RESEARCH PROGRESS IN MECHANICAL AND MANUFACTURING ENGINEERING
e-ISSN: 2773-4765

RPMME

Vol. 5 No. 1 (2024) 602-609
https://publisher.uthm.edu.my/periodicals/index.php/rpmme

Fabrication of Alumina-Zirconia Foam Using Foam Replication Method

Muhammad Akif Azi Rahimee[1], Fazimah Mat Noor[1*]

[1] *Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia, 86400, Parit Raja, Johor, MALAYSIA*

*Corresponding Author: fazimah@uthm.edu.my
DOI: https://doi.org/10.30880/rpmme.2024.05.01.070

Article Info

Received: 15 January 2024
Accepted: 15 July 2024
Available online: 15 Sept. 2024

Keywords

Alumina, Zirconia, foam replication method, kaolinite, composite foam, open pores, interconnected pores, compositions, microstructure

Abstract

Phase-stabilized zirconia (ZrO_2) and aluminium oxide (Al_2O_3) are popular structural materials due to their high wear resistance, biocompatibility, and corrosion resistance. This thesis explores the fabrication of Alumina Zirconia foam through the foam replication method. The study investigates the effects of different compositions of alumina (Al_2O_3), zirconia (ZrO_2), and kaolinite on the properties of alumina-zirconia composite foam. The objectives of this research are to fabricate alumina-zirconia ($Al2O_3+ZrO_2$) composite foam with open and interconnected pores, investigate the physical and mechanical properties of $Al_2O_3+ZrO_2$ composite foam with different compositions of Al_2O_3, ZrO_2, and kaolinite, and observe the microstructure of $Al_2O_3+ZrO_2$ composite foam with different compositions of Al2O3, ZrO_2, and kaolinite. Five different compositions (C1 to C5) were prepared and analysed. The methodology involves a series of steps, including slurry mixing, PU foam dipping and squeezing, drying, sintering, and testing, including microstructure analysis, density and porosity test, compression tests, and followed by data analysis. Microstructure analysis was done to analyse the size and shape of pores, struts, and pore distribution. The results showed that composition C2 with 22.5wt% alumina powder, 22.5wt% kaolin powder, 5 wt% zirconia powder, 2.5 wt% PEG powder, 2.5 wt% CMC powder, and 45 wt% distilled water had a strong structure with minimal shrinkage but showed significant compression strength compared to other compositions.

1. Introduction

Ceramics, polymers, metals, and composites are four different classifications of biomaterials. Ceramics are less dense and more resistant to corrosive conditions, making them useful in various applications. Phase-stabilized zirconia (ZrO_2) and aluminum oxide (Al_2O_3) are commonly used as structural materials due to their high wear resistance, good biocompatibility, and excellent corrosion resistance [1]. However, they often experience unexpected failures due to slow fracture expansion. This study aims to improve the properties of alumina- zirconia composite foam by adding zirconia. The objectives include fabricating alumina-zirconia composite foam with open pores, investigating the physical and mechanical properties of $Al_2O_3+ZrO_2$ composite foam with different compositions, and observing the microstructure of $Al_2O_3+ZrO_2$ composite foam.

2. Literature Review

The literature review discusses various materials, including porous ceramics, alumina, zirconia, and foam replication. Porous ceramics have high permeability and surface area, making them ideal for various applications [2]. Open pores connect to the outside of the material, while closed pores connect to the inside and may contain fluid [3]. Penetrating pores are an example of open pores. Alumina ceramics are of interest due to their compressive strength, low density, high hardness, Young's Modulus, excellent high-temperature oxidation resistance, and good thermal stability [4]. However, they have high intrinsic brittleness, which limits their application and development [5]. A-phase Al2O3 has been extensively used in engineering disciplines due to its exceptional phase stability, high hardness, electric characteristics, and thermal properties [6]. Zirconia, also known as (ZrO2), is a polymorphic compound with three different shapes depending on temperature [7]. It is well-known for its mechanical, thermal, optical, and dielectric properties, making it one of the most frequently used dental restorative materials [8]. Glazing techniques have the potential to improve bonding between non- etchable ZrO2 ceramics and resin cement and tooth substrates [9]. Kaolin, a Chinese term meaning "high ridge," is used in various applications such as fire bricks, coated paper, and ceramics [10]. The foam replication method, created by Schwartzwalder et al., involves creating ceramic foams with high porosity by adjusting the slurry's viscosity and polymeric foam parameters [11]. The density of the ceramic slurry has a major effect on the properties of ceramic foam [12].

3. Methodology

The process starts with the preparation of raw materials, including Alumina Powder, Zirconia Powder, Kaolin Powder, Polyethylene Glycol (PEG), Carboxy Methyl Cellulose (CMC) Powder and Distilled Water. These materials are mixed using a mechanical stirrer to form a slurry. The slurry undergoes PU Dipping and Squeezing before being dried in an oven. After drying, it is subjected to a sintering process at 1400°C to form Alumina Zirconia Foam (Al_2O_3-ZrO_2). The success of the fabrication is evaluated through characterizations including microstructure analysis, density tests and porosity tests, shrinkage tests, and compression tests. Data from these tests are analyzed to conclude the effectiveness and quality of the fabricated alumina zirconia foam. The composition of alumina has been set as:

Table 1 *Composition of Al2O3-ZrO2*

Material / Composition	PEG (wt%)	CMC (wt%)	Al_2O_3 (wt%)	H_2O (wt%)	ZrO_2 (wt%)	Kaolin (wt%)
C1	2.5	2.5	25	45	0	25
C2	2.5	2.5	22.5	45	5	22.5
C3	2.5	2.5	20	45	10	20
C4	2.5	2.5	17.5	45	15	17.5
C5	2.5	2.5	15	45	20	15

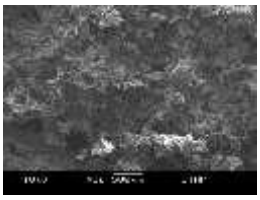
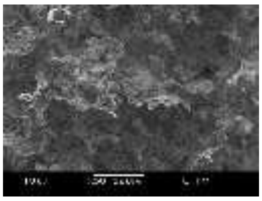
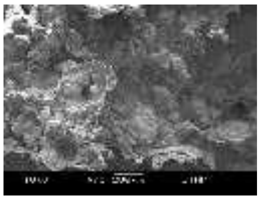
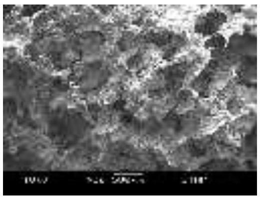
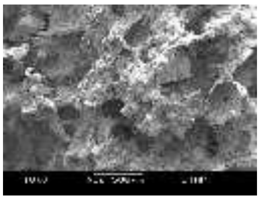
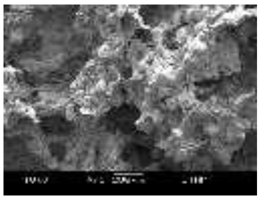
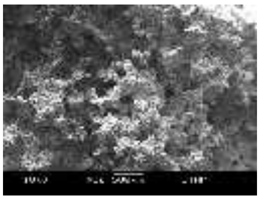
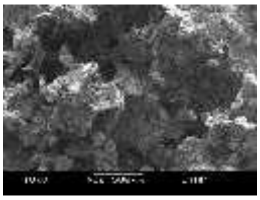
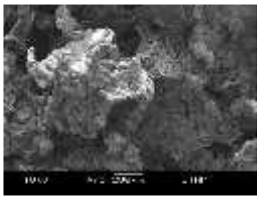
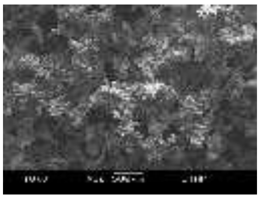
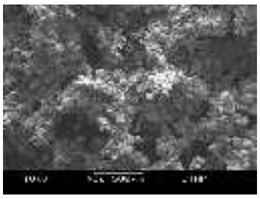
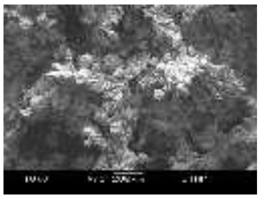
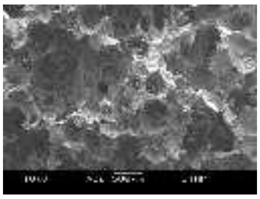
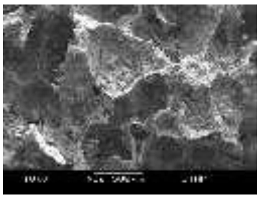
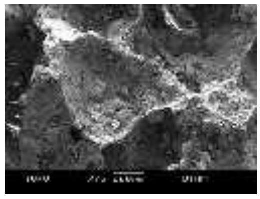
Microstructure analysis is used to observe the types of pores and the structure inside of the Alumina-Zirconia foam produced. The density and porosity of the foam are characterized using the Archimedes Method with ASTM B962-15 standard. Shrinkage analysis is done to determine the foam's shrinkage before and after the sintering process. Compression test is used to determine the compressive strength of the foam. The higher the Alumina composition in the slurry, the higher the shrinkage percentage found in the Alumina-Zirconia foam. The suitable solvent used for the Alumina slurry is distilled water. The higher the Alumina composition in the slurry, the lower the density of Alumina-Zirconia foam, but the higher the porosity on Alumina-Zirconia foam for both solvents.

4. Result and Discussion

4.1 Microstructure Analysis

The microstructure analysis of five compositions and three different zoom levels of SEM showed that the microstructure of ceramic foam is characterized by its size, shape, distribution, and connectivity of pores. Composition C1 has the highest density and lowest shrinkage, while composition C3 has the highest porosity and shrinkage but the lowest density and compression strength. The absence of ZrO_2 might contribute to a less intricate structure, potentially affecting its mechanical properties. This is consistent with its low shrinkage rate as a denser structure is less prone to shrinking. C2 SEM images show a balanced microstructure with moderate pore size and distribution, leading to higher mechanical strength while maintaining some level of porosity. C2 exhibits a more complex and denser microstructure due to the introduction of 5wt% ZrO_2. This could explain the highest maximum force observed in compression tests, as the combination of Al_2O_3, kaolin, and ZrO_2 results in enhanced structural integrity. Composition C2 has the lowest porosity but the highest compression strength, with a balanced microstructure with moderate pore size and distribution. Compositions C4 and C5 have intermediate properties, with C4 having 15wt% ZrO2 and C5 having the highest ZrO2 content. SEM images provide insights into the pore size, distribution, wall thickness, and overall morphology of each composition's microstructure, allowing correlation between mechanical properties and microstructural characteristics. C4 has 15wt% ZrO_2 and shows an increase in structural complexity but not enough to surpass C2's mechanical strength. The interconnected struts are more pronounced but may not be optimally balanced with pore size.

Table 2 *SEM Figure*

Composition	Microstructure analysis (SEM Zoom)		
	x30	x50	x75
C1			
C2			
C3			
C4			
C5			

4.2 Density and Porosity Test

The average porosity percentage of the five compositions is shown in the Fig. 1.

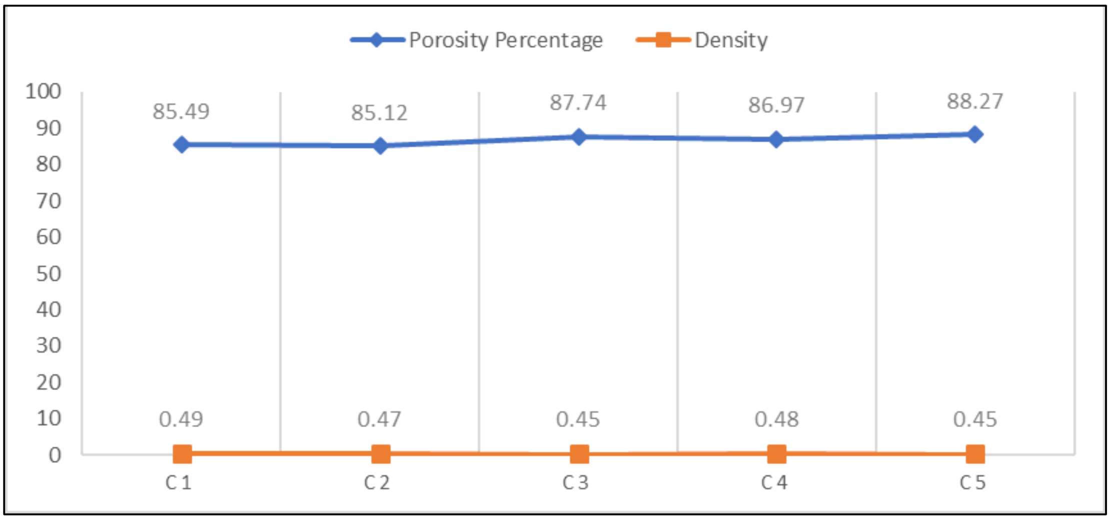


Fig. 1 *Porosity and density*

The porosity percentage of C5 is the highest, at around 88%, while the porosity percentage of C2 is the lowest, at around 85%. C1, C3, and C4 porosity percentages are between 80% and 86%. The high porosity percentage of C5 is due to the absence of ZrO_2, which is known to reduce porosity. The porosity percentage decreases as the weight percentage of ZrO_2 increases, which is consistent with previous studies.

The average density of three samples for each composition is shown in Fig. 1. The density of C3 and C5 are the lowest, at 0.45 g/cm^3, while the density of C1 is the highest, at around 0.49 g/cm^3. The density of C2 and C4 are between 0.47 g/cm^3 and 0.48 g/cm^3. The low density of C3 is due to the high porosity percentage, which is consistent with the results of previous studies. The density increases as the weight percentage of ZrO_2 increases, which is also consistent with previous studies.

4.3 Shrinkage Test

In general, Al_2O_3+ZrO_2 is sintered at temperatures ranging from 1200 to 1600 degrees Celsius. A phase shift occurs in ZrO_2 when its temperature rises over 1170 °C. Therefore, the temperature of the sintering process needs to be above 1350 ° C to have a suitable amount of sintering for the produced sample. This is based on the degree of success of the ZrO_2 interactions throughout the sintering process. The shrinkage test results for each composition were determined by measuring the dimensions of each sample and taking the average value for each composition. The comparison average values for the shrinkage test are different because of the composition of the mixtures, pore size, cutting method for the foam in the procedure before the sintering, and some human and apparatus errors.

The passage discusses the intricate details of the sintering process for a material composed of aluminium oxide (Al_2O_3) and zirconium oxide (ZrO_2). The sintering temperature range specified for Al_2O_3+ZrO_2 spans from 1200 to 1600 degrees Celsius. Notably, a crucial phase shift in ZrO2 occurs when its temperature surpasses 1170 °C, necessitating a sintering temperature exceeding 1350 °C for a successful outcome in the produced samples. The focus extends to the evaluation of dimensional changes through shrinkage tests. These tests involve measuring the dimensions of each sample and calculating the average value for compositions labelled C1 to C5. The comparison of average shrinkage values unveils discrepancies attributed to factors such as the mixtures' composition, pore size variations, the cutting method employed for the foam before sintering, and the potential influence of human and apparatus errors. The intricacies of these factors are acknowledged as contributors to the observed differences in shrinkage test results, emphasizing the complexity and precision required in the sintering process. Fig. 2 visually represents the average shrinkage values, providing a concise summary of the variations across different compositions and shedding light on the impact of the experimental considerations. Fig. 2 shows the average of shrinkage test for C1 to C5:

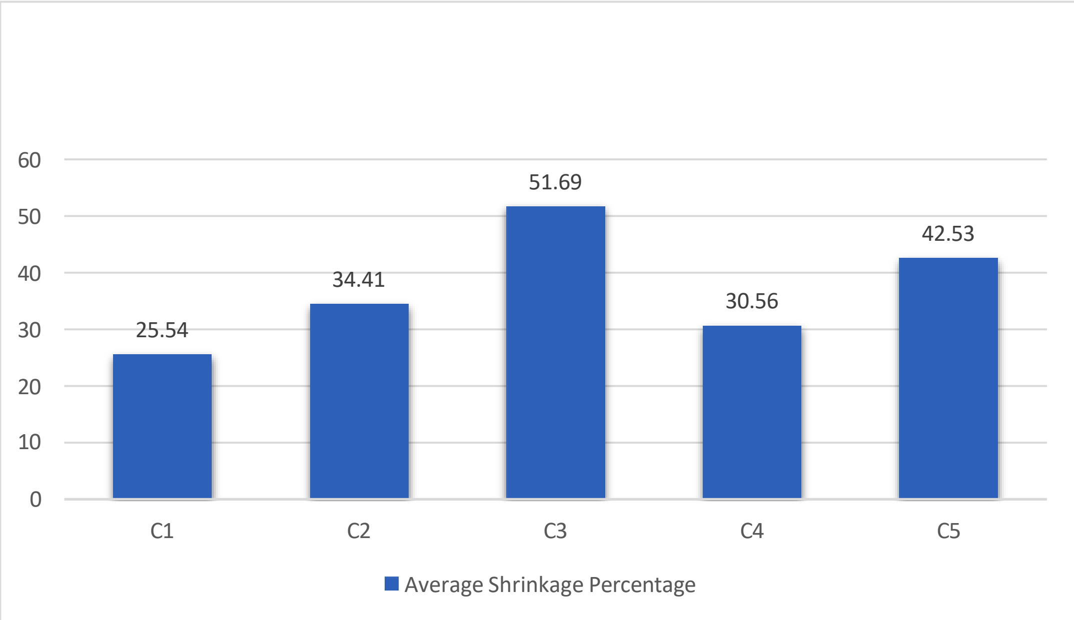


Fig. 2 *Shrinkage test*

The composition affects not only the shrinkage but also other properties like density and porosity. The increase in ZrO_2 content from C1 to C5 correlates with a decrease in density, indicating that ZrO_2 might contribute to porosity. The highest porosity in C5 can be associated with its high ZrO_2 content of 20wt.%, leading to an expanded structure during sintering. In summary, the variations in shrinkage are influenced by the balance between Al2O3, kaolin, and particularly ZrO_2 content. Higher amounts of ZrO_2 seem to reduce density while increasing porosity and brittleness [13].

The varying alumina, kaolin, and zirconia compositions in each sample can be attributed to the shrinkage results. C1 has equal parts of alumina and kaolin but no zirconia, resulting in moderate shrinkage. C2 slightly increases zirconia 5wt.%, which could contribute to increased binding and reduced shrinkage. C3 has higher zirconia content 10wt.% but shows significant shrinkage due to its brittleness as indicated by the low compression test result. Besides, C4 and C5, with even higher zirconia content, show reduced shrinkage compared to C3 because the increased amount of zirconia might be improving the structural integrity despite increasing brittleness.

4.4 Compression Test

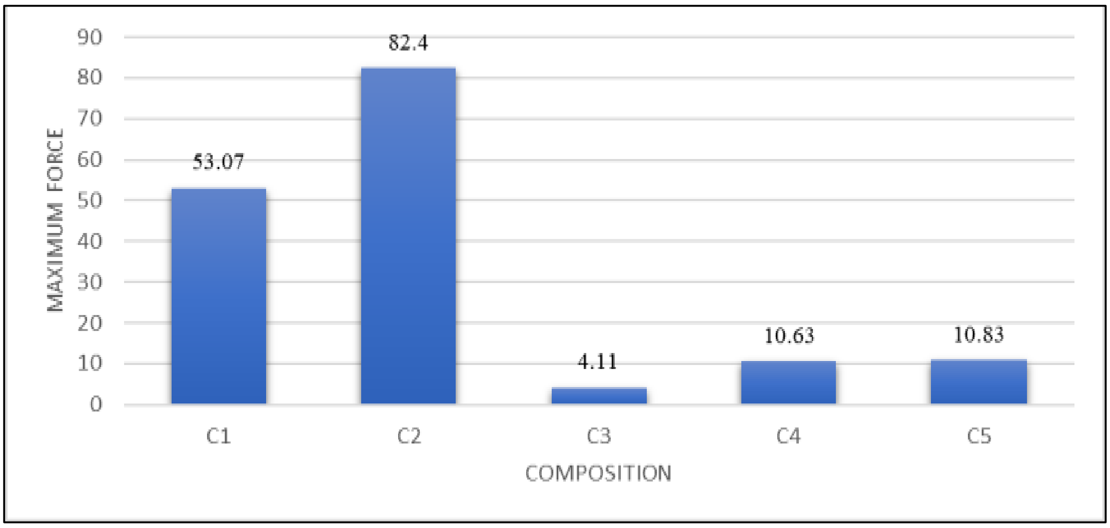


Fig. 3 *Compression test*

The compression test results show that the maximum force required to compress foam increases with the weight percentage of ZrO_2. Composition C1, which lacks ZrO_2, has the lowest force at around 53.07N. Composition C2, which contains 5wt% ZrO_2, has a significant increase at around 82.40N. Compositions C3, C4, and C5, which contain 10wt.%, 15wt.%, and 20wt.% ZrO_2, have maximum forces between 4.11N and 10.83N. The result can be justified as C1 and C2 samples exhibited higher maximum forces due to their higher densities resulting from lower ZrO_2 content. C3 and C5 could be, despite having similar densities, there's a significant difference in their compression strengths due to variations in their compositions affecting structural integrity. C4 serves as a middle ground with moderate ZrO_2 content leading to intermediate mechanical strength [14].

Fig. 3 was based on the average value for the three tests on each sample of composition that has been set. From the graph, the lowest strength for the compression test is C3, with a value of 4.11N, which is average, while the highest strength is C2, with a value of 82.40. The compression test results showed that the value of the maximum force of C1 is 53.07N, C4 is 10.63N, and C5 is 10.83N. The composition of C2 had the highest value of maximum force, while the lowest value was observed for C3. The results show that the maximum force required to compress the foam increases as the weight percentage of ZrO_2 increases. Composition C1, which contains no ZrO_2, exhibits the lowest maximum force required to compress the foam at around 53.07N. Composition C2, which contains 5wt% ZrO_2, significantly increases the maximum force required to compress the foam at around 82.40N. Compositions C3, C4, and C5, which contain 10wt.%, 15wt.%, and 20wt.% ZrO_2, respectively, exhibit maximum forces required to compress the foam between 4.11N and 10.83N. The results suggest that the introduction of ZrO_2 enhances the compressive strength of the foam, but there appears to be an optimal composition beyond which additional ZrO_2 does not translate into increased strength - rather, it may lead to decreased performance due possibly to structural or molecular imbalances.

5. Conclusion

In conclusion, the study successfully conducted tests and methods to manufacture Al_2O_3+ZrO_2 composite foam using foam replication. The results showed distinct characteristics for each composition, with a balanced combination of Al_2O_3, kaolin, and ZrO_2 achieving optimal mechanical properties. Pore size, strut thickness, and interconnectedness are crucial factors in determining these properties. Composition C2, which contains alumina powder, is 22.5 wt.%, kaolin powder is 22.5 wt.%, and zirconia powder is 5 wt.%, PEG powder 2.5 wt.%, CMC powder 2.5 wt.%, distilled water 45 wt.% had a strong structure with minimal shrinkage but showed significant fragility compared to other compositions. The porosity percentage and density of the foam were affected by the weight percentage of Al_2O_3, kaolin, ZrO_2, CMC, PEG, and distilled water. The introduction of ZrO_2 known as its enhanced the foam's compressive strength, but there was an optimal composition beyond which additional ZrO_2 did not translate into increased strength. Comparing the other sample to other compositions, however, reveals a significant degree of fragility even at its lowest point for composition C3 contained alumina powder are 20 wt.%, kaolin powder are 20 wt.%, zirconia powder 10 wt.%, PEG powder 2.5 wt.%, CMC powder 2.5 wt.%, distilled water 45 wt.%.

Acknowledgement

The authors would like to thank the Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia for the support in conducting the research.

Conflict of Interest

The authors declare that there is no conflict of interest regarding the publication of the paper.

References

[1] Boniecki, M., Sadowski, T., Gołębiewski, P., Węglarz, H., Piątkowska, A., Romaniec, M., Krzyżak, K., & Łosiewicz, K. (2020). Mechanical properties of alumina/zirconia composites. Ceramics International, 46(1), 1033–1039. https://doi.org/10.1016/j.ceramint.2019.09.068

[2] Lee, S., Lee, C. Y., Ha, J. H., Lee, J., Song, I. H., & Kwon, S. H. (2021). Enhancing compressive strength of reticulated porous alumina by optimizing processing conditions. Applied Sciences (Switzerland), 11(10). https://doi.org/10.3390/app11104517

[3] Oh, S. T., Chang, S. Y., & Suk, M. J. (2012). Microstructure of porous Cu fabricated by freeze-drying process of CuO/camphene slurry. Transactions of Nonferrous Metals Society of China (English Edition), 22(SUPPL.3). https://doi.org/10.1016/S1003-6326(12)61787-7

[4] Liu, D., Zhang, G., Gui, K., Han, W., Niu, J., & Wang, M. (2023). Fabrication of strong and tough

alumina ceramic with isotropic textured microstructure at low temperature. Journal of Materials Research and Technology, 24, 5159-5164, https://doi.org/10.1016/j.jmrt.2023.04.122

[5] Yang, S., Fu, X., Han, N., Wei, P., Fan, L., Dai, Y., & An, L. (2023). Strengthened the dense alumina ceramics by flash treatment. *Ceramics International*. https://doi.org/10.1016/j.ceramint.2023.03.161

[6] Ishizaki, K., Komarneni, S., & Nanko, M. (1998). Porous Materials (Vol. 4). Springer US. https://doi.org/10.1007/978-1-4615-5811-8

[7] Treccani, L., Yvonne Klein, T., Meder, F., Pardun, K., & Rezwan, K. (2013). Functionalized ceramics for biomedical, biotechnological and environmental applications. In *Acta Biomaterialia* (Vol. 9, Issue 7, pp. 7115–7150). Elsevier Ltd. https://doi.org/10.1016/j.actbio.2013.03.036

[8] Ambilkar, S. C., Kapgate, B. P., Das, A., Mandal, S., Maji, P. K., Singh, S., Kasilingam, R., Gedam, R. S., & Das, C. (2023). Precise role of zirconia to boost up the mechanical, thermal, viscoelastic, dielectric, and chemical resistance properties of natural rubber-nitrile rubber blend. European Polymer Journal, 194. https://doi.org/10.1016/j.eurpolymj.2023.112163

[9] Khalile, N., Petit, C., Meunier, C., & Valdivieso, F. (2023). Hybrid microwave sintering of Zirconia Toughened Alumina in a multimode cavity – Influence of the content of 3 mol% yttria-stabilized zirconia and sintering configurations. Open Ceramics, 14, 100371. https://doi.org/10.1016/j.oceram.2023.100371

[10] Prasad, M. S., Reid, K. J., & Murray, H. H. (1991). Kaolin: processing, properties and applications. In *Applied Clay Science* (Vol. 6).

[11] Muhamad Nor, M. A. A., Hong, L. C., Arifin Ahmad, Z., & Md Akil, H. (2008). Preparation and characterization of ceramic foam produced via polymeric foam replication method. Journal of Materials Processing Technology, 207(1–3), 235–239. https://doi.org/10.1016/j.jmatprotec.2007.12.099

[12] Sutygina, A., Betke, U., & Scheffler, M. (2020). Open-Cell Aluminum Foams by the Sponge Replication Technique: A Starting Powder Particle Study. Advanced Engineering Materials, 22(5). https://doi.org/10.1002/adem.201901194

[13] Afuza, A. A., Hadzley, A. B., Norfauzi, T., Umar, A. A., Faiz, M., Naim, F., & Thongkaew, K. (2020). Analysis of particles size distribution on the agglomeration and shrinkage of alumina-zirconia compacts. International Journal of Nanoelectronics and Materials, 13, 277-284.

[14] Sedyono, J., Rusli, M. R., Noor, F. M., & Ismail, A. (2021). Effects of Al2O3 composition on the physical properties of Al2O3 foam prepared by foam replication method. *International Journal of Engineering Trends and Technology*, *69*(1), 179–184. https://doi.org/10.14445/22315381/IJETT-V69I1P227