STRUCTURE AND SOME PROPERTIES OF COMPACTED AND SINTERED Cu- Al_2O_3 PRODUCTS

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الملخص

تم بنجاح تحضير مجموعة من المواد المركبة من النحاس (Cu) وأكسيد الالومنيوم (Al_2O_3) باستخدام تقنية المساحيق. تحتوي هذه المواد المركبة على النحاس (Cu) كمادة أساسيه لينة ومرنة للترابط وأكسيد الالومنيوم (Al_2O_3) كمادة قصفة وثانوية بنسب حجميه مختلفة تتراوح ما بين 0.0 إلى 20.0 % بهدف دراسة تأثير نسبة اكسيد الالومنيوم وزمن التلبيد على الخواص الفزيائية والميكانيكية لهذه المجموعة من المواد المركبة.

خلطت هذه المساحيق ميكانيكيا وضغطها (كبسها) باستخدام قالب أحادي تحت ضغط يصل إلى (451 MPa). تم تلبيد المنتج بعد عملية الكبس عند درجة حرارة (670 °C) في جو خامل من غاز الارجون (Argon gas) لفترات زمنية متفاوتة كما تم قياس بعض الخواص الفيزيائية والميكانيكية لعينات من النحاس (Cu) النقي وللعينات المركبة التي تحتوي على نسب مختلفة من أكسيد الالومنيوم (Al_2O_3).

أظهرت نتائج هذا البحث أن كثافة المادة المركبة تتناقص قبل وبعد التلبيد مع الزيادة في نسبة أكسيد الالومنيوم (Al_2O_3). بالإضافة إلى ذلك فإن المسامية تتزايد مع زيادة نسبة أكسيد الالومنيوم (Al_2O_3). كما لوحظ أن الزيادة في زمن التلبيد تؤدي إلى زيادة في الصلادة ونقص في المسامية.

ABSTRACT

A series of Cu-Al_2O_3 composites were successfully prepared using powder metallurgy techniques. The composites consisted of pure copper (Cu) powder (99.8 %) as a soft ductile matrix and alumina powder (Al_2O_3) as a hard second phase with volume fraction varied from 0.0 % up to 20.0 %, in order to investigate the effect of alumina volume fraction and sintering time on the physical and mechanical properties of these composites. The composite powders were mechanically mixed and pressed under single action die at a compaction pressure of 451 MPa and sintered at 670°C in an inert atmosphere (Argon gas) for different time intervals.

Some physical and mechanical properties were measured for compacted and sintered copper powder, as well as for the composites with different volume fractions of

Al_2O_3. It was observed that the green and sintered densities decreased as the volume fraction of Al_2O_3 increased. It was also observed that the percent porosity increased as the volume fraction of Al_2O_3 increased. On the other hand, a decrease in porosity and an increase in hardness were observed with increasing the sintering time.

KEYWORDS: Cu-Al_2O_3 Composites; Copper Powder; Alumina Powder; Argon Gas.

INTRODUCTION

In many engineering products, different types of phases (often called material components) are mechanically or metallurgically bonded together in order to optimize their properties and behavior under service condition.

There are two major reasons for the revived interest in composite materials. One is the ever-increasing demand for higher performance in many product areas especially in the aerospace, nuclear energy and aircraft fields. The second reason, which is the most important one on the long run, is that the composites concept provides scientists and engineers with a promising approach to design, rather than to select a material to meet the specific requirements of an application.

The overall behavior of a finished product is even more dependent on their structure than is the behavior of single-phase materials. The properties are affected by the size, distribution, and particle morphology of the phases. In other words, the properties will depend on the bond strength between different phases, the shape and amount of each phase and the properties of each phase. The dispersion-strengthened metal matrix composites have been a subject of many research articles [1-4]. Cu-Al_2O_3 dispersion-strengthened composites have good mechanical properties at room and elevated temperatures, providing superior creep resistance compared with copper alloys. Oxide dispersion-strengthened copper is an effective method to increase copper strength without remarkable decrease in electrical and thermal conductivity [4]. Mechanical alloying was found to be more suitable in producing alumina dispersion strengthened copper composites [1]. Alumina content of 5 wt.% or less was found to be suitable to achieve equilibrium in both hardness and electrical conductivity[1]. The micro-composites show better density and hardness at higher sintering temperatures. This behavior is the other way around in nano-composites. The wear resistance is higher for nano-composites than micro-composites under the same conditions [5]. In their study of the 95 % Cu with 5 % Al_2O_3 composite [6], they found that after 12 hours of milling the particle size was reduced from 2.531 nm to 1.7343 nm and the material have reached a steady state. The lattice strain has been increased as a result of reduction in particle size. Mechanical properties may also be enhanced by decreasing the particle size and increasing the lattice strain.

The objective of this investigation was to prepare a series of Cu–Al_2O_3 composites and investigate the effect of increasing Al_2O_3 volume fraction and sintering time on some physical and mechanical properties of the resulting material.

EXPERIMENTAL PROCEDURE

A series of composites were prepared using the powder metallurgy techniques. The composites prepared were metal–nonmetal particulate composites with copper being the matrix (particle size 40 μm), and the second phase was metal oxide being alumina (particle size in the range 53-150 μm).

The general formula used for preparing the mixture was (100-X) Cu, where X– expresses the volume fraction of alumina in the mixture. The amount of X varied from 0.0 % up to 20.0 %. The total weight of sample was 30 grams. The theoretical density ($\rho_{theoritical}$) of the composite was calculated for different volume fraction by using the law of mixtures. The powder mixture was mixed by magnetic stirring motor to ensure uniform distribution. The mixing time was limited to one hour to avoid over mixing which may decrease the particle size.

The mixed Cu-Al_2O_3 powders were mechanically compacted using cold pressing technique utilizing a single action compaction die. The compaction pressure was 451 MPa. All the produced compacts were in the form of a small circular disc shaped of 3.2 cm in diameter and 0.5-0.71 cm in thickness.

The final stage for obtaining the composites was the densification step. Each series of Cu-Al_2O_3 particulate composite compacts were sintered at a constant sintering temperature of 670 ºC in a protective atmosphere of argon for different sintering times of 0.5, 1 and 2 hours, the sintering temperature was selected to be 670 ºC (≈ 0.7 of the absolute melting temperature of copper). This was done to optimize the required sintering time for composites.

Physical properties measurements

The green density was calculated according to the formula:

$$\rho = m/v$$

Where:

ρ = green density in (gm/cm^3)

m= mass of compacts in (gm)

v = volume of compacts ($\pi r^2 t$) in (cm^3); r and t are the radius and thickness of the obtained disc respectively

The density of sintered compacts was determined using the same principle of calculation.

The percent porosity present within the composite disc specimens was calculated according to the formula:

$$\% \text{ porosity} = \frac{\text{Theoretical density} - \text{Sintered density}}{\text{Theoretical density}} \times 100$$

Hardness measurements

Rockwell hardness tester was used to measure the hardness of compacts sintered for various time intervals. Rockwell B scale was adopted in all cases using 10 kg as an initial load and 90 kg as the main load. Four readings were taken for each sample and the mean hardness values were reported.

Metallographic investigations

Samples were ground on different emery papers, starting with coarse grade 120, down to grade 1200. They were then polished using alumina paste. For microstructure examination alcoholic $FeCl_3$ (5 gm ferric chloride + 95 ml methanol + two drops of hydrochloric acid) etching reagent was used. The microstructure was observed and photographed by means of an optical microscope equipped with photographic facilities.

RESULTS AND DISCUSSION

Green density

The green density of the compacts was calculated as mass over measured volume. A decrease in green density with increasing volume fraction of Al_2O_3 was expected and could be justified by the rule of mixture. Where the light alumina particles ($\rho_{alumina}$= 4 gm / cm³) were replacing heavy copper particles (ρ_{copper}= 8.96 gm / cm³). Generally, the green density is lower than the calculated theoretical density as shown in Figure (1).

Sintered density

The sintered density of the produced compacts with different volume fraction of Al_2O_3 sintered for various time intervals of (0.5 hr, 1 hr, and 2 hrs) was calculated. It was observed that, with increasing alumina volume fraction, the sintered density shows a gradual reduction, as shown in Figure (1). Such reduction is due to the squeezing effect and the interface Cu /Cu, Al_2O_3 / Cu, Al_2O_3 /Al_2O_3 change. It is also clear that the densities of sintered compacts are greater than the densities of un-sintered compacts. This can be explained by the reduction in surface energy, dimension shrinkage of the composites and internal voids during sintering process [1]. Such difference increases with increasing sintering time as well as with Al_2O_3.

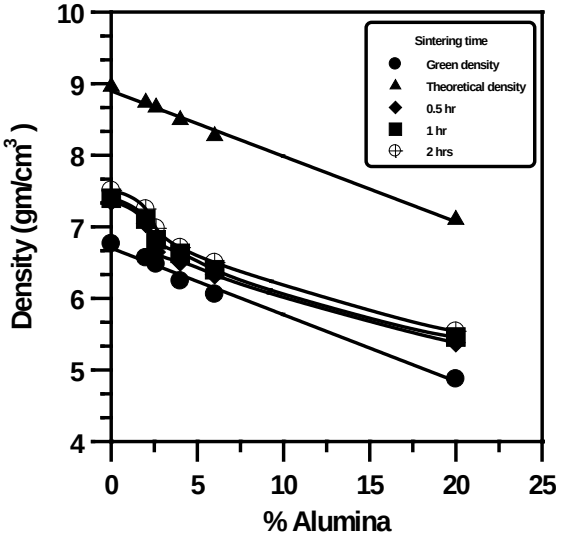


Figure 1: Effect of alumina content and sintering time on the density of produced compacts

Green porosity

The values of green porosity are mainly dependent on the load of compaction and the content of the second phase. It is evident that the first addition of the alumina phase affects the green porosity much more than further additions. Thus, the first 4 % of Al_2O_3 increases the porosity by 9 % while the further 16 % additions of alumina increase the porosity only by 7 %. This fact could be explained by the fact that the portion of the interface Al_2O_3 /Cu boundaries sharply increase on the expense of the Cu/Cu interface boundaries with the first portions of Al_2O_3 added to the powder mixture. Some boundaries may accumulate pores retarding their squeezing out during compaction. In such a way, the percentage green porosity builds up rapidly with the addition of Al_2O_3 particles.

Further additions of Al_2O_3 particles increase the probability of Al_2O_3 / Al_2O_3 interfaces and under the compaction action many of these brittle particles may fracture and fill the pores surrounding them giving rise to a lesser increase in porosity as shown in Figure (2)**.**

Sintered porosity

The amount of porosity present in the sintered product was related to its density; it decreases with increasing density. The more dense material shows lower porosity. That occurs during sintering where excessive shrinkage and green growth may cause reduction in pores size. The results are given in Figure (2).

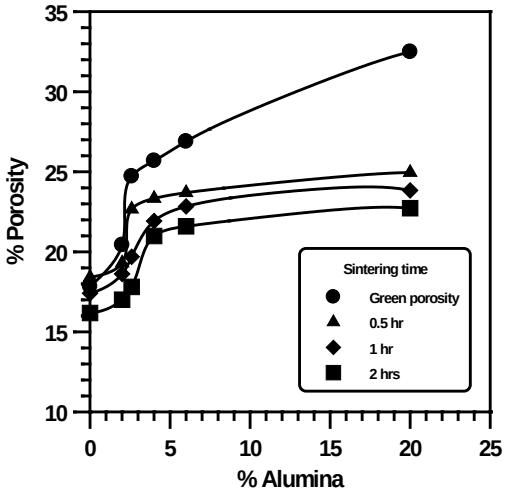


Figure 2: Effect of alumina content and sintering time on the percentage porosity of Produced compacts

Hardness

The obtained results show a slight increase in the hardness of the compacts produced with different Al_2O_3 volume fractions. It is clear that the hardness increases as the volume fraction of alumina increases as predicted in Figure (3).

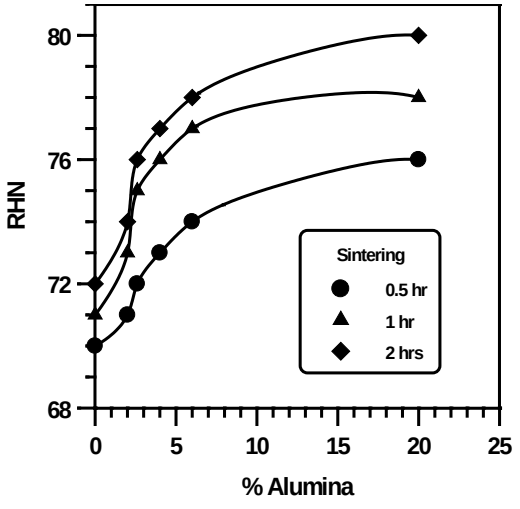


Figure 3: Effect of alumina content and sintering time on the hardness of produced Compacts

This was probably due to the effect of the relatively hard particles interface surrounded by soft copper matrix. Considering the relationship between hardness and sintering time, Figures (4), the obtained results show that hardness increases as the sintering time increases with almost the same rate. That is probably due to the decrease in porosity as the sintering time increases.

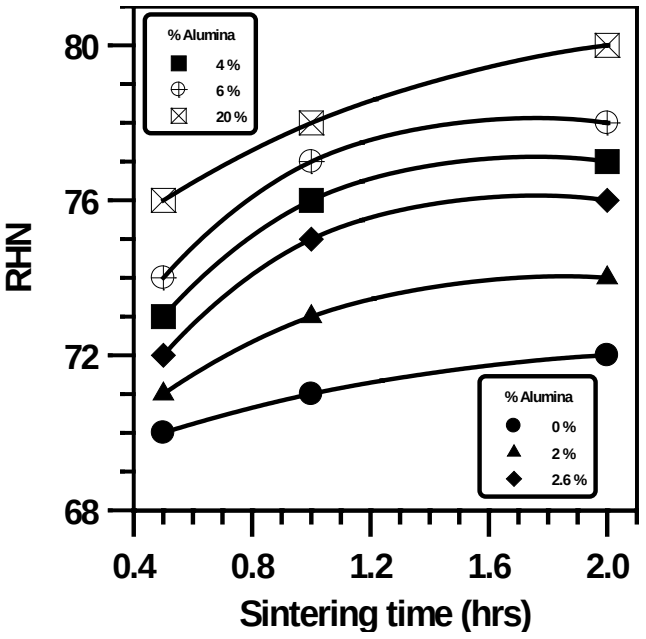


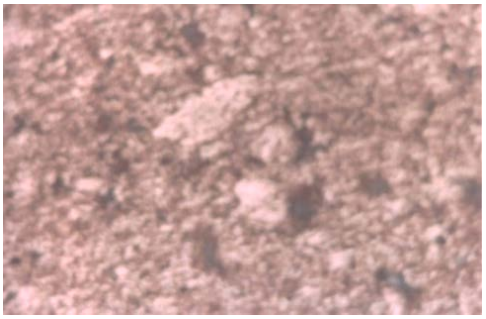
Figure 4: Effect of sintering time and alumina content on the hardness of produced compacts

GENERAL REMARKS

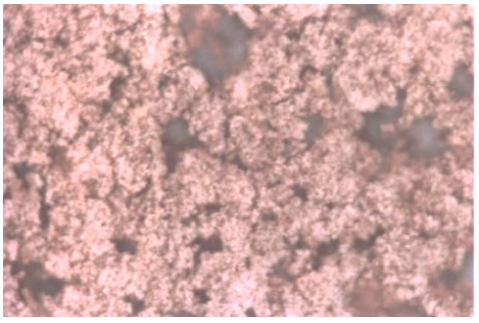
As it has been mentioned before, increasing the volume fraction of Al_2O_3 shows an increase in the hardness and porosity of the sintered product. Such increase in hardness was found to be more pronounced as the sintering time increases. This can be attributed to the decrease in porosity as the sintering time increases.

The sintered density was found to decrease as the volume fraction of Al_2O_3 increases, which is in an agreement with the increase in porosity. On the other hand, the sintered density was found to increase with increasing sintering time due to the decrease in porosity of the product.

The effect of Al_2O_3 content, on the properties of the compacts, was found to be more effective in lower percentage range and leveling off at higher percentage. That could be due to a decrease of soft matrix, which may lead to lower work hardening rate. The microstructure of produced compacts, are shown in Figures (5) and (6). A good agreement between observed changes in mechanical and physical properties was observed. The copper oxide layers form a continuous net work surrounding the matrix copper grains. Such oxide may undergo chemical changes during sintering. This oxide may also, somehow, affect the measured properties. Figures (5) and (6) show the appearance of porosity for two samples where Al_2O_3 content and sintering time increases.

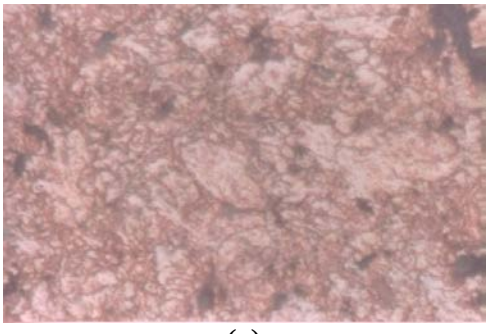


(a)

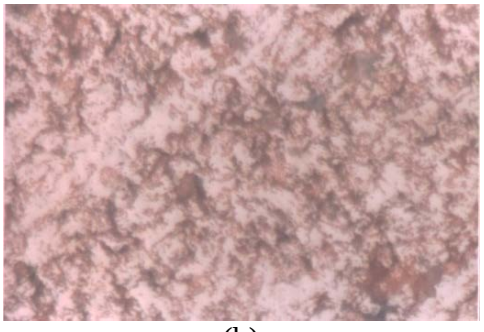


(b)

Figure 5: (a) Microstructure of 6 % Al_2O_3 sample sintered for (1 hr) (200 X)
(b) Microstructure of sample 20 % Al_2O_3 sintered for (1 hr) (200 X)



(a)



(b)

Figure 6: (a) Microstructure of 2.6 % Al_2O_3 sample sintered for (0.5 hr) (1000 X)
(b) Microstructure of 2.6 % Al_2O_3 sample sintered for (2 hrs) (1000 X)

CONCLUSIONS

Based on the results obtained in this paper, the following conclusions can be drawn:

- The green and sintered densities appear to follow a similar trend. As the volume fraction of alumina increased both green and sintered densities decrease. The theoretical values of densities also decrease as the volume fraction of Al_2O_3 increases.
- The hardness was found to increase slightly with increasing of the volume fraction of Al_2O_3.
- Increasing the volume fraction of Al_2O_3 was found to increase the percentage porosity.
- The sintering time has a minor effect on the density and hardness of sintered compacts.

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