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Effect of Hot Rolling on Microstructure and Mechanical Properties of Stir Cast AZ 61 Alloy with Minor Additions

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Abstract

Objective: The microstructure and mechanical properties of modified AZ 61 alloy -graphene composite (1wt. %, and 2wt. % graphene) were examined under various conditions of temperature of rolling, number of roll pass, and the amount of deformation. **Method:** The rolling operation was carried out in a laboratory scale rolling with the initial stock temperature of 400^0C. The rolling temperatures were varied and kept at 350^0C 300^0C, and 250^0C. After a few roll passes, the rolled stocks were reinserted within the furnace to make up for the temperature drop. The rolling was continued until the amount of thickness reduction reached 35%. The number of passes varied between 1-4. It was observed that the influence of rolling temperature was quite sensitive to the number of passes to reach the target deformation ~35%. **Finding:** The study on the structure and properties of hot rolled graphene-reinforced AZ-61 magnesium alloy with minor additions of scandium and calcium is scarce in the literature. **Novelty:** In the present investigation attempts are made to probe into the effect of graphene reinforcement in AZ-61 alloy that is compositionally modified by minor additions of scandium and calcium with the expectation of a higher level of strengthening. The results showed that maximum hardness values ranged with in 101HV to 106HV whereas, the maxima of tensile and yield strength were found to be 266 .82 MPa and 102 MPa at an elongation percent of 25%. By refining the microstructure, dynamic recrystallization enhanced the samples' strength and ductility. AZ 61 graphene composite with minor additions of scandium and calcium achieves excellent structural homogeneity and mechanical properties after hot rolling with 2 wt. % graphene rolled at 350^0C.

Keywords: AZ61 Alloy; Rolling Temperature; Graphene; Composite; Tensile Strength; Deformation Percentage

1 Introduction

Due to their high specific characteristics, magnesium alloys may find use as structural materials As a result, magnesium alloys are emerging as the first choice for application in the aerospace and transportation sector. The problem identified regarding magnesium composite is limited wettability between the ceramic nanoparticles and molten magnesium matrix is the primary challenge to be overcome when fabricating magnesium matrix nanocomposites with outstanding mechanical characteristics. Because magnesium melt is more flammable than other metal melts, it poses a greater risk during alloy melting, making it harder to scatter nanoparticles in the melt. This makes it very difficult to create a magnesium matrix nanocomposite. However, their restricted slip systems at ambient temperature give their hexagonal closed-pack (hcp) crystal structure a low formability. Parameters in the rolling process of Mg alloys should be tightly managed to avoid fracture and minimize edge fractures. However, due to a few numbers of available slip systems coupled with the basal texture generated during deformation, magnesium alloy shows poor ductility at low or room temperature. In order to enhance the formability of magnesium alloys, a number of techniques are employed. These include the increase in working temperature, refinement of grains along with the modification of texture, the mechanical parameters of cross-rolled samples including a maximum tensile strength of 106 MPa and a maximum allowable length change before delamination of 7.5%. When cross rolled, samples are heated to temperatures as high as 400°C for an hour, the ultimate tensile strength drops by 10.4 % while the elongation increases by 60 %[(1)]. However, low mechanical properties, and high work hardening rate are still the concerns in the production of sound magnesium strips. Suitably controlled rolling process and rolling temperature is expected to improve the properties of the finished product.

Hot rolling experiments were conducted on AZ31 magnesium alloy specimens at an initial rolling temperature of 350 °C, and the grain distribution and edge damage of the strip after rolling by a controlled rolling reduction in different regions were analyzed in light of the MAS rolling and cross rolling theories. As a result of research into the effects of cross-variable thickness rolling on magnesium alloy, edge curves were developed to reduce edge cracks and refine grains. The findings showed that edge cracks in magnesium alloy might be mitigated using variable thickness rolling in conjunction with the curvature of the edge. In addition, the findings showed that various edge curves may realize the optimal grain refinement in various places throughout the rolling process, resulting in diverse degrees of grain refinement at the strip edge[(2)].

The impact of rolling deformation on the microstructure and characteristics of magnesium alloy has been studied by a number of researchers. Comparing the microstructure of pure Mg and AZX211, the latter's grain size was found to have been reduced by more than half, from 24.5 μm to 10 μm. The AZX211 also included a notable number of dispersed participants, which were later determined to be $(Mg,Al)_2Ca$. It was shown that limited slip system has been responsible for poor formability and ductility of Mg-alloys at room temperature. For this reason, it is necessary to adopt hot or warm rolling for producing a flat work piece from Mg-alloy. In the rolling process, the thickness of Mg-alloy is generally lowered by multiple number of roll passes with a small amount of deformation being given at each pass; intermediate annealing is required to increase the total deformation at minimum edge cracking. The above stated constraints have limited the production and hence the wide spread usage of magnesium alloy flat products. Although some of the shortcomings of conventional rolling could be overcome by the novel rolling methods reported elsewhere, it had been difficult to implement the techniques for industrial manufacturing[(3)].

In order to determine the best craft, additional research was done to examine how traditional rolling was affected by characteristics like temperature and rolling reduction. It is reported by researchers that confirmed that the rolling temperature had a significant impact on the workability of magnesium alloy. Magnesium-based materials are commonly thermally deformed during the rolling process. It is possible to increase the amount of deformation with less energy expenditure because too hot rolling's low deformation resistance. Some flaws in the cast metal may be removed by hot rolling, and the metal's density and strength will be enhanced through the process. Hot rolled products have inconsistent qualities and cannot be precisely regulated to meet mechanical property specifications. Magnesium matrix composites with nanoparticle reinforcement have only been subjected to hot rolling a handful of times. To optimize the Freudenthal fracture criteria for predicting crack initiation and propagation during hot rolling by conducting a quantitative analysis of damage distributions generated by rolling deformation in the transverse direction (TD) for twin-roll casted (TRCed) AZ31 Mg alloy. Edge-cracking behaviors were studied by finite element modeling (FEM) simulations and laboratory testing during the rolling process at temperatures between 250^0C and 400^0C and reduction rates between 30% and 45%. Cracking depth along the TD rises with decreasing rolling temperature and rising reduction rate, and 45° crossed shear cracking is found to be the dominant fracture mechanism of edge cracks in the longitudinal (rolling direction-normal direction) section of rolled sheets[(4)]. The rolling temperature ranged from 250°C to 400°C, and the rolling speed averaged 0.5 m/s. On a sheet of magnesium-alloy AZ31B. It was demonstrated that, while rolling at higher temperatures, edge fractures can be considerably reduced if the direction of rolling is switched by 90° between each successive pass. The mechanism of fracture tip propagation was studied in detail at 350°C, with results suggesting that a broad area of grain boundaries between finer grains may improve the crack propagation resistance. AZ31 magnesium alloy was studied while it was subjected to a cyclic expansion extrusion with an asymmetrical extrusion cavity (CEE-AEC) to learn more about its microstructure, textural development, and mechanical characteristics. Three passes resulted in a refined microstructure of the deformed samples, with an average grain size of 6.9 μm for the alloys in the asymmetrical cavity area. As the number of passes is increased, the maximum intensities of the basal textures rise, and the basal textures are deflected as a result of the deformation. The alloy's tensile yield strength (TYS), ultimate tensile strength (UTS), and elongation-to-failure in the asymmetric cavity area are 146 MPa, 230 MPa, and 29.7 % after 3 passes, respectively[5]. To make sheets of fine-grained ZK60 alloy, a dual equal channel lateral extrusion (DECLE) technique with several passes was used. After 3 and 5 passes of DECLE and subsequent extrusion, the coarse grain structure of the annealed sample (size: 68 μm) transformed into fine grains of 6.0 μm and 5.2 μm, respectively. Temperatures ranged from 200^0C to 300^0C, and strain rates from 0.003 to 0.33 seconds-1 were used for the standard penetration test (SPT). Strip defects like edge cracking and uneven structure lower magnesium alloy yield due to the alloy's close-packed hexagonal (HCP) crystal structure and the inconsistency of metal flow and temperature changes in the deformation zone during rolling. So it appears difficult to produce wrought products on a large scale. Thus, evolving improved rolling techniques is essential for enhancement of rolling forming performance of magnesium alloy; to combat with the problem of edge cracking, emergence of new thermo mechanical can be noted in the efforts of the previous workers. Rheological stress increases with strain rate and decreases with rising deformation temperature in cast roll AZ31 magnesium alloy, which exhibits a linear drop in work hardness with increasing strain rate and deformation temperature. As temperature and strain rate rise, the microstructural twins that govern deformation become less robust. Over time, the grain angles change, the crystal orientation differences within the grains increase, and the grain slip system grows. As a consequence, the material becomes significantly softer, the number of dynamically recrystallized grains increases, and the underlying texture weakens. Moreover, researchers have been observed to have applied a variety of processing techniques to alter the microstructure of the Mg-alloys so as to be able to undertake high plastic deformation. At temperatures between 300°C and 350°C, the average grain size of high speed rolled (HSRed) materials decreases because to the reduced area fraction of coarse undynamic recrystallized grain (unDRXedgrains), but increases at 400°C due to the increased dynamic recrystallized grain (DRXedgrain) size. Twin formation and twin-induced dynamic recrystallization (TDRX) activity were stimulated at higher temperature rolling, leading to a higher area percentage of dynamic recrystallized grain (DRXed grains). The highest pole intensity in the dynamic recrystallized region moved from the normal direction (ND) to the rolling direction (RD) as the rolling temperature decreased because to the strong shear deformation caused by the Hart Scott Rodina process[2] . It may be mentioned that for the sake of improving the formability of magnesium alloys and to significantly increase its' yield strength, ultimate tensile strength and ductility properties at room temperature, a number of techniques, including equal channel angular processing (ECAP), single roll drive rolling (SRDR), two stage rolling and multistep rolling thermomechanical process were adopted. Based on the idea of a single variable, the effect of pulsed electric current strength, frequency, and width on edge cracking of AZ31B magnesium alloy plate cold rolling was examined. Experiments showed that using a pulsed electric current to help prevent edge cracking reduced the problem significantly, with the inhibitory impact increasing dramatically for higher values of the current parameters. The specifications of the pulsed electric current needed to prevent any edge cracking were 4800 A, 500 Hz, and 50 s. Edge fracture statistics, rolling load variation, and microstructure analysis all pointed to a rolling process under pulse electric current in which non-thermal effects were more influential than thermal ones. Grain refinement and basal plane weakness are other side effects of electro plastic rolling. Magnesium strip yield and tensile strengths improve along with a drop in hardness when the current parameter is increased. With the increase in current intensity, pulse frequency, and width, the number and depth of edge cracks on the AZ31B magnesium alloy strip surface are effectively inhibited, and the rolling force is also reduced. Moreover, the influence of current intensity and pulse frequency is the most significant. Electro-plastic rolling increases the number of fine grains and shear bands, presenting a blanket structure. The number of shear bands increases, so the amount of strain experienced by a single shear band decrease.

The as-cast ZEK100 (Mg-1.2Zn-0.35Zr-0.17Nd, in wt%) magnesium alloy was hot rolled at temperatures between 350°C and 450°C on the laboratory 50 tons reversible mill with heated rollers to create the 1.5-1.7 mm thick sheet. Rolling temperature had an effect on the properties of the sheet, with the tensile strength decreasing from 257 to 228 MPa and the tensile yield stress decreasing from 237 to 185 MPa when the rolling temperature was increased from 250^0C to 450^0C. The grain size of the alloy changed as the rolling temperature went from 350°C to 450°C, with the largest expansion happening between 400°C and 450°C[6].

It is well known that the application of Mg alloy is limited by its low wear resistance and corrosion resistance. In order to take care of this inherent deficiency in strength and wear properties of Mg-alloys, attempts have been made by others to reinforce AZ-61 alloys by various reinforcements viz. oxides, borides, carbon fiber, glass fiber, and these reinforcements could improve some property or other[7]. On the contrary graphene, a single layer 2D graphitic material is known to bear tremendous application potential in the advanced technology area. This can enhance strength properties, conductivity property, corrosion resistance and lubricating efficiency of the matrix material. It is reported by previous researchers that graphene nanosheets reinforcement could influence the mechanical properties of AZ-alloys under compressive load and a much higher corrosion resistance is obtainable due to graphene nanosheets reinforcement[8]. Graphene as reinforcing agent, is advantageous over the carbon nanotube because of its 2D structure; this is why, graphene is being tried as the reinforcement material, in Mg alloys; this is meant to overcome the inherent difficulty of carbon nano tube reinforcement into metal matrix which is due to the agglomeration of tendency of carbon nano tubes. The high aspect ratio and Van-der Waal force of attraction make carbon nano tube vulnerable to agglomeration; moreover, carbon nanotubes are quite prone to structural damage, under harsh processing conditions like mechanical alloying. Previous research have demonstrated that reinforcing graphene in a magnesium alloy matrix was able to bring about considerable improvement in strength properties along with the resistance to corrosion. It is reported that graphene insures a better wettability in AZ alloys and this is why the particle -matrix interface does not act as the source of premature crack nucleation. Due to good wettability. There are reports of graphene reinforcement in AZ-31 alloy through the conventional stir casting route. These composites reinforced with 1.5 wt. % graphene were extruded at 500^{0}C and such extruded material could achieve yield strength of 187 MPa, ultimate tensile strength, 285 MPa at a 12% elongation. Although a lot of efforts were made to reinforce Mg-alloys with carbon nano tubes (CNTs), only limited improvement in properties could be noticed due to its' tendency for agglomeration and damage of carbon nano tubes. On the other hand, 0.3 wt.% graphene with its characteristics of high modulus of elasticity and fracture strength (1 TPa and 125 GPa respectively) could enhance the strength of pure Mg from 187 MPa to 197 MPa and the UTS from 219 MPA to 238 MPa respectively of Mg-graphene composite. Powder metallurgy and hot extrusion were used to create AZ31 alloy composites reinforced with graphene nanoplatelets (GNPs). It was investigated how the extruded GNPs/AZ31 composites' microstructures, mechanical characteristics, and wear performance changed depending on the GNPs content (0.5, 1.0, and 2.0 wt. %). The results showed that the basal plane texture and grain refinement of the AZ31 matrix metal were both decreased due to the inclusion of GNPs. Both Vickers hardness and tensile yield strength were improved by less than 1.0 wt.% GNPs in GNPs/AZ31 composites, while elongation remained within an acceptable range. Compared to unreinforced AZ31, the composite made from 1.0GNPs/AZ31 has a higher Vickers hardness (4.9%) and tensile yield strength (9.5%). Composites' elongation was also comparable to AZ31 base alloy[9] . An overlay mould was laid at an initial temperature of 400°C to conduct the hot rolling experiment on AZ31 magnesium alloy. Different reductions in the middle and edges of the magnesium alloy were realized using the cross rolling process and the rolling theory developed by the Mizushima automatic plan view pattern control system (MAS), and the impact of the regional controlled reduction rolling on the edge cracks and microstructure gradient of the magnesium alloy was studied. The maximum edge fracture depth of the rolled piece has been demonstrated to be lowered by 56.85 % using this rolling method, and there is a weakening tendency in the base surface texture of the strip edge, with the density decreasing from 23.97 % to 17.48 % after regular flat rolling. The unequal deformation of the sheets is mirrored in the RD direction as a gradual transition from a smoother edge to a rougher center. It provides a theoretical foundation for the variable thickness rolling of the magnesium alloy strip and is therefore applicable to the manufacturing of metal moulds requiring substantial edge reductions, such as mobile phone shells.

Multi-pass cross-rolling was used to reduce edge cracking, however this process required multiple passes. Pre-rolling is performed in which effectively reduces the maximum thickness with each pass and weakens the strong basal texture through twinning deformation. There is a clear anisotropy in the mechanical characteristics of as-rolled magnesium alloy sheets. Multi-pass hot rolling of AZ31 magnesium alloy sheets of 4 mm thickness utilising varied single pass rolling reductions yielded sheets with good performance and allowed for the investigation of the influence of microstructure, texture, mechanical characteristics and mechanical anisotropy. The grain structure was significantly improved by many passes of hot rolling. From ingots with a grain size of 250 μm, the as-rolled sheets have a grain size of less than 30 μm. Grain refinement and texture intensity are responsible for the increase in mechanical parameters from 83.4 MPa to 120 MPa in yield strength, 218.2 MPa to 230 MPa in ultimate tensile strength, and 8.9% to 18%.

In light of available information, it appears that graphene used as a reinforcing agent in Mg- composite exert a positive influence on the microstructure and mechanical properties of the matrix composite. However, studies on the structure and characteristics of graphene-reinforced AZ-61 magnesium alloys containing trace amounts of scandium and calcium are rare. Importantly, the AZ-61 alloy derives its strength from $Mg_{17}Al_{12}$; due to a good combination of strength and toughness, AZ-61 alloys are targeted for structural application in various industrial sectors.

Wrought AZ-61 alloys research has not taken off yet, unfortunately. There is a dearth of information in the literature concerning the structure and properties of AZ-61 alloys and composite with present research as discussed in (Table 1). It is possible that the substructure of AZ-61 alloy can be stabilized through the precipitation of hardening phases during controlled rolling. Therefore, the alloy's toughness might be improved even further. Additionally, Al_3Sc is well-known for being a superior precipitation hardener[10]. As a result, investigating whether or not a small amount of scandium added during rolling of AZ-61 alloys can create the precipitating phase, Al_3Sc, to achieve much higher strength and toughness, seems like an interesting undertaking. Therefore, the finding of this research are to determine how to successfully handle the cracking issue in stir cast AZ-61 alloy - graphene composites that have been subjected to thermo mechanical treatment at varying finish rolling temperatures to observe if formation age hardening phases like Al_3Sc and Al_2Ca/Mg-Ca. is accentuated. The low quality of AZ61 Magnesium means it can't be used in many structural applications. Magnesium alloys are reinforced using a wide range of reinforcing materials and sizes to improve their thermal and mechanical properties. To address the issue of the hot rolling process for AZ 61-graphene composite, while there is some literature regarding graphene-free AZ-31 and AZ-91, but no reports exist on hot-rolling AZ-61 alloy-graphene composites at varying temperatures. In order to get a high-performance magnesium composite, studying the effects of hot-rolling on the microstructure and characteristics of AZ-61 alloy-graphene composites is highly relevant. In this paper, we will examine the significant development of graphene-reinforced lightweight AZ 61 matrix composites for hot rolling mechanisms. Graphene-reinforced hot-rolled AZ 61 magnesium matrix composites with minor additions of scandium and calcium will be discussed, along with their microstructure and mechanical characteristics.

Table 1. Comparison on mechanical properties of AZ61alloys and composite with present research

Materials	Process	0.2%YS (MPa)	UTS (MPa)	% Elongation	Hardness	Ref.
AZ 61 Graphene Composite	Stir Casting and Hot Rolling at 350°C	102.18	266.82	28	107 HV	Present research
AZ61 alloy	Cyclic expansion extrusion with asymmetrical extrusion cavity	124	230	29.7	NA	(11)
AZ61 alloy	Equal-channel angular pressing (ECAP) and electropulsing treatment (EPT)	100	260	7	NA	(12)
AZ61 alloy	120 °ECAP (Route A at 483 K)	195	232	15.2	NA	(13)
AZ61 alloy	Friction stir processing	72	243	17.6	NA	(14)
9AZ61 alloy	450°C rolling	148	229	18	NA	(15)
AZ61 alloy	Forging + heated treated	170	259	20.6	NA	(16)
AZ61/SiCp Magnesium Matrix Micro- and Nano-Composites	Extrusion and Subsequent Annealing	136.4	220	6.4	NA	(17)
AZ61/20 μmSiC	Sintered	NA	NA	NA	71	(18)
AZ61/Al_2O_3/SiC hybrid composite	ECAP processing	110.3	250.1	19.3	61.2 ± 4.6	(19)
AZ 61- T4- 0.1% CNT	Powder Metallurgy	79.3	141.4	14.98	NA	(20)
AZ61/SiCp Mg MMCs cast	Gravity casting method using the stirring process	166.64	100.64	3.34	NA	(21)

2 Methodology

2.1 Raw materials

The graphene powder were purchased from Platonic Nanotech Private Limited, Dist –Godda, Jharkhand, The average thickness and lateral dimension of the supplied graphene nanoplates of >99% purity, are reported to have been 5-10nm and 5-10μm respectively. Aluminum-2wt. % scandium master alloy with >99 purity, granular calcium, zinc and manganese with >95% purity) was supplied by MatRics, The Technological Solution, Kanyakumari , Tamil Nadu. The nominal composition of the AZ 61 alloy is discussed in Table 2.

Table 2. Chemical Composition of Experimental AZ61 Alloy

Element content (wt%)						
Mg	Al	Zn	Mn	Ca	Sc	C
88.07	6.04	1.30	0.20	0.49	0.18	3.72

2.2 Experimental procedure

Modified AZ 61 alloy matrix composites reinforced with graphene are prepared by stir casting method. Three different composites containing (0 wt. %, 1wt. %, and 2wt. %) of graphene (GNPs) were prepared for further processing by hot rolling. Melting of the matrix alloy was carried out in the electric resistance furnace under the suitable flux cover to avoid oxidation. In the process of preparation of alloys, commercially pure magnesium (99.5% purity) was taken as the starting material along with the (6 wt. %) of aluminum, scandium (0.2 wt.% Sc) and manganese (0.5 wt.%).

The materials were taken in a clay graphite crucible and the subjected to melting in an electrical resistance heated pot furnace. Melting temperature was kept at $780 \pm 15°C$ and after complete meltdown, metallic zinc (1 wt. %) and calcium (0.5 wt.%) were plunged into the melt. The addition of different percentages of GNP powder was done in the molten bath which was kept under continuous stirring. The furnace had a separate arrangement of stirring and the stirring speed was kept at 600 rpm. Stirring was done for about 20 minutes to obtain a homogenous distribution of GNPs. Slag 30 (perlite ore) is used to separate the slag from the molten metal by way of making it highly viscous. The slag was skimmed off and the melt casting was done in the ultra-low carbon interstitial free steel moulds of size 200 mm x 20 mm x 20 mm rectangular bar.

The cast samples were cut into pieces, each 70 mm in length. Hot rolling was carried out in a laboratory scale rolling mill at 34% thickness reduction. The sample size was 70 mm x 20 mm x 20 mm, before the rolling operation. The proposed methodology is mentioned in (Figure 1). The rolling operation was carried out in a laboratory scale rolling with the initial stock temperature of 400^0C. The rolling temperatures were varied and kept at 350^0C 300^0C, and 250^0C. After a few roll passes, the rolled stocks were reinserted within the furnace to make up for the temperature drop. The rolling was continued until the amount of thickness reduction reached 35%. The number of passes varied from 1-4. It was observed that the influence of rolling temperature was quite sensitive to the number of passes to reach the target deformation ~35%. The rolling temperature affected the achievable hardness and tensile strength values. Hot-rolled samples were subjected to microstructural characterization and study of the mechanical properties. The hardness of different samples, processed under different schedules at different temperatures was measured in a Vickers hardness testing machine; all the measurements were carried out under a normal load of 100 gf and with a dwell time of 10 s. An average of five consistent readings is accepted as the representative hardness of the alloy. Tensile tests were carried out in an Instron testing machine of model no. 5967. All the tests were carried out at room temperature and at strain a rate of 0.1mm/min. The tensile test samples were prepared in accordance with E8-ASTM specification. The tensile properties and hardness of as-cast AZ 61 alloy are discussed in Table 3. Test samples were prepared in an EDM machine along the longitudinal direction; this was followed by smoothening and removal of adherent scale or unevenness by using 120-grade silicon carbide paper.

For optical metallographic analysis, the mechanical grinding of samples was done in successively finer grades of emery paper starting from 120 grit size to 2500 grit size; at each step, the samples are washed in running water in order to remove the dirt and debris adhering to the surface. This also serves the purpose of cooling of the heated samples. After grinding, the samples are polished in cloths by using successively finer diamond suspension from 9 μm to 1μm, which is followed by polishing with alumina suspension of size 0.5 μm. After polishing the samples are sonicated in an ultrasonic bath to further clean the surface and finally these are dried. The dried samples are etched in a mixture of 10 ml distilled water+1.5g picric acid+5ml acidic acid+25ml ethanol composition, the effect of rolling on the microstructure and hence the properties of AZ-61 alloy composite were assessed.

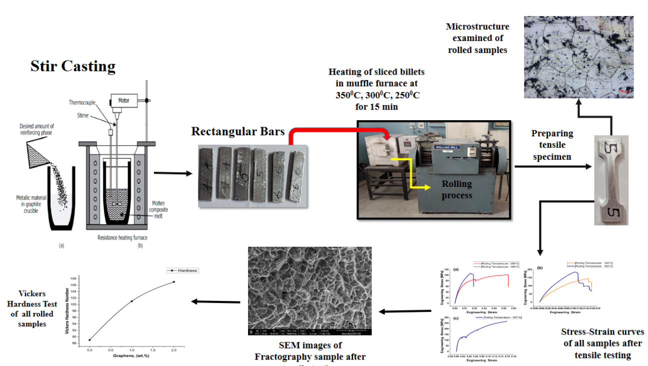

Fig 1. The proposed methodology in the present research

Table 3. Tensile properties and Hardness of as-cast AZ 61 alloy

0.2% YS (MPa)	UTS (MPa)	Elongation (%)	Vickers Hardness (HV)
90.58	109.19	10.3	84.69

3 Results and Discussion

3.1. Microstructures of rolled samples

The optical micrographs of AZ-61 – graphene composite rolled at 300^0C are shown in (Figure 2 a-c). There is no qualitative difference between the optical micrograph of a 1% graphene-reinforced sample and that of 2% graphene reinforcement. The only marginal difference may be observed to be in the amount of graphene particles which seems to be a little higher in the 2 wt.% sample. It is clear that rolling at 300^0C gives rise to fully recrystallized structure (Figure 2 c). Graphene is seen to have been distributed within the micrograph but many such black particles are found to be located at the grain boundaries. The precipitates of $Mg_{17}Al_{12}$ are likely to be formed due to the ageing effect which is caused by holding the samples at the rolling temperature; the precipitation process might have also been aided by the rolling deformation which entices precipitation of $Mg_{17}Al_{12}$. There are reports that this type of precipitate is formed in scandium-treated Al-6Mg alloy at temperatures 350^0C to 400^0C[(22)]. The higher magnification image in (Figure 3) shows that apart from the grain boundary phase there are some intragranular particles. It is not possible to say which particle is which. It is hypothesized that the embedded graphene particle of hexagonal structure can very well act as the nucleation sites for nucleation of Mg-rich phase which is also of closed packed hexagonal structure. This is why the graphene particles are seen to be present at the triple points. The presence of graphene at triple points is a clear indication that the nucleation Mg-rich phase is heterogeneously nucleated at the graphene sites. On the contrary, the intragranular particles are supposedly the precipitates of $Mg_{17}Al_{12}$ that were formed due to the ageing effect. An intermetallic compound, Mg_2Ca, with a high melting point is formed when Ca is added to magnesium alloys, which is observed in (Figure 2 a) to enhance the composite hardness. X-ray diffraction pattern of AZ 61 alloy are shown in (Figure 5). However, much more elaborate studies with high-resolution microscopy are required to make a convincing comment on the nature of the second phase. Microscopically, the AZ 61 graphene composite has a finer and more uniform microstructure thanks to the addition of graphene, suggesting that this addition will facilitate dynamic recrystallization during hot rolling. To understand the situation in a better manner, the as-cast microstructure of AZ-61 alloy reinforced with 2 wt.% graphene is presented in (Figure 4). It is revealed that the graphene particles are seated at the triple points. Due to the addition of scandium the dendritic structure is eliminated. It is apparent that graphene particles act as the heterogeneous nucleation sites of the Mg-solid solution. This corroborates the structure of the rolled sample. It is their conjectured that the black regions at the grain boundaries delineate graphene particles whereas, the intragranular particles are the precipitate phases of the $Mg_{17}Al_{12}$ and Al_2Ca. Moreover, (Figure 4) discerns the presence of twins representing the characteristic deformation mode of hexagonal close packed alloys. Furthermore, the hot rolled AZ 61 graphene composite achieves a favourable compromise between yield strength and plane-strain fracture toughness. Despite the shown efficiency of thermo-mechanical methods in enhancing mechanical characteristics, As a consequence of the thermo-mechanical processing involved in rolling, the magnesium strip grain structure was refined, which improved its mechanical properties. Rolling at higher temperatures, from 250°C to 350°C, coarsens the alloy's grain size, with the most expansion occurring at 300°C as shown in (Figure 3). More passes for multi-pass hot rolling at different temperatures means more cumulative deformation, which in turn means more dynamic recrystallization and finer grains. However, the inclusion of graphene nanoparticles and an increase in precipitated second phases at the grain boundaries during multi-pass hot rolling may effectively pin the recrystallized grain borders and halt the development of recrystallized grains. During hot rolling, DRX considerably improves the microstructure of the AZ 61 graphene composite. The volume percentage and grain size of the dynamic recrystallized (DRXed grains) in the rolled composite both increase as the rolling temperature rises. Grain boundary shearing occurs often in the early stages of hot rolling, resulting in the development of serrated borders and homogeneous local strain gradients along the initial high-angle grain boundaries. Due to limited slip systems the hexagonal close packed material is known to employ twinning as the alternate means of plastic deformation. The traces of these twins are noticed in the (Figure 4). The presence of alloying element in the AZ 61 alloy are confirmed by Energy Dispersive Spectroscopy (EDS) analysis are shown in (Figure 6).

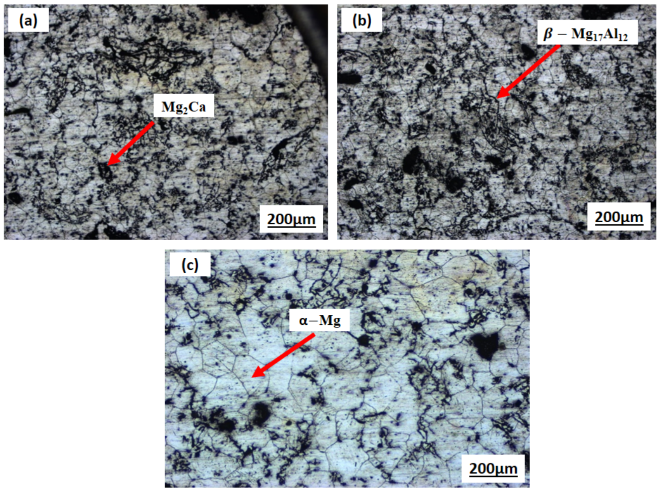

Fig 2. (a) AZ61-1% graphene sample rolled at 300^0C, (b) and (c) AZ61-2% graphene sample rolled at 300^0C

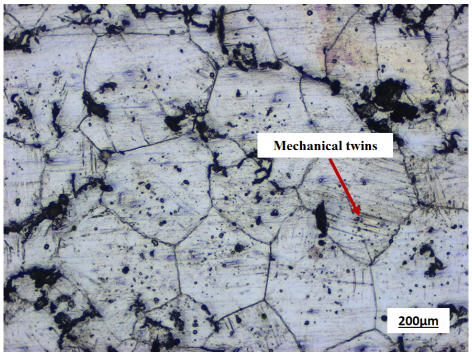

Fig 3. Higher magnification micrograph of AZ61-2% graphene sample rolled at 300^0C

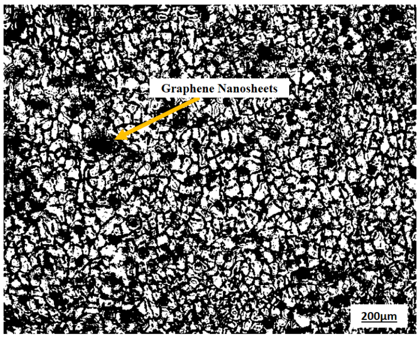

Fig 4. Photomicrograph of stir cast AZ61-2% graphene composite before rolling

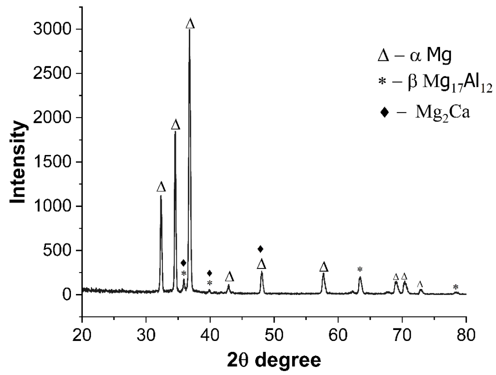

Fig 5. X-ray diffraction (XRD) pattern of the Modified AZ 61 alloy

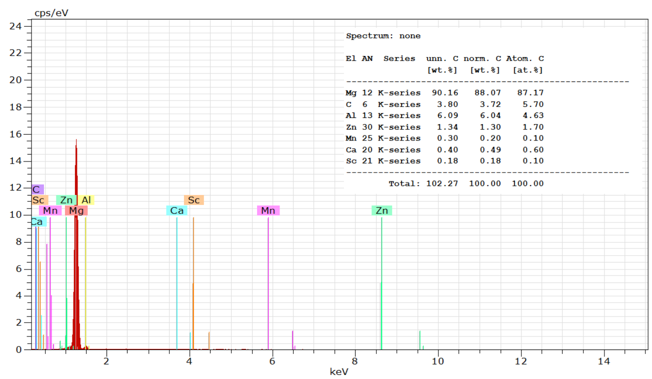

Fig 6. Energy Dispersive Spectroscopy (EDS) analysis of Modified AZ 61 alloy

3.2 Mechanical properties

The mechanical properties of modified AZ 61 alloy and its composites rolled at different temperatures are furnished in Table 4. The table shows the results of rolled material without graphene reinforcement and that reinforced with 1 wt. % graphene. As there has been the paucity of samples only limited test reports are reported to depict an indicative picture. The Vickers hardness testing method has been used to study the hardness values of different treated samples. The hardness measurement results reveal an increase in the hardness of the modified AZ 61 alloy and its composites with 1%, graphene reinforcement with decreasing temperature of rolling (Table 4). However, the hardness of AZ61-2 wt. % composite remains more or less constant with increasing rolling temperature (Figure 8). It is known that rolling at lower temperature should yield precipitates of finer size than for higher rolling temperature. Moreover, the dislocation density in material rolled at a lower temperature is supposedly higher than that in case of rolling at a higher temperature. Moreover, it is observed that the hardness of the composite is higher than the pristine alloy. For example, the hardness values of 1wt.% graphene reinforced AZ61 composite is higher than the AZ61 alloy without reinforcement when the either material is rolled at the same temperature, say, 300°C. Thus, the hardness values of 1 wt. % graphene reinforced sample and that without reinforcement (when rolled at 300°C) are found to be 100.82 HV and 88.94 HV respectively. While higher rolling temperature suffers from loss in hardness, the higher amount of graphene content enhances the hardness value of the experimental material, the hardness of 2wt. % graphene reinforced sample shows a near constancy of hardness with variation in rolling temperature. This is ascribed to the mutually opposing effect of rolling temperature and reinforcement content as reasoned earlier. The grain boundary phases restrict the movement of the grain boundaries and hence prohibits grain growth; this aid in the retention of fine-grained microstructure.

In addition, tensile strength of as extruded graphene nanoplatelets-AZ61 composite was investigated at temperatures ranging from 75 °C to 225 °C with initial strain rate of $2 \times 10^{-3} S^{-1}$. The results show that total fracture strain increases and tensile yield strength decreases with increasing testing temperature. The increased fracture strain at high temperature is mainly attributed to significant grain refinement and uniform particle distribution[23]. From the present investigation the results of tensile testing, it is observed that the yield strength and ultimate tensile strength of 1 wt. % graphene composite increases with rolling temperature; a similar observation can be made with the alloy without any reinforcement. This, suggests that at least for the low reinforcement content the strength properties depend on the age hardening in alloys. As the rolling temperature is increased, more precipitation takes place and the strength increases. The supposition of increase in precipitate density at increasing rolling temperature is supported by the increase in yield strength which is sensitive to the small size of the precipitates. If the rolling temperature is further increased, the particle coarsening comes to play and fine particles become larger. This accentuates Orowan bypassing mechanism to be operative. Thus, there may be lowering of yield strength with simultaneous enhancement of ductility. This fact can be verified from (Figure 9) in which it is clearly noticeable that increasing ageing temperature increases the UTS with concurrent diminution in yield strength. The lowering of yield strength is explained by the inevitable enlargement of particle size at higher rolling temperature; particle coarsening changes the deformation mode from cutting mechanism to bypassing mechanism; the former enhances yield strength whereas the latter is responsible for higher ductility at the cost of yield strength. UTS is not a structure sensitive property as yield strength is and hence dispersion hardening along with composite hardening continue to raise the ultimate tensile strength value. In the case of 2 wt. % graphene reinforced composite the effect of rolling temperature is shown in (Figure 10). It can be noticed from the figure that the change in yield strength with the change in rolling temperature is not significant. It is further observed that tensile strength and percent elongation increases with increasing rolling temperature. The hot rolled tensile characteristics of the graphene composite AZ 61 rolling at higher temperatures increases the 0.2% proof yield strength (YS) and ultimate tensile strength (UTS) of the material while increasing the elongation-to-fracture marginally. According to reports [24], the Mg-1Ca-1Zn-0.6Zr casting alloy (T6 condition, peak aged at 200 °C) has a YS of 145MPa, a UTS of 241MPa, and an elongation of 9%. It is evident that hot rolling significantly increases the tensile strength of the AZ 61 graphene composite alloy. The ultimate tensile strength in 2 wt.% graphene composite is 266.82 MPa when as well as fine grain size due to the material is rolled at 350^{0}C. The material's mechanical performance is often measured in terms of its yield strength. The yield strength of alloys with Sc in them is affected by the amount of Sc present, the amount of other micro alloying elements present, and the processing circumstances. The relationship between yield strength and Sc content over time is seen in Table 4. The yield strength has increased dramatically in recent studies. The composite hardening due to higher reinforcement content, higher degree of dispersion strengthening and the fine grain size as obtained by recrystallization are responsible for high tensile strength of the composite. It is also interesting to note that the composite with 2 wt. % graphene exhibits excellent ductility as characterized by 25 % elongation. Such high ductility is attributed to the small size of recrystallized grains. The engineering stress -strain diagram of the 2wt.% composite shows a high strain hardening rate and it seems that recovery process viz. recrystallization occurs fast enough to keep pace with strain hardening; this is the reason for high elongation percent for this composite rolled at high temperature. The tensile fractured sample was subjected to fractography in Scanning Electron Microscope to understand the operative mechanism of deformation. The fractograph (Figure 7) of AZ61 – 2 wt.% graphene reinforced composite shows a mixed mode of fracture. The fractograph is characterized by and large by the presence of dimples and cleavage at places. It is conjectured that cleavages are initiated by the reinforcing graphene and ductile dimples are responsible for crack propagation through the matrix alloy. It is evident that the fracture surface had several big and small plastic dimples, a feature of ductile rupture. Dimples were also seen, and they correspond well with those described in matrix alloy. The presence of inclusions, dispersoids, precipitates, or second phases is often linked to the development of dimples. The nucleation and expansion of voids, which lead to the creation of dimples during deformation, may be influenced by these particles. Thus, the dimples are almost the same size as the particles. As a result, the tiny bumps on the fracture surface could be traced back to the fine precipitates in the as-hot-rolled microstructure. The substantial intermetallic phase,Mg_2Ca, was clearly linked to the ductility in the heat rolled AZ 61 graphene composite. Increased ductility in wrought AZ 61 graphene composite would result from breaking up these big Mg_2Ca particles and spreading them out more evenly following heat deformation.

Table 4. Mechanical Properties of rolled modified AZ 61 alloy and composite (1wt. % Graphene)

Materials	**Rolling Temperature (°C)**	**0.2% YS (MPa)**	**UTS (MPa)**	**Elongation (%)**	**Vickers Hardness (HV)**
AZ 61 alloy (0% GNP)	250	66.24	101.82	7.2	101.05
AZ 61 alloy (0% GNP)	300	90.75	106.59	2.5	88.94
AZ 61 composite (1% GNP)	300	68.57	184.43	4.8	100.82
AZ 61 composite (1% GNP)	350	128.91	144.46	4.9	91.91

It can also be observed that addition of GNPs and its increasing contents leads to increase in Vickers hardness. The AZ31-3GNP composite exhibits the highest value of hardness (HV = 68.9). Thus, GNPs may contribute toward increased micro-hardness of AZ31 alloy However, if we compare the present work with Ref. [25], we found that from Figure 6 shows the relation

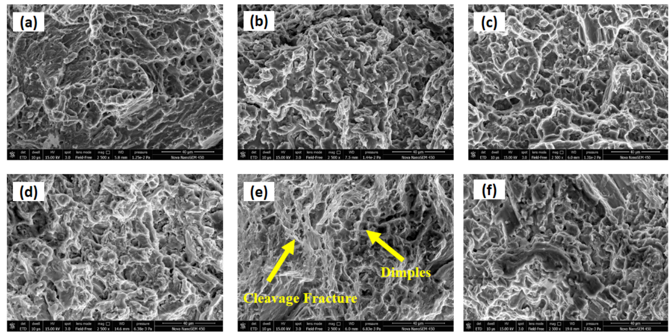

Fig 7. Fractograph of tensile samples a) AZ 61 alloy rolled at 250^0C, b) AZ 61 alloy rolled at 300^0C, c) AZ61-1wt.% graphene composite rolled at 300^0C, d) AZ61-1wt.% graphene composite rolled at 350^0C, e) AZ61-2wt.% graphene composite rolled at 250^0C, f) AZ61-2wt.% graphene composite rolled at 350^0C

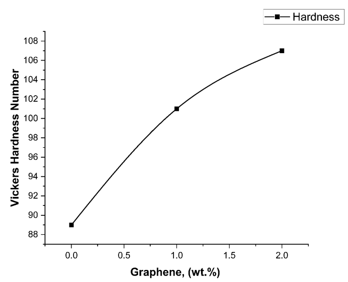

Fig 8. Curve for weight percentage composition of Graphene content v/s Vickers Hardness Number at 300^0C rolling

between the amount of reinforcement and hardness of the composite after rolling at 300^0C. It is clear that with addition of 2 wt% amount of graphene enhances the hardness (107 VHN) of the composite. This is quite common in a two phase mixture as the hardness of the mixture is decided by the weightage average of the constituent phases. Graphene is much harder that the Mg- matrix alloy and hence increasing the graphene content the amount to increase in overall hardness of the material.

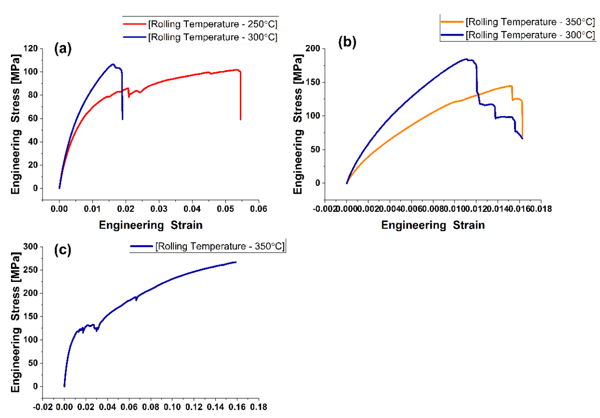

Fig 9. (a) Stress- Strain curves of AZ 61 alloy (0% GNP) at rolling temperature 250°C and 300°C, (b) Stress- Strain curves of AZ 61composites with (1% GNP) at rolling temperature 350°C and 300°C, and (c) Stress- Strain curves of AZ 61composites with (2% GNP) at rolling temperature 350°C

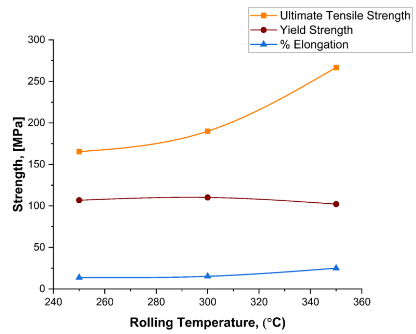

Fig 10. Strength of (2 wt. %) graphene reinforced composite v/s rolling temperature

4 Conclusion

The effect of rolling variables on the microstructure and mechanical properties of modified AZ 61 alloy-graphene composites have been examined. From the results of the study, it is concluded that the composite samples rolled at 350°C attains a good combination of strength and ductility. Higher amount of deformation has led to finer recrystallized grains. According to microstructural analysis, graphene addition acts as grain refiner for AZ 61 graphene composite. Optical microscopy analysis confirmed the even distribution of GNP throughout the composite matrix. Graphene reinforcement enhances the hardness of the alloy in accordance with the amount of reinforcement content. Thus, for a specific rolling temperature, say, 300°C the hardness value increases with an increase in graphene content. This research shows the structural refinement is possible by graphene reinforcement in a scandium added AZ61 alloy. Graphene act the nucleating agent for heterogeneous nucleation of grains in AZ61 alloy. The crystallographic match between reinforcement agent, graphene, and Mg-solid solution, induces the nucleation of new grains. The hot rolled tensile characteristics of the graphene composite AZ 61 rolling at higher temperatures increases the 0.2% proof yield strength (YS) and ultimate tensile strength (UTS) of the material while increasing the elongation-to-fracture marginally. It is inferred that a strength of 266.82 MPa at 25% elongation is achievable in AZ61-2wt. % graphene composite after rolling at 350°C. While the reinforcement leads to cleavage due to its sheet structure, the ductile matrix promotes micro-void coalescence thereby producing ductile dimples in the fracture surface. Overall, AZ 61 graphene composites were made using a stir casting process, and their structural design resulted in improved mechanical characteristics. The hot rolling technique that followed ensured that the GNPs were evenly distributed on the Mg. Graphene was successfully bonded to the Mg matrix and was incorporated deeply inside the matrix. Comparison of literatures with the present work confirmed the enhancement potential of GNPs in AZ 61 graphene composite which is achieved excellent structural properties after hot rolling. Magnesium-based composites with graphene reinforcement, known as AZ 61, are scarce. Lightweight, and mechanical integrity make novel AZ 61magnesium alloy graphene composites a potential replacement for conventional magnesium alloys and composites in the aerospace and automotive industries. Vehicle weight, petrol consumption, and carbon dioxide emissions are all areas where the automotive sector might benefit from weight reduction technologies like AZ61 alloy graphene composite that is compositionally modified by minor additions of scandium and calcium.

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