

## **EFFECT OF COMPOSITION AND SINTERING TEMPERATURES ON THE PROPERTIES OF STAINLESS STEEL (SS316L) - HYDROXYAPATITE (HAP) FOAM**

Rajasakaran Yarshine Rani, Sufizar Ahmad\*, Umira Asyikin Yusop, Rizamarhaiza Muda, Hamimah Abdul Rahman, Azzura Ismail and Fazimah Mat Noor

Department of Manufacturing Engineering, Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia, 86400 Parit Raja, Batu Pahat, Malaysia

\*sufizar@uthm.edu.my

---

**Abstract.** SS316L stainless steel is one of the most commonly available commercial biomaterials due to its ease of biocompatibility and long-standing performance in the human body. In this study, SS316L-Hydroxyapatite (HAP) foam was fabricated using the slurry method with composition of 60 wt.% SS316L. The hydroxyapatite powder compositions were varied in the range of 1 wt.% to 10 wt.% while the binders' composition such as Polyethylene Glycol (PEG) and Carboxymethyl Cellulose (CMC) were fixed at 2.5 wt.%. The balance of the composition was distilled water which was used as a solvent. Polyurethane (PU) foam was used as a sacrificial template. The morphology analysis of the SS316L-HAP foam was carried out using Scanning Electron Microscope (SEM). The effects of HAP composition and sintering temperatures of 1150 °C and 1350 °C on the SS316L-HAP foam properties such as pore size, modulus elasticity and yield strength were investigated. The result of yield strength and modulus elasticity were obtained in the range of 3.01 MPa-18.85 MPa and 0.02 GPa-0.49 GPa, respectively. The pore size was found in the range of 100 µm to 386 µm while the cancellous bone yield strength and modulus elasticity are between 4.1 MPa-68 MPa and 0.02 GPa to 5 GPa, respectively. The SS316L-HAP foam produced has properties and microstructure almost similar with the cancellous bone. Hence, it has a great potential for biomaterial application.

**Keywords:** SS316L foam, hydroxyapatite, composition, sintering temperatures

---

### **Article Info**

Received 29<sup>th</sup> March 2022

Accepted 9<sup>th</sup> June 2022

Published 23<sup>rd</sup> December 2022

Copyright Malaysian Journal of Microscopy (2022). All rights reserved.

ISSN: 1823-7010, eISSN: 2600-7444

## Introduction

Stainless steel has become the primary material used to produce medical devices [1]. Notably, the use of stainless steel in orthopedic surgery opened and exposed a wide range of new opportunities in treating bone failures. The most commonly employed stainless steel alloy used for a bone implant is the SS316L grade due to its good corrosion resistance, lower cost, excellent fabrication properties, availability, and less expensive than cobalt-chromium alloys, pure Ti, and Ti alloys. Furthermore, it is resistant to a wide range of corrosive agents due to its high Cr content [2]. Until now, SS316L has been extensively used as a biomaterial to fabricate joint replacements, stents, orthopedic pins, fracture plates, screws and other implants [3].

Hydroxyapatite ( $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ ) (HAP) is a member of calcium phosphate ceramics. Tricalcium phosphates and bio glass ceramics are other members of this family and are commonly utilized as bone substitute materials. Due of its structural and functional closeness to the mineral composition found in bones and teeth, HAP is preferred over other calcium phosphates [4]. Solid scaffolds with varying porosities that may be made from HAP are excellent for cell adhesion, migration, and bone formation [5]. Furthermore, HAP has low mechanical qualities, such as low fracture toughness and brittleness, limiting its use in load-bearing applications [6].

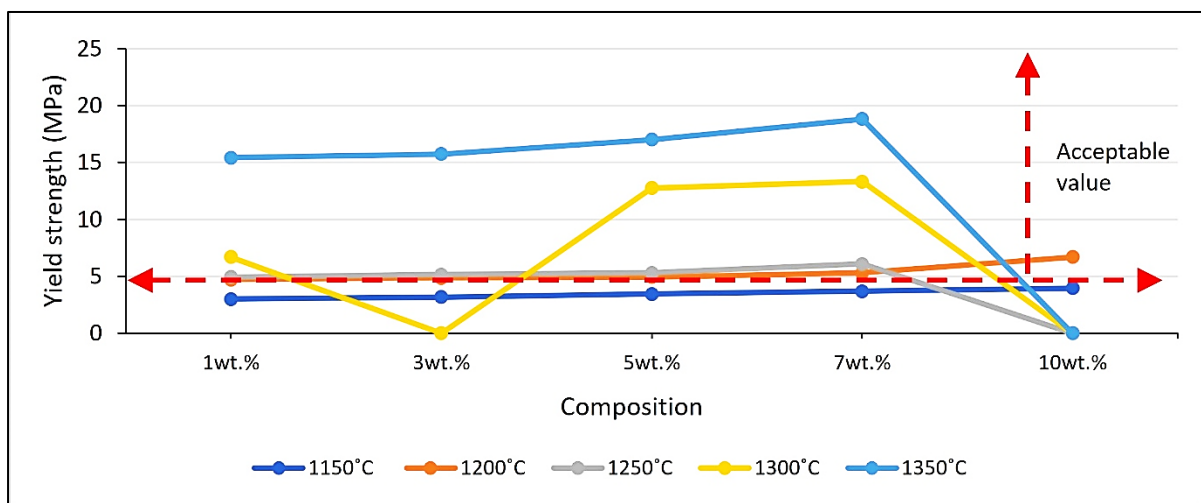
However, to broaden the application, the pure SS316L implant required improvement, and HAP is an option as coating materials for biomedical implants due to the possibility of simulating bone calcium phosphate formation on the implant surface, improving implant fixation and bone growth. As a result, tough metal-ceramic composites are made of metallic materials to improve mechanical properties [7]. The HAP and SS316L composites could provide a strong biomaterial for biomedical implant applications [8]. The metallic materials are employed to manufacture strong metal-ceramic composites with improved physical properties [6]. Stainless steel SS316L and HAP have been chosen as the main raw material in this study. The effect of HAP composition and sintering temperatures on the properties of the fabricated foams was investigated.

## Materials and Methods

SS316L-HAP foam was fabricated using slurry method [7]. The SS316L powder was mixed with HAP, PEG, CMC and distilled water to form a slurry. Polyurethane (PU) foam was used as a template. The PU foam was cut into a cylindrical shape with a height 27 mm and a diameter of 13 mm. Various amount of HA compositions (1 wt.%, 3 wt.%, 5 wt.%, 7 wt.%, and 10 wt.%) were used. The SS316L-HA slurry was mixed using a mechanical stirrer for 2 hours [9]. The PU foam was immersed in a slurry for 15 minutes and the excessed slurry was removed manually. The samples were dried in a drying oven for 24 hours at 40°C. Then, the samples were sintered at five different temperatures (1150 °C, 1200 °C, 1250 °C, 1300 °C and 1350 °C) in a vacuum furnace. The compression test was carried out using the universal testing machine to determine the yield strength and modulus elasticity in accordance with the international standard ISO13314:2011. SEM was used to perform microstructural analysis and pore size measurements on SS316L-HAP foams [10,11].

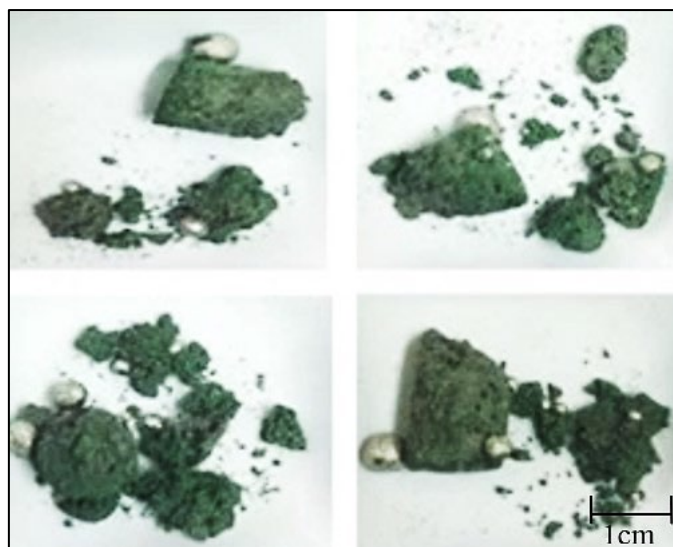
## Results and Discussion

Compression test was performed to identify the mechanical properties of SS316L-HAP foam. Figure 1 shows the average yield strength of SS316L-HAP foam increases with composition and sintering temperatures. The maximum value of yield strength was 18.85 MPa obtained from a sample containing 7 wt.% HAP and sintered at a temperature of 1300 °C, while the lowest yield strength was 3.01 MPa obtained from sample containing 1 wt.% of HAP and sintered at 1150 °C. Thus, the higher the sintering temperature, the higher yield strength attained. This is because, as the sintering temperature rises, heavy atoms migrate towards connective surfaces, causing sintering necks to grow and interparticle spacing reduced [10,11]. However, at the composition of 10 wt.% HAP, the yield strength reduced due to high slurry viscosity. It may be difficult for high viscosity slurry to penetrate the deepest part of the PU foams.



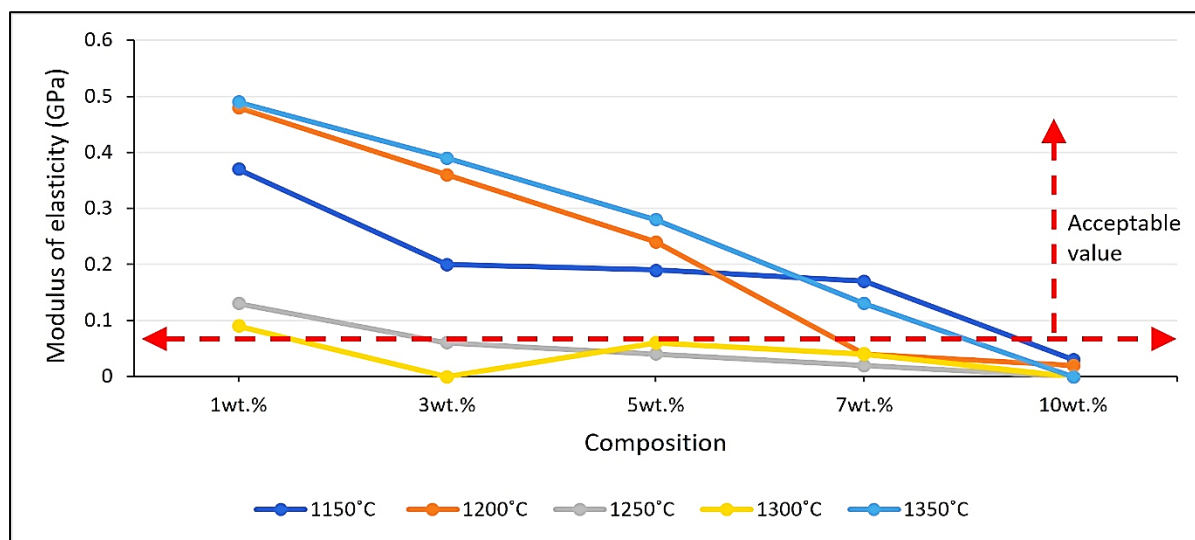
**Figure1:** Average yield strength of SS316L-HAP foam sintered at 1150 °C, 1200 °C, 1250 °C, 1300 °C and 1350 °C

Furthermore, inconsistent trend was observed for samples with 10 wt.% HAP. The sample collapse during and after the sintering process because not fully sintered (Figure 2). The sample size and shape also did not fulfill the international standard for mechanical testing. The colour of sample also changes to green colour after the sintering process. It showed that the sample was oxidized and not suitable for other testing [3]. In addition, the sample sintered at 1300 °C containing 3 wt.% HAP did not pass further mechanical testing (yield strength and modulus elasticity). Other than that, this situation also can be related to the increasing of relative density and decreasing of sample porosity that will contributed to the increasing of yield strength value [12]. According to [13], the yield strength value range that can be accepted for cancellous bone is around 4.1 MPa to 68 MPa.



**Figure 2:** SS316L-HAP foam after sintering at 1300 °C

Modulus elasticity was performed to identify the mechanical properties of SS316L-HAP foam. Three results of modulus elasticity were determined for each parameter throughout this study. Figure 3 shows the average modulus elasticity of SS316L-HAP foam sintered at 1150 °C, 1200 °C, 1250 °C, 1300 °C and 1350 °C. The maximum value of modulus elasticity was 0.49 GPa with 10 wt.% HAP, sintered at a temperature of 1150 °C, while the lowest modulus elasticity was 0.02 GPa with 3 wt.% and 5 wt.% of HAP and sintered at 1350 °C and 1300 °C.

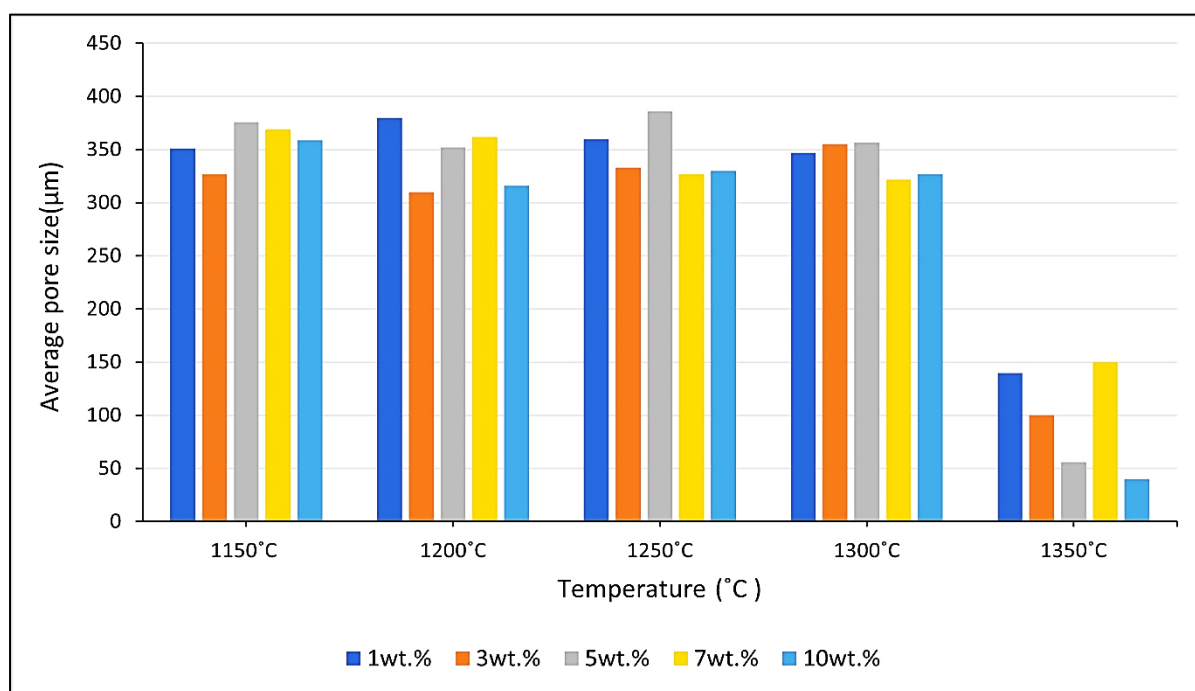


**Figure 3:** Modulus elasticity of SS316L-HAP foam sintered at 1150 °C, 1200 °C, 1250 °C, 1300 °C and 1350 °C

However, the result of modulus elasticity is not consistent with the sintering temperatures. In this present study, the modulus elasticity of SS316L-HAP foam was successfully performed in the range 0.02 GPa-0.49 GPa at sintering temperature from 1150 °C to 1350 °C. Furthermore, it is also known that various factors have affected the failure and structural defects of the SS316L-HAP foam. According to [14], the modulus elasticity could

be adjusted by changing the porosity level. The range can be accepted to cancellous bone of modulus elasticity is between 0.02 GPa to 5 GPa [15].

The pore size of SS316L-HAP foam was obtained from the image of SEM microstructure [16]. Through this method, all the samples were cut into cross sections surface using a wire cut machine. Figure 4 shows the average pore size for all SS316L-HAP foam samples and the sizes are all in the range of 40  $\mu\text{m}$  to 386  $\mu\text{m}$ . Pore size is critical for bone implant application, so implant materials should have appropriate pore sizes to allow cell infiltration and bone tissue growth into the pores. Aside from that, a strong interfacial bonding between the bone and metal implants also was critical [17]. Because the average pore sizes obtained by all the samples ranged from 200  $\mu\text{m}$  to 500  $\mu\text{m}$ , the new tissue would be unable to develop an efficient blood supply [18].



**Figure 4:** Average pore size of SS316L-HAP foam

As shown in Table 1, when the composition increases, the closed pore structure increases, and the open pores decreases. As the composition increases, the strut collapses because the slurry becomes viscous and difficult to penetrate the PU foam to fill the voids. From the SEM micrograph, the foam containing 1 wt.% to 5 wt.% were suitable composition at all the sintering temperatures. Previous research [9,19] supported this by stating that increased solid loadings produced a thicker and more viscous slurry while also producing more closed pores. Increased closed pores, especially at the highest composition, could be caused by an excess of slurry that is not completely eliminated from the PU template due to liquid films spanning the pore or strut cells [2]. The viscosity of the slurries is related to the difference in the size of the pores for each sample with the variable of composition which the result shows the high composition was viscous and the slurry cannot be impregnation into the PU foam and the pore size large and brittle thus the pore interconnectivity becomes poor. As a result, these findings are consistent with previous research [20], which discovered that slurries play an important role in establishing proper foam structure during the impregnation process. However, based on the morphology study, all the sintering temperatures (1150 °C to

1350 °C) are suitable for producing SS316L-HAP foam. Varying amounts of composition influenced the size of the pores. However, at 7 wt.% to 10 wt.% HAP, the foam was brittle due to high viscosity, and this result was supported by other researchers [20].



**Table 1:** SEM micrograph of SS316L-HAP foams using 1 wt.%, 3 wt.%, 5 wt.%, 7 wt.% and 10 wt.% of HPA composition at temperature of 1150 °C – 1350 °C

Temperature/ HPA	1wt%	3wt%	5wt%	7wt%	10wt%
1150°C					
1200°C					
1250°C					
1300°C					
1350°C					

## Conclusions

The SS316L-HAP foams with different compositions of HAP and sintering temperatures of 1150 °C, 1200 °C, 1250 °C, 1300 °C, and 1350 °C was successfully produced. The results revealed that the composition of HAP affects the modulus elasticity and yield strength. Thus, it is clear that modulus elasticity and yield strength are interrelated. In addition, the modulus elasticity of SS316L-HAP foam increased and yield strength decreases as composition increases. As the sintering temperature increases, the pores size decreases thereby leading to an increase in yield strength.

## Acknowledgements

This research was made possible by funding from research grant number GPPS-H583 and Grant MDR-H501 provided by the Universiti Tun Hussein Onn Malaysia for its support.

## Author Contributions

All authors contributed toward data analysis, drafting and critically revising the paper and agree to be accountable for all aspects of the work.

## Disclosure of Conflict of Interest

The authors have no disclosures to declare

## Compliance with Ethical Standards

The work is compliant with ethical standards

## References

- [1] Zhu, W., Su, Z., Guo, J., Li, K., Chen, K., Li, W., Yi, A., Liao, Z., Luo, Y., Hu, Y., Xu, Y., Lin, Q. & Meng, X. (2022). Preparation and characterization of Diamond-Like Carbon (DLC) film on 316L stainless steel by Microwave Plasma Chemical Vapor Deposition (MPCVD). *Diamond and Related Material*, 122, 108820.
- [2] Godbole, N., Yadav, S., Ramachandran, M. & Belemkar, S. (2016). A review on surface treatment of stainless-steel orthopedic implants. *International Journal of Pharmaceutical Sciences Review and Research*, 36(1), 190-194.
- [3] Noor, F. B. M. (2018). Foam replicated porous 316L stainless steel based on Taguchi method for biomedical applications (D. Eng. Thesis, Universiti Teknologi Malaysia) pp. 22-44.



- [4] Dutta, S. R., Passi, D., Singh, P. & Bhuibhar, A. (2015). Ceramic and non-ceramic hydroxyapatite as a bone graft material: A brief review. *Irish Journal of Medical Science (1971)*, 184(1), 101-106.
- [5] Ahmad, S., Muhamad, N., Muchtar, A., Sahari, J., Jamaludin, K. R., Ibrahim, M. H. I., Nor, N. H. M. & Murtadhahadi. (2010). Pencirian titanium berbusa yang dihasilkan pada suhu pensinteran yang berbeza menggunakan kaedah buburan. *Sains Malaysiana*, 39(1), 77-82.
- [6] Ahmad, S., Muhamad, N., Muchtar, A., Sahari, J., Jamaludin, K. R., Ibrahim, M. H. I. & Nor, N. H. M. (2011). Optimisation of sintering factors of titanium foams using Taguchi method, *International Journal of Integrated Engineering*, 2(1), 1–6.
- [7] Noor, F. M., Jamaludin, K. R. & Ahmad, S. (2017). Physical and mechanical characteristics of porous SS316L for biomedical implant. *Solid State Phenomena*, 268, 374-378.
- [8] Rafter, M. F. M., Ahmad, S., Ibrahim, R., Hussin, R. & Taib, H. (2015). Development of stainless steel (SS316L) foam with different composition using compaction method. *Advanced Materials Research*, 1087, 86-90.
- [9] Noor, F. M., Jamaludin, K. R. & Ahmad, S. (2017). Fabrication of porous stainless steel 316L for biomedical applications. *MATEC Web of Conferences*, 135, 7.
- [10] Tan, R., Yoo, J. & Jang, Y. (2020). Engineering approaches to create antibacterial surfaces on biomedical implants and devices. In *Racing for The Surface: Pathogenesis of Implant Infection and Advanced Antimicrobial Strategies*. Li, B., Moriarty, T. F., Webster, T. & Xing, M. (Springer Cham. Switzerland), pp. 313-340.
- [11] Baino, F. & Verne, E. (2017). Glass-based coatings on biomedical implants: A state-of-the-art review. *Biomedical Glasses*, 3(1), 1-17.
- [12] Balasubramanian, R., Nagumothu, R., Parfenov, E. & Valiev, R. (2021). Development of nanostructured titanium implants for biomedical implants – A short review. *Materials Today: Proceedings*, 46(2), 1195-1200.
- [13] Vignesh, M., Kumar, G. R., Sathishkumar, M., Manikandan, M., Rajyalakshmi, G., Ramanujam, R. & Arivazhagan, N. (2021). Development of biomedical implants through additive manufacturing: A review. *Journal of Materials Engineering and Performance*, 30, 4735-4744.
- [14] Branquinho, M. V., Ferreira, S. O., Alvites, R. D., Magueta, A. F., Ivanov, M., Sousa, A. C. Amorim, I., Faria, F., Fernandes, M. H. V., Vilarinho, P. M. & Mauricio, A. C. (2021). In vitro and in vivo characterization of PLLA-316L stainless steel electromechanical devices for bone tissue engineering- A preliminary study. *International Journal of Molecular Sciences*, 22(14), 7655.
- [15] Xie, F., He, X., Cao, S. & Qu, X. (2013). Structural and mechanical characteristics of porous 316L stainless steel fabricated by indirect selective laser sintering. *Journal of Materials Processing Technology*, 213(6), 838-843.

- [16] Yang, D., Zhang, Y., Song, X., Chen, Y., Shen, Z. & Yang, C. (2016). Effects of sintering temperature and holding time on porosity and shrinkage of glass tubes. *Ceramics International*, 42(5), 5906-5910.
- [17] Vivanco, J., Aiyangar, A., Araneda, A. & Ploeg, H. L. (2012). Mechanical characterization of injection-molded macro porous bioceramic bone scaffolds. *Journal of the Mechanical Behavior of Biomedical Materials*, 9, 137-152.
- [18] Purnama, A., Hermawan, H., Couet, J. & Mantovani, D. (2010). Assessing the biocompatibility of degradable metallic materials: state-of-the-art and focus on the potential of genetic regulation. *Acta Biomaterialia*, 6(5), 1800-1807.
- [19] Muda, R., Azmi, M. A., Mahzan, S., Elwalwal, H. M., Ahmad, S. & Taib, H. (2018). Effect of SiO<sub>2</sub> solid loading and sintering temperatures on the physical properties of SiO<sub>2</sub>-NiO foam. *Key Engineering Materials*, 791, 37-44.
- [20] Nazaruddin, N. A., Rahim, P. S. A., Muda, R., Kamal, W. M. H. A. A., Azmi, M. A., Taib, H. & Ahmad, S. (2017). The effect of different binder compositions in fabricating silica foam (SiO<sub>2</sub>) via replication method. *Journal of Mechanical Engineering*, 4(5), 53-62.