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Preparation and Characterization of γ-Al_2O_3/Al Composite

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Abstract:

A cermet (ceramic-metal) composite have been prepared from alumina (γ-Al_2O_3) reinforced with aluminum (Al) for the concentrations of (0, 10, 20, 30, 40, & 50) wt. %Al. The cermet was formed by single axial pressing, sintered in vacuum atmosphere. Compaction behaviors were studied in solid state sintering at sintering temperatures (400, 450, & 550) °C, sintering times (2, 4, & 6) hrs., and forming pressures (5, 10, 15) MPa, also in liquid phase sintering at (800 °C). The cermet was characterized by x-ray diffraction (XRD) and by scanning electron microscope (SEM), also physical and mechanical properties have been studied. SEM results showed the Al flowing inside the ceramic body due to uniform distribution of Al particles and due to sintering temperature (800 °C) have been used. XRD results showed that there is no new phase formed between Al and Al_2O_3 after sintering due to there is no chemical reaction but physical reaction between them. The existence of Al enhanced the physical properties as the sintering (time & temperature) and pressure increase. Hardness values increased to 30 wt. % Al then decreased due to Al ductility. The oxide layer on the friction surfaces played important role for increasing wear rate.

Keywords: cermet, composite, ᵞ-alumina.

تحضير ودراسة خصائص المتراكب γ–Al_2O_3/Al

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الخلاصة:

تم تحضير المتراكب السيرميتي (سيراميك–معدن) من الالومينا (γ–Al_2O_3) المدعمة بالألمنيوم (Al) بالتراكيز (0، 10، 20، 30، 40، 50)Al%. شكل السيرمت بفعل الضغط باتجاه واحد، ولبد في جو مفرغ من الهواء. تم دراسة سلوكيات الرص بحالة التلبيد الصلب عند (400، 450، 550) م° و (2، 4، 6) ساعات وكذلك (5، 10، 15) ميكاباسكال، وكذلك تم دراستها عند تلبيد الطور السائل عند (800 م°). تم تشخيص السيرمت بواسطة حيود الاشعة السينية (XRD)، و المجهر الالكتروني (SEM)، وكذلك تم دراسة الخواص الفيزيائية والميكانيكية. اظهرت نتائج الفحص المجهري (SEM) انسياب الالمنيوم داخل الجسم السيراميكي وذلك تبعا للتوزيع المنتظم لجسيمات الالمنيوم وكذلك درجة حرارة التلبيد المستعملة (800 م°)، واظهرت نتائج (XRD) ان ليس هنالك تكون طور جديد بين الالمنيوم و الالومينا بعد التلبيد نسبة الى عدم حدوث تفاعل كيميائي بين المادتين فقط حدوث تفاعل فيزيائي بينهما. وجود الالمنيوم حسن الخواص الفيزيائية بزيادة كل من درجة حرارة التلبيد وزمن التلبيد و الضغط. قيم الصلادة ازدادت حتى التركيز 30Al% وبعدها تبدا القيم بالنقصان و ذلك نسبة الى مطيلية الالمنيوم. الطبقة الاوكسيدية المتكونة عند السطوح المحتكة لعبت دور مهم في زيادة معدل البلى.

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Introduction:

Composite materials are multiphase materials obtained through the artificial combination of different materials in order to attain properties that the individual components by themselves cannot attain [1]. The term cermet is used to describe a ceramic matrix material having metallic particles dispersed in the ceramic body. The term is also used to describe the reverse, that is, a metal matrix having dispersed therein refractory or ceramic materials which impart ceramic-like characteristics to the cermet. Oxide ceramics are strong and resistant to both creep and oxidation at high temperatures. However, for many applications, they are brittle and susceptible to thermal shock failure. On the other hand, metals, which are ductile and less prone to thermal shock failure are susceptible to destructive oxidation at high temperatures [2]. Alumina (Al_2O_3) possesses favorable physical and chemical properties such as high strength, hardness, elastic modulus and excellent resistance to thermal and chemical environments, However; its applications are somewhat limited because of poor toughness and inferior thermal resistance. On the other hand, most structural ceramics present poor electrical conductivity. It has been reported that the incorporation of some amounts of small-size metal particles into an Al_2O_3 matrix, can significantly improve both the toughness and electrical properties [3], while Light metals like aluminum, titanium, magnesium are gaining use as structural materials in engineering components. Their low density combined with reasonable tensile strength and ductility has led to their extensive use in the field of aviation and automotive industries, however; they have low wear, erosion and abrasion resistance which limit their use For, many of the engineering applications [4].

Jose G.miranda et al. (2009) had produced Al_2O_3 matrix cermets from mechanically mixed powders containing different metals (Al, Fe or Ti) as reinforcement, the powder mixture was compacted to cylindrical samples, which are subjected to pressure –less sintering 1500 °C for 1 h. for all types of matrices. It was found that microstructure of cermets with Al and Fe, consisted of equally distributed metallic particles in the Al_2O_3 matrix, also that incorporation of a ductile metal in to a hard ceramic matrix improves its fracture toughness [5].

H. Dakhel in 2007 prepared composites series of alumina and aluminum for different weights. Physical, thermal, and mechanical properties have been studied, it has been concluded that the brittleness of alumina decrease with the increase of aluminum metal, which gives us higher mechanical strength, thermal conductivity, and lower porosity [6].

M. Taher in 2008 produced metal matrix composite by powder metallurgy process, the matrix is aluminum while silicon oxide used as reinforcement for different weight percentages, it has been concluded that the density increases after the sintering process, also from XRD he found that there is no new phase after sintering. The properties measured were increased with the increase of added particles, also, the properties measured were increasing with the increase of sintering temperature except at 530 °C [7].

This paper aims to study the compaction behaviors for the (γ-Al_2O_3/Al) cermet composite for different concentrations, by test the physical, microstructural, and mechanical properties for this composite and see which properties are useful for the industrial application.

Experimental Work:

Materials have been used in this study were γ-alumina with purity of 99.98, and aluminum with purity 99.99, the concentrations of aluminum were chosen as (0, 10, 20, 30, 40, 50) wt % from alumina. The mixing process was done by the magnetic stirrer for 2 hours. The forming process included the pressures (50, 100, and 150) bar according to the literature survey and experiment, and the sintering process included the temperatures (400, 450, 550, and 800) °C and times (2, 4, and 6) hrs.

The samples were analyzed by x-ray diffraction, the microstructure was characterized by the scanning electron microscopy. The physical properties (practical density and porosity) were obtained using the equations (1) [by the application of Archimedes rule] and (2) [8] respectively:

$$\boldsymbol{Practical\ Density} = \frac{mass}{volume}\ \mathbf{(gm/cm^3)} \qquad \mathbf{(1)}$$

$$\emptyset[\%] = \left(\mathbf{1} - \frac{\rho_{\ sintering}}{\rho_{\ theoretical}}\right)\mathbf{100}\% \qquad \mathbf{(2)}$$

$\emptyset$: Value of total porosity.

$\rho_{\ sintering}$: Sintering density.

$\rho_{\ theoretical}$: Theoretical density, which is calculated from the equations (3) [6]

$$\frac{1}{\rho_{th}} = \frac{Xm}{\rho_m} + \frac{Xr}{\rho_r} \quad (3)$$

Where:
ρ_{th} theoretical density for mixture (gm/cm^3).
ρ_m theoretical density for alumina (3.9 gm/cm^3).
ρ_r theoretical density for aluminum (2.7 gm/cm^3).
Xm weight percent for alumina powder.
Xr weight percent for aluminum powder.

The hardness of the samples were tested by shore D hardness, the shore D apparatus is used by hand which has an indenter pressed by the force of hand on the sample surface and the hardness value will appear as a number on the electronic screen of the apparatus.

wear rate is calculated from the equation (4) [6] :

$$\boldsymbol{Wear\ Rate} = \frac{\Delta W}{S_p} \quad (4)$$

Where :
ΔW: Difference in weight of the specimen before and after test (gm).
S_p: Total sliding distance in (cm) which is calculated by equation (5):

$$\mathbf{S_p = 2\pi r N_D t} \quad (5)$$

Where:
r: Distance from the center of the sample to the center of the disc in (cm), which was (7cm) in this study.
N_D: Disc rotational speed in (r.p.m.).
t: Time of rotating in minute.

Results and Discussion:

Figure-1 represents the samples prepared at sintering temperature (400, 450, and 550) °C with forming pressure (5) MPa and sintering time (2) hrs., this figure was shown that the sintering density is increasing with the increase of (aluminum content in the prepared composites, sintering temperature except at the sintering temperature of (400) °C which showed decreasing in practical density with increase in porosity percent), in other hand, the porosity percent decreases with the increase of aluminum content in the prepared composites. Also, the samples prepared with 50% of aluminum content and with sintering temperature of (550) °C showed the less porosity percent and higher practical density.

Figure-2 represents the samples prepared at sintering times (2, 4, and 6) hrs., forming pressures (5) MPa and sintering temperature (550) °C, this figure shown that the sample prepared at (550) °C with aluminum content of 40% at sintering time of (6) hrs. gives the best value of practical density and the lowest bulk porosity percent. Also the increase in (Al) powder content results to decline the sintering densities at sintering times (4, and 6) hrs. because of increasing porosity percent at those sintering times, while sintering density increases at sintering time (2) hrs. because the porosity percent at that sintering time has the lowest value.

The behaviors in Figures-1 and 2 can be explained as : Sintering time and sintering temperature of the cermet composite (Al_2O_3/Al) samples may effect in one direction, which means, when the sintering time increase at specific sintering temperature it could make the better mechanical (physical) adhesion (physical adhesion happens between the particles of the two materials in the compacted body without any change in the chemical compositions of each material due the influence of the sintering time, sintering temperature and forming pressure on the materials particles.) happens in the compacted body. Also this adhesion could happen when the sintering temperature increases at specific sintering time [7].When the sintering temperature increases, the movement of the powder particles will increase also, which results in weakening the yield resistance of the powder particles which goes to increase the surface tension to shrink the voids existed in compacted body, also the increase of sintering time results in increasing the total amount of flowing, diffusion, and recrystallization at the contacted surfaces between the particles.

Figure-3 represents the samples that prepared at the forming pressures (5, 10, and 15) MPa, sintering temperature (550) °C and sintering time (4) hrs., it's obvious from this figure the increment in sintering density values with the increase of the forming pressure, and the sintering density increases with the increase of aluminum content at the forming pressures 10 and 15 MPa, while the sintering density is decreasing at 5 MPa for Al content greater than 40% wt.

This behavior could be happened because, when a pressure is applied on the powder during the forming that results to increase the adhesion force between the surfaces of particles through the expulsion of air and restricted gases.

The increasing of pressure results to deform the particles as a sequent of pressure applied to them, and then the primary contact points become larger to become contact areas. So the internal atomic adhesion forces will increase with the increase of forming pressure [7], which results to increase the density and decrease the porosity percent.

Figures-1, 2 and 3 were represent solid state sintering.

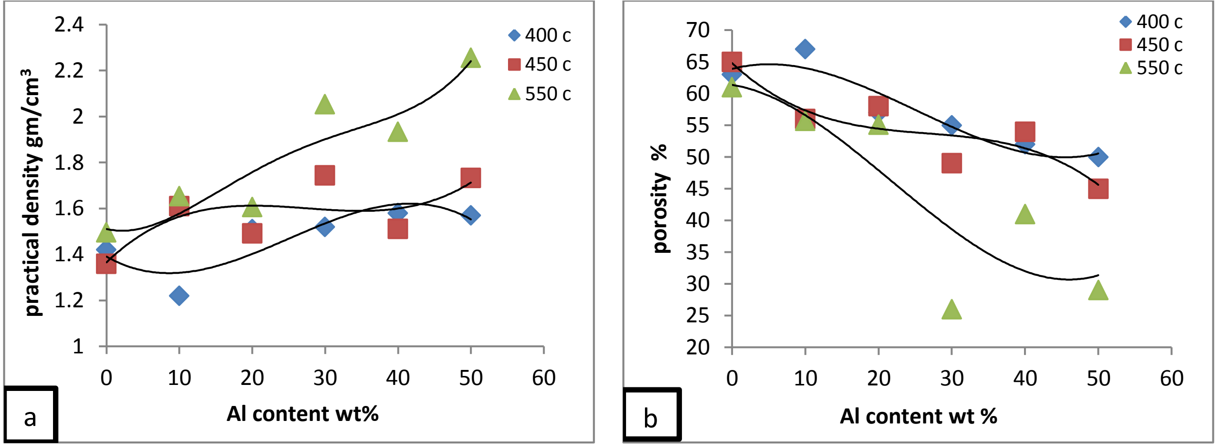


Figure 1- the effect of Al content on a- sintering density, b- porosity at (400, 450, and 550)°C.

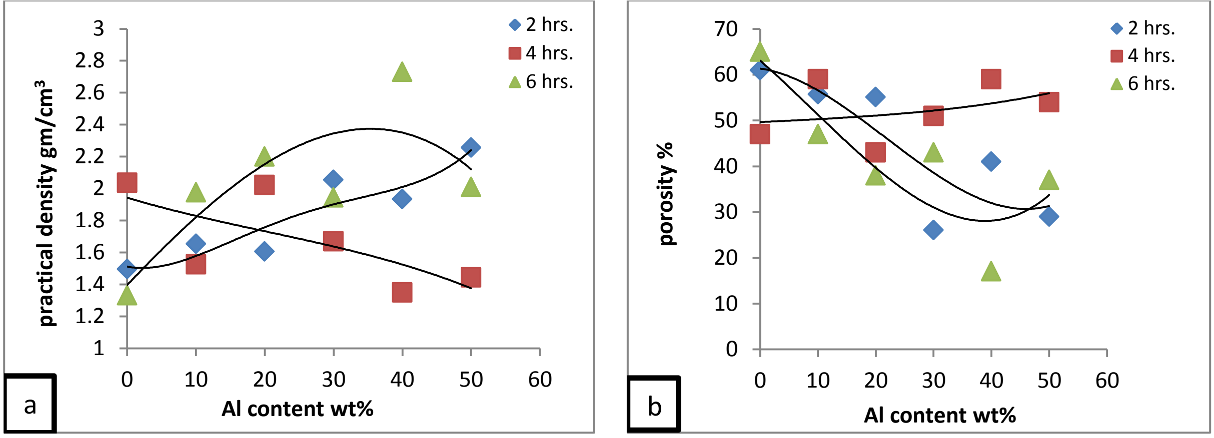


Figure 2- The effect of Al content on a-sintering density, b- porosity at (2, 4, and 6) hrs.

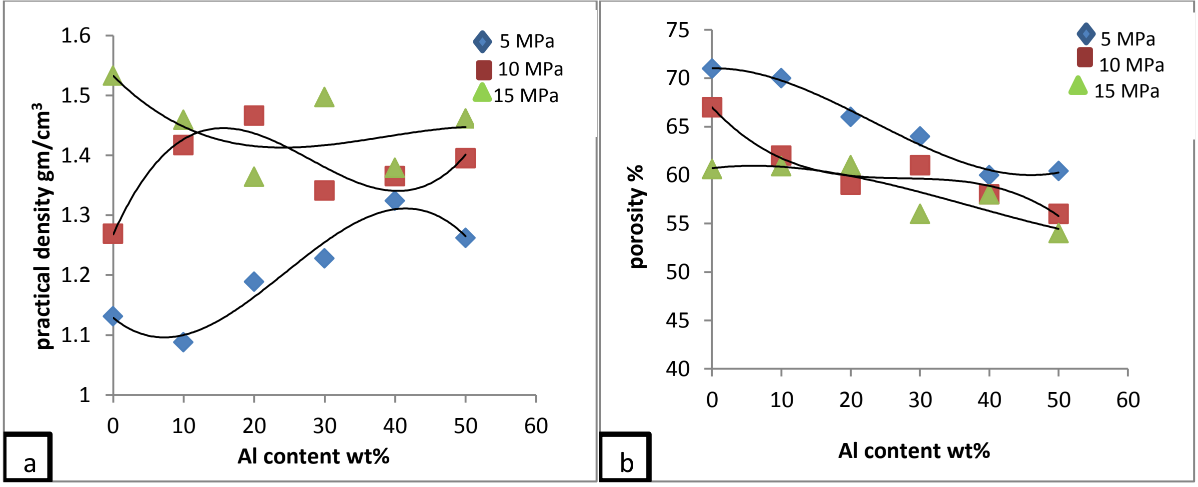


Figure 3- The effect of Al content on a-sintering density, b- porosity at (5, 10, and 15) MPa.

Liquid phase sintering chosen to be at the sintering temperature 800 °C with the best forming pressure and sintering time for the aluminum contents (0, 10, 20, 30, 40, and 50) %.

Figure-4 represents the samples prepared at 800 °C, 2 hrs., 15 MPa, from this figure the sintering density increases with the increase of aluminum metal content, and the sintering density values in this situation are higher than that of solid state sintering indicated by Figures-1, 2 and 3, that is because the sintering process is done at 800 °C which is higher than the aluminum melting point which is (660 °C), which results to melt the aluminum particles, interring that melt in to the brittle material porosities due to surface tension force, which results to give the samples higher density in Figure-4a and lower density in Figure-4b.

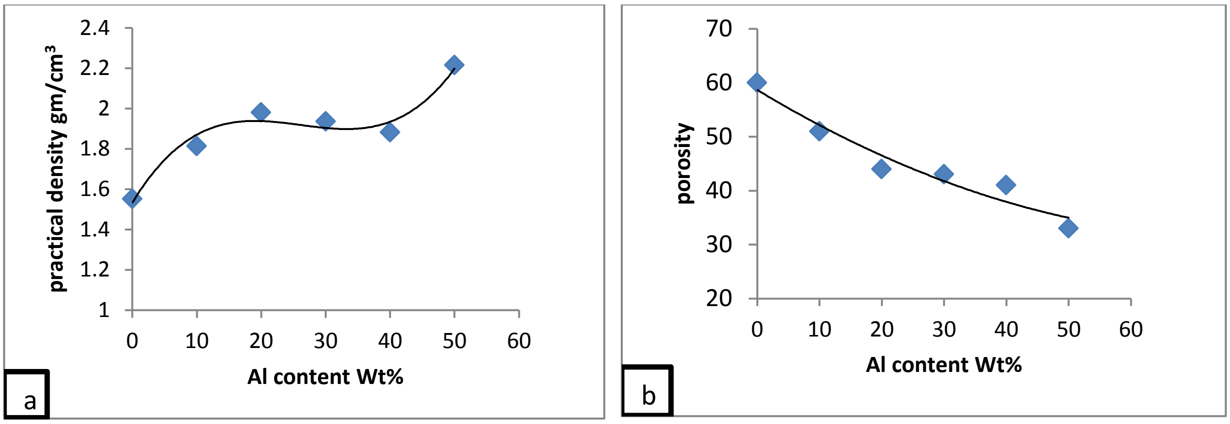


Figure 4- The effect of Al content on a- sintering density, b- porosity at 800 °C.

Figure-5(a) shows XRD of γ-Al_2O_3 powder in room temperature, three major peaks appear at 2Θ = 67.0221°, 45.7503° and 42.6421, (b) shows XRD of γ-Al_2O_3 from PCPDFWIN software no.(100425), it has three major peaks at 2Θ = 67.032°, 45.862° and 37.603°. There is good agreement in the peak positions between (a) and (b), also, it has been noticed from (a) there is no crystalline phase in alumina that indicates that the alumina is amorphous.

Figure-6(a) shows XRD of aluminum metal powder at room temperature, three obvious peaks were detected at 2Θ = 38.5605°, 44.8018 and 65.1644°, it has good match with the peaks positions in Figure-6(b) which represents XRD of Al from PCPDFWIN software no.(040787) where its three peaks appeared at 2Θ = 38.472°, 44.738° and 65.133°.

Figure-7(a) shows XRD of sintered γ-Al_2O_3 at 800 °C for 2hrs. at forming pressure (15) MPa, (b) shows XRD of (γ-Al_2O_3-50%Al) sintered at 800 °C for 2hrs. at forming pressure (15) MPa.

In Figure-7(a) the sintered alumina has different peaks positions than in Figure-5(a), where became more sharper and less peaks number than in (5a), that transformations in phases in alumina after sintering is due to thermal scattering of x-ray. So in Figure-7(a) the peak at 2Θ = 38.3693° represents γ-alumina phase while the peak at 2Θ = 44.6084° represents γ, δ-alumina phases, and the peak at 2Θ = 64.9945° represents γ, δ, Θ-alumina phases. These phases are overlapped due to their approximated densities, and that makes it harder to separate hide alumina multiphase's and that agree with [9].

In Figure-7(b) it has been noticed that there are no aluminum 3 peaks in spite of its real existence with 50% percent, that because of thermal expansion of aluminum lattice when the sintering temperature increases, Al peak positions keep moving towards lower 2Θ angles, also, Al intensities decrease with the increase of the temperature until they disappear above 690 °C that's what Dudi and others reached to [9], which concurs with this situation due to the used sintering temperature (800) °C. It have been noticed there are no new phases between alumina and aluminum due to, there is no chemical reaction between them, only the mechanism of physical adhesion between the alumina and aluminum melt.

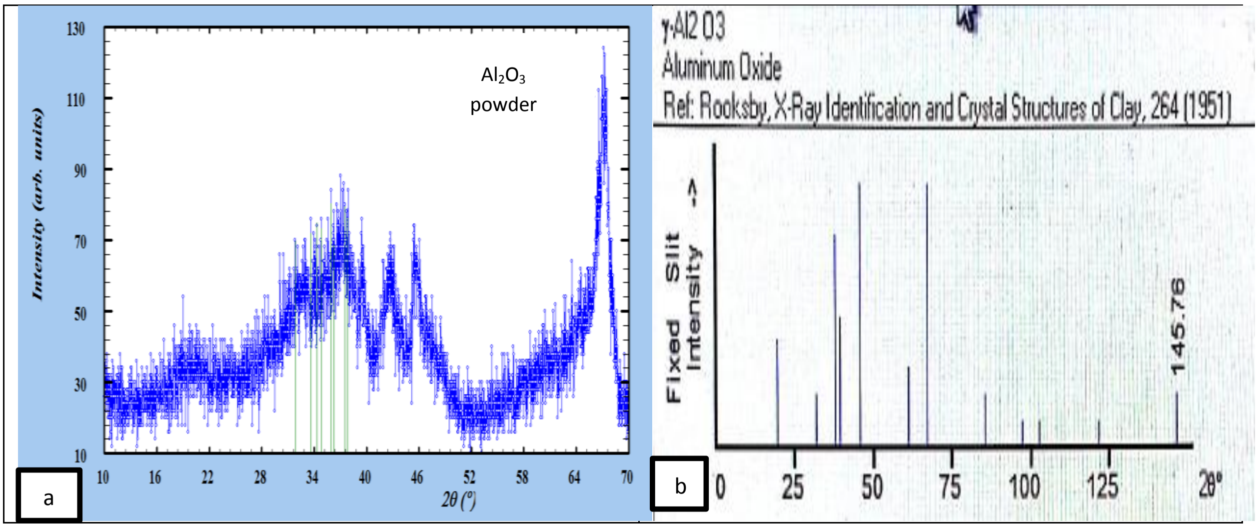


Figure 5- (a) XRD of aluminum oxide (γ-Al_2O_3) powder, (b) ASTM card of γ-alumina x-ray diffraction.

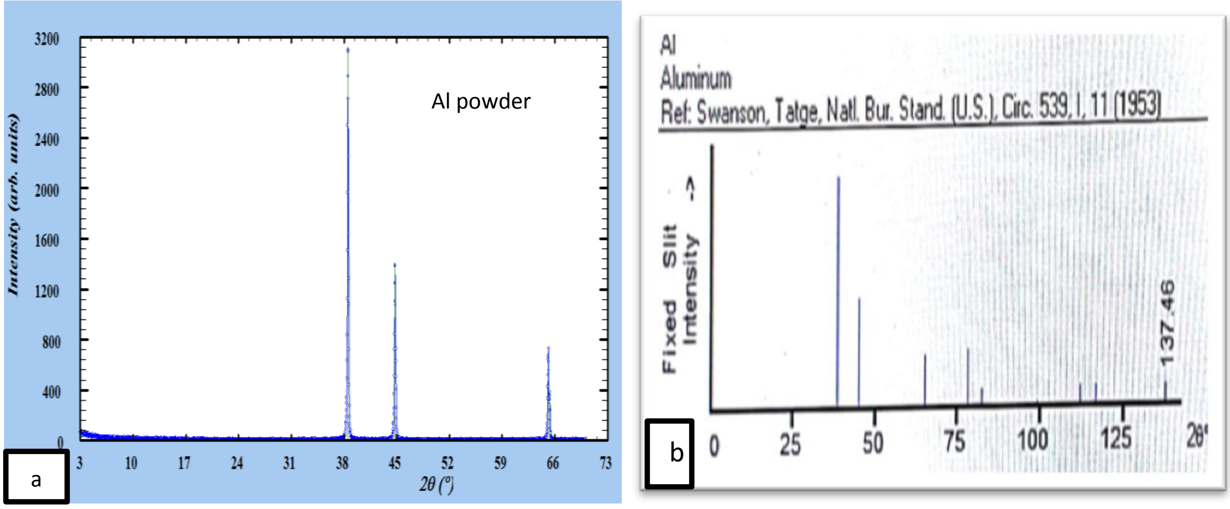
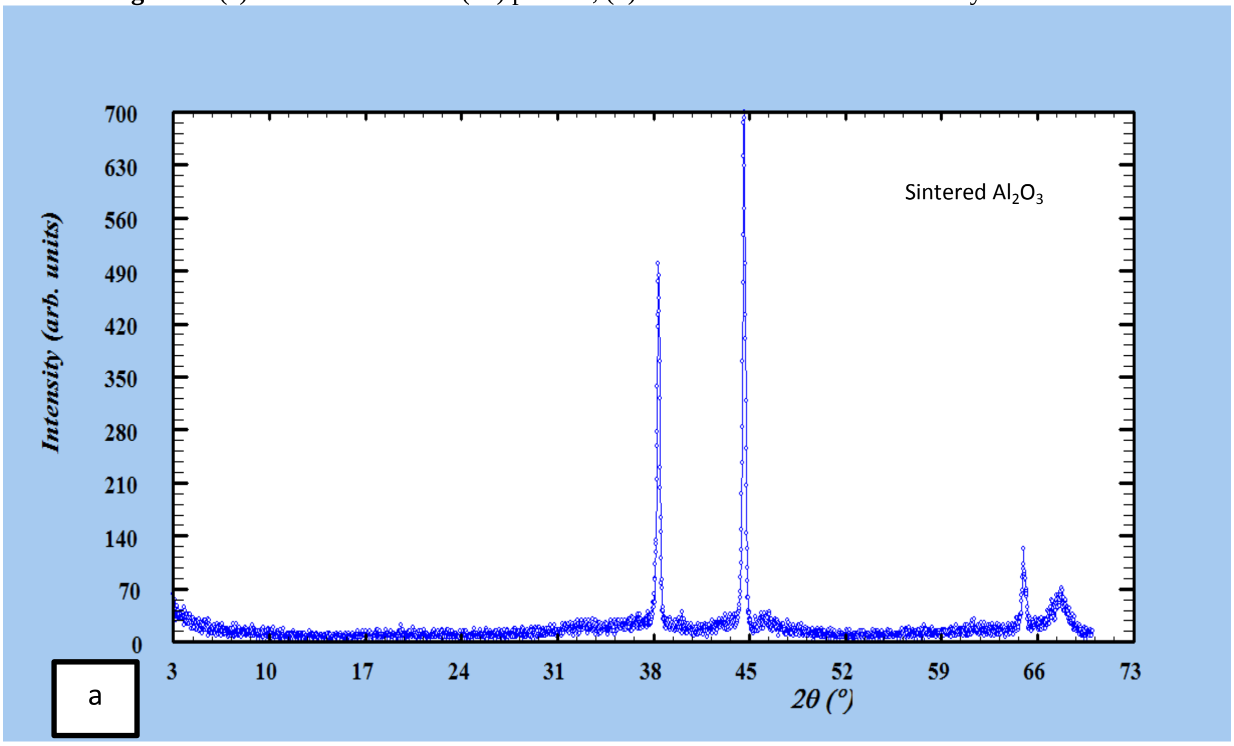


Figure 6- (a) XRD of aluminum (Al) powder, (b) ASTM card of aluminum X-ray diffraction.



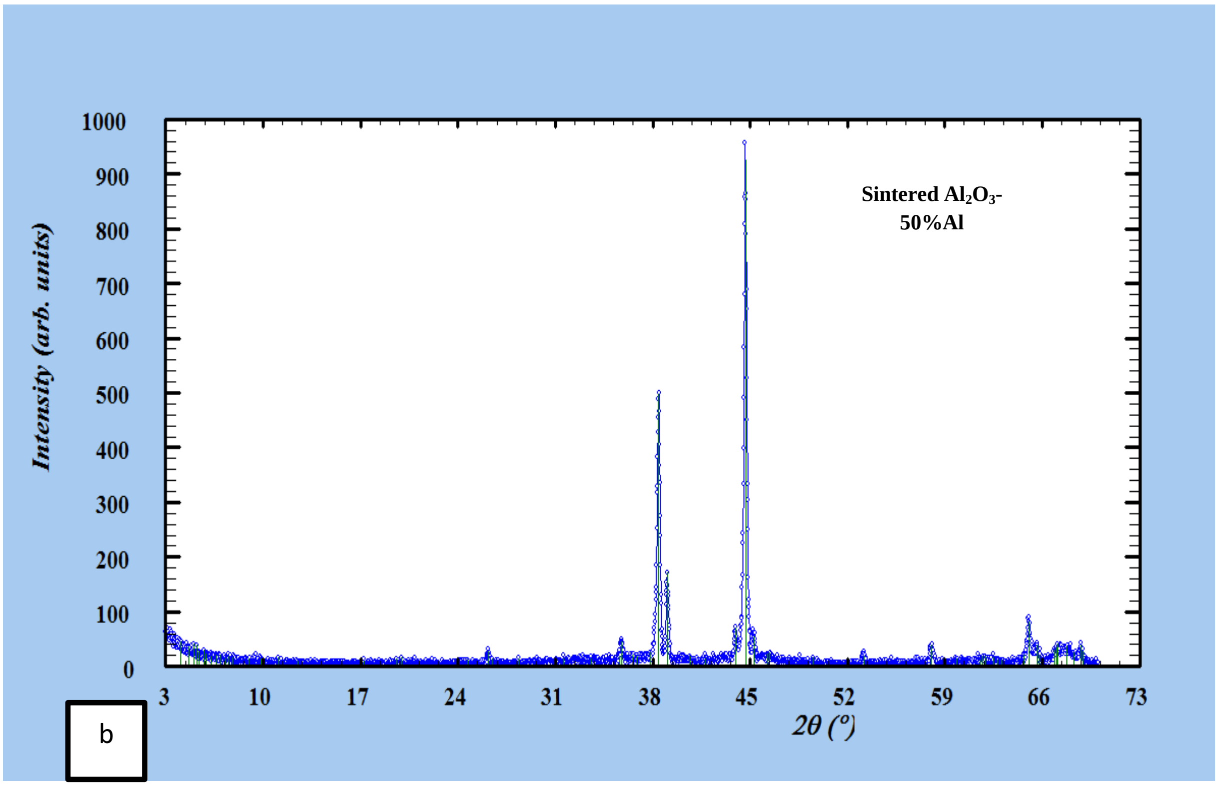


Figure 7- (a) XRD of sintered Al_2O_3, (b) XRD of sintered γ-Al_2O_3-50%Al.

Figure-8 (a) shows the result of the scanning electron microscopy (SEM) for the cermet composite (γ-Al_2O_3-50%Wt Al) pressed at 15 MPa, sintered at 800 °C for 2 hours in vacuum, from this figure its noticeable the porosity and the aluminum flowing between the alumina particles and the uniform distribution between them where they formed what looks like conducting islands (aluminum) separated by isolated areas (alumina). This is a sequent of the sintering temperature that crosses the aluminum melting point (660°C), this can be explained because of the high porosity ratio that make the metal's melt to inter the ceramic porosities because of the capillary force of the metal.

Figure-8 (b) represents the source [10] that depended on to recognize the used materials in this work.

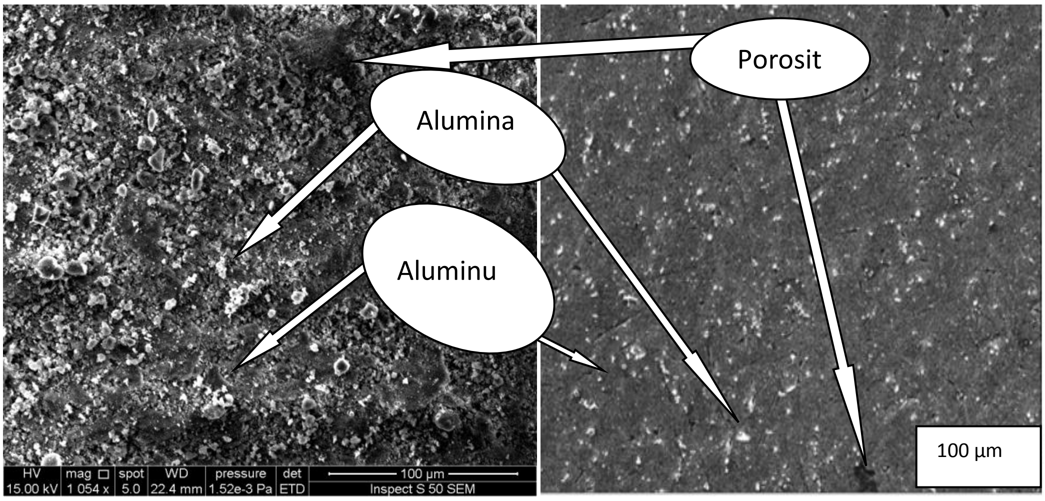


Figure 8- SEM image for γ-Al_2O_3/Al composite at 100 µm for alumina of (a) 50% wt, (b) 8% (volume fraction).

Figure-9 shows the shore D hardness results for the cermet composites prepared at 800 °C, 15 MPa, 2 hrs. . The increase of hardness with the increase of aluminum content have been noticed, which is an indicator of the increase of toughness of the cermet body with the increase of Al content, one of the reasons for this behavior could be because of the decrease in the volumetric ratio of porosities, which they act as defects inside the cermet composite body, which sometimes help to failure when the cermet body is forced to mechanical load. Its noticeable that hardness value starts to decrease after the concentration of 30%wt Al that because of Al ductility nature [6].

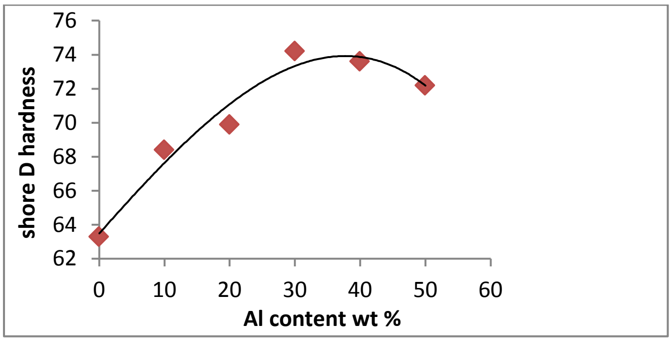


Figure 9- The effect of Al content on the shore D hardness.

The wear behavior for these composite materials has a big and direct influence on the safety of the contact parts in the rotary systems, if this behavior changed it might be effect on the efficiency of these rotary systems, so, mechanical tests should be done to check how far the wear rate is affected by other variables, like applied load, operating time….etc.

The wear test is tested on the sample which has the properties of (Al_2O_3-50%wt Al, 800 °C, 2hrs., 150 bar), this sample has been chosen because it has equal percent of Al and Al_2O_3 which means more quantity of Al than Al_2O_3 because as it known the density of Al is less than the density of Al_2O_3 [11] that enables the sample to hold itself and give the optimum wear resistance more than the other samples which have less Al percent.

This prepared cermet composite leaves wear dribs equal to (0.111) gm at time of (61) sec. during slipping on a disk of cast iron. So, the existence of aluminum in the cermet composite helps to increase this composite wear resistance , where the aluminum considers one of metals conducting to heat as is mentioned before, that results to increase the friction surfaces temperature, which makes layer of aluminum oxide on this composite interface between the rotational parts, so it is predictable to have a lower wear rate because the aluminum oxide layer is attached on the friction surfaces, which protect the friction surfaces and increase its wear resistance.

Conclusions:

1. The increase in sintering process wouldn't happen unless the chosen temperature makes the liquid phase happen.
2. Aluminum metal (Al) powder plays the role of binding material in forming process of alumina (Al_2O_3) powder, and increases the mechanical properties, where the mechanical binding process for the cermet composite compressed components was accomplished, that indicates to the forming of a structure or internal web of aluminum that binds all the cermet composite particles with each other, in addition to its role in electrical conducting.
3. Aluminum particles partially gathered together between each other to create metallic structure holds the alumina particles together inside the compacted body to give it the mechanical toughness which basically relies on the cohesion and coherence of the metal particles.
4. High percent of aluminum particles on the friction surface influence on the stability of wear rate values, that because of forming a metal oxide layer on the surface which effects greatly on the wear rates stability.

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