

Preparation and Mechanical Characterization of Cu-Al₂O₃ Functionally Graded Material for electrical contact applications

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Abstract

In this study, five-layered stepwise Cu/Al₂O₃ as functionally graded materials have been prepared from copper powder with with five percentage of alumina powder as (5, 10, 20, 30 and 40 Wt%) by using powder metallurgy technique. Mixing of copper (34.32 μ m) and alumina (1.439 μ m) powders for two hours and then several disk sample with dimensions (14mm diameter and 10 thickness) have been compacting at different compacting stresses (550, 650 and 750 MPa). However, sintering of specimens for three hours at 850 C° under vacuum about 10⁻³ torr has been achieved. The Porosity and density before and after sintering have been calculated. X-ray diffraction analysis showed that tests before and after sintering is similar, there is no new phase formed after sintering. Scanning electron microscopy technique is used to characterize the surface of each layer. Hardness test showed slight linear graded microhardness across the layers without any obvious jumps throughout the thickness. Pin on disc method have been used in determining the wear or material removal of prepared FGM samples. In addition, it was examined thermal conductivity and the electrical resistance have been done in preparing samples from composite and functionally graded material. From the experimental results, it is clear that hardness results change in each layer by producing FGM according to the percentage of the harder constituent (i.e. Al₂O₃) from 53 to 113 Hv. While Thermal conductivity decrease with the increasing addition weight percentage for α -Al₂O₃. And Electrical resistance increased when alumina content increased for (5% to 40%). Wear rate improved with the increasing additive percentage of alumina from 0.507mm³ for layer 1 to 0.15mm³ for layer five at 15N and 25 min. Smoothly gradual change of the composition in the Cu/Al₂O₃ FGM can eliminate the microscopic interface, such as a result as that traditional Cu-Al₂O₃ joint.

Keywords: Cu/Al₂O₃, functionally graded materials, powder metallurgy technique, Material characterization of FGM.

الخلاصة

في الدراسة الحالية، تم تحضير خمسة طبقات من نحاس/الومينا كمادة متدرجة الوظائف بواسطة تقنية ميتالورجيا المساحيق، مسحوق النحاس مع خمسة نسب وزنية مختلفة من مسحوق الألومينا [5, 10, 20, 30 and 40 wt%] خلطت لمدة ساعتين وكبست بضغوط مختلفة [550, 650 and 750 MPa]. تم تحضير عينات قرصية مختلفة بأبعاد 14mm القطر وسمك 10mm، وتم تلييد العينات المحضرة عند درجة تلييد 850C° وزمن مكوث 3hr حيث تم التلييد في فرن انبوبي مفرغ من الهواء. تم حساب المسامية والكثافة قبل وبعد عملية التلييد، تحليل الاشعة السينية وضح ان الفحص قبل وبعد عملية التلييد متشابه وهذا يعني انه لا يتكون طور جديد بعد عملية التلييد. قد تم دراسة فحوصات مثل الصلادة والبلى الانزلاقي الجاف والمقاومة الكهربائية والموصلية الحرارية وكذلك تمت فحوصات البنى المجهرية بواسطة المجهر الالكتروني الماسح. ومن خلال النتائج المستحصلة من الفحوصات في هذه الدراسة، نتائج الصلادة برهنت تدرج الخواص عبر طبقات المادة المتدرجة الوظائف عند ضغط 650MPa حيث تزداد مع زيادة النسبة الوزنية للالومينا، بينما التوصيل الحراري يقل بزيادة الالومينا والمقاومة الكهربائية تزداد مع زيادة النسبة الوزنية للالومينا و مقاومة البلى تتحسن مع زيادة النسبة الوزنية للالومينا. التغير التدريجي السلس في التركيب في Cu/Al₂O₃ FGM يمكنه القضاء على الحد الفاصل الناتج عند ربط Cu/Al₂O₃ تقليديا.

الكلمات المفتاحية: - نحاس/الومينا، المواد المتدرجة وظيفيا، تقنية البودر متلورجي، خصائص المواد المتدرجة وظيفيا.

Introduction

Many structural components encounter severe conditions and, so, need material performance that changes with location within the component. For example, the body of a gear must be strong, while its surface should be solid and wear resistant, and the body of a turbine blade should be strong, tough and creep-resistant, while on the other hand its outer surface should be refractory and oxidation-resistant (Ghafil, 2011).

It is well known that abrupt transitions in materials composition and features within a component often result in sharp local concentrations of stress, whether the stress is applied internally or externally. Another known this is that these stress concentrations are greatly lessened if the transition from one material to the other is made gradually (Maricel, 2011).

Functionally Graded Material (FGM) belongs to a class of advanced material characterized by variation in properties as the dimension varies. The overall properties of FMG are unique and different from any of the individual material that forms it (Mahmoud *et.al.*, 2012).

FGM is frequently a mixture of two particular material phases: e.g. Ceramic and metal with the variation of the volume fraction (Tran *et.al.*, 2014). Because of contrasts of thermal and mechanical properties in ceramics and metals, residual stresses develop in regions near the ceramic-metal interfaces during the manufacturing and under thermal and mechanical loading in service. These stresses influence the performance and the lifetime of the ceramic-metal bonded systems and can lead to breaking within ceramic, plastic distortion accompanied by the formation and growth of voids in metal and: or ceramic metal decoration (Grujicic *et.al.*, 1998).

From literature review, it is observed, two types of functionally graded materials FGMs were studied. The first type was metal/metal FGM, such as Steel/Al, Al/Si (Nemat-Alla1 *et.al.*, 2011; Zhongtao and Tingju 2008). The second type was metal/ceramic FGM, such as Al/SiC and Al_2O_3/Ti , Cu/ Al_2O_3 , Cu/Nbc, Al/ Al_2O_3 (Kumar1 and Chandrappa, 2014; Abdulamer, 2012; Strojny-Nędza and Agata, 2016; Shiri *et.al.*, 2015; Sanuddin, 2012). the aim of this type was to increase the wear resistance and hardness, in addition, don't cracking or failure under thermal stresses when functionally graded materials FGMs were used instead of welding.

In this study, an attempt is made to prepare five-layers functionally graded Cu/ Al_2O_3 with gradient of Al_2O_3 from 5, 10, 20, 30 and 40 wt.% by using powder metallurgy technique. Furthermore, different parameters have been studied such as: α - Al_2O_3 weight percentage, Different compacting pressure, Dry sliding wear parameters.

Experimental Part

In this work, elemental powders of copper and alumina used to prepare the functionally graded Cu/ Al_2O_3 with gradient of Al_2O_3 from 5, 10, 20, 30 and 40 wt.% By using powder metallurgy technique. Mixing of copper (34.32 μm) and alumina (1.439 μm) powders for two hours and then several disk sample with dimensions (14mm diameter and 10 thickness) have been compacting at different compacting stresses (550, 650 and 750 Mpa). However, sintering of specimens for three hours at 850 C° under vacuum about $10^{-3} torr$ has been achieved. The samples surface subsequently ground, polished and then characterize with scanning electron microscopy. Compositions for each layer of elemental powders used in this study have been shown in the Table 1.

Table 1 Weight composition for each layer in FGM.

Layer Number	Layer weight Percentage Cu%	Layer weight Percentage Al ₂ O ₃ %	Layer weight in gm			
			Cu	Al ₂ O ₃		
1	95	5	2.5383	0.1336	2mm	95%Cu+5%Al ₂ O ₃
2	90	10	2.3349	0.2594	2mm	90%Cu+10%Al ₂ O ₃
3	80	20	1.9517	0.4879	2mm	80%Cu+20%Al ₂ O ₃
4	70	30	1.5993	0.6854	2mm	70%Cu+30%Al ₂ O ₃
5	60	40	1.2779	0.8519	2mm	60%Cu+40%Al ₂ O ₃

The density and porosity was calculated before sintering according to (1, 2) equations (Abid ,2008).

$$\rho_g = m_g / V_g \quad (1)$$

Where:

ρ_g : is the green density (g/cm³), and m_g : mass of green compacted (g)

V_g : volume of green compacted (cm³).

$$P_g = (1 - \rho_g / \rho_{th}) \times 100 \quad (2)$$

Where:

P_g =green porosity (%), ρ_g = green density (g/cm³)

ρ_{th} =theoretical density of mixed powders(g/cm³)

and after sintering according to ASTM B328, in order to determine the porosity and density of sintered samples the following procedure was followed:

1-After drying at 100°C for 5 hours in a vacuum furnace the sample was weighed, and the weight represent mass A.

2-the sample is completely immersed in Paraffin oil with density 0.8 g/cm³ and an evacuating system (to decrease the pressure) for 30 minutes at room temperature.

3-the impregnated (soaked) sample has been weighed in air, and the weight was mass B.

4- Mass C was determined by weighing the impregnated sample in water.

5- The porosity and density have been calculated by the following equations (3, 4)

[ASTM B – 328(2003)]:

$$P = \left[\frac{B-A}{(B-C)D^\circ} \times 100 \right] D_w \quad (3)$$

$$D = \left[\frac{A}{B-C} \right] D_w \quad (4)$$

Where:

D° =the density of the used oil

D_w = the density of water

Appropriate grinding and polishing were carried out before subjecting the samples to the hardness test. The test was conducted on Micro Vickers hardness device using the weight of (300g) for 10 sec with a square-base diamond pyramid. Then reading were done along each layer.

The Vickers microhardness (H.V.) is defined as follows:

$$HV = 1.854 P / d^2 \quad (5)$$

Where

P = applied load Kg.

d = average length of the diagonal mm².

Pin on disk concept was used to study dry sliding wear, The specimen to be tested was set as a pin against a rotating steel disc at a radius of 5mm. The rotating speed of the disc was fixed at 300 rpm, while the loads were (5N, 10N, 15N). The sample is weighted after (5, 10, 15, 20, and 25) min to determine the dry sliding wear rate according to equation (6). The test method has been covered according to [ASTM G 99-04"].

$$\text{Volume loss (cm}^3\text{)} = \text{weight loss (g)} / \rho \text{ (g/ cm}^3\text{)} \quad (6)$$

Where: Δw : Weight lost = weight before the test – weight after the test.

samples of 14mm in diameter that prepared by powder metallurgy are used to show the effect of additive percentage on the electrical resistance. The measurement method depended on Ohm's law from the equation (7) to calculate the electrical resistance.

$$\sigma = 1/\rho \dots \dots \dots (7)$$

thermal conductivity was conducted by using a thermal system which includes two disks of copper. The sample was placed within the upper heating disk. It was placed between the two copper disks. Then the required temperature degree was selected as (100°C) to measure the thermal conductivity coefficient then, the reading results, that represent the voltage of the upper and lower copper disks in both cases; cooling and heating, were recorded. Heat transfer by conduction shown in this equation:

$$Q_{\text{cond}} = -KA \frac{dT}{dX} \dots \dots \dots (8)$$

The thermal conductivity coefficient was determined based on the equation below.

$$K = mc \frac{hp}{\pi(R_B)^2(V1-V2)} + \frac{2hp+RP}{(2RP+2hp)\left(\frac{\Delta v}{\Delta t}\right)} \dots \dots (9)$$

K: thermal conductivity coefficient (w/m.k).

m: mass of copper plate (0.824 kg).

C: specific heat capacity of copper plate is 3.805×10^2 (kj/kg.k).

hp, Rp: thickness and radius of copper plate are 7.01mm, 32.5mm respectively.

RB: radius of specimen is 8mm.

v1, v2: voltage of thermocouple 1,2 at heating.

Δv : difference in voltage at cooling.

Δt : difference in time in cooling

Results And Discussion

Figure(A): is shown that if the compacting pressure increases, the green density increases too, until it reaches a certain limit at which any further increase in the pressure has no or little effect on its value. This can be attributed to the fact that powder under compaction pressure is subjected to the force that contributes in plastic flow, which increases compact density so the preferred pressure is determined as 650 MPa for all layers of FGMs. And **Fig 1 B:** indicates that an increase in compacting pressure causes a decrease in the green porosity due to increase in the contact points between particles that reduce voids.

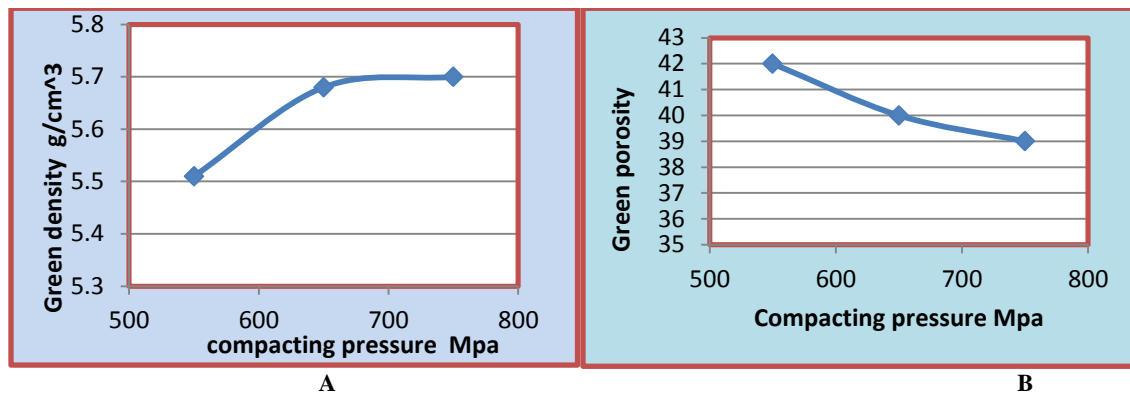


Fig. 1: A: Green density of FGMs compacts as a function of compacting pressure. B: Green Porosity of FGMs compacts as a function of compacting pressure.

Fig 2 A: shows the effect of additions α -Al₂O₃ on the porosity after sintering. the porosity increase with α -Al₂O₃ increases As we know The main disadvantage of copper matrix composites reinforced with aluminum oxide particles is residual porosity, which influences the material properties, and pores that are present within the area of the ceramic phase. One can observe that α -form of aluminum oxide powder (α -Al₂O₃) shows a strong tendency to form agglomerates at the preparation stage of Cu and Al₂O₃ powder mixtures, which results in residual porosity in composites. **Fig 2 B:** show the effect of compacting pressure on true porosity. When the pressure increases, the porosity decreases because of the increasing the agglutination atoms.

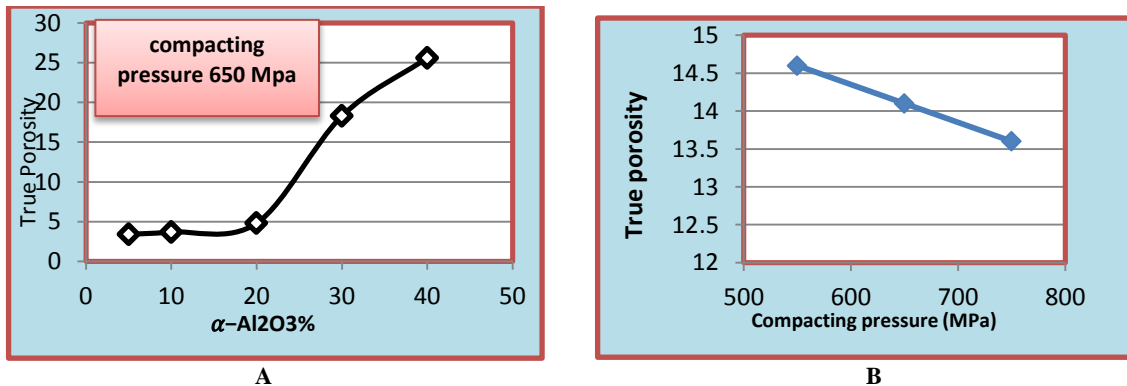


Fig. 2 A: Effect of additions α -Al₂O₃ on the porosity. B: Effect of compacting pressure on true porosity for samples of FGM.

Fig. 3 A and B: show the X-ray diffraction results for layer (70%Cu+30%Al₂O₃) before and after sintering process. From results of tests before and after sintering is similar, there is no new phase formed after sintering, this indicates that there is no solubility between copper and alumina This means that the strengthening here by dispersion and there is no depositions.

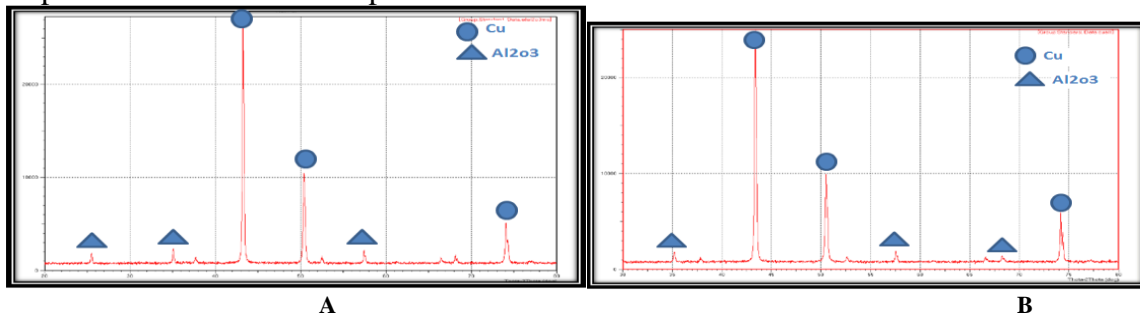


Fig. 3 A: X-ray diffraction pattern of layer (70%Cu+30% Al₂O₃) before sintering process. B: X-ray diffraction pattern of layer (70%Cu+30%Al₂O₃) after sintering.

Fig. 4, and Fig 5: show the SEM images for Layers each one individually and FGMs sample using a compacting pressure of 650 Mpa with different magnifications. From these images it is seen that the distribution and existence of additive particle and pores through the sample surface. Note that additive particle distributed uniformly in the copper matrix, Through the SEM test for functionally graded sample as shown in **Fig.5** seen that the existence of a gradation clear for layers within the sample functionally graded, and also the presence of the overlap between the layers, this is proof of the gradation properties.

Hardness results

The results show that the hardness increases with the increasing the adding percentage because of the added material particles (α -Al₂O₃) work to obstruct and prevent the movement of dislocations on the sliding planes and due to the fact that the rise in additive content make the ability of the indenture of the hardness tester to hit additive particles increase and the additive particles has high hardness compared with copper hardness. This result has been corresponding with (Li Yu *et.al.*,2009).

Fig. 6 A: shows the measured hardness for layers (Cu-Al₂O₃ composite) at compacting pressure 650 MPa, and **Fig.6 B:** for FGMs sample using various values of compacting pressure. Figure (4.14) shows Vickers microhardness in each layer was measured at 1mm interval from the first to fifth layer. The interface between each layer was monitored carefully in this work. All samples generally show an interesting graded microhardness across the thickness. Dependent on the microstructure of those samples, material properties vary with position within the gradient and can be used to tailor functionality. Removing the sharp interface by an interlayer compositional gradient. However, residual stresses still develop at the new heterogeneous interfaces introduced by the composite compositional gradient.

It is realized that when the compacting pressure increases the hardness increases. Increasing pressure works on minimizing the pores and increases the density of the sample and leads to increase of the hardness.

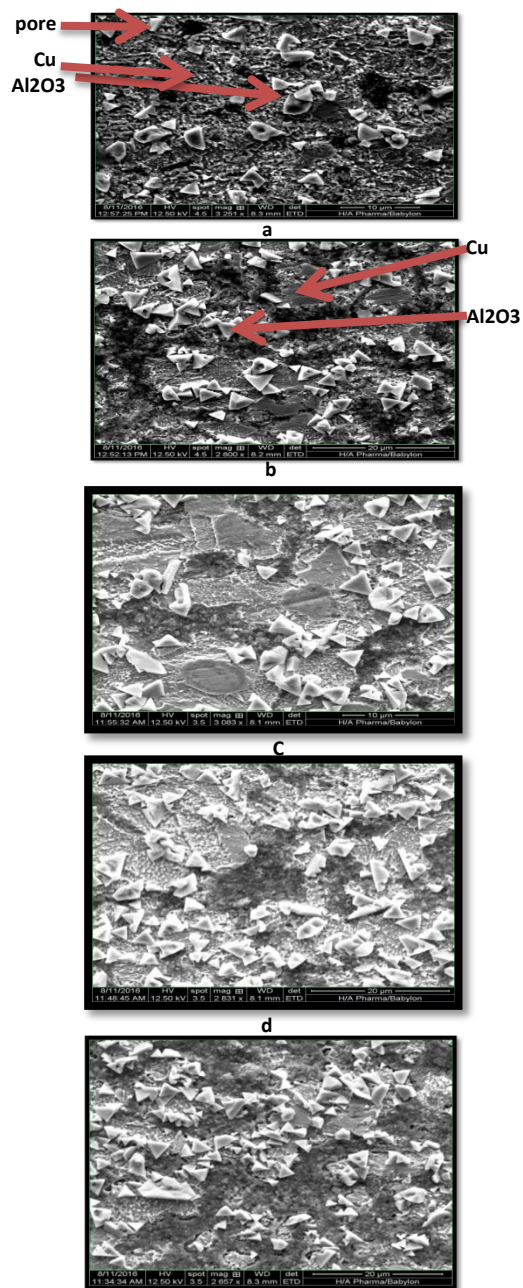


Fig. 4: SEM images of layers (a) 5% Al₂O₃ (b) 10% Al₂O₃ (c) 20% Al₂O₃ (d) 30% Al₂O₃ (e) 40% Al₂O₃.

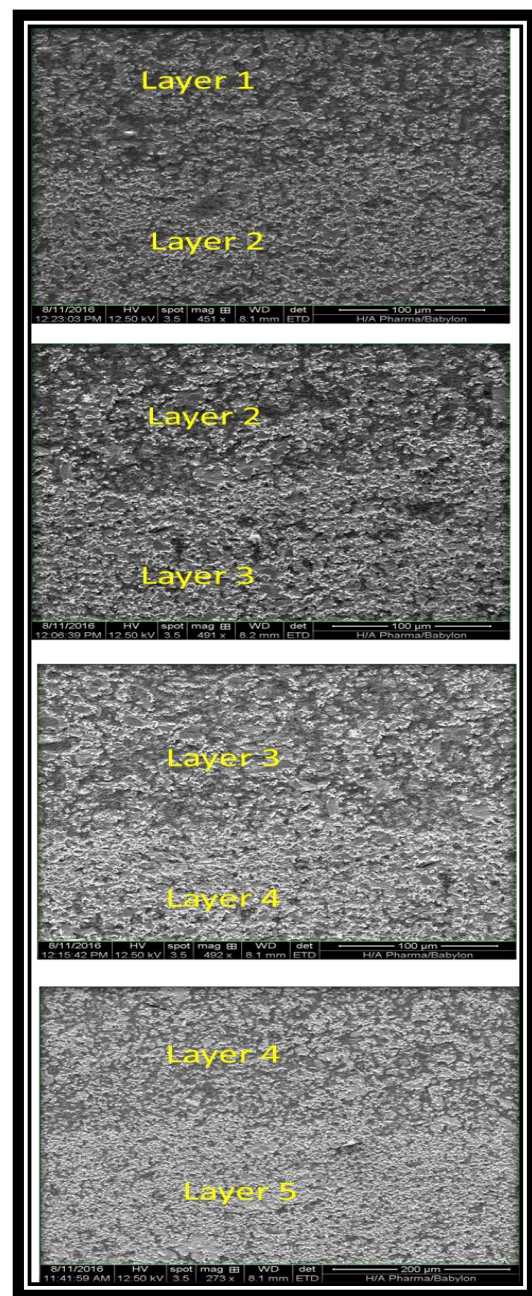


Fig. 5: SEM images for FGMs samples

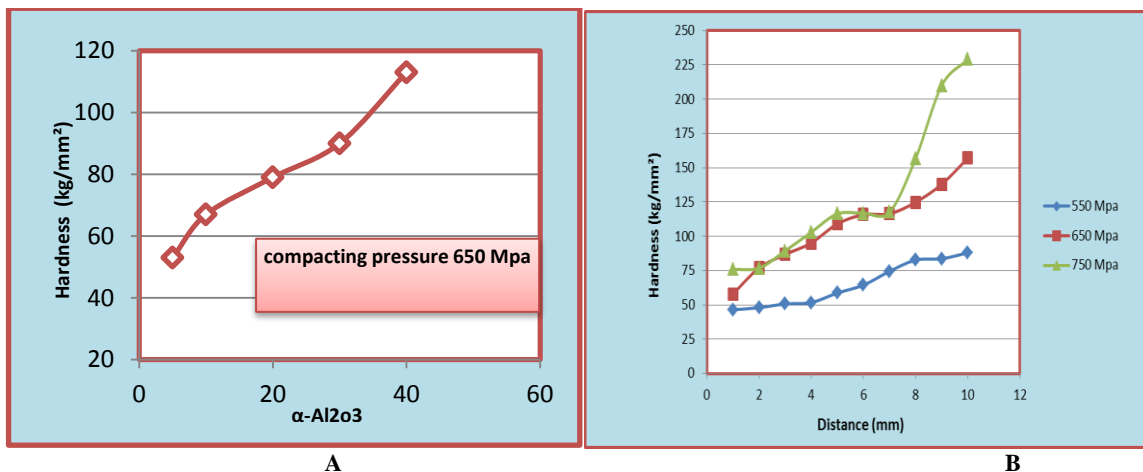


Fig.6 A: Hardness for Layers each one individually using a compacting pressure of 650 Mpa. **B:** Hardness as a function of distance of FGMs from one layer (5% Al₂O₃) at various compacting pressures.

Wear results

Pin on disc method have been used in determining the wear or material removal of prepared FGM samples. All the prepared samples are subjected to different loads (8, 10 and 15 N) at room temperature and different times (5, 10, 15, 20 and 25 min.) As shown in **Figures(7-13)**.

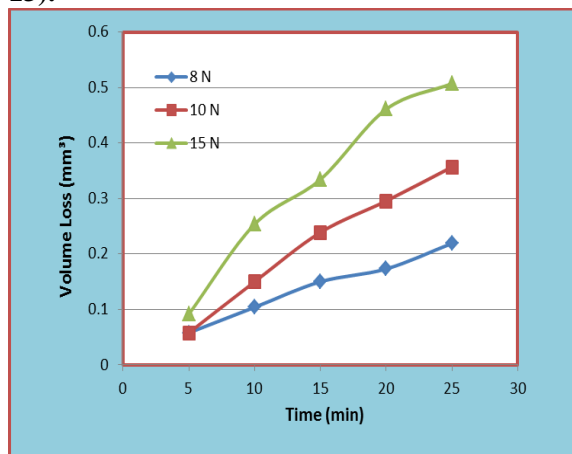


Fig. 7: Volume loss as a function of time for layer 1

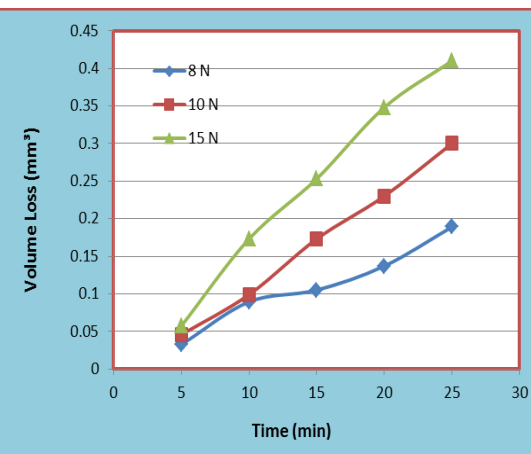


Fig. 8: Volume loss as a function of time for layer 2

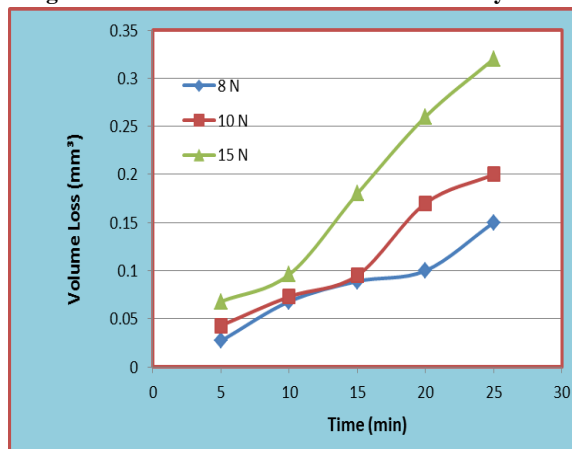


Fig. 9: Volume loss as a function of time for layer 3.

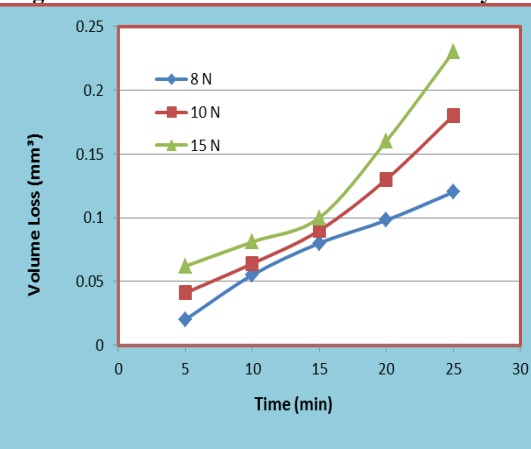


Fig.10: Volume loss as a function of time for layer 4.

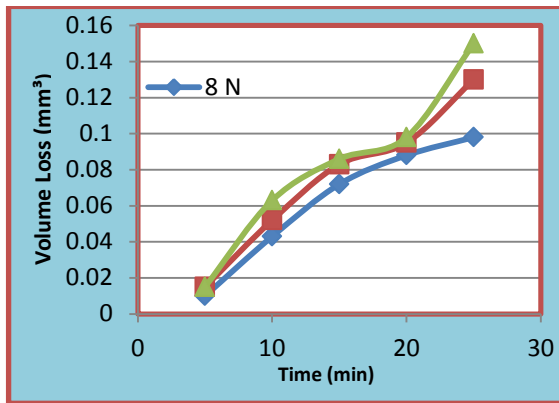


Fig. 11: Volume loss as a function of time for layer 5.

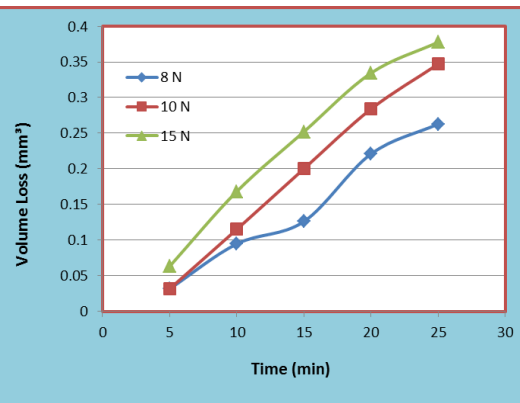


Fig. 12: Volume loss as a function of time for FGM.

It is noticed that the volume loss due to wear decreases when the percentage of alumina increase. For layer1 (Cu-5% Al₂O₃), the Volume loss is (0.22) mm³, whereas for layer2 (Cu-10% Al₂O₃), the volume loss decreased to (0.18) mm³, whereas for layer3 (Cu-20% Al₂O₃), the volume loss is (0.15) mm³, whereas for layer4 (Cu-30% Al₂O₃), the volume loss was decreased to (0.12) mm³, and for layer5 (Cu-40% Al₂O₃) the Volume loss was decreased to (0.09) mm³, at 8 N load and 25 min. Similar trends in volume loss for all other loads are observed.

The wear is highest in layer1 [Cu-5%Al₂O₃], which is due to the layers with relatively less alumina content tend to wear more when compared to the layers with high alumina content. The decrease in weight loss with increasing percentage of alumina can be attributed to the presence of hard alumina particles adhered to the composite. Al₂O₃ being inherently harder than Cu, hence the wear resistance of the composites is higher than native Cu.

However, as the number of Al₂O₃ microparticles increase, the resistance to the penetration of abrasive particles increases (Hardness increases with increases in the reinforcement content) and hence the wear depth decreases. The drastic reduction in wear rate may be attributed by (1) enhancement in hardness of the composite reinforced by Al₂O₃ particles and (2) greater reduction of direct load contact between the Cu/Al₂O₃ composite surface and disk in comparison with pure Cu due to load bearing component action of hard Al₂O₃ particles. This result has been corresponding with [DASH and Khushbu (2014)]. Hence, Cu–Al₂O₃ composite with high alumina content exhibit better wear resistance at elevated pressures. In FGM as shown in fig. 12 the Volume loss between the first layer and the fifth layer, meaning it is the middle between them, Volume loss case be less than the first layer and the second, but more than the fourth and fifth layer, meaning it has a resistance to wear suitable and moderation between layers.

The volume loss increased from (0.03) mm³ to (0.26) mm³ for FGM with increasing time from (5-25 min), at 8N, as in Fig. 12 and also for all layers, because of the friction between the surfaces increases, then increase the heating and softening of the copper matrix of the Cu-Al₂O₃ composite increases and the particles tend to remove from the surface, therefore increases material removal rate lead to increase the wear rate. The wear rate contributed in plastic deformation of the surface. The results of wear tests on all samples with varying loads (8, 10 and 15 N) are illustrated in previous Figures. The Volume loss, increased for FGM with increasing load from (8-15 N), and also for all layers. During the dry sliding motion, the temperature at the interface increases with the increase in the pressure because of increase the friction force. The damaged surface has been studied due to the adhesion wear for FGM and photos which have taken by the SEM

with different magnifications as shown in the **Fig. 13** and **Fig. 14**. It is clear that some pores have been found in the wear surface which may be from the action of Al_2O_3 hard particles. Alumina particles act as abrasive in the copper matrix during dry sliding wear, so that grooves and pores with sliding direction appear in the scanning electron microscopy images.

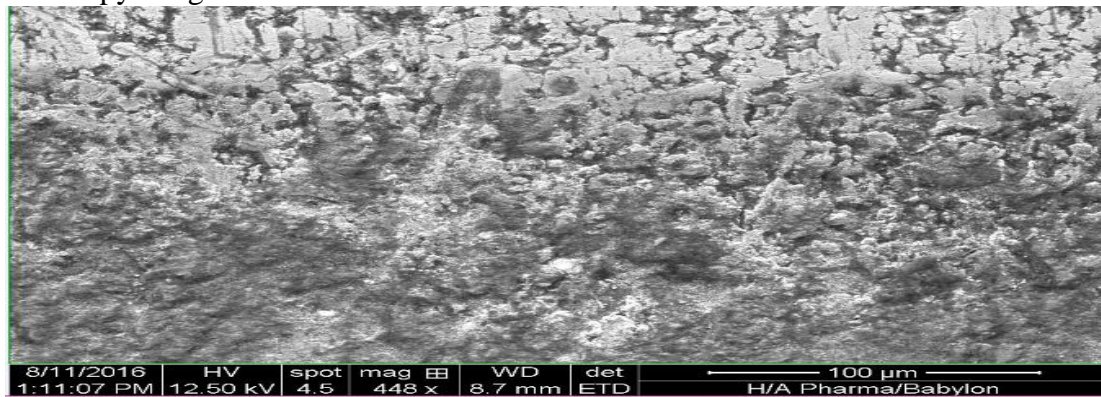


Fig. 13: Shows the damaged surface due to the adhesion wear for FGM.

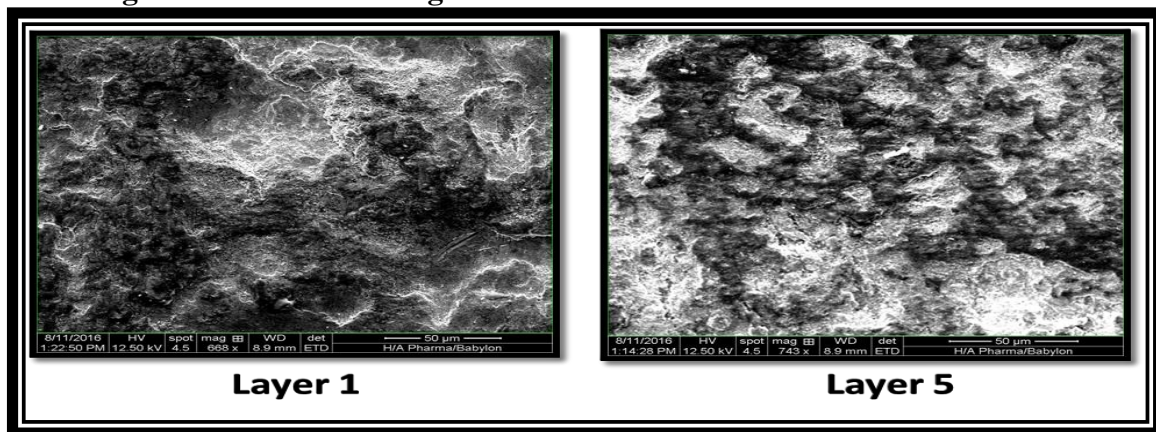


Fig. 14: Shows the damaged surface due to the adhesion wear for layer1 and layer5.

Electrical resistance results

The electrical resistance test had been carried out in layers, each one individually prepared at compacting pressure 650 Mpa. And for samples of FGMs prepared at different compacting pressure.

Fig.7 show that increasing the additives percentage leads to increase the electrical resistance of all samples, because the additive particle works as obstacles to the movement of electrons which is carrier electric charge that leads to increase the random and electrons welter and decrease the free flight time for the carrier electron. This result has been corresponding with(Shih-Hsien *et.al.*, 2012).

α - Al_2O_3 which has an effect on electrical resistance property because it is an insulator ceramic material. And also there is an increase in porosity that leads to increase the electrical resistivity because pores act as an obstacle to electrical movement.

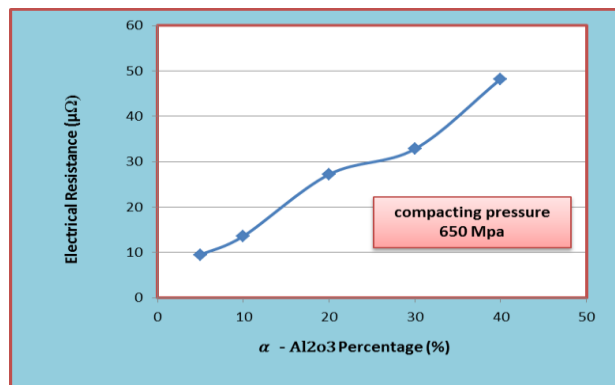


Fig.7: Effect of additions α -Al₂O₃ on the electrical resistance.

Fig.8 shows the effect of compacting pressure on electrical resistance. It can be noticed that when the pressure increases, the electrical resistance decreases and increasing the electrical conductivity. This is due to the decrease of the voids and pores between particles which work on block the movement of electrons that carry the charges and decrease the free flight of electrons.

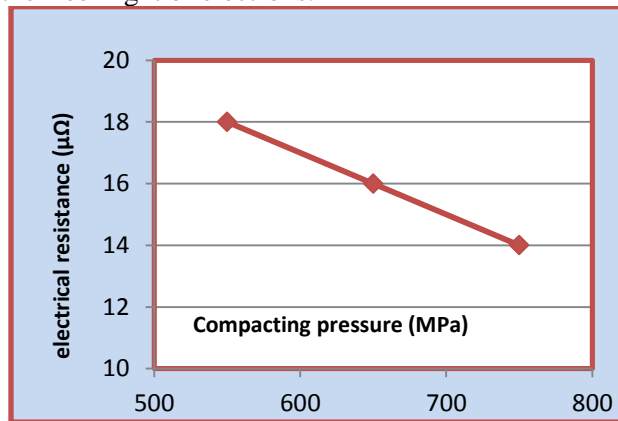


Fig.8: Effect of pressure on electrical resistivity for FGMs samples.

Thermal conductivity results

Fig.9 show the results of thermal conductivity test. It is obvious that the values of thermal conductivity varied with the presence of Al₂O₃ percentage, the addition of Al₂O₃ lead to decrease in thermal conductivity due to The presence of pores in the structure of composites fundamentally affect the obtained thermal conductivity values. Pores present between aluminum oxide grains, which are barriers for the heat transport in a material. This result has been corresponding with (Strojny-Nędza *et.al.*, 2016).

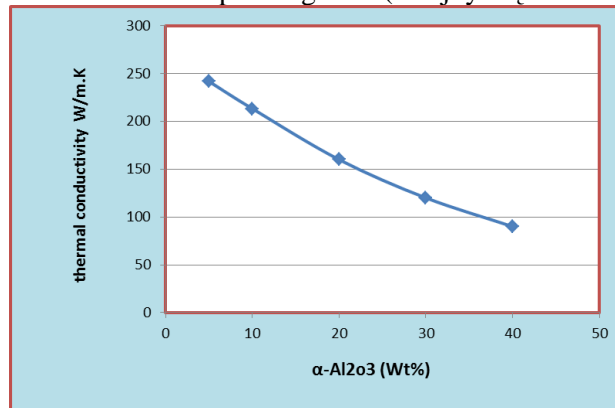


Fig. 9: Shows the thermal conductivity varied with the presence of Al₂O₃ percentage.

Conclusions

From the experimental, several conclusions may be drawn as follows:

1. The best compacting pressure for α -Al₂O₃ addition was 650 Mpa.
2. Hardness number is changed in each layer of produced FGM according to the percentage of the harder alumina content.
3. Porosity increase with increasing of addition percentage of α -Al₂O₃.
4. Wear resistance, improved with the increasing of α -Al₂O₃ percentage.

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