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Preparation and Microstructural Characterization of Five-layered Aluminium-Aluminium Oxide Functionally Graded Material

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Abstract. In this research, the metallic phase Aluminium (Al) and the ceramic phase Aluminium Oxide (Al_2O_3) were combined in different proportions for the preparation of five-layered Al- Al_2O_3 functionally graded material. Delamination or crack is the common problem at the metal-ceramic interface because of the large difference in material properties. Therefore, the main objective of this research was to investigate the defects within the layer during processing of the graded structure. The compositions of different weight percentages of five-layered structure are 100% Al+0% Al_2O_3 , 95% Al+5% Al_2O_3 , 90% Al+10% Al_2O_3 , 85% Al+15% Al_2O_3 and 80% Al+20% Al_2O_3 . During preparation of the graded composite structure, conventional two-step sintering cycle was followed. Heating rate $2^\circ\text{C}/\text{min}$, sintering temperature 630°C and sintering time 3 hours were maintained. It was observed that there was uniform distribution of Al_2O_3 ceramic particle in the Al matrix with the minimum of agglomeration, all interfaces were almost parallel with layers boundary and gradual transition occurred from first layer to fifth layer which confirmed the proper preparation processes of the five-layered graded composite structure. In addition, it was evident that there was no existence of crack within any layer or interface of the prepared composite structure.

Keywords: aluminium; aluminium oxide; functionally graded material; five-layered; characterization

1. Introduction

The Currently the development of Functionally Graded Material (FGM) is gaining popularity in various manufacturing sectors for diverse engineering applications such as aerospace, automotive and armor parts. To achieve the desired properties, metal and ceramic can be combined in different proportions for the preparation of FGM using different processing methods such as centrifugal casting, laser sintering, deposition, powder metallurgy, thermal spraying etc. The new generation FGM is a way to solve practical problems and this graded composite material is suitable for multiple functions with high performance / efficiency [1-4]. FGM can be prepared into two types of graded structure which are continuous structure and stepwise structure. For continuous graded structure, the composition and microstructure change continuously with the position while for stepwise structure, composition and microstructure vary in a stepwise manner, with distinguished interface between discrete layers. Powder metallurgy is the best method to prepare stepwise FGM in order to achieve the intended properties while



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continuous graded structure can be prepared by centrifugal force [5]. Processing the FGM by powder metallurgy mainly consists of powder mixing, powder stacking, powder compacting and sintering of green compact. Powder compaction and sintering are the most important steps during the process. On the other hand, there are some parameters that need to be considered to avoid cracks or camber in the sample because of residual stress that occur in sintering and thermal expansion between the successive layers [6]. At the initial stage, die compaction of powder involves particle arrangements which are influenced by the physical properties of powder such as particle size, particle shape and density of powder particle. The properties and the quality of the particles are the essential factors that control the compressibility behavior of the powder. Moreover, work hardening of the particles is influential factor that affects the hardness of the end product. The green density of compact has direct influence on the densification and porosity of the product and its strength [7].

Aluminium based composite materials are popular in the present era and these composites have the potential for their outstanding physical, mechanical and tribological properties [8]. On the other hand, aluminium oxide possesses high stiffness and hardness, high wear resistance, good strength and high chemical inertness [9]. Using laser melting deposition, composite materials of multi-layered graded structure were prepared and it was revealed that strong bonding between the deposited material and the substrate was formed [10]. Aluminium based graded structures were analyzed [11] and it was clarified that hardness of outer part was higher as compared to middle part or inner part. In addition, strength of outer part was higher compared to that of inner part. Aluminium based functionally graded composite panels were studied both theoretically and experimentally [12]. In this investigation, thermo-mechanical behavior / bending of the composite panel was analyzed under different loading conditions. The obtained results showed that theoretical predictions were in good agreement with the experimental results.

In this research, five-layered Al-Al₂O₃ graded composite specimen was prepared considering 0% to 20% Al₂O₃ concentration (first layer to fifth layer) with 5% increment for the successive layers. During sintering, two-step profile was followed and 3 hours sintering time was maintained. Microstructural investigations were carried out layer by layer and at the interface. In addition, hardness of each layer was examined in this study.

2. Experimental procedure

2.1. Sample preparation

High purity Al powders (HmBG, Germany) with 20-40 µm average size and Al₂O₃ powders (Sigma Aldrich, USA) with 40-60 µm average size were used for the preparation of the five-layered graded composite specimen. Based on the molecular weight and percentage composition of each type powder, the amount of each type powder was measured for each layer. Both powder materials were sieved and then mixed together depending on each layer composition. After that the mixed powders were blended allowing sufficient time in order to achieve good homogeneous mixture. In preparation of five-layered aluminium-aluminium oxide (Al-Al₂O₃) graded composite specimen, the metal and ceramic powders were mixed according to different compositions and the ratios of metal:ceramic are 100:0, 95:5, 90:10, 85:15 and 80:20 (first layer to fifth layer). The arrangement of five-layered FGM sample is shown in Figure 1. The different compositions of raw powders were stacked layer by layer in a cylindrical steel die of 30 mm diameter. After that, the uniaxial pressure was applied and increased slowly step by step until it reached 30 ton at room temperature using hydraulic press (TOYO: Model TL30, capacity 30 ton) to the stacked powders.

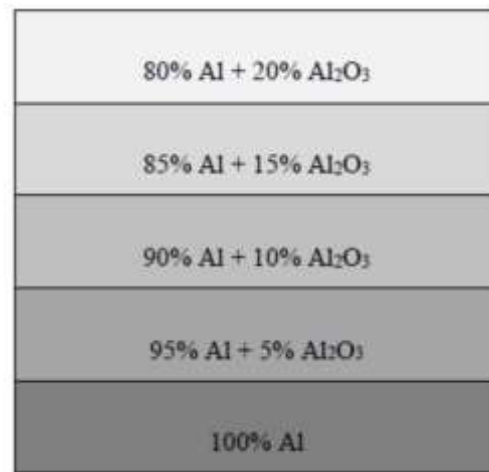


Figure 1. Arrangement of five-layered functionally graded Al-Al₂O₃

Then, the green compact sample was allowed for the sintering process in a pressureless sintering furnace (Nabertherm; made in Germany) using two-step sintering cycle as shown in Figure 2 [13][14]. In this research, 630°C sintering temperature was applied. Based on the Figure 2, during first step sintering cycle, the temperature increased from 30°C to 150°C with the heating rate 2°C/min. At 150°C, the holding time one hour was maintained and after that, the heating process was continued with same heating rate until the sintering temperature reached 630°C. In this second step of sintering cycle, the sintering time was maintained for three hours. Then, the specimen was allowed to cool in the furnace in order to reach room temperature.

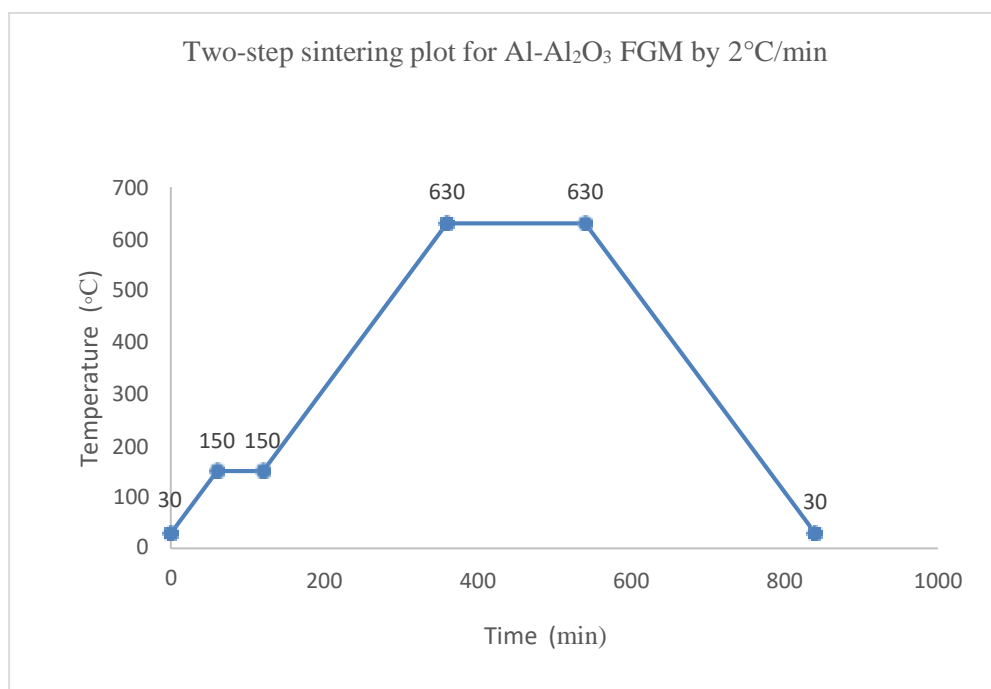


Figure 2. Two-step sintering cycle of Al-Al₂O₃

2.2. Sample characterisation

To measure the shrinkage of the sample, the diameter of each layer of the sample before and after sintering was measured by using vernier caliper [15]. The average of ten measurements was taken for each layer diameter. In order to examine the gradation of Al-Al₂O₃ FGM in details, the sample was cut using a diamond cutter in cross-section of sample and along the layer. After that, the sample was ground and polished along the cross-sectional direction. For the microscopic observation, the sample was mounted as shown in Figure 4. Then the sample was observed using field emission scanning electron microscopy (FESEM) (made in USA) in backscattered mode.

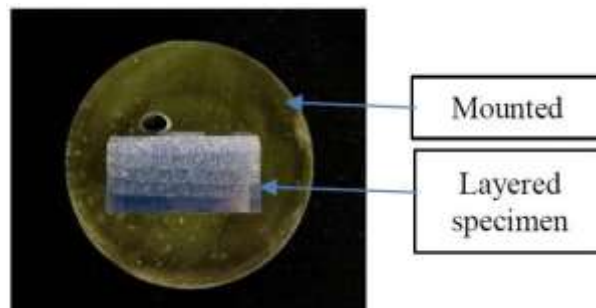


Figure 3. Sample Al-Al₂O₃ after mounted

2.3. Vickers microhardness measurement

The Vickers microhardness test was performed according to ASTM E384 Standard. The microhardness was measured using Vickers microhardness tester (Wilson Hardness: Model 402 MVD, made in USA). Hardness was measured on each layer of the sample along the longitudinal axis under the test load 300 gf (2.94 N) and a dwell time of 15 seconds for each layer. Ten measurements were carried out at a distance of about one mm interval avoiding any neighboring indentation effect. From the measured data values, the average hardness value was calculated for each layer of the five-layered specimen.

3. Result and discussion

3.1. Sintering effect

The linear shrinkage of specimen can be explained by the reduction in diameter of the green specimen after the sintering process. It was observed that due to shrinkage of the green specimen during sintering, the sintered sample was reduced in diameter. In green compact, the diameter of the Al-Al₂O₃ specimen was measured as 30 mm and it decreased to 29 mm after sintering as shown in Figure 4. After sintering process it was found that the weight of the sintered specimen was decreased as compared to the weight of green specimen. The weight loss occurred due to the evaporation during sintering process, where oxygen diffuses at high temperature. Based on the Table 1, the weight loss for the five-layered Al-Al₂O₃ graded specimen is 7.16%, and this weight loss resulted because of isothermal holding time for 3 hours as similar to previous work [16].

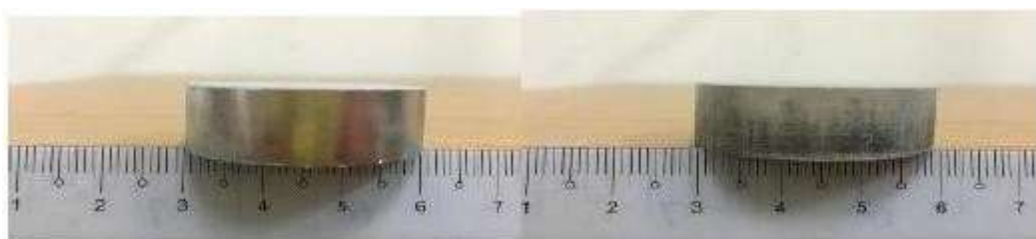


Figure 4. Diameter before and after sintering: 30mm - 29mm

Table 1. Weight loss% of sample Al-Al₂O₃

Specimen	Weight Before sintering	Weight After sintering	Weight loss %
Five-layered Al-Al ₂ O ₃ FGM	16.19g	15.03g	7.16

3.2. Microstructural observation of each layer and interface of adjacent layers

Figure 5 shows the FESEM image of each homogeneous layer in backscattered mode and the microstructural studies were performed along the polished cross-sectional surface of the five-layered graded composite material of Al-Al₂O₃. Different types of elements such as aluminium oxide (dark area), the aluminium matrix (light grey area) and the pores (black spots) were observed from second layer to fifth layer [17]. Based on the microstructure, it is understood that the ceramic particles were almost uniformly distributed in the matrix. The clear presence of pores was observed in the fourth and fifth layers containing 85%Al+15%Al₂O₃ and 80%Al+20%Al₂O₃ (Figure 5 (d) and Figure 5(e)). The pores are shown as tiny black spots. It is believed that because of increased ceramic addition, the apparent viscosity of metallic matrix increases which in turn increases the air entrapment in the layer, and this is the reason of increased porosity in the fourth and fifth layers [18]. It is clear that the ceramic content (dark area) exists in the second layer and increases layer by layer up to fifth layer and the ceramic particles were distributed almost uniformly in the aluminium matrix. From the microstructural observation, it is noticed that there is a gradual increment of ceramic content which confirms good mixing and preparation process of the five-layered graded composite specimen.

Figure 6 exhibits the FESEM images of interface of adjacent layers of five-layered sample. From the observation it is apparent that two adjacent layers are clearly separated by the interface (marked by the dotted line) which is distinct. During processing of the graded composite specimen in a stepwise way, it is evident that continuity of structure is maintained although the ceramic content increases to the next layer. Moreover, any macrocrack or microcrack was not identified / observed within any individual layer or at any interface. This indicates that after sintering process, good interfacial bonding between aluminium and aluminium oxide was established in the five-layered composite specimen. From the obtained results it is apparent that transition occurred step by step from the first layer to fifth layer. It was also noticed that each interface in between the layers was almost straight and parallel to the adjacent layers which proved that powder stacking process for the five-layered graded specimen was properly carried out [19-20]. In addition, microstructural observations revealed that delamination did not occur at or near the interface proved that good interfacial bonding was established for the five-layered composite specimen sintered at 630°C for 3 hours sintering time using two-step sintering cycle. Considering these sintering parameters, increased porosities (black spots) were observed in fifth layer than that in third or fourth layer. The high content of porosity in the fifth layer might be decreased considering a longer sintering time [21].

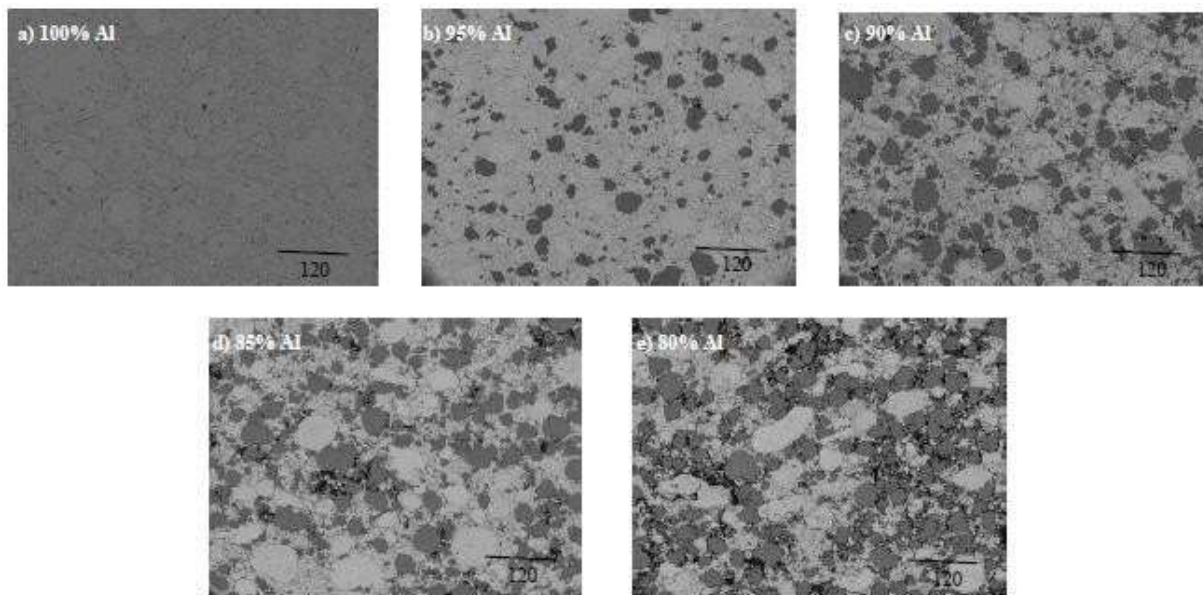


Figure 5. Microstructure of Al-Al₂O₃ graded composite layers a)100% Al at 1st layer b)95% Al+ 5% Al₂O₃ at 2nd layer c)90% Al+10% Al₂O₃ at 3rd layer d)85% Al+15% Al₂O₃ at 4th layer and e)80% Al + 20% Al₂O₃ at 5th layer.

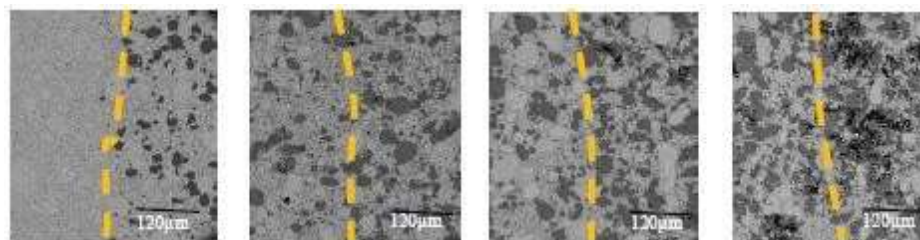


Figure 6. Five layered Al-Al₂O₃ graded composite: Micrographs of interface: (a) 1st-2nd layer interface (b) 2nd-3rd layer interface (c) 3rd-4th layer interface (d) 4th-5th layer interface.

3.3. Vickers microhardness

Figure 7 exhibits Vickers microhardness test results along thickness direction of five-layered Al-Al₂O₃ graded composite specimen. The obtained hardness at first layer (100% of Al) is 32.72 HV and at second layer (5% Al₂O₃), the hardness increased to 33.7 HV. For the third layer (10% Al₂O₃), the hardness also increased to 35.1 HV and for fourth layer (15% Al₂O₃), the hardness further increased to 37.23 HV. However, the fifth layer that contains 20% of Al₂O₃ shows small increase in hardness to 38 HV. From the obtained results, it is apparent that increasing trend of hardness with increasing reinforcement of Al₂O₃ in five-layered graded composite specimen occurred. It is concluded that, the hardness of each layer of the five-layered graded composite specimen increases with the increase in Al₂O₃ content. This is due to the addition of hard ceramic material into soft and ductile metallic phase.

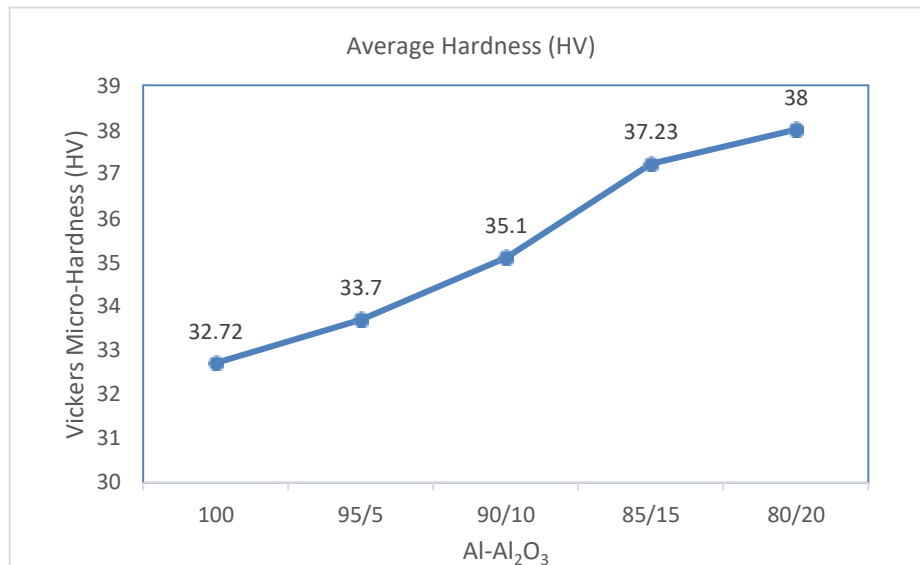


Figure 7. Vickers microhardness of each layer of Al-Al₂O₃ FGM

4. Conclusion

Five-layered Al-Al₂O₃ functionally graded composite specimen was prepared through powder metallurgy route and two-step sintering cycle. Sintering temperature 630°C and sintering time 3 hours were used. In microstructural characterization it was evident that ceramic particles are almost uniformly distributed in the metallic matrix and there is a gradual increment of ceramic content from second layer to fifth layer. It was noticed that no macrocrack or microcrack is identified / observed within any individual layer or at any interface. It was also evident that transition occurs step by step from the first layer to fifth layer. Microstructural observations confirmed that there is no delamination at or near the interface for the five-layered composite specimen. Experimental data revealed that layer hardness increases continuously in the direction of increasing ceramic content although hardness of fifth layer increases slightly.

5. References

- [1] Naebe, M., & Shirvanimoghaddam, K. (2016). Functionally graded materials: A review of fabrication and properties. *Applied Materials Today*, 5, 223-245.
- [2] Watanabe, Y., Hattori, Y., Sato, H. (2015) Distribution of microstructure and cooling rate in Al-Al₂Cu functionally graded materials fabricated by a centrifugal method, *Journal of Materials Processing Technology* 221, 197–204.
- [3] Wu, K., Scheler, S., Park, H.S., Porada, M.W (2013) Pressureless sintering of ZrO₂-ZrSiO₄/NiCr functionally graded materials with a shrinkage matching process, *Journal of the European Ceramic Society* 33, 1111–1121.
- [4] Khoddami, A.M., Sabour, A., Hadavi, S.M.M. (2007) Microstructure formation in thermally- sprayed duplex and functionally graded NiCrAlY/Yttria-stabilized zirconia coatings, *Surface and Coatings Technology* 201, 6019-6024.
- [5] Udupa, G., Rao, S. S., & Gangadharan, K. V. (2014). Functionally Graded Composite Materials: An Overview. *Procedia Materials Science*, 5, 1291-1299.
- [6] Sun, L., Sneller, A., & Kwon, P. (2008). Fabrication of alumina/zirconia functionally graded material: From optimization of processing parameters to phenomenological constitutive models. *Materials Science and Engineering: A*, 488(1-2), 31-38.
- [7] Al-Qureshi, H. A., Soares, M. R. F., Hotza, D., Alves, M. C., & Klein, A. N. (2008). Analyses of the fundamental parameters of cold die compaction of powder metallurgy. *Journal of Materials Processing Technology*, 199(1), 417-424.

- [8] Erdemir, F., Canakci, A., & Varol, T. (2015) Microstructural characterization and mechanical properties of functionally graded Al₂O₃/SiC composites prepared by powder metallurgy techniques. *Transactions of Nonferrous Metals Society of China*, 25(11), 3569-3577.
- [9] Zhang, J., Yang, S., Chen, Z., Wyszomirska, M., Zhao, J., & Jiang, Z. (2019). Microstructure and tribological behaviour of alumina composites reinforced with SiC/graphene core-shell nanoparticles. *Tribology International*, 131, 94- 101.
- [10] Zhang, C.H., Zhang, H., Wu, C.L., Zhang, S., Sun, Z.L., Dong, S.Y. (2017) Multi-layer functional graded stainless steel fabricated by laser melting deposition, *Vacuum* 141, 181-187.
- [11] Radhika, N., Raghu, R. (2016) Development of functionally graded aluminium composites using centrifugal casting and influence of reinforcements on mechanical and wear properties, *Transactions of Nonferrous Metals Society of China* 26, 905-916.
- [12] Lin, Q., Chen, F., Yin, H. (2017) Experimental and theoretical investigation of the thermo- mechanical deformation of a functionally graded panel, *Engineering Structures* 138, 17–26.
- [13] Latiff, M.I.A., Nuruzzaman, D.M., Basri, S., Ismail, N.M., Jamaludin, S.N.S. and Kamaruzaman, F.F. (2018) Preparation and characterization of 6-layered functionally graded nickel-alumina (Ni- Al₂O₃) composites, *IOP Conf. Series: Materials Science and Engineering*, Vol. 342, pp.1–8.
- [14] Yusefi, A. and Parvin, N. (2017) Fabrication of three layered W-Cu functionally graded composite via spark plasma sintering, *Fusion Engineering and Design*, Vol. 114, pp.196–202.
- [15] Shahrjerdi, A., Mustapha, F., Bayat, M., Sapuan, S.M. and Majid, D.L.A. (2011) Fabrication of functionally graded hydroxyapatite-titanium by applying optimal sintering procedure and powder metallurgy, *International Journal of the Physical Sciences*, Vol. 6, No. 9, pp.2258–2267.
- [16] Xu, Z., Hodgson, M., Chang, K., Chen, G., Yuan, X., & Cao, P. (2017) Effect of Sintering Time on the Densification, Microstructure, Weight Loss and Tensile Properties of a Powder Metallurgical FeMn-Si Alloy. *Metals*, 7(3).
- [17] Kamaruzaman, F. F., Nuruzzaman, D. M., Ismail, N. M., Hamedon, Z., Iqbal, A. K. M. A., & Azhari, A. (2018) Microstructure and properties of aluminium-aluminium oxide graded composite materials. *IOP Conference Series: Materials Science and Engineering*, 319.
- [18] Bhattacharyya, M., Kumar, A.N. and Kapuria, S. (2008) Synthesis and characterization of Al/SiC and Ni/Al₂O₃ functionally graded materials, *Materials Science and Engineering: A*, Vol. 487, Nos. 1– 2, pp. 524–535.
- [19] Canakci, A., Varol, T., Özkaya, S. and Erdemir, F. (2014) Microstructure and properties of Al- B₄C functionally graded materials produced by powder metallurgy method, *Universal Journal of Materials Science*, Vol. 2, No. 5, pp. 90–95.
- [20] Kamaruzaman, F.F., Nuruzzaman, D.M., Chowdhury, M.A., Jamaludin, S.N.S., Basri, S. and Ismail, N.M. (2019) Characterisation of four-layered Al-Al₂O₃ functionally graded material prepared through powder metallurgy and pressureless sintering, *International Journal of Materials and Product Technology*, Vol 59, No. 1, pp. 48-62.
- [21] Almomani, M.A., Shatnawi, A.M. and Alrashdan, M.K. (2015) Effect of sintering time on the density, porosity content and microstructure of copper – 1 wt. % silicon carbide composites, *Advanced Materials Research*, Vol. 1064, pp. 32–37.