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Effect of yttria-stabilized zirconia content on morphology, mechanical properties and *in-vitro* bioactivity behaviour of powder metallurgy stainless steel 316L

Ahmad Farrahnoor[1,a*], Mahfuzah Zainudin[1,b], Nur Antasya Amany Md Nor Azman[1,c] and Nur Annesa Wilda Faudzi[1,d]

[1]Centre for Mechanical Engineering Studies, Universiti Teknologi MARA, Cawangan Pulau Pinang, Permatang Pauh Campus, 13500 Pulau Pinang, Malaysia

[a*]farra728@uitm.edu.my, [b]mahfuzah035@uitm.edu.my, [c]antasyaazman@gmail.com, [d]nesawilda@gmail.com

Abstract

Among the metallic materials, stainless steel 316L (SS316L) is widely used in hip and knee replacement surgery due to its cost-effectiveness, good mechanical properties, and biocompatibility. However, the stress shielding phenomenon provokes bone resorption and consequent adverse effects on prosthesis fixation because of its high Young's modulus. Furthermore, the weak mechanical bond with the bone tissue due to its biological inertness delays the healing process. By incorporating various yttria-stabilized zirconia (YSZ) contents (i.e. 2, 3, and 4 wt.%), SS316L/YSZ composite was fabricated using the powder metallurgy method. Powder blends consisting of SS316L and YSZ were milled at a speed of 200 rpm for 2 h using 10 mm in diameter hardened steel balls with a ball-to-powder ratio (BPR) of 10:1. All composites were compacted at 200 MPa and sintered at 800 °C for 2 h. Morphological observations, mechanical characterizations and bioactivity tests were performed to evaluate the feasibility of the SS316L/YSZ composite as an implanted biomaterial. Results indicated that all YSZ contents presented Young's modulus of the composite ranging between 33.91 and 21.71 GPa, overcoming the problem of stress shielding. The compressive strength of SS316L/YSZ composite decreased remarkably from 208.92 to 5.43 MPa with increasing YSZ content. *In-vitro* biological evaluation tests confirmed that with increasing YSZ content, the rate of apatite deposition on the SS316L/YSZ composite's surface had an increasing trend after four days of immersion in Hank's Balanced Salt Solution (HBSS). Based on the results, the powder metallurgy-fabricated SS316L/2YSZ composites have the desired mechanical properties and good osteointegration, making them feasible materials for orthopedic implant applications.

Keywords: bone implant, apatite, bioactivity, yttria-stabilized zirconia, stainless steel

1. Introduction

In recent years, there has been a growing trend in research focusing on enhancing the functionality of biomedical implants. Metallic implants such as stainless steel 316L (SS316L) have been commonly used by clinicians to treat fractures and bone defects. However, it shown limitations in terms of long-term durability. For example, Young's modulus of SS316L ranges from 189 to 205 GPa [1]. The implant must have Young's modulus in the range of 10 to 40 GPa, similar to that of cortical bone, in order to distribute the load evenly to the connecting bones [2]. The similarity in Young's modulus of the bone and implant is crucial in reducing the stress shielding effect, which can often lead to degradation and loosening of the tissue surrounding the implant.

Additionally, SS316L is an inert material that fails to form strong chemical bonds with human bone. The development of bioactive materials that can form a chemical link with bone has been proposed to address these issues. For instance, a combination of the metallic substrate SS316L with the bioactive material yttria-stabilized zirconia (YSZ) is suggested. YSZ offers excellent biocompatibility, bone conductivity, and bone bonding properties, with minimal risk of causing allergic reactions in the human body [3], [4]. Furthermore, YSZ has a low load-bearing capacity, which limits its application without reinforcement. By combining the advantages of SS316L and YSZ, a metal implant can be created with improved material properties.

The investigation of SS316L/ceramic composites has recently attracted increasing attention and is being considered for bone implant applications. However, to our knowledge, the effect of YSZ content on the mechanical properties and bioactivity behavior of SS316L produced through high-energy ball milling and powder metallurgy has not been explored. By varying the YSZ content as a reinforcement material in the SS316L matrix up to 4 wt.%, this study aims to determine the optimum YSZ content that can enhance the desired properties of the composite.

2. Materials and Method

2.1 Preparation of SS316L/YSZ composite

This study utilized SS316L powder (Sandvik Osprey) and YSZ (Sigma Aldrich). YSZ contents of 2, 3, and 4 wt.% were selected to investigate their effects on SS316L, aiming to adapt the composite for implantation in human bodies. The milling process involved mixing SS316L and YSZ in the desired ratio using a high-energy planetary ball mill at 200 rpm for 2 h (Table 1). This method is an alternative means of producing metallic and ceramic powder particles in the solid state. Polyvinyl alcohol (PVA) served as a process control agent (PCA), effectively dissipating heat, preventing excessive cold welding, and enhancing milling efficiency. The milling occurred in an argon atmosphere, utilizing a 250 mL hardened steel vial and 10 mm diameter hardened steel balls. The ball-to-powder (BPR) weight ratio was set at 10:1. Prior to pressing, the weighed powder was filled into a pressing die. The powder was then compacted using a pressing machine (Specac) under a pressure of 200 MPa, and the compacting process was held for 2 min. After compaction, the resulting green pellets were transferred into a tube furnace for sintering. During sintering, the composites were heated at a rate of 10 °C/min in an argon gas flow until they reached a temperature of 800 °C. The sintering duration was 2 h. Subsequently, the composites were allowed to cool inside the furnace.

Table 1: Composite composition

Composite ID	SS316L content (wt.%)	YSZ content (wt.%)
SS316L/2YSZ	98	2
SS316L/3YSZ	97	3
SS316L/4YSZ	96	4

2.2 Characterization of SS316L/YSZ composite

The compressive strength and Young's modulus of the SS316L/YSZ composite were evaluated using a universal testing machine (Shimadzu AG-IS-50kN) with a crosshead speed of 1 mm/min. The cylindrical composites with a diameter of 10 mm and thickness of 10 mm were prepared for the measurements, following ASTM E9-09 standards (standard test methods of compression testing of metallic materials at room temperature). The surface morphology of the SS316L/YSZ composite was observed using a scanning electron microscope (SEM, TM3030 Tabletop Microscope with SwiftED3000). For the immersion testing, all the sintered composites were immersed in Hank's Balanced Salt Solution (HBSS), which has similar ion concentrations to human blood plasma. The HBSS was manufactured by Gibco, Life Technologies (14175-095). The composites were immersed individually in 30 mL of HBSS with an initial pH value of 7.4 and placed in a water bath (model: TWB-30D) at 37 °C. The immersion period lasted for four days, during which the SS316L/YSZ composites were kept in HBSS without agitation to maintain a stable chemical composition and pH of the solution.

3. Result and Discussion

3.1 Raw materials

Fig. 1 shows the powder morphology of SS316L and YSZ, with SS316L having spherical particles while YSZ exhibits an irregular shape.

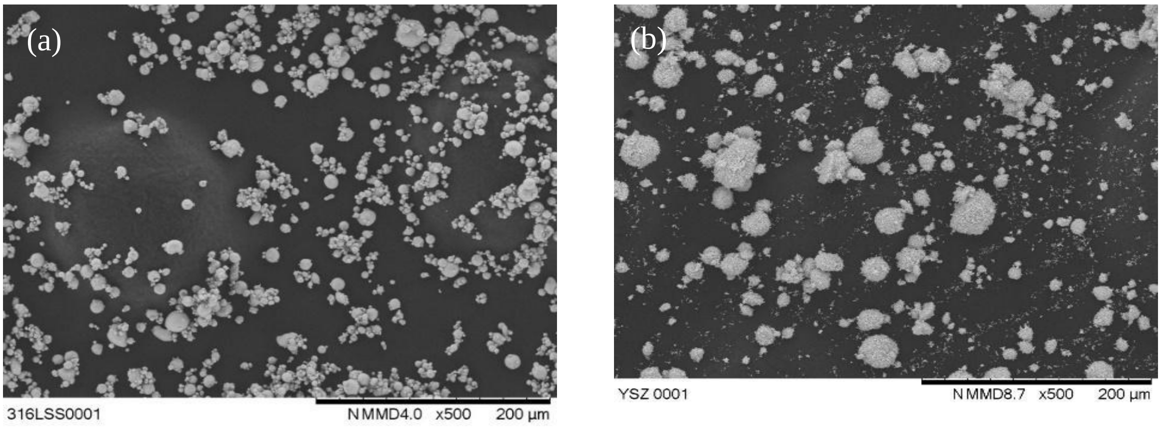

Fig. 1: SEM micrographs of (a) SS316L powder, and (b) YSZ powder (500x magnification)

3.2 Morphological observation of as-sintered SS316L/YSZ composite

Fig. 2 displays a series of SEM micrographs illustrating the changes in surface morphology of SS316L with varying YSZ contents after sintering at 800 °C. The surfaces revealed some isolated pores and unmelted particles. The presence of unmelted particles indicates that the sintering temperature used was insufficient to cause significant melting necessary for effective bonding and necking between the particles. Fig. 2(a) demonstrates a slightly denser surface appearance after sintering, indicating the best mechanical resistance under these conditions. The powders undergo deformation under compaction pressure, leading to an increased contact area between the particles. This increased contact area enhances the bonding between the SS316L and YSZ phases, promoting better interfacial adhesion and mechanical strength. However, YSZ exhibits strength under compression but is weak under tensile and shear stress. The property of YSZ becomes relevant when considering the effect of increasing YSZ content in the composite. Consequently, with a higher YSZ content, the powders become more difficult to deform during compaction. As a result, it becomes challenging to achieve uniform densification, leading to a rougher surface appearance after sintering. This rough surface is characterized by a significant number of pores within the composite. Apart from that, with an increase in YSZ content, the distribution of pores also increases due to the weak interfacial bonding between the metal (e.g., SS316L) and ceramic (e.g., YSZ) phases. As the YSZ content increases, more ceramic particles are dispersed within the metal matrix. The dissimilarity in the nature of the metal and ceramic leads to challenges in forming a strong and continuous bond at their interface during the sintering process. The difference in thermal expansion coefficients and crystal structures between the metal and ceramic phases further contributes to the weak bonding. As a result of this weak interfacial bonding, the formation of pores becomes more prevalent. During sintering, areas of poor bonding can create voids within the composite leading to increased porosity. These pores act as stress concentrators and reduce the composite's overall mechanical strength and structural integrity. Therefore, increasing the YSZ content in the SS316L/YSZ composite results in a composite with a rougher surface and a higher number of pores, adversely affecting its mechanical properties.

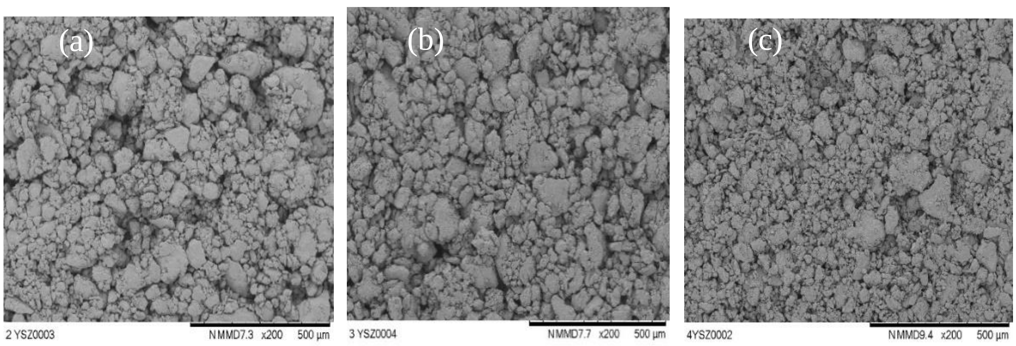

Fig. 2: SEM micrographs of as-sintered SS316L/YSZ composite with different contents of YSZ : (a) 2 wt.%, (b) 3 wt.%, and (c) 4 wt.% (200x magnification)

3.3 Mechanical properties of as-sintered SS316L/YSZ composite

Fig. 3 shows the variation in compressive strength for the SS316L/YSZ composite. Among the composites tested, the one containing 2 wt.% YSZ exhibited the highest compressive strength, measuring 208.92 MPa. On the other hand, the composite with 4 wt.% YSZ displayed the lowest compressive strength at 21.71 MPa. The increase in YSZ content resulted in a decrease in compressive strength. The reduction in strength can be attributed to the poor interfacial bonding between the metal-ceramic phases, as YSZ is a brittle material. It can be postulated that an increase in YSZ content may facilitate and promote the transformation from the tetragonal to the cubic phase. This may result in significant microstructural instabilities within the composite, causing localized stress concentrations that drastically reduce mechanical properties [5]. Additionally, the changing pattern of Young's modulus follows a similar trend to the compressive strength. The Young's modulus was calculated by determining the slope from the stress-strain curve. Consequently, Young's modulus decreases significantly with increasing YSZ content. The composite with 2 wt.% YSZ achieved the highest Young's modulus at 33.91 MPa while composite with 4 wt.% YSZ exhibited the lowest, measuring 5.43 GPa. The addition of YSZ content allows for changes in atomic bonding intensity, which affects the interfacial bonding between SS316L and YSZ. The weak interfacial bonding leads to increased porosity and compromised stress transfer between the phases, resulting in a lower Young's modulus.

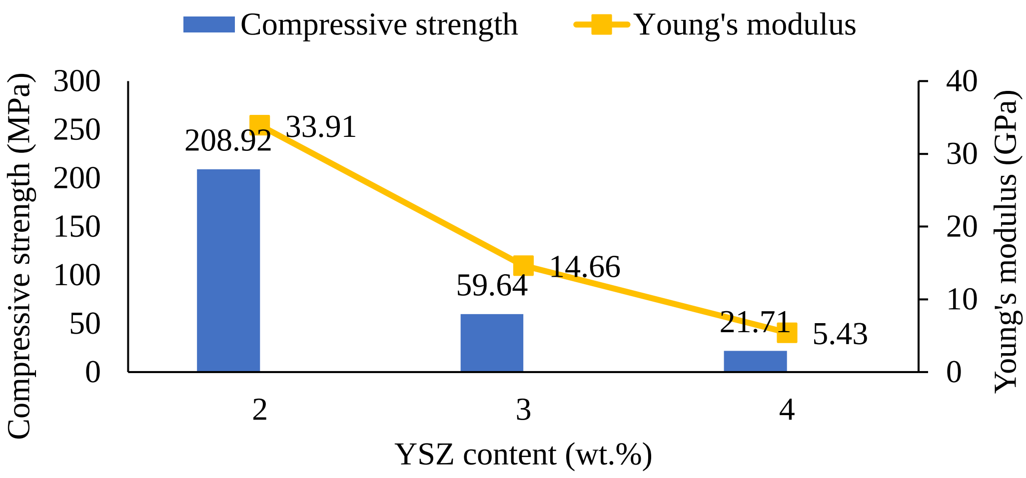

Fig. 3 : Compressive strength and Young’s modulus of SS316L/YSZ composite at various YSZ content

3.4 Apatite deposition on as-immersed SS316L/YSZ composite

Fig. 4 shows SEM images of the SS316L/YSZ composite surfaces after four days of immersion in HBSS. All composites showed similar morphologies of globule apatite, similar to the apatite crystal obtained by Nakamura M. et al. [6], who observed the growth of apatite in the YSZ when immersed in a physiological solution for four and six days. With increasing YSZ content, the entire surface of the composite becomes coated with a progressively denser apatite layer. This revealed that YSZ induced the nucleation of apatite crystals during immersion. A greater amount of apatite deposition was identified at 4 wt.% YSZ.

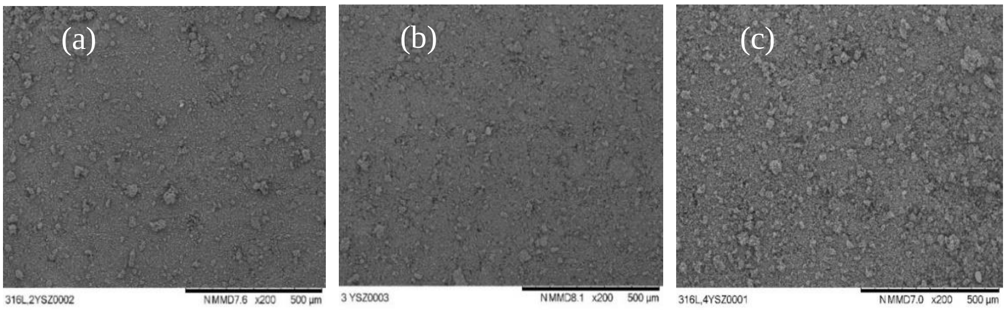

Fig. 4 : SEM micrographs of SS316L/YSZ with different contents of YSZ : (a) 2 wt.%, (b) 3 wt.% and, (c) 4 wt.% after immersion in HBSS for four days (200x magnification)

3.5 Weight change

With the increase in YSZ content, all the composites demonstrated an increase in weight gain as shown in Fig. 5. The 316LSS/2YSZ composite exhibited a few apatite crystals weighing approximately 1.13 g, followed by 316LSS/3YSZ with 1.14 g, and the SS316L/4YSZ composite showed a substantial amount of apatite with 1.15 g. The highest weight gain was demonstrated by 4 wt.% YSZ (5.00%) while 2 wt.% YSZ exhibited the lowest weight gain (1.00%). This bone-like apatite formation ultimately promotes osseointegration occuring at the contact between the implant and bone tissues [7], [8]. The reason behind this phenomenon lies in the important role played by YSZ content in promoting the growth of an apatite layer on the composite's surface after exposure to HBSS. In a study reported by M.R.N. Liyana et al. [9], they investigated the bioactivity of YSZ-Al_2O_3/10HAP with 30 wt. % and 60 wt. % of YSZ content. They observed the apatite formation on the surfaces of the composite only after nine days of immersion in physiological solution. This indicates that in the present study, the growth of apatite on SS316L/YSZ composite may still contribute to its improved bioactivity even at low YSZ content.

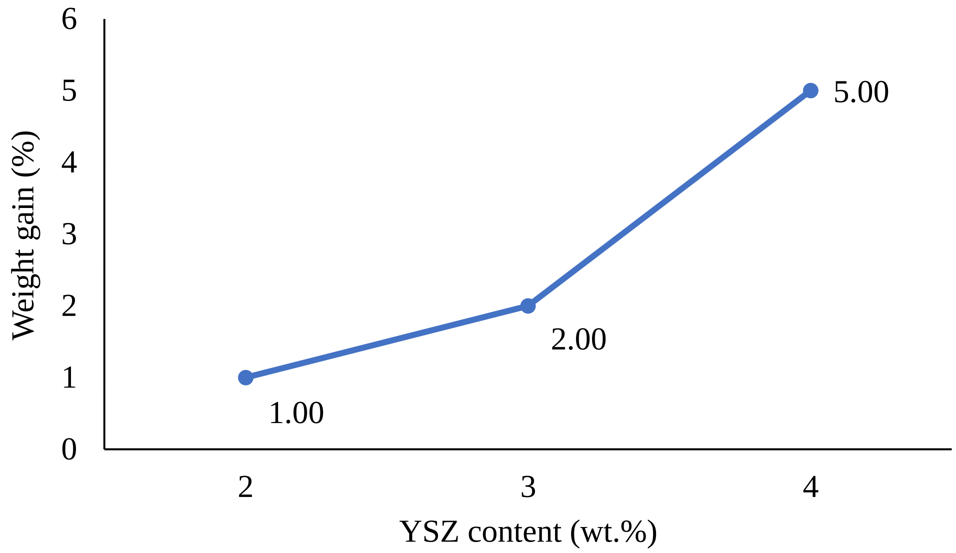

Fig. 5 : Measured weight gain percentage after immersion in HBSS for four days related to various YSZ content in SS316L/YSZ composite

4. Conclusion

i. The successful fabrication of SS316L/YSZ composite was achieved through a process involving mechanical alloying, powder metallurgy, compaction, and sintering.
ii. According to the results obtained, the composite containing 2 wt.% YSZ exhibited the most appropriate compressive strength (208.92 MPa) and Young's modulus (33.91 GPa), comparable to human cortical bone. Cortical bone has a compressive strength of 100 to 280 MPa, and Young's modulus ranging from 10 to 40 GPa. This suggests that SS316L/YSZ composite has the potential to serve as an artificial implant option.
iii. The immersion test in HBSS revealed abundant apatite formation on the surface of all composites, demonstrating good bioactivity of SS316L/YSZ composite.
iv.

Acknowledgement

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