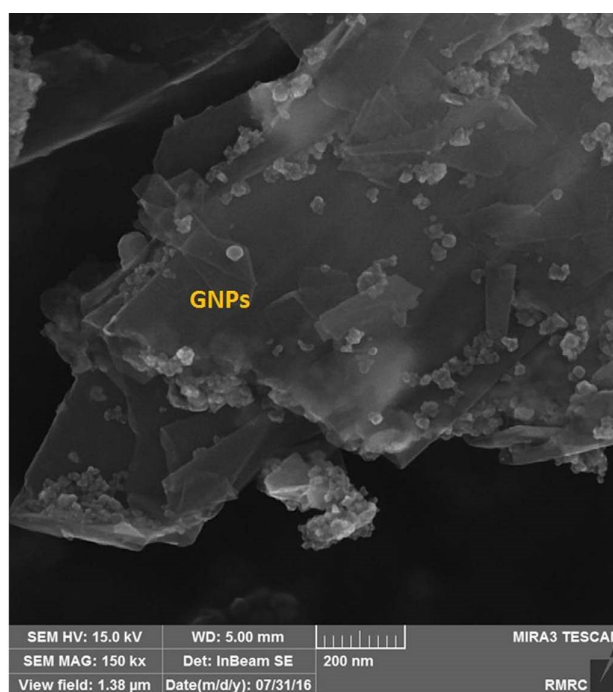


Fig. 1. SEM images of Exfoliated GNPs.

The chemical composition of the AA7068 aluminum alloy used as matrix material in this work is given in Table 1. The AA7068 aluminium alloy ingots cut into various small pieces and then placed into a graphite crucible. The graphite crucible was placed in an electrical resistance. Melting of aluminium alloy was done by heating it to a temperature of ~ 750 °C. Then, stirring of AA7068 alloy melt was accomplished for 10-15 min with the help of a mechanical stirrer (500 rpm) and 60 s with 2000w ultrasonic waves to homogenise the uniform temperature throughout followed by addition of pre-heated aluminum powder and GNPs composite with different wt.% in the metal melt. Nanocomposites specimens have been prepared with of 0.1, 0.3, 0.5, 0.7 and 1 wt.% GNPs. The heat numbers of different GNPs are listed in the Table 2. After successful addition of GNPs and uniform mixing throughout, the composite melt was poured into a permanent mold designed and fabricated according to ASTM B557M-10 standard (see Fig. 2). The advantage of using permanent mold is the provision of appropriate uphill filling system and feeding design. This design will have a low turbulence manner of fluid flow resulting in reduced air entrapment and porosity in the fabricated composite specimens.

Table 1. Chemical composition of the AA7068 primary ingots (wt.%).

Al	Zn	Mg	Cu	Ti	Mn	Fe	Si
Rem	8.2	3	2.46	0.1	0.1	0.16	0.03

Table 2. Specimens numbers of the master alloys.

Specimens no.	GNPs (wt%)
S0	—
S1	0.1
S2	0.3
S3	0.5
S4	0.7
S5	1

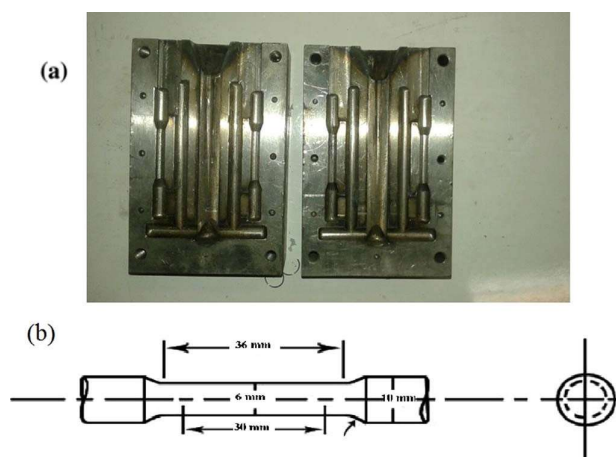


Fig. 2. Tensile specimen geometry and dimensions (a) cast iron mold and (b) tensile sample dimensions.

For microstructural studies, optical microscope equipped with an image analysis system (Clemex Vision Pro. Ver.3.5.025), HRTEM (High-resolution transmission electron microscopy, make: Philips CM200 at an accelerating voltage of 200 kV) and SEM (Make: Cam Scan MV2300) equipped with an energy dispersive X-ray analysis (EDX) have been used. The cut nanocomposite sections were polished using SiC based abrasive papers and then etched by Keller's reagent (2 ml HF, 3 ml HCl, 5 ml HNO₃ and 190 ml H₂O) to reveal the structure. The average grain size of the nanocomposite specimens were measured in

accordance with the ASTM: E112 standard. Phase identification was performed by X-ray diffraction method (Make: Philips PW 1830). To study the effect of heat treatment (T6 conditions) on mechanical properties, the as cast AA7068 alloy and nanocomposite samples were heated at 460 °C for 24 h and water quenched. Finally the quenched samples were tempered at 120 °C temperature for 24 h and were furnace cooled [2].

Before tensile testing, extrusion processes was applied to the T6 heat treatment specimens. Subsequently the alloy is hot extruded by using a hydraulic press at a ram speed of 1 mm/s at 320 °C from round initial ingot with 29 mm diameter to exit section bar with extrusion ratios (which is the ratio of the initial cross-section area of the billet to the final cross-sectional area after extrusion) of 6:1. Extrusion process was carried out with using graphite base oil between metal and dies.

Tensile testing on all the nanocomposite samples was performed at room temperature using KOOPA universal testing machine at the strain rate of 1 mm/min. Four test bars were tested for each nanocomposite sample and the average value is reported here. Hardness test was carried out according to ASTM E10 standard to check resistance of nanocomposite towards the plastic deformation (Make: ESEWAY 7500 CCD universal hardness tester, 30 kg force and indenter 2.5 mm diameter). Dry sliding wear tests were conducted in accordance with ASTM G99 standard using a pin-on-disc set up at room temperature (see Fig. 3). The counterface used was DIN 100Cr6 steel disk with a hardness of 62 HRC. The pins 5 mm diameter and 20 mm length were in a conformal contact with the steel disk. All the samples were tested at a rotational speed of 250 rpm, which corresponds to a speed of 0.5 ms⁻¹, under nominal loads of 20 and 40 N. A fixed sliding distance of 1500 m was chosen for testing all

the samples. The mass of the all specimens before and after the wear test were recorded using an electronic balance (Make: GR200-AND) with an accuracy of 0.1 mg.

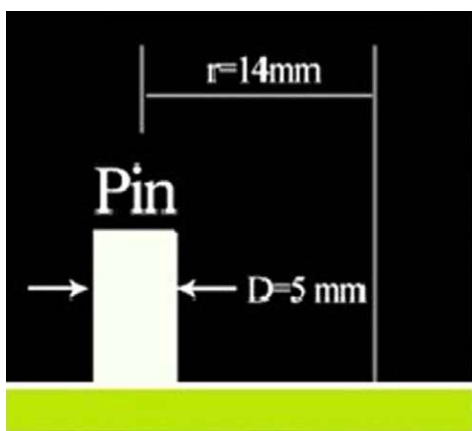


Fig. 3. Schematic of pin-on-disk configuration.

3- Results and Discussion

3-1- Microstructural Characterization

The initial preform of Al-GNPs nanocomposite was prepared by mechanical alloying and powder metallurgy technique. The initial particle size of pure aluminum powder before ball milling was found to be $\sim 45 \mu\text{m}$ and the morphology was spherical. After 2 h of milling, the particle size of aluminum powder decreased by milling process, which will have better dissolution and lower agglomeration during stir casting. Fig. 4 shows the TEM micrographs of morphology and size of flake shaped Al-GNPs nanocomposite powders after 2 h of milling. The morphology of the aluminum powders after milling change to flaky shape due to intense plastic deformation (as shown in Fig. 4-e). The GNPs were found to be entrapped in between these flattened aluminium powder particles and were distributed uniformly throughout them. Many studies have shown that mechanical alloying technique is very effective for uniform dispersion of nanosized reinforcements in the metal matrix [22,23].

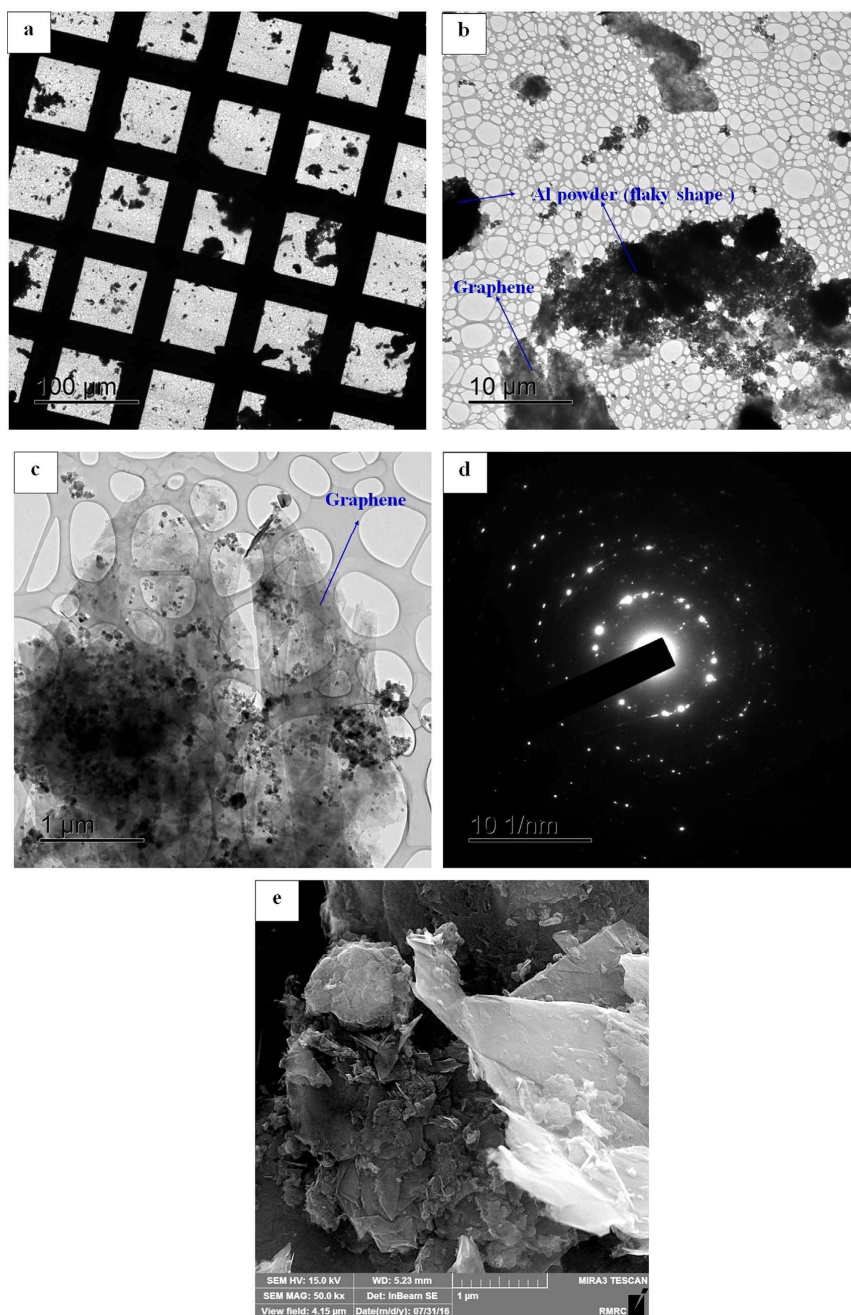


Fig. 4. TEM micrograph of (a) and (b) powder morphology of Al-25 wt.% GNPs (c) and (d) high magnification image of Al-25 wt.% GNPs associated with selected area diffraction (SAD) pattern of specified region and (e) SEM micrograph of Al-25 wt.% GNPs powder morphology after 2 h of milling.

It is obvious that dissolution of small powders in the melt is much easier relative to the larger powder particle preform ones. Besides, formation of nanodispersions is mainly caused by two phenomena. During insertion of powder preforms into the AA7068 melt, dissolution of aluminum powders occurs, which act as carriers of GNPs to the melt and also protect them from any contact with the surface of the melt. After dissolution, GNPs are released in the AA7068 melt. In addition to this, ultrasonic vibration prevents agglomerated particles from being sintered. It has been observed that aluminum powders which are in the inner part of the preforms act as a binder at 700 °C avoiding separation and release of GNPs, which only could be separated by means of ultrasonic vibration during stirring.

Fig. 5 shows the influence of various amounts of GNPs on the grain refinement of AA7068 matrix. Several mechanisms have been proposed for the grain refinement process which includes influence of secondary particles on the metal matrices. The average grain size of unreinforced AA7068 aluminum alloy was found to be in range of 550 μm . The addition of GNPs to AA7068 matrix has significantly refined the coarse columnar primary $\alpha\text{-Al}$ grains to fine equiaxed $\alpha\text{-Al}$ grains of average size of 70 μm . The main reason is that these nanoplatelets act as nucleating agents during the solidification of $\alpha\text{-Al}$ grains. With increasing content of GNPs, these nanoplatelets pin the grain boundaries and increase the grain refinement, deflect cracks and cause increase of strength in nanocomposite. When wt.% of GNPs is more than 0.5 wt.%, the grain boundaries have reached saturation and effect of grain refinement diminished. Fig. 6 shows the SEM micrographs of as cast microstructures of unreinforced and varying weight percentage of GNPs reinforced AA7068 matrix nanocomposites. The micrographs clearly show there is significant change in dendrite morphology of the AA7068 aluminum alloy after adding GNPs. The microstructures of alloy

revealed a rosette-like microstructure of primary α -Al grains solid solution surrounded by interdendritic secondary phases. In comparison with GNPs added specimens, unrefined specimens showed coarser morphology. From Fig. 6, it is noticeable that GNPs and ultrasonic assisted stir casting increases the number of grain boundaries and therefore promotes a more homogeneous distribution of intermetallic precipitates. The optimum content of GNPs that can be used to reinforce AA7068 matrix is 0.5 wt.%. XRD analysis was undertaken for phase identification the unreinforced and GNPs reinforced AA7068 nanocomposite (samples S0 and S3) which are shown in Fig. 7. The XRD results showed the presence of α -Al in the as cast alloy as the major element in the nanocomposite. The presence of GNPs was also confirmed from the XRD pattern as shown in Fig. 7b.

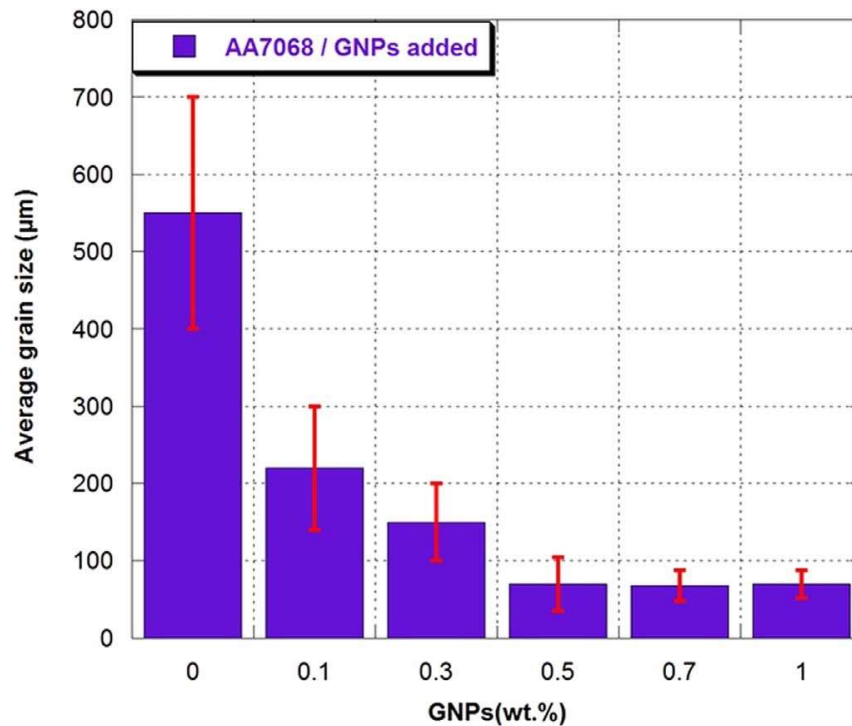


Fig. 5. Grain size of AA7068 with various GNPs contents.

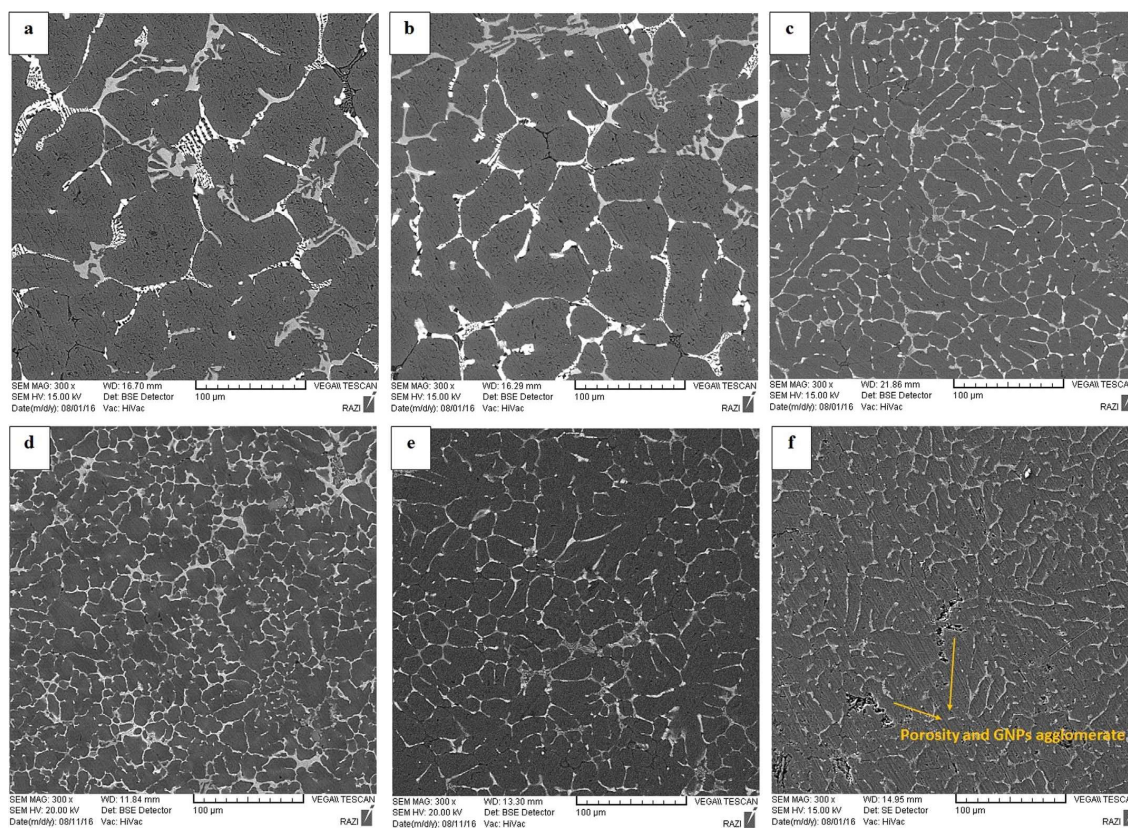


Fig. 6. SEM images of microstructures of AA7068, with (a) 0.0 wt.% GNPs, (b) 0.1 wt.% GNPs, (c) 0.3 wt.% GNPs, (d) 0.5 wt.% GNPs, (e) 0.7 wt.% GNPs and (f) 1 wt.% GNPs.

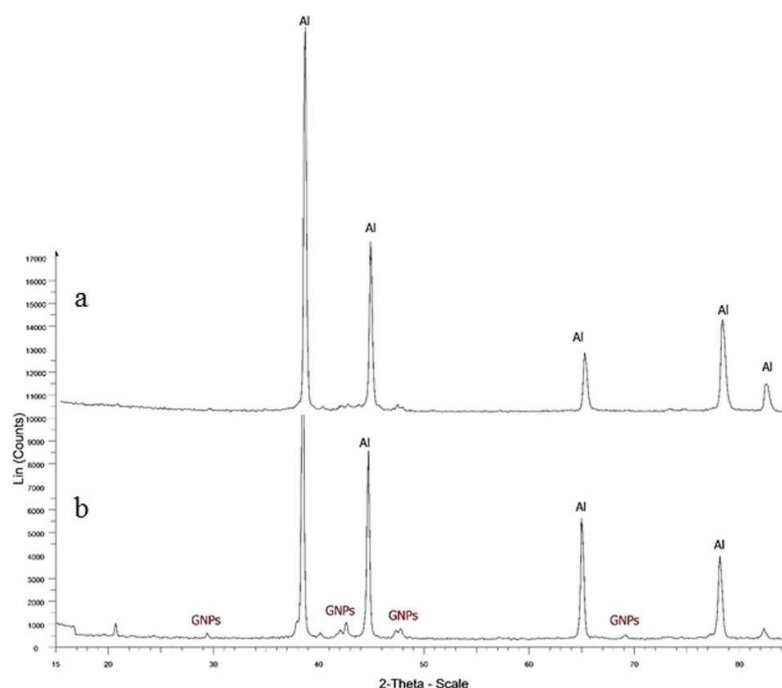


Fig. 7. XRD pattern of (a) AA7068 and (b) AA7068-GNPs.

Fig. 8 shows SEM micrograph of unreinforced AA7068 as cast alloy. The lamellar eutectic structures like α -Al and η -MgZn₂ phases were clearly seen from the micrographs. It is important to note that the solubility of η -phase for copper is relatively high, which is observed in the eutectic structure. In addition to this, the EDS carried out on this alloy showed that the presence of MgZn₂ intermetallic which indicates that magnesium and zinc contents are higher than the average level of chemical composition in the alloy. It is obvious that the MgZn₂ dissolved the Cu and Al elements, and formed the Mg(Zn,Cu,Al)₂ phase, which is consistent with the present literatures [8]. The segregation of solute that occurred during ultrasonication assisted casting led to the high concentration of Cu, Mg and Zn in the interdendritic eutectic regions. It is well known that the diffusion velocity of copper is lower than zinc and magnesium, which results in the higher concentration of copper in the regions of eutectic structures during solidification. The driving force for the phase transformation to

Al_2CuMg from $\text{Mg}(\text{Zn,Cu,Al})_2$ phase must be super saturation of copper in the regions of eutectic structures, which makes Al_2CuMg a stable phase. Figs. 9 and 10 show the distribution of GNPs in the AA7068 matrix in as cast condition. The nanocomposite with the 0.5 wt.% of GNPs showed that the nanoplatelets were uniformly distributed in AA7068 matrix compared to that of 1 wt.% GNPs reinforced nanocomposite. As the GNPs content increases beyond 0.5 wt.%, the chances of clustering is very high and is quite evident from the Fig. 6-f. Agglomeration of GNPs can be observed in the year the nanocomposite with 1 wt.% of GNPs. These agglomerates cause grain boundary embrittlement and porosity, resulting in weaker strength.

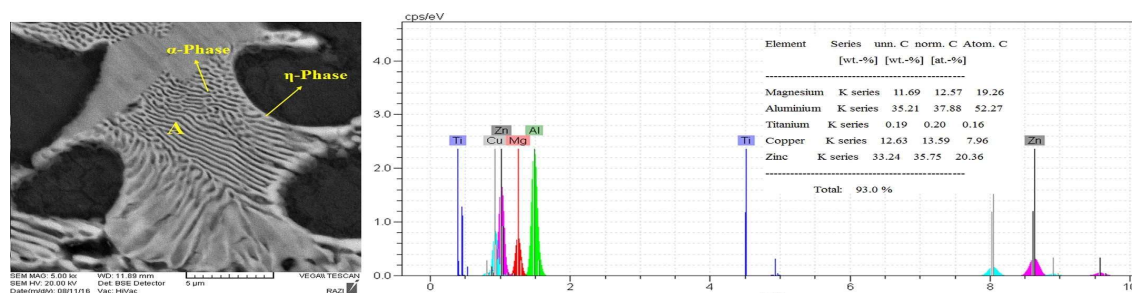


Fig. 8. Microstructures and EDX analysis of eutectic phase in as-cast AA7068 specimen.

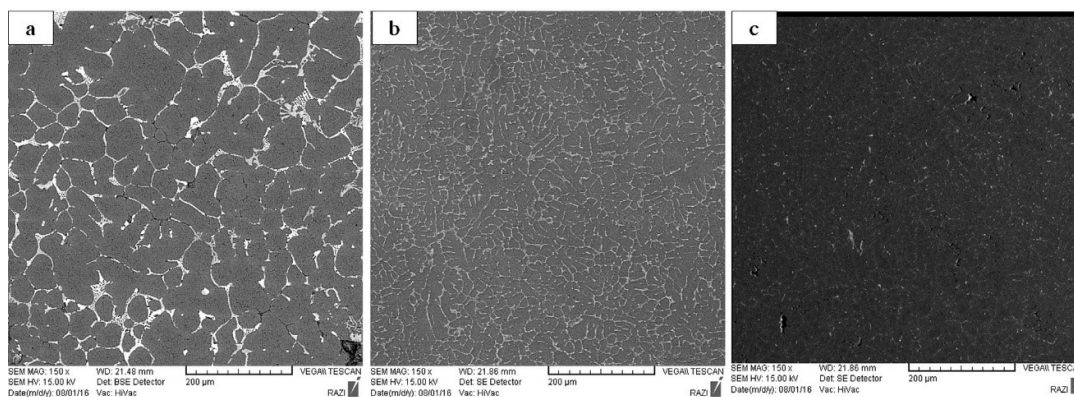


Fig. 9. SEM back-scattered images, showing the microstructures of the AA7068 alloy with (a) 0.0 wt.% GNPs and (b) 0.5 wt.% GNPs, (c) 0.5 wt.% GNPs after T6 heat treatment.

3-2- Solidification Mechanisms

Three class of models has been developed regarding incorporation of particles into a solidifying metal matrix, namely (i) kinetic models predicting velocity of the solid/liquid interface which is critical for the transition from particle pushing to engulfment, (ii) thermodynamic models [24] corresponding to classical heterogeneous nucleation theory and the last one (iii) models based on the ratio of the thermophysical properties of the particles/reinforcements and the melt [25,26]. The latter model predicts the incorporation ability in general, that means dependency of incorporation behaviour on the processing conditions. The last two models demonstrate that thermal conductivity of particles incorporated into the liquid matrix is directly related to change of the interface shape from convex to concave as the solidification of the metal matrix does depend upon the particles/reinforcement present [24-28].

In fact, thermal conductivity of the will have significant effect on temperature gradient ahead of the solidification front. Therefore, reinforcements will contribute towards the removal of the heat necessary for further solidification and consequently inhibits metal matrix grains from further growing. As we know that the thermal conductivity of GNPs is very high which help in removal of excess heat necessary for the solidification of the metal matrix grains to grow larger and there by effectively inhibiting the grain growth. So as these grains are not allowed to grow further, one can observe from the SEM micrographs shown in Fig. 6 and 9, that with the increase in GNPs content the grain size is decreasing until 0.5 wt.%, beyond which the refinement is not so significant. Fig. 10 shows the element mapping of AA7068-0.5 wt.% Gr nanocomposite, in which all the major alloying elements are shown with different colour codes. The EDS mapping confirms the presence of GNPs by showing carbon element mapping.

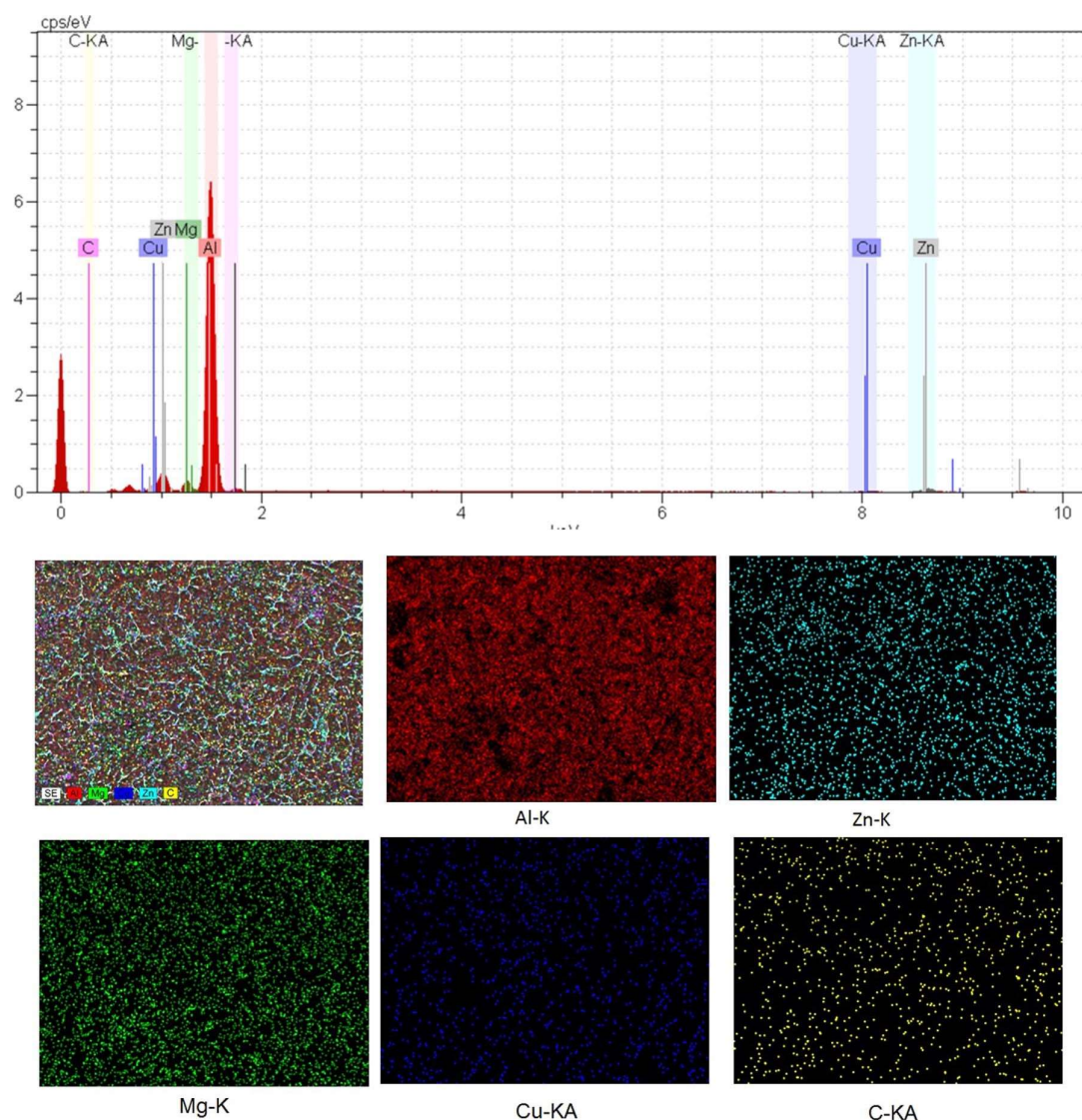


Fig. 10. Energy dispersive X-ray and map analysis of the AA7068-0.5 wt.% GNPs nanocomposite and distribution of the major elements.

3-3- Hardness results

Fig. 11 shows the hardness results of the unreinforced and GNPs reinforced AA7068 matrix with 0.5 wt.% GNPs before and after solutionizing, aging (T6 heat treatment) and extrusion process. As it is shown in Fig. 11, the average hardness of the specimens increases

from 115 BHN to 136 BHN and 139 BHN to 175 BHN after adding 0.5 wt.% GNPs for samples before and after T6 heat treatment and extrusion process, respectively. Adding of GNPs, T6 heat treatment and extrusion process make about 52% enhancement in hardness compared to that of unreinforced aluminum alloy. The slight improvement in hardness of the nanocomposite specimens could be a result of the effective dispersion of GNPs in the matrix. As expected, the hardness values of the nanocomposites were found to be higher than those for the unreinforced ones. This may be due to the solid solution hardening of AA7068 alloy by secondary solid elements and presence of GNPs at elevated holding time and temperature. The Fig. 11 confirms in the end that both alloy and nanocomposite are sensitive to T6 heat treatment.

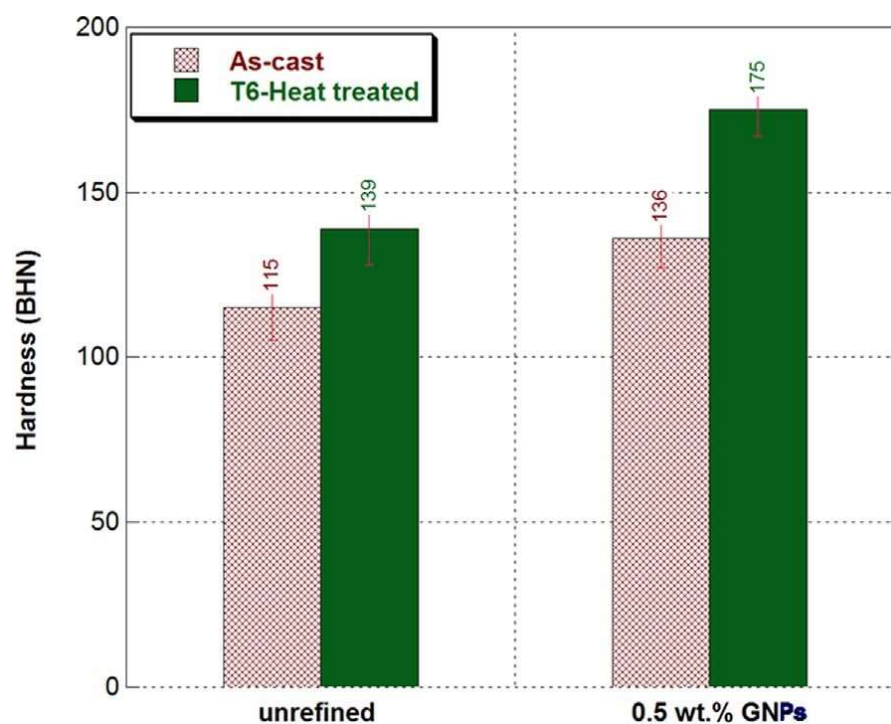


Fig. 11. Hardness of samples for unrefined and refined by 0.5 wt.% GNPs.

3-4- Tensile Properties

Fig. 12 shows the ultimate tensile strength (UTS) values of the AA7068-0.5%GNPs nanocomposite before and after heat treatment and extrusion process. From comparison of UTS and elongation values it is observed that the addition of 0.5 wt.% GNPs was the most effective amount to enhance tensile properties of the castings when compared to that of other GNPs wt.%. The tensile strength increases with increasing wt.% of GNPs but beyond 0.5 wt.% the strength values start to drop. Though the drop is observed in strength values after 0.5 wt.% but they are significantly higher than that of AA7068 alloy. As it is shown in Fig. 12, the average ultimate tensile strength of the specimens increases from 263 MPa to 372 MPa and 495 MPa to 615 MPa after adding 0.5 wt.% GNPs for samples before and after T6 heat treatment and extrusion process, respectively. Mechanical properties of the AA7068 alloy mainly depend on the GNPs, shape, size, wt.%, dispersion in the metal matrix and the distribution of the intermetallics in the interdendritic region [3]. The first reason for this improvement is high probably due to the extensive grain refinement caused by the GNPs, which also helps in finer distribution of second phases like intermetallics. According to Hall–Petch theory we know that finer the grains, the higher the strength we will get. So, in present work a comparison between grain size and strength was done and studied. But, the strengthening of AA7068 matrix cannot be contributed by GNPs alone, especially after heat treatment or precipitation hardening. So observed results tell us that the dependence of the strength to grain size can be due to multiple factors.

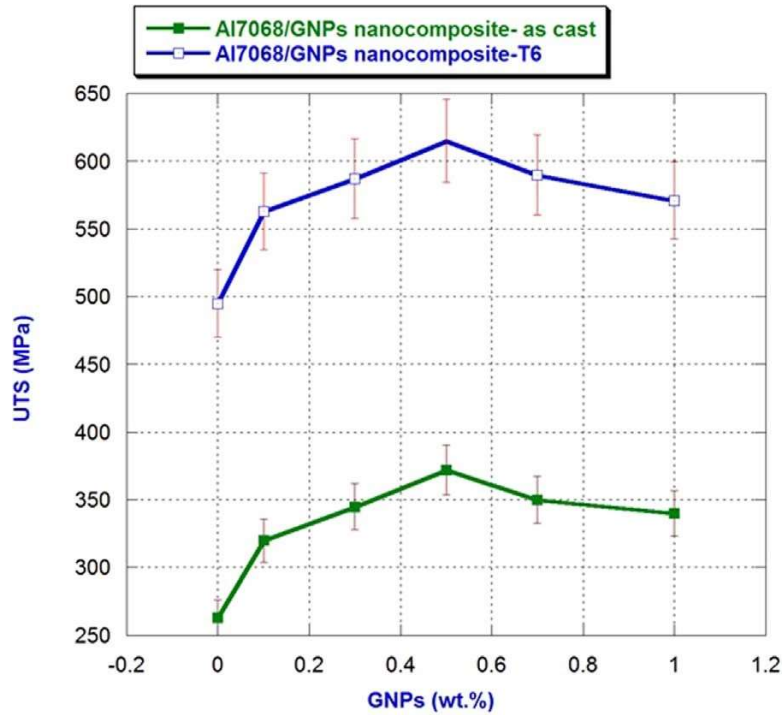


Fig. 12. Mechanical properties of AA7068 nanocomposite at the different conditions.

Some studies found that the high improvement in the strength of GNPs nanocomposites was due to various strengthening mechanisms, which includes stress dispersing, crack deflection, bridging, and branching (see Fig. 13) [29]. The strong interfacial bonding between GNPs and AA7068 matrix can increase the load-transfer efficiency and eliminate the destruction of stress concentration. In addition, the GNPs provide a higher resistance to crack propagation. When the initiate at the surface of tensile sample and start propagating inside the matrix grain but when it encounters the GNPs, the crack will be inhibited from further movement until the load increases. Further increase in loading during testing will force the crack to deflect as shown in Fig. 14. The crack branching or deflection mechanism could increase the path of crack development, which improves the mechanical strength of AA7068 matrix. Moreover, the crack bridging mechanism of GNPs could absorb the more energy

effectively, when the composite is under external load. The whole mechanical map is presented in Fig. 13. In a word, due to the high tensile strength and high Young's modulus, GNPs can effectively reduce the stress concentration and prevent the development of the cracks, thus enhancing the mechanical strength of the AA7068 matrix. These results are helpful in understanding the reinforcing effect of nanosized reinforcements like GNPs used for metal matrix composites.

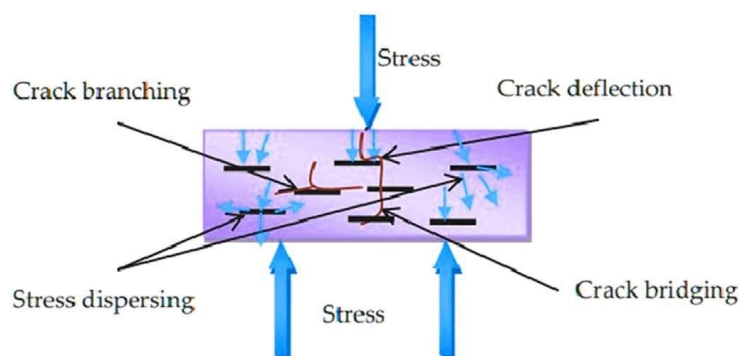


Fig. 13. Scheme of AA7068-GNPs nanocomposite under load.

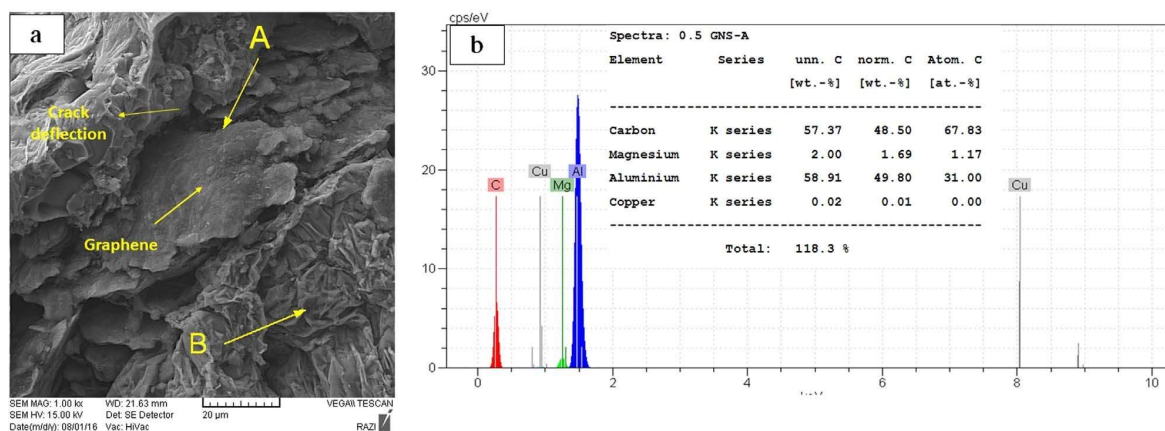


Fig. 14. SEM micrograph of (a) the fractured AA7068-0.5 wt.% GNPs specimen and (b) EDX of GNPs in fracture surface.

3-5- Fractography of tensile specimens

Figs. 15a and 15b shows SEM micrographs of the fracture surface of as cast AA7068 alloy after tensile testing. Fracture surface observations do reveal the presence of dendritic structures. This indicates inter-dendritic cracking as the main fracture mechanism operating in the case of as cast samples. During solidification process of alloy, the alloying elements get segregated at the inter-dendritic regions. These regions help for propagation of micro-cracks during loading and result in the failure of the samples. Further casting defect such as shrinkage pores has also been seen in alloy. These shrinkage pores favour for the formation of local micro-voids leading to linkage of micro-cracks and causing failure of the samples. Figs. 15c and 15d shows the fracture surfaces of the GNPs-refined specimens at higher magnifications. The AA7068 nanocomposite exhibited ductile fracture behaviour with fine dimples observed the fracture surface (see Figs. 15c and 15d). In Fig. 14 GNPs shows in the fracture surfaces. The main reason for this improvement is high probably due to the reduced grain size of the castings, leading to a finer distribution of second phases (i.e. intermetallics) and crack deflection in matrix of nanocomposite in AA7068 alloy.

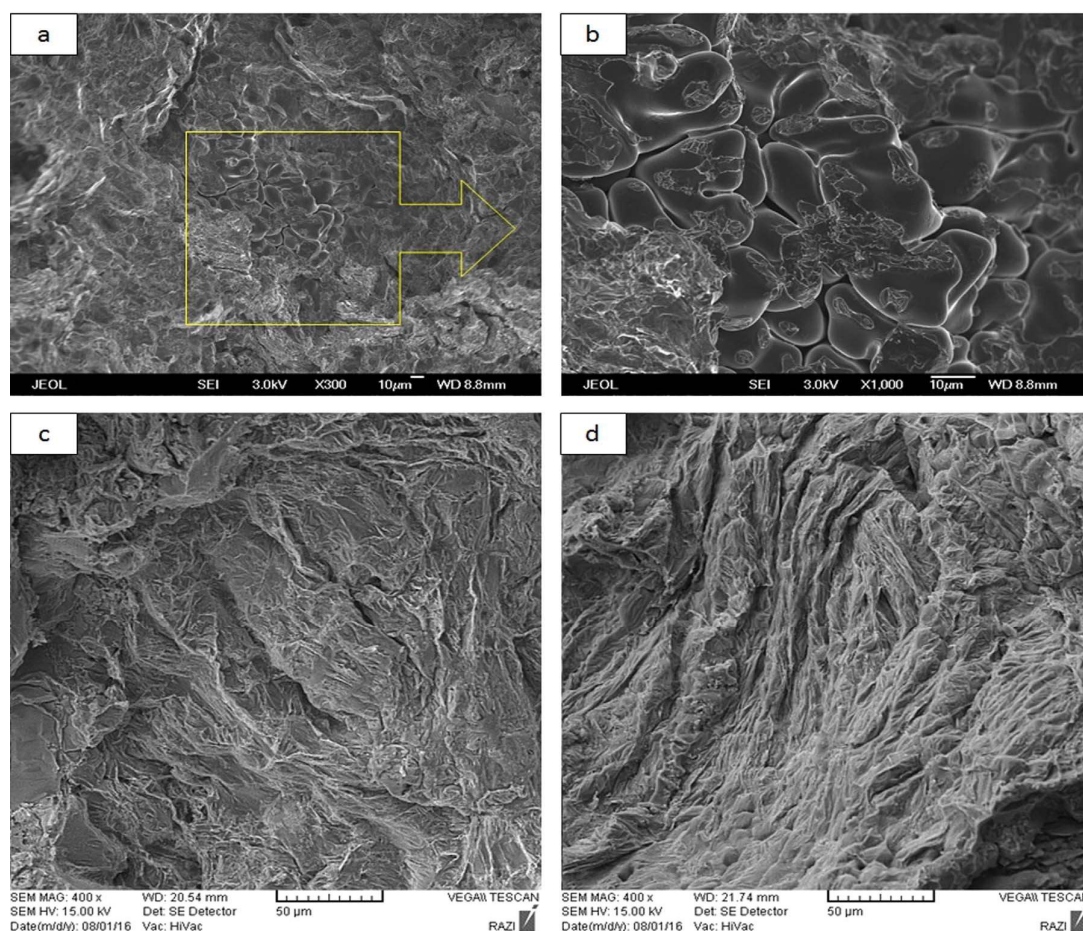


Fig. 15. Fracture surfaces of (a-b) AA7068 alloy and (c-d) AA7068-0.5 wt.% GNPs.

3-6- Wear behavior of AA7068/GNPs nanocomposite

Fig. 16 shows the variation of coefficient of friction (COF) with normal load for AA7068 alloy and nanocomposite before and after T6 heat treatment and extrusion process. As it is shown in Fig. 16, the COF of the specimens decreases from 0.26 to 0.25 and 0.23 to 0.21 after adding 0.5 wt.% GNPs for samples before and after T6 heat treatment and extrusion process, respectively. The results indicate that the COF decreases with increasing normal loads in all cases. The results show that the COF change significantly by adding 0.5 wt.% of GNPs to the AA7068 matrix because of sufficient amount of solid lubricant available

at the contact surface. On the other hand, heat treated nanocomposite with 0.5 wt.% GNPs had lowest COF compared to that of as cast and heat treated alloy and nanocomposite.

The wear results for AA7068 alloy and nanocomposite with and without T6 heat treatment and extrusion process is shown in Fig. 17. The amounts of weight loss against sliding distance for all the samples are plotted. This plot was obtained at constant normal loads (20 N and 40 N) with a constant rotation speed of the counter disk of 250 rpm. As it is shown in Fig. 17, the weight loss of the specimens decreases from 13.5 mg to 6.5 mg and 8 mg to 4 mg after adding 0.5 wt.% GNPs for samples before and after T6 heat treatment and extrusion process, respectively. Adding of GNPs, T6 heat treatment and extrusion process make about 237% decreases in weight loss compared to that of unreinforced aluminum alloy. It observed that the amount of weight loss has increased by increase in sliding distance showing a linear trend. Comparison between unreinforced and nanocomposite samples before and after T6 heat treatment and extrusion process shows that the addition of GNPs to AA7068 matrix has reduced the weight loss in comparison with unreinforced AA7068 alloy. The decrease in weight loss is mainly attributed to uniform distribution and strong interfacial bonding of GNPs with AA7068 matrix.

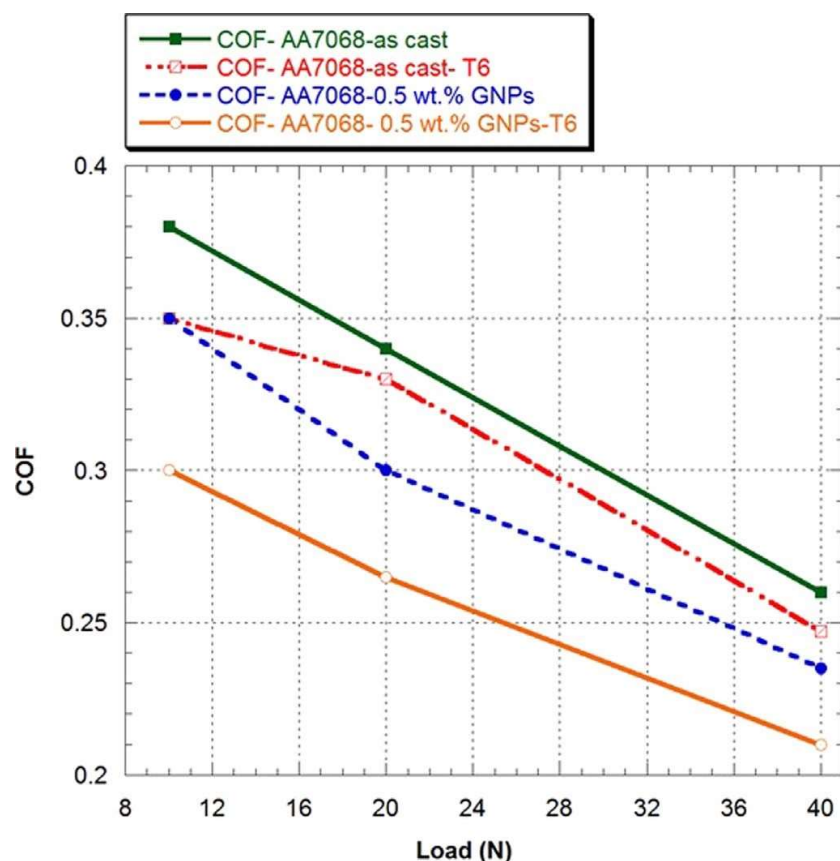


Fig. 16. COF values of the nanocomposite as function of load amount.

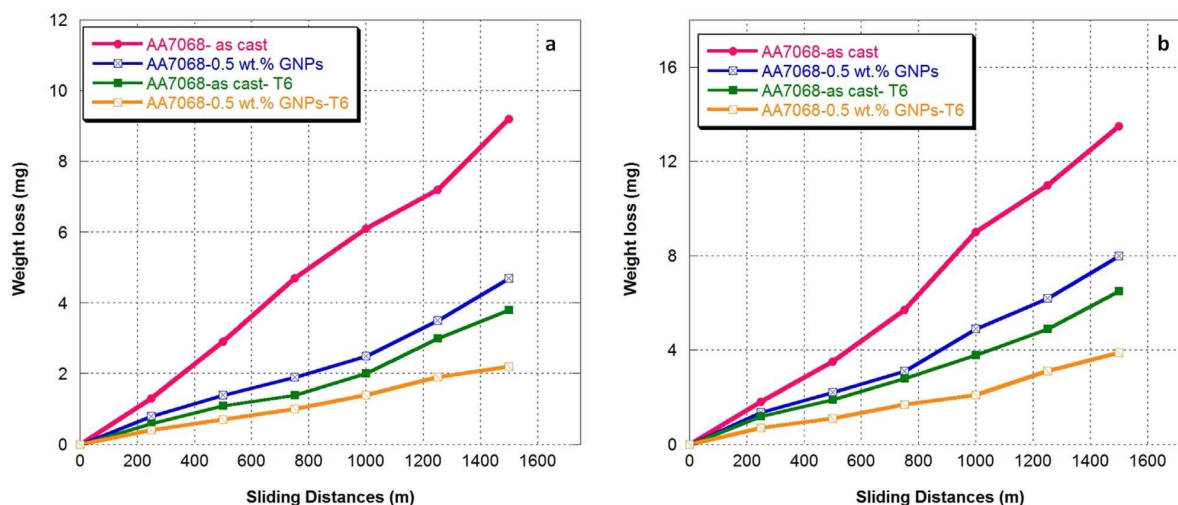


Fig. 17. Weight loss as a function of sliding distance for unrefined and refined nanocomposites with and without T6 heat treatment, (a) 20 N and (b) 40 N.

An interesting observation was that the T6 heat treated AA7068 alloy had better wear resistance compared to that of unheat treated AA7068-0.5%GNPs nanocomposite. Probably due to the reduced grain size of the castings, leading to a finer distribution of second phases in AA7068 alloy compared to that of AA7068-0.5wt%GNPs nanocomposite. Wear surfaces of unrefined and nanocomposite specimens by 0.5 wt.% GNP with and without T6 heat treatment and extrusion process are shown in Fig. 18. It can be seen a mild wear regime grooves the wear mechanism of the all alloys clearly showing plastic deformation and delamination characteristics. Under an applied load of 40 N, two different regions are distinguished on the worn surface: cavities or craters and smooth regions marked as A and B in Fig. 18, respectively. In the smooth regions, the surface disclosed fine grooves and ploughing which suggest that abrasive wear was prevailing mechanism in these regions. Detached layer in the form of craters or cavities indicates locally adhesive wear due to the formation of and breaking of micro-welds during sliding. In addition, the wear rate of refined alloy by 0.5 wt.% GNP with T6 heat treatment and extrusion process are lower than unrefined in all applied loads. It seems that during the early stages of sliding, delamination wear is the dominant mechanism for the unrefined and nanocomposite specimens by 0.5 wt.% GNP with and without T6 heat treatment and extrusion process. For unreinforced AA7068 alloys with coarse dendritic morphology can be easily broken and contribute to the weight loss.

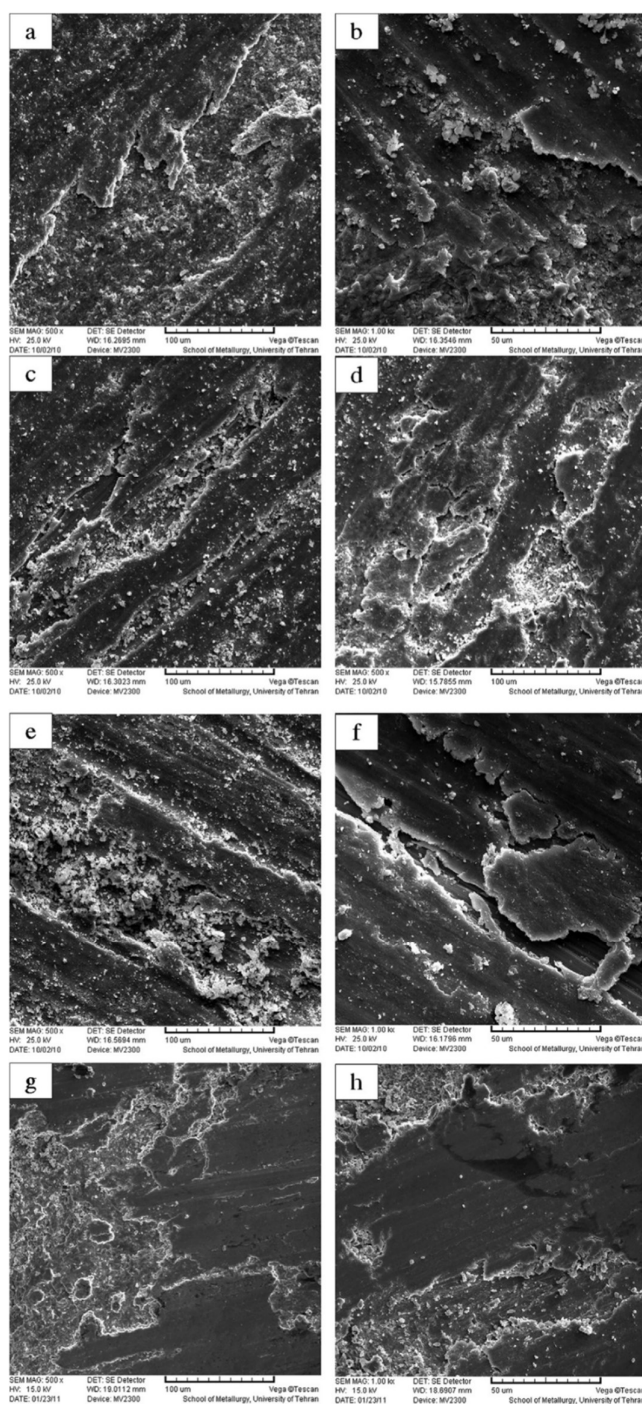


Fig. 18. Wear surface of the AA7068 after 1500 m distance with 40 N load during wear process of (a) and (b) unrefined, (c) and (d) refined by 0.1 wt.% GNPs, (e) and (f) refined by 0.5 wt.% GNPs, (g) and (h) refined by 0.5 wt.% GNPs after T6 heat treatment.

4- Conclusions

In this study, 7068 aluminum matrix nanocomposites reinforced by GNPs was synthesized and the microstructural, T6 heat treatment, mechanical and tribological behavior of these nanocomposites was investigated. The experimental findings are summarized as follows:

1. Stir casting method with ultrasonic waves is very effective for improvement in mechanical properties of 7068 aluminium alloy matrix GNPs reinforced composites and achieves uniform distribution of GNPs in the aluminium matrix.
2. The optimum amount of nanoparticles is 0.5 wt.% GNPs that nanocomposite exhibits tensile strength of 372 MPa and 615 MPa before and after T6 heat treatment and extrusion process, respectively.
3. Adding of 0.5 wt.% GNPs, T6 heat treatment and extrusion process make about 134% enhancement in tensile strength compared to that of unreinforced aluminum alloy.
4. When the wt.% GNPs reaches more than 0.5 wt.%; agglomeration of GNPs at the grain boundaries causes embrittlement, porosities, less interfacial bonding and so decrease in mechanical properties.
5. The coefficient of friction of the specimens decreases from 0.26 to 0.25 and 0.23 to 0.21 after adding 0.5 wt.% GNPs for samples before and after T6 heat treatment and extrusion process, respectively.
6. The SEM investigation of the worn surfaces had shown that abrasive wear was the main wear mechanism in these composites.
7. The lowest wear rate and coefficient of friction of the AA7068 with 0.5 wt.% GNPs is attributed to self-lubricating behavior of the nanocomposites.

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