INFLUENCE OF MULTI-WALLED CARBON NANOTUBES (MWCNTs) CONTENT ON THE MORPHOLOGICAL AND MECHANICAL PROPERTIES OF TITANIUM-HYDROXYAPATITE (Ti-HA) COMPOSITE

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ABSTRACT

Despite the promising properties of titanium-hydroxyapatite (Ti-HA) composites in biomedical applications, their mechanical performance needs further enhancement. The incorporation of multi-walled carbon nanotubes (MWCNTs) has been proposed to improve these properties, however, their tendency to agglomerate at higher concentrations may negatively impact the composite's performance. Ti-HA was successfully reinforced with MWCNTs using a process involving mechanical alloying, powder metallurgy, compaction, and sintering. This study investigated the effect of MWCNTs content, specifically 5 and 10 wt.%, on the morphological and mechanical properties of Ti-HA-MWCNTs composite. Every composition was evaluated for its compressive strength and Young's modulus. The values obtained for 5 wt.% were 176.39 MPa and 66.82 GPa, respectively. For 10 wt.%, the values were 61.50 MPa and 23.05 GPa. The porosity values were determined to be 15.19% for 5 wt.% and 26.02% for 10 wt.%. The decline observed with the addition of 10 wt.% MWCNTs was associated with the MWCNTs agglomeration within the Ti-HA matrix, and the weak interfacial bonding between them. Additionally, the density measurements yielded 3.40 g/cm³ for 5 wt.% and 2.76 g/cm³ for 10 wt.%. These findings emphasise the importance of optimising dispersion techniques and improving interfacial bonding strategies in order to fully utilise MWCNTs' reinforcing potential in Ti-HA composites.

Keywords: *Titanium, bone implant, MWCNTs, hydroxyapatite, agglomeration.*

1.0 INTRODUCTION

Every year, osteoarthritis and fractures necessitate over two million bone transplants worldwide. Consequently, metal implants are frequently employed as supportive materials within the field of orthopaedics [1]. There has been a growing interest in the development of metal implants with enhanced mechanical strength. The implant must effectively facilitate the transmission of loads to bone tissues over an extended duration when inserted into the human body. This characteristic is crucial when prostheses are employed to replace damaged hard tissues. Among all commonly used matrix materials in metal matrix composites, titanium (Ti) matrix has experienced significant advancements in recent years, driven by the increasing demand for improved implant materials with enhanced mechanical properties, corrosion resistance and biocompatibility [2]. Nevertheless, a significant obstacle that must be addressed is the reduction of the disparity between the Young's modulus of cortical bone (ranging from 10-40 GPa) and existing Ti implants (ranging from 100-120 GPa [3, 4]. The stress shielding phenomenon occurs, causing bone resorption and, as a result, premature implant failure due to loosening within the host bone. As a result, additional improvements are required to accurately reproduce the mechanical behaviour of cortical bone. Aside from that, Ti is bio-inert, meaning that no cellular reactions occur and the implants are simply encapsulated, impeding the growth of native tissue.

While the majority of metals demonstrate this phenomenon, ceramics like hydroxyapatite (HA) ($Ca_5(OH)(PO_4)_3$) possess chemical constituents that bear a resemblance to those found in human bone. In orthopaedics, HA is often utilised as a replacement for bone or as a constituent of composite materials. Ceramic HA exhibits low mechanical properties, such as low fracture and strength toughness due to its highly brittle nature. Therefore, it is not suitable for standalone application in weight-bearing regions of the human body. The integration of Ti with HA has been proposed to enhance the apatite-forming ability of inert implants while preserving the mechanical strength of Ti as bone implant material. However, the addition of HA alone will not effectively address the mismatch in Young's modulus between Ti implants and natural bone.

Carbon nanotubes (CNTs) possess the shape of a graphite sheet rolled into a cylinder and can have various structures, such as, multi-walled CNTs (MWCNTs), single-walled CNTs (SWCNTs), and rope nanotubes. Generally, MWCNTs are more prevalent than SWCNTs due to their superior chemical stability and reduced production expenses. CNTs possess exceptional chemical durability, mechanical strength, thermal properties, and electrical properties due to their nanoscale structure [5]. Therefore, they are of interest not only in electrical and mechanical applications but, also in biomedical applications. The elasticity coefficient of CNTs is approximately 1 TPa, and the tensile strength is about 60 GPa [6]. Several studies have attempted to improve the properties of Ti by incorporating CNTs so that they are comparable to cortical bone. Liu et al. [7] fabricated high strength CNTs/Ti composites without sintering using high-energy planetary ball milling, by combining cold compaction and hot extrusion. A superior tensile strength of 1262 MPa was achieved due to the effects of grain refinement and secondary-phase strengthening, as a result of enhanced CNT integrity. This demonstrates the potential of nanotube reinforcement in improving the mechanical behaviour of Ti alloys. Meanwhile, Nezhad et al. [8] conducted a study on nanocomposites consisting of hydroxyapatite/titanium nanotube/carbon nanotubes (HA/TNT/CNTs) having varied CNTs content (e.g. 1, 1.5, and 2 wt.%). They reported that an increase in the CNTs content resulted to a substantial increment in nano/micromechanical properties, biocompatibility, and tribological properties. In another study, Dabees et al. [9] investigated the effect of MWCNTs content (e.g. 1, 2, 3, 4, and 5 wt.%) on hybrid composite. The study discovered that 5 wt.% MWCNTs had strong physical bonding. However, to optimize mechanical properties, the ideal content of MWCNTs in the matrix must be determined. When the MWCNTs content exceeds a particular threshold, the nanotubes start to aggregate into clusters, compromising the mechanical characteristics. This supports the findings of Jagannatham et al. [10] who developed composites by milling with various MWCNTs contents (e.g. 0.5, 1, 1.5, and 2 wt.%), followed by sintering and hot extrusion. They observed that adding MWCNTs up to 1 wt.% improved tensile, hardness, and dry sliding wear performance of the manufactured composites, while adding more than 1 wt.% MWCNTs, decreased the mechanical and tribological properties. Despite these promising findings, there remains a knowledge gap regarding the optimal content of MWCNTs to achieve both desirable mechanical properties with cortical bone. Furthermore, most prior studies have focused on MWCNTs additions up to 5 wt.% while the effects of higher MWCNTs contents, particularly those between 5 and 10 wt.% on the mechanical performance of Ti-based composites remain largely unexplored.

The primary obstacles to achieving optimal utilisation of CNTs as a reinforcing agent in metal matrix composites pertain to the formidable van der Waals forces between CNTs particles, which impede the attainment of uniform dispersion, as well as the hydrophobic nature of CNTs, establishing a weak bonding between the matrix and the CNTs. The observed decrease in strength regarding CNTs may be assigned to the agglomerate's formation, which is facilitated by their one-dimensional nature and susceptibility to coagulation. Numerous researchers have endeavoured to address this issue by employing high-energy planetary ball milling, a technique capable of delivering sufficient energy to overcome the van der Waals force constraint [11, 12, 13]. Consequently, CNTs exhibit enhanced dispersion characteristics within the Ti powder matrix. In another study, Feijoo et al. [14] demonstrated that the low-energy ball milling approach does not properly scatter MWCNTs bundles and does not accomplish adequate dispersion and assimilation in~~to~~ the metal matrix. In addition, it is imperative to employ chemical treatment to introduce hydrophilic functional groups onto the surface of the MWCNTs. This method permits MWCNTs to disperse and eliminate nanotube agglomeration, thereby enhancing the bond between MWCNTs and the matrix. Nitric acid (HNO_3) and a mixture of HNO_3 and sulphuric acid (HNO_3:H_2SO_4) can be used as reagents [15, 16]. These aforementioned studies have established the foundation for examining the effect of reinforcement materials on the mechanical properties of Ti alloys. Nonetheless, less study has investigated the combined effects of higher MWCNTs content (5-10 wt.%) and HA on the compressive strength and Young's modulus of Ti-based composited for potential implant applications.

In this study, the Ti-HA-MWCNTs composite was fabricated using mechanical alloying and powder metallurgy techniques. The morphological and mechanical properties of a composite containing MWCNTs ranging from 5 to 10 wt.% were investigated. The novelty of this work is that it investigates the effect of a relatively higher MWCNT content on the mechanical properties of the composite with cortical bone, an area that has not received adequate attention in previous literature. The primary goal of this study is to create a Ti-HA-MWCNT composite with mechanical properties comparable to cortical bone, specifically Young's modulus and compressive strength. By addressing these gaps, this study aims to contribute valuable data for the development of load-bearing implants with improved performance and durability.

2.0 METHODOLOGY

2.1 Raw materials

The starting materials were commercially pure Ti powder (cp-Ti, obtained from Strem), hydroxyapatite (HA, obtained from Sigma-Aldrich), and MWCNTs (obtained from Hasrat Bestari Sdn Bhd). The mean particle size of cp-Ti powder was 139.02 μm, while HA powder had a particle size of 10.36 μm. MWCNTs with a purity of over 95%, an outer diameter of more than 10 ± 1 nm, and lengths ranging from 1 to 5 μm were used as the reinforcing material.

2.2 Acid treatment

The MWCNTs were modified using the acid treatment recommended by Zaine et al. [17]. Other chemicals used included analytical reagent grade H_2SO_4 and HNO_3, both of which had a purity of 95-98%. Merck supplied all of the chemicals. The MWCNTs were heated for 6 h at 100 °C in a controlled environment of HNO_3:H_2SO_4 (3:1 v/v). The mixture was permitted to undergo cooling at ambient temperature, subsequently by filtration as well as repeated washing with distilled water until the pH of the water approached 7.0. The MWCNTs were dried in an oven at 80 °C for one night to remove any volatile solvents.

2.3 Preparation of Ti-HA-MWCNTs composite

Figure 1 depicts the composite preparation procedure for Ti-HA-MWCNTs. The powders of Ti, HA, and MWCNTs were ball-milled with stainless-steel balls in a stainless-steel jar character a high-speed planetary milling machine (Fritsch Pulverisette, P-5 planetary mill machine) at 200 rpm in order to achieve homogeneous mixing. The ball-to-powder weight ratio was fixed at 10:1, and the milling time was set to be 2 h. To avoid excessive cold welding of the powders, a process control agent (PCA) of n-heptane was added. Furthermore, the mixed powder was compacted into 10 mm diameter pellets with a Specac hand press machine at 400 Mpa pressure. The compaction process consolidates the particles of Ti, HA, and MWCNTs and prepares the sample for the subsequent sintering procedure. The sintering temperature was set to 1000 °C for a duration of 2 h, with a heating rate of 10 °C/min. The samples were subsequently cooled to room temperature in a furnace in order to create a composite of Ti-HA-MWCNTs that is homogeneous and dense.

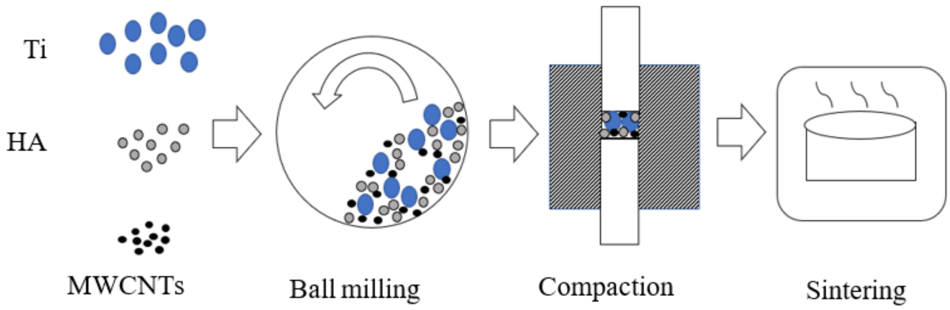

Figure 1. Fabrication of Ti-HA-MWCNTs composite using mechanical alloying and powder metallurgy route

2.4 Materials characterization

The sintered samples' microstructures were observed using a scanning electron microscope (SEM, TM3030 Tabletop Microscope with SwiftED3000). The compression properties were determined using a universal testing machine (Shidmadzu AG-IS-50kN) at a crosshead speed of 1 mm/min. Moreover, the sample possessed a diameter of 10 mm and a thickness of 10 mm, following the guidelines of ASTM E9-09. Three samples of each material type were examined in order to determine compressive strength and Young's modulus. Archimedes' method was used to calculate the bulk density of the samples following sintering. The relative density is useful for representing densification, as shown in Equation 1. As shown in Equation 2, it has an inverse relationship with porosity. The average porosity was calculated by taking three readings for each sample.

$$\text{Relative density (\%)} = \frac{\text{Measured density}}{\text{Density}_{\text{ROM}}} \ x\ 100\% \quad (1)$$

$$\text{Porosity (\%)} = 1 - \text{Relative density (\%)} \quad (2)$$

3.0 RESULTS AND DISCUSSION

3.1 Density measurement

Figure 2 shows the density measurement of the Ti-HA-MWCNTs composite, demonstrating its relationship with the increasing MWCNT content. The addition of MWCNTs clearly reduced the composite density slightly. It should be noted that the highest density was achieved at 5 wt.% MWCNTs. However, when the MWCNTs content exceeded 5 wt.%, the density declined. The lowest density was achieved with 10 wt.% MWCNTs, indicating that MWCNT content has a significant impact on composite density. The decrease in composite density as MWCNT content increased could be attributed to the MWCNTs' relatively low density.

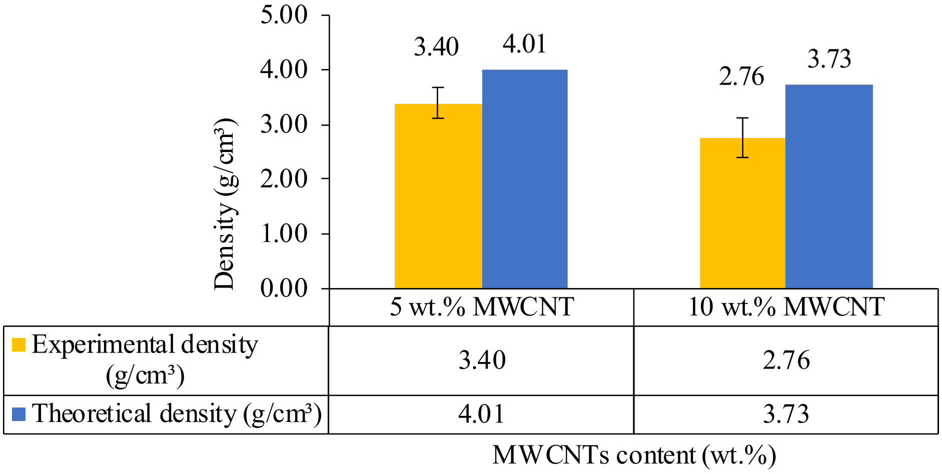

Figure 2: Experimental density and theoretical density of 5 wt.% MWCNTs and 10 wt.% MWCNTs

Ti, HA, and MWCNTs had respective densities of 4.51 g/cm^3, 3.156 g/cm^3, and 1.7 g/cm^3. When MWCNTs were introduced into the composite, they displaced a portion of the matrix material, which resulting in a lower overall density. As the content of MWCNTs increased up to 10 wt.%, a greater volume of low-density MWCNTs was incorporated into the composite, contributing to the decrease in density. MWCNTs' lower density compared to matrix materials caused a dilution effect, resulting in a reduction in overall material density. This trend is consistent with the general principle that when a higher percentage of lower-density reinforcements is used, the overall density of the composite decreases. Additionally, the increased in MWCNTs content aggravated agglomeration, causing pores to increase in number and expand in size [18]. As a result, the densification of the composite was hindered. This is in line with a study performed by Kim et al. [19], who confirmed that the addition of 5 to 10 wt.% MWCNTs reduced the density of composite, starting from 5.98 g/cm^3 to 5.76 g/cm^3 which was caused by MWCNTs agglomeration.

3.2 Porosity measurement

Figure 3 shows the porosity of the Ti-HA-MWCNTs composite. The lowest porosity was achieved with 5 wt.% of MWCNTs (15.19%). The porosity increased to 26.02% as a result of further enhancement of MWCNTs content at 10 wt.%. Several factors could have contributed to the increase in porosity. One possible explanation is the agglomeration of

MWCNTs within the composite. The higher the content of MWCNTs, the greater the likelihood of their agglomeration. When MWCNTs agglomerate, they form clusters within the composite, leaving gaps between the clusters. These voids help to increase the composite's overall porosity. The dispersion of MWCNTs within the composite matrix is a subsequent factor influencing porosity.

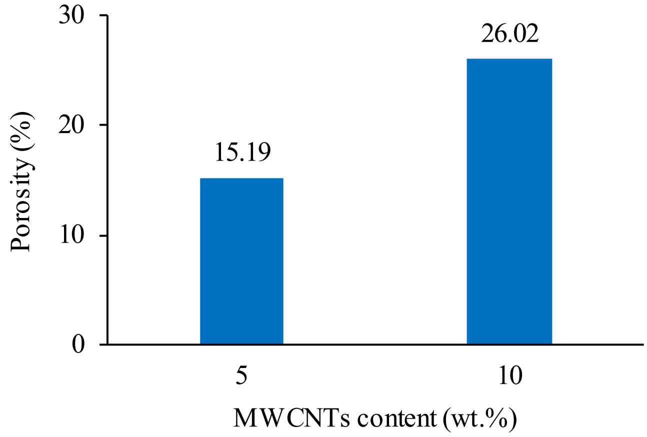

Figure 3: Porosity of 5 wt.% MWCNTs and 10 wt.% MWCNTs

As the content of MWCNTs increases, the difficulty of achieving uniform dispersion increases. Inadequate dispersion may result in an uneven distribution of MWCNTs, with regions having higher concentrations and others having lower concentrations. The variations in concentration may cause void formation in the composite, increasing its overall porosity. As a result, if the MWCNT content exceeds 10 wt.%, there is a greater likelihood of agglomeration and poor dispersion of MWCNTs, resulting in increased porosity and larger voids. The negative impact of porosity on composite mechanical properties has been well documented. Higher porosity can weaken the material, lowering its compressive strength and Young's modulus. Furthermore, porosity affects composite density, with higher porosity resulting in a lower density. To improve the mechanical performance of the composite, it is critical to optimise the dispersion of MWCNTs and reduce its porosity.

3.3 Morphological observation of Ti-HA-MWCNTs composite

Figure 4 depicts the SEM morphology of a composite consisting of Ti-HA-MWCNTs with varying MWCNT contents. When the MWCNTs content is 5 wt.%, the MWCNTs are evenly distributed across the surface of the Ti-HA composite, as shown in Figure 4a. The observation indicates that the ball milling process effectively disperses the MWCNTs onto the Ti-HA particles. However, with a content of 10 wt.% MWCNTs, the microstructure exhibits minimal changes (Figure 4b).

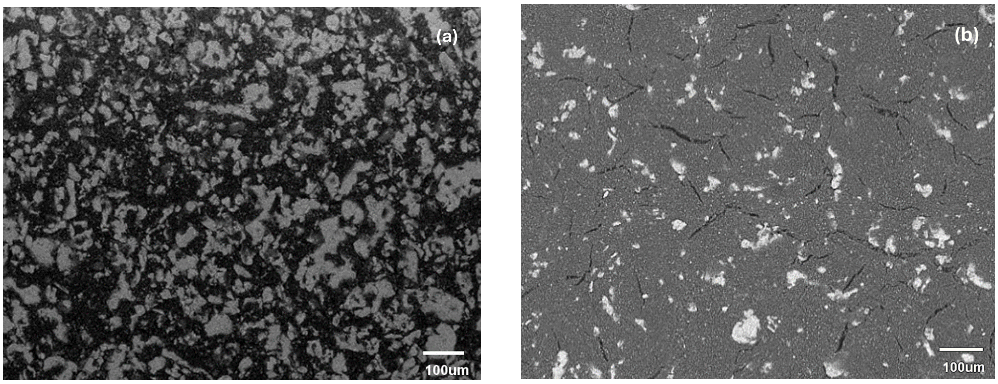

Figure 4: SEM image of Ti-HA composite with different MWCNTs content: (a) 5 wt.% MWCNTs, and (b) 10 wt.% MWCNTs at magnification of 200x

Higher MWCNT content causes an increase in black spots within the microstructure, corresponding to MWCNT clusters in the mixed powder. The incident was caused by an excessive amount of MWCNTs. Thus, the ball milling dispersion could no longer be distributed evenly because there were too many MWCNTs, resulting in the formation of MWCNT clusters, as shown in Figure 5. The thread-like architecture of MWCNTs can create a network-like structure by interweaving and forming van der Waal and π bonds [20]. As a result, the van der Waals forces reflect the difficulty in characterising the dispersion of MWCNTs, which is one of the primary causes of MWCNT entanglement. This indicates that the distribution of 10 wt.% MWCNTs during the production step was only partially successful in achieving a composite with a high MWCNTs content. Xu et al. [21] reported similar experimental outcomes, wherein an excessive amount of MWCNTs content leads to poor ball milling dispersion and caused more black spots in the microstructure. In such cases, agglomeration and porosity become unavoidable [22, 23].

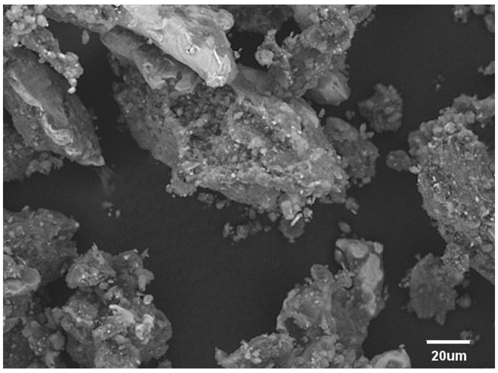

Figure 5: Agglomeration in 10 wt.% MWCNTs after milling at magnification of 1000x

Higher MWCNTs content leads to weaker internal bonding strength at the MWCNTs cluster, making it more likely that it will serve as a source of cracks, as shown in Figure 6. When the composite undergoes deformation, areas of high stress concentration can develop. The presence of stress concentration during deformation can reduce the ability of material to withstand further deformation before breaking, cracking and the degradation of fracture and strength in composites. Note that this phenomenon collaboratively strengthens the reduction in both the strength and Young's modulus of the composite.

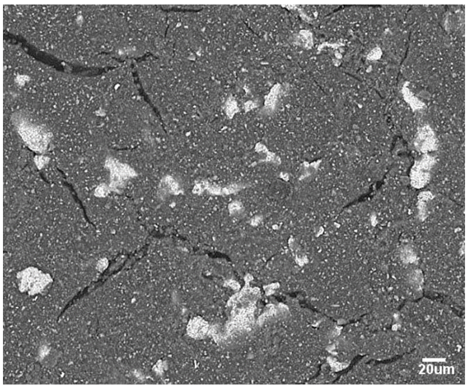

Figure 6: Higher occurrence of cracks in Ti-HA consisting of 10 wt.% MWCNTs after sintered, indicating a potential structural weakness, at magnification of 500x

3.4 Mechanical properties of Ti-HA-MWCNTs composite

Figure 7 shows the trends observed in the compressive strength and Young's modulus of Ti-HA-MWCNTs composite as a function of MWCNTs content. The study's findings revealed a consistent pattern in compressive strength and Young's modulus for composites. Specifically, both parameters exhibited a decrease as the content of MWCNTs rose to 10 wt.%. Moreover, the composite material comprising 5 wt.% MWCNTs established the highest compressive strength in comparison to the other compositions tested. Specifically, it achieved a compressive strength of 176.39 MPa, along with Young's modulus of 66.82 GPa. The observed increase could be attributed to process hardening that occurred during ball milling. This was due to the high energy imparted by the ball milling process, leading to the encapsulation of MWCNTs within the matrix. In the present scenario, the MWCNTs exhibited a bridging function within the matrix, facilitating load transfer between the Ti and HA via a bridging mechanism. The composite's compressive strength was increased due to the suppression of plastic deformation and fracture processes in the matrix. On the other hand, MWCNTs are preferentially distributed at grain boundaries, blocking them and refining the grains, resulting in higher-density and uniformly dispersed dislocations and increasing composite strength [24, 25]. However, when the MWCNTs content exceeded 5 wt.%, both compressive strength and Young's modulus decreased significantly. This finding aligned with Kumar et al. [26], who examined the effect of 0, 1, 3, 5, and 7 wt.% MWCNTs on hybrid nanocomposites fabricated by ball mixing process and followed by compression moulding. They found that 5 wt.% MWCNTs was the optimal content for improving the mechanical properties of hybrid nanocomposites. The rationale behind this phenomenon was that when the quantity of MWCNTs surpasses a specific threshold, the nanotubes have a tendency to amass and form agglomerates, resulting in clusters.

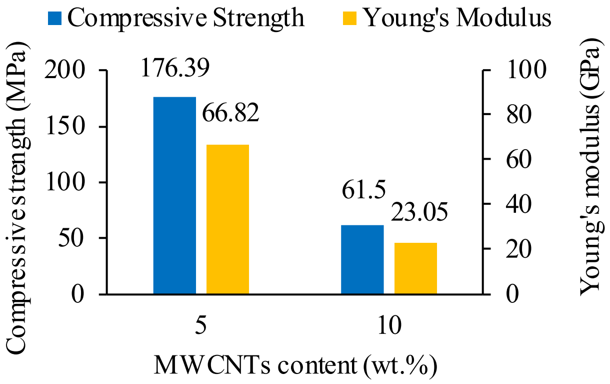

Figure 7: The effect of MWCNTs content on compressive strength and Young's modulus of Ti-HA composite

The interfacial area between the matrix and reinforcement was reduced by the agglomeration of MWCNTs. As a result, the load transfer strengthening capability of MWCNTs was compromised, which leads to elevated stress concentration and the formation of cracks, thereby compromising the mechanical properties. Hence, the decline in compressive strength may be attributed to the presence of weak interfacial bonding and the agglomeration of MWCNTs, which exist as flaws and cracks within the Ti-HA matrix. Inevitably, poor distribution occurred, and agglomeration and porosity formed at a higher content of MWCNTs [27]. The reason behind this phenomenon was that agglomeration gave rise to internal stresses and defects, which function as micro voids, resulting in a significant reduction in mechanical properties. Consequently, achieving a uniform distribution of MWCNTs within the matrix material and preserving their structural integrity were crucial factors in enhancing the mechanical characteristics. This enhancement could be assigned to the effective transfer of loads at the interface between the MWCNTs and the matrix.

4.0 CONCLUSION

This study investigated the effect of increasing the MWCNT content on the mechanical properties of Ti-HA composite. The study's findings can lead to the following conclusions:

1. Mechanical alloying and powder metallurgy methods were used to successfully fabricate Ti-HA-MWCNTs composites.
2. The composite with 5 wt.% MWCNTs (176.39) had higher compressive strength than that with 10 wt.% MWCNTs (61.50 MPa). The agglomeration of MWCNTs in the composite containing 10 wt.% MWCNTs was the primary cause of its low strength. Agglomerated MWCNTs can cause stress concentration sites and weak points in the matrix, increasing its susceptibility to cracking under external load. It is also possible to argue that the heterogeneous distribution of MWCNTs acted as a defect, resulting in regions with higher nanotube concentrations. This resulted in crack initiation and reduced the composite's mechanical properties.
3. The higher porosity of 10 wt.% MWCNTs (26.02%) compared to 5 wt.% MWCNTs (15.19%) led to a decrease in Young's modulus. The Young's modulus of 5 and 10 wt.% MWCNTs was 66.82 GPa and 24.05 GPa, respectively.

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