ORIGINAL ARTICLE

Fabrication and Characterization of Mg-Zn Alloys Reinforced with Carbon Nanofiber for Orthopaedics Implant Application: A Study on the Different Compositions

Muhammad Al Kahfi Edrus[1], Muhammad Sallehuddin Mohd Azman[1], Herman Tuminoh[1], Ahmad Kafrawi Nasution[2], Muhammad Hanif Ramlee[1,3]

[1] Bioinspired Devices and Tissue Engineering Group (BIOINSPIRA), School of Biomedical Engineering and Health Sciences, Faculty of Engineering, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia.
[2] Department of Mechanical Engineering, Faculty of Engineering, Muhammadiyah University of Riau, Pekan Baru, Riau 28294, Indonesia.
[3] Medical Devices and Technology Centre (MEDiTEC), Institute of Human Centered Engineering (iHumEn), Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia.

ABSTRACT

Introduction: Biodegradable materials, such as Mg-based, Fe-based, and Zn-based, bring as much attention as bone-implant materials due to its biocompatibility and biodegradability characteristics. Among them, the Mg is the most abundant elements in human body and primarily found in the bones. However, the Mg has a lower mechanical properties and resistances to fracture compared to the other biodegradable and non-biodegradable metals. Therefore, the aim of this study was to develop a possible biodegradable material made of Mg-Zn alloys reinforced with carbon nano fiber (CNF) and later tested with several testing procedures. **Methods:** The powder metallurgy method (PM) was utilized to fabricate a total of 24 samples of Mg-Zn alloys reinforced with 1.0%wt, 1.2%wt, 1.4%wt, 1.6%wt, 1.8%wt and 2.0%wt of CNF. The PM method was involved with the process of grinding using ball milling, compaction under 400MPa pressure and sintered under 400 °C. Compression testing was done to measure the mechanical strength meanwhile scanning electron microscope (SEM) and energy-dispersive X-ray spectroscopy (EDS) was used to identify the microstructural of samples. **Results:** From this study, it was found that Mg-Zn alloys with 1.6 wt% of CNF produce the highest Young's modulus (2687.91MPa) with acceptable yield strength (84.91MPa). For microstructural analysis, the results showed a compact surface for 1.2, 1.4 and 1.6 wt% of CNF and non-homogeneous structure of all the samples. **Conclusion:** In conclusion, this study has successfully shown the promising use of Mg-Zn-CNF composite as new materials for implant in terms of suitable strength and structure.

Keywords: Biodegradable materials, Magnesium composite, Carbon nanofiber, Reinforcement particle, Powder metallurgy

Corresponding Author:
Muhammad Hanif Ramlee, PhD
Email: muhammad.hanif.ramlee@biomedical.utm.my
Tel: +607-5558480

INTRODUCTION

It is commonly known that magnesium (Mg) and its alloys have been well known as one of the most promising biomaterials for biomedical application in medical practitioners (1). This is due to the fact that Mg alloys strongly show biodegradable and bioabsorbable properties, plus it is safe to be used in human body with minimum side effect (2). When the Mg alloys is placing inside the human body, it should be noticed that this material can be degraded in many ways such as through protein interaction with Mg, electrochemical reaction (3), alloy compositions (4) and surface conditions (5). Thus, by going through this degradation process, it can avoid the need essentials of a second surgery, to prevent potential infections and additional pain for patients (6). However, the yield strength of Mg alloys of 170 MPa is less favourable in terms of mechanical properties compared to that other non-biodegradable materials used in clinics and hospitals (7). Even though the use of Mg alloys as a potential material for the human implant body was preferable, but it has a poor mechanical performance particularly in orthopedics applications (8,9).

One of the most favourable methods to increase the mechanical properties of Mg alloys is to incorporate reinforcement particle into the components of Mg alloy (10). The reinforcement particles are used to increase the strength and also enhance the corrosion resistance of the composite (11–14). Nowadays, carbon particle is a versatile element and usually used as a reinforcement particle for metal alloy due to their ability to increase the mechanical properties of any materials (15). In general, the use of nanoparticle such as carbon nanofibers (CNF) as a reinforcement particle is one of possible solutions in increasing the strength of a metal matrix (14). According to Orawan model (16), the Orawan strengthening effect will be increased by reducing the particle size of a composite. It is also reported the effect could significantly increase when CNF particles increased, at the same time the resistance to fracture (17) and the microhardness (18) were also increased. Moreover, it has a great potential as reinforcement particle due to their high surface area to volume ratio, nanoscale diameter, and mechanical properties (14,15). On top of that, the CNF is additionally is an animate elements of collagen fibrils (0.1– 8 µm in distance across) in the bone which helps in its regeneration.

The current biodegradable materials for orthopaedics implants such as iron based (19,20), zinc based (21–23) and Mg based (24,25) are not yet incorporated with the CNF element. Therefore, a fundamental study is needed to investigate the potential of CNF element that mixed with Mg alloys for developing a potential biodegradable and bioabsorbable implant that comprise with optimum mechanical strength. In this recent study, a development of Mg-Zn alloys with six different compositions of CNF was conducted and later was analysed via compression test, scanning electron microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDS). Our hypothesis is that the reinforcement of CNF to Mg-Zn alloys could improve the mechanical performance by increasing Young's modulus and compressive yield strength of that materials.

MATERIALS AND METHODS

Synthetisation of Mg-Zn reinforced with CNF

Powder metallurgy is a synthesising process composed of three main processing steps including the ball mills, compacting the mixture into a mold, and then sintering. First, all the powder is mechanically ground using ball mills. Next, the powder is compacted. To achieve the same compression ratio across more complex pieces, it is often necessary to use lower punch and upper punch. Finally, near its melting point, the compacted powder is exposed to high temperature called sintering.

At first, carbon nanofiber (CNF) with different compositions (1.0, 1.2, 1.4, 1.6, 1.8 and 2.0 wt. %) were dissolved in absolute ethanol using ultra sonication for 1 hr. At the same time, Mg-Zn powders were mixed in absolute ethanol using mechanical agitator. Next, the CNF solution was dropped into the Mg-Zn powder slurry and obtained a composite. The composite mixture was further mixed for 30 min to obtain the homogeneity.

The mechanical agitated composite was then filtered and vacuum dried at 60^{o}C for 12 hr. In order to increase the bonding between CNF and Mg-Zn matrix, the composite have been undergone mechanically milled using high-energy Fritsch Pulveristte P-5 planetary mill (250 RPM, 4 h) under argon atmosphere, where 20 mm stainless steel balls were used during the process. The milling process was paused for 6 min after every 15 min. The stainless-steel mold was loaded with weighted composite powder mixture. The mold and punch set-up were placed on an Instron machine with 400 MPa compaction pressure for 5 minute holding time (26). Next, the composite was placed inside the tubular furnace and underwent 4 hours of solid state sintering in inert (Argon) atmosphere using Compact Split Tube Furnace BS-1200-50X (27). Before sintering process can start, the argon gas was flow to the tube furnace for 15 minutes in order to ensure there was no pollution and oxygen inside the tube.

Characterisation of Mg-Zn reinforced with CNF

The microstructural analysis for the Mg-Zn alloys reinforced with CNF was performed using Scanning Electron Microscopy (SEM), TM 3000 model. Once the location of the composites was fulfilled with the condition of the specifications, they were placed in the chamber of SEM. To allow its output, the SEM was evacuated, then the specimen was magnified with 15kV electron shooting in the methodical mode under 50X.

The mechanical properties of Mg-Zn alloys reinforced with CNF, in terms of Young's modulus and yield strength were measured through the compression test. It was performed at room temperature with a speed rate of 2.00 mm/s according to the ASTM E9-2009 standard (28). A triplicate of each composition (1.0, 1.2, 1.4, 1.6, 1.8 and 2.0 wt. %) was tested to obtain the average result. The dimension (height x diameter) of every sample was approximately 5 x 10 mm.

RESULTS

Microstructural Analysis

The microstructural and surface comprehension of Mg-Zn alloys with CNF was investigated. Figure 1(a-f) shows SEM images for all compositions of the Mg-Zn alloys reinforced with CNF. Based on the figures, the compounds possessed some non-homogeneous microstructural properties. These heterogeneous structures show the diversity composition that existed in the composite with different compositions of the CNF. The addition of CNF to the composite effect the surface morphology of the samples. However, at low and high percentage of CNF, no significant differences were spotted, as shown in Figure 1(a), (e) and (f). Based on these images, it was found that the samples are not well compacted and pores. This may lead to a weak bonding between particles and will affect the mechanical strength of composites. The opened crack surface presents in these images will render the composites low mechanical properties. Whilst, image (b), (c) and (d) shows smoother

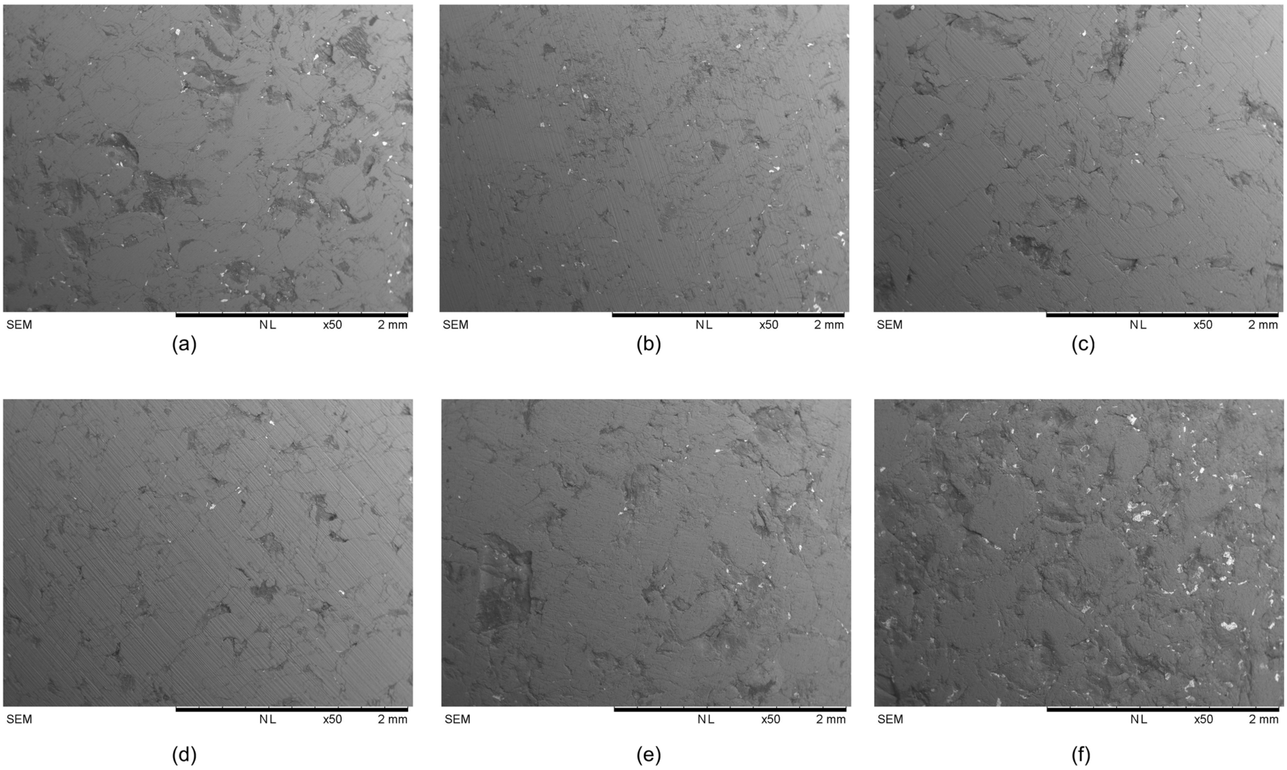

Figure 1: The morphology of Mg-Zn reinforced with (a) 1.0wt%, (b) 1.2wt%, (c) 1.4wt%, (d) 1.6wt%, (e) 1.8wt% and (f) 2.0wt% of CNF under 50X SEM magnification. All the samples were polished using silicon carbide paper before the SEM analysis.

surfaces and containing less pore compared the others.

Figure 2(a-f) shows the specific elements inside the composite from EDS analysis. The height peaks in EDS diagram show the quantification of each elements concentration that are present in the composites. Based on figures, there are three main elements in the composite (Mg, Zn and CNF) where these can indicate that there was no contamination occurring during the fabrication of Mg-Zn alloys reinforced with CNF. All elements released less than 2.0 keV of x-ray energy. The peak of Mg element was the highest recorded and also shows the highest percentage of weight measured recorded followed closely by C and Zn. For all cases, the Mg element released the highest energy of x-ray after the electron bombarded the surface while C has emitted the lowest x-ray energy. Other than that, the additional of CNF into the lattice of the Mg-Zn alloys caused the interfacial layer between Mg-Zn alloys and CNF. It made C easily to detect and the distribution of CNF on the surface is higher compared to Zn.

Strength analysis

Figure 3 shows the stress-strain curve of Mg-Zn alloys

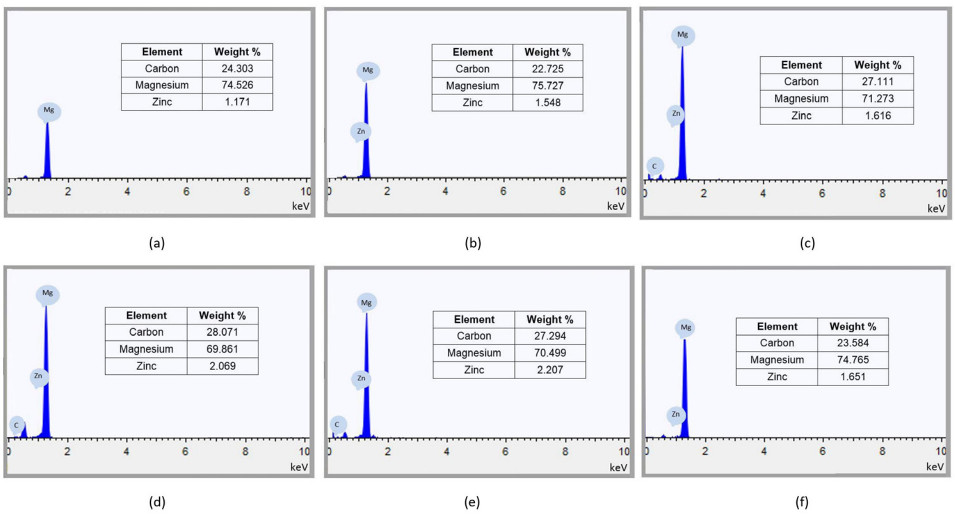

Figure 2: EDX analysis corresponding to Mg-Zn reinforced with (a) 1.0wt%, (b) 1.2wt%, (c) 1.4wt%, (d) 1.6wt%, (e) 1.8wt% and (f) 2.0wt% of CNF.

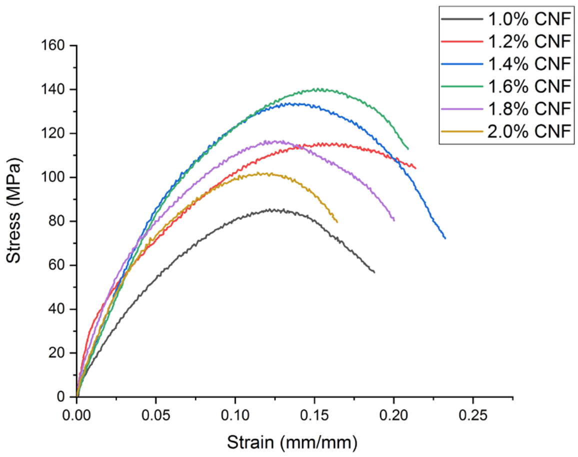

Figure 3: Compression stress-strain curve for Mg-Zn alloys with 1.0wt%, 1.2wt%, 1.4wt%, 1.6wt%, 1.8wt% and 2.0wt% of CNF respectively. The mechanical properties of the strength Mg-Zn reinforced with CNF were measured by the compression test according ASTM E9-2009 standard.

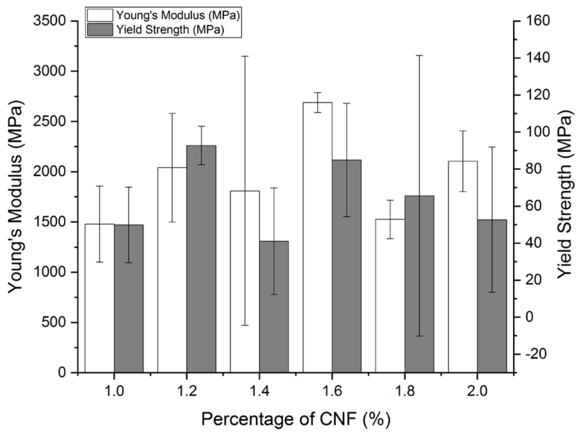

Figure 4: Young's modulus and yield strength of Mg-Zn reinforced with 1.0wt%, 1.2wt%, 1.4wt%, 1.6wt%, 1.8wt% and 2.0wt% of CNF respectively.

reinforced with CNF for different composition. The findings from analysis found that all curves behave likes ductile of metal properties. In general, the composites were initially elastic and yielded with increased pressure where the stress was remained on the plateau after yielding point while the strain continued to rise until ended with densification. For each composite, a triplicate compression test was done and examined to obtain the average and standard deviation of yield strength and Young's modulus. Based on the curves, 1.6 wt% CNF showed the highest value of maximum stress while 1.0 wt% CNF was the lowest. Other than that, the stress-strain curve for other compositions are lies in between these two curves. Besides, the proportional limit elastic zone of each curves is varying with increasing of CNF content. Beyond this limit, all curves obey the Hooke's law. Based on Figure 3, 1.0 wt% of CNF recorded the lowest area under the curve followed by 2, 1.2, 1.8, 1.6 and 1.4 wt% of CNF. It shows that 1.4 and 1.6 wt% of CNF have highest ability to regain its original shape after the force released. Moreover, 1.0 and 2.0 wt% of CNF were recorded the lowest plastic zone area compare to the other composites.

Figure 4 shows the average Young's modulus and yield strength for all composites with different composition of CNF. In general, the average Young's modulus value was increased when CNF concentration increased from 1.0 wt% (1478.97 MPa) to 1.2 wt% (2040.35 MPa), however it decreased when CNF concentration increased from 1.2 wt% to 1.4 wt% (1809.74 MPa). The increasing pattern was similar happened when the CNF concentration increased from 1.4 wt% to 1.6 wt% (2687.9 MPa), unfortunately decreased again substantially when the CNF concentration increased from 1.6wt% to 1.8 wt% (1525.79 MPa). As the CNF concentration is increased from 1.8 wt% to 2.0 wt% (2104.12 MPa) Young's modulus show some increment, however, the yield strength continued to decrease. Based on the figure, 1.6 wt% of CNF is the stiffest composite compared the others due to the highest elastic modulus. Obtained from our experimental results, an analysis of maximal stress feature in a material without causing plastic deformation was conducted to determine the compressive yield strength. The 0.2 % offset method was used to find the yield strength by choosing two arbitrary points that can be assumed to be in elastic region. Based on findings, a zig-zag pattern graph was obtained from this analysis and 1.2 wt% of CNF recorded the highest average compressive strength of 92.72 MPa compared to the other composites.

DISCUSSION

It is found that the Mg-Zn alloys with 1.6 wt% CNF exhibit the highest average Young's modulus (2687.90 MPa) and as the second largest average value of yield strength (84.91 MPa) as compared to other compositions. When the concentration of the CNF element is low (e.g. 1.0 wt%), the material's strength would mainly come from the Mg-Zn alloys and the weakening effects of the Mg-Zn and CNF interfaces would dominate, which resulted in strength decreased for the 1.2 wt%. By increasing concentrations of CNF, the reinforcing effect became increasingly important and overriding the weakening effect of the interfaces of Mg-Zn and CNF. The strength decreases from 1.6wt% to 2.0wt% should be due to the agglomeration of CNF and unwell mixture during the ball milling process. This can be observed based on the reduction of weight percentages from EDS results in a correlation with the depletion of CNF composition from 1.6 wt% to 2.0 wt%, and this was also reducing the yield strength. According to Preetkanwal et al. (29), the percentage of reinforcement particle in metal composite is depend on the rule of mixture. Based on this approach, the more reinforcement particle in a metal matrix may increase the elastic modulus of a composite (30). However, a low and high percentage of

CNF in Mg-Zn matrix in this study could be classified as immoderate and could not be used in structural application due to the unstable of composite to fulfil its function (31,32). In other study conducted by Slipenyu et al. (33) found that the excessive of silicone carbide in aluminium matrix caused the bad adhesion between reinforcement particle and the metal matrix. The rule of mixture explained where it was due to the load not effectively distributed and the mixture carry more load (34). Therefore, the low and high percentage of CNF in Mg-Zn matrix could be seen as not working properly and the mixture are unstable.

In the fabrication process of CNF composites, the reinforcing phase easily agglomerated. For a comparison, the results from other studies on porous magnesium composites combined with 1.5 wt% CNF has the highest average yield strength (74 MPa) and the largest average ultimate compressive stress (94 MPa) among the others (35). Our findings are similar to that of results where the maximum average strength was obtained for 1.6 wt% CNF. As compared to other study, it is found that pure Mg has Young's modulus of 1860 MPa (36) in which much lower than our findings for 1.6 wt% CNF (2687.90 MPa). From this recent study, we found that the most favourable option for the composition is 1.6 wt% CNF where the Young's modulus was highest among others. As reported by other studies, the highest modulus could provide sufficient strength and stability for treating fracture bone (37,38). However, all new materials that have been developed by many researchers are having less elastic modulus than natural bone (maximum of 23GPa) (36). This shows that more future studies are needed to develop a strong material that similar to real bone of human. In terms of yield strength, previous published articles reported that for pure Mg, porous Mg-1.5 wt% CNF and Mg-(4,6) Zn have the yield strength of 29.88, 74 and 100-235 MPa, respectively (27,35,36,39). Based on recent studies, we found that the yield strength of our composites was in range of 50 to 90 MPa (Figure 4) where these values are almost similar to other biodegradable implants. In order to prevent from brittle fracture of implant in human bone, a high yield strength compared to the human bone is needed. The finding from this study was quite similar to the previous studies done by Caroline et al. (40) where they concluded that the yield strength of femur bone for young people is at 110 ± 30 MPa through compression test. Nevertheless, the difference values may be due to fact that the experiments conducted by them are not similar to that of methods that we applied in this study. Some limitations have been considered where there were only 6 different compositions have been investigated. On top of that, there were only three samples for each composition have been tested using compression method. In the future studies, it is suggested that five replicates measurements can be performed to undergo strength analysis so that the results might be more interesting (28).

CONCLUSION

Results from this finding showed that the carbon nanofibers (CNF) can become a possible reinforcement element to increase the strength of magnesium alloys where the highest Young's modulus was 1.6 wt% CNF. This shows that the study has been successfully meet the main objective where to develop possible biodegradable material for implant application. However, further studies need to be investigated to prove and support the findings.

ACKNOWLEDGEMENTS

This research is fully supported by the Fundamental Research Grant Scheme (vote no. 5F135) under Ministry of Higher Education Malaysia, Universiti Teknologi Malaysia under Tier 2 Research University Grant (vote no. 15J84) and The Ministries of Research, Technology and Higher Education Republic of Indonesia through Director of Research and Community Service with contract number: 009/L10/AK.04/KONTRAK-PENELITIAN/2019.

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