EFFECT OF SINTERING TEMPERATURE ON PHYSICAL PROPERTIES AND CORROSION BEHAVIOR OF COMPACT (AL-4.5%CU- 1.5%MG) ALLOY

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ABSTRACT:

This work aims to study the effect of sintering temperatures on physical properties (density and porosity) and corrosion behavior of compact(Al-4.5%Cu-1.5%Mg) alloy in 3.5% NaCl solution at room temperature using cyclic polarization method. Powder technology was used to prepare this alloy by mechanical mixing of Al, Cu and Mg powders in a ball mill for 6 hours to ensure a homogenous distribution of powders in the mixture. Mixed powder was compacted by using single action pressing at (450 MPa) then followed directly by sintering process at different temperatures (520,540,560,580 and 600ºC) for one hour in vacuum furnace under the effect of inert gas condition. Density ,porosity and microhardness measurements were performed to find the best sintering temperature. Tafel extrapolation method and cyclic polarization tests were conducted for all sintered samples. It was shown that the highest density and micro hardness were at sintering temperature 560ºC. It was found that the pitting corrosion resistance enhances with increasing sintering temperature up to 560ºC and afterword it drops again at higher temperatures (580 and 600ºC).

Keywords: sintering temperature ,powder technology, corrosion, Al-Cu-Mg alloy.

دراسة تأثير درجة حرارة التلبيد عل الخواص الفيزياوية والسلوك التاكلي لمكبوسة من سبيكة (ألمنيوم- 4.5% نحاس –1.5% مغنيسيوم)

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الخلاصة :-

يهدف هذا البحث الى دراسة تأثير درجة حرارة التلبيد على الخواص الفيزياوية (الكثافة والمسامية) والسلوك التاكلي لسبيكة مكبوسة من (ألمنيوم –4.5% نحاس-1.5% مغنيسيوم) في محلول 3.5% كلوريد الصوديوم في درجة حرارة الغرفة بطريقة الاستقطاب الحلقي (الدوري) باستخدام جهاز المجهاد الساكن . حيث تم خلط المساحيق من الألمنيوم ،النحاس ،المغنيسيوم بواسطة طاحونة الكرات لمدة 6 ساعات للحصول على مسحوق متجانس. تم كبس الخليط بعدها بضغط أحادي الاتجاه تحت ضغط (450MPa) بواسطة مكبس هيدروليكي . لبدت المكبوسات بدرجات حرارة مختلفة ولمدة ساعة واحدة لكل درجة حرارية في فرن كهربائي مفرغ (في جو غاز الاركون). تم قياس الكثافة والمسامية والصلادة الدقيقة للعينات وكذلك اجري فحص التآكل بطريقة تافل للاستقطاب والاستقطاب الدوري للعينات. لوحظ أن أعلى كثافة حقيقية وصلادة مجهرية كانت عند درجة تلبيد (560 ºC). وكذلك لوحظ ان مقاومة التآكل النقري للسبيكة الملبدة تتحسن مع زيادة درجة حرارة التلبيد إلى حد (560ºC) ثم تنخفض بعد ذلك عند الدرجات الحرارية الأعلى منها .

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1- INTRODUCTION :-

Aluminum alloys are used in advanced applications because of their combination of high strength, low density, durability, machinability, availability and the cost is also very attractive compared to competing materials **[Girisha& Sharma,2012]**. The (Al-Cu-Mg) alloy considered one from the high strength alloys which is used in many engineering applications which needs high strength like gears, rotating shafts, pins, valves, bolts, nuts and other parts of aircrafts and computer structures.This alloy distinguished by good corrosion resistance and could be improved by heat treatments which could give more strength and hardness **[Khairia,2012]**.The production of (Al-Cu-Mg) alloy by casting process have widely used in different industries but the casting method including many defects, such as porosity, cracks and inclusions. Therefore the production of(Al-Cu-Mg) alloys by powder metallurgy which have many advantages and uses in engineering industries. Aluminum (P/M) parts are used for their improved material characteristics or in some cases (e.g. complex shapes) because of their lower production cost. In most of the applications the P/M parts are used because of one (or more) of the following properties like high Young's modulus, low density, high room temperature strength, better high temperature strength and better wear resistance **[Sudhichan , 2014]**. Other applications are also found in automotive components. The need for light, corrosion resistant materials to get a strong, higher temperature materials and this type of materials promote the interest for P/M aluminum parts. The strong point of P/M aluminum parts is probably the wide variety of alloy compositions which can be prepared, offering in principle the possibility of achieving a desired combination of properties**[Smith,1981]**. A lot of these alloys are currently still under investigation.

[Mohammed Sallab ,2002] investigated to design dies for parts made from powder metallurgy and to produce different alloys including Al-Cu and Al-Cu-Si.It has found that sintered density was greater than green density, because of dimensional changes during sintering process and the Al-4.3Cu alloy gave optimum properties among the produced alloys **.** **[Suhalya Noori,2006]**studied the effect of the precipitation heat treatment on the corrosion resistance of (Al –Cu-Mg) alloys in 3.5% NaCl solution. It was found that the corrosion resistance decreases (or increase corrosion rate) of (Al –Cu-Mg) alloys in 3.5% NaCl solution after performing the precipitation heat treatment and artificial aging at 190C°.This is due the presences of precipitated hardened phases of (Al_2Cu) and (Al_2CuMg) distributed in aluminum matrix which act as cathodic sites and increase aluminum dissolution lead to pitting corrosion.

[Cooke et al.,2012]developed an Al–Cu–Mg alloy with a low Cu/Mg ratio suitable for press and sinter powder metallurgy (PM) processing. It was found a novel Al–Cu–Mg alloy with a low copper to magnesium ratio was successfully developed and processed through press and sinter PM technology.

[Raji,2014]investigated the influence of density on Al-Cu composition during compaction process using powder metallurgy. It was found the effect of tapping on powder composition for various taps showed an increase in the tap density. There are many researches and development works have been done to study the effect of powder technology process (mixing , compaction and sintering) variables on physical and mechanical properties such as mixing time, compact pressing ,time and sintering temperature**[Dhokey et al.,2013]**,[**Hogg**,**2009],[Li Sun et al . ,2009]**.

The relationship between sintering temperature and corrosion behavior of (Al –Cu-Mg) alloy and also the relationship between true density and corrosion rate or pitting corrosion parameters (ipass,Epass, Eb,ib, Ep, ip) during the cyclic polarization test. The aim of this work is study the effect of sintering temperature on corrosion behavior of compact (Al – 4.5%Cu-1.5%Mg) alloy in 3.5% NaCl solution.

2- EXPERIMENTAL WORK :-

Material used

The materials used for preparation of compact (Al –Cu-Mg) alloy are Al powder, Cu powder, Mg powder. The materials properties used are listed in Table (1).

Sample Preparation

Mixing is the most common pre–compaction step in powder metallurgy. Theoretically any composition can be prepared starting from elemental powders. In this experiment 9.4 g of aluminum powder is mixed with 0.45 g of copper and 0.15 gm magnesium powder with addition of zinc stearate of (0.5%) as a binder and a lubricant mixture. Mixing was carried out in a ball mill type (Jar rotating by motor) which have alumina balls. The number of balls are 20 balls with ratio 20:1 weight of powder to weight of balls for mixing time of 6 hours at speed of 650 rpm in order to get a homogeneous mixture. The homogeneous mixing will improve the sintering ability, ejection of compact and strength of the compact. Uniaxial cold compaction process was carried out on mixed powders to obtain good compaction and to produce the green billet with few porosity. More than one compaction experiment was made at different pressures of (150,250,350 and 450)MPa to choose the appropriate pressure. The powders were pressed at (450MPa) for all samples used in this work. A cylindrical one direction action die with 20mm hole diameter and (20 ton) capacity of electric hydraulic press, for, incubation time(15 min) in each applied pressure was used. Sintering process was carried out in electric tube furnace type (MITI CORPORATION GSL)with maximum temperature of (1600^{o}C) with using argon of purity 99.99%. **Figure (1)** shows heating and cooling cycle of sintering process which was carried out in sintering furnace with Ar flow rate 2L/min and at different temperatures of (520,540,560,580 and 600) ºC.

3- TESTS AND INSPECTIONS :-

3.1 Density and Porosity Measurements

The final true density and porosity for all sintered samples were measured according to ASTM D 792 standard which is based on Archimedes principle. The specific gravity of material is given by equation(1) and the porosity is given by equation (2) **[Muna& Mohammed ,2015].** The sample was first weighed in air, and then immersed and weighed again in a liquid with a known density, and the density of the sample was finally calculated from equation (1) and equation (2) for porosity. This process was carried out at room temperature, and the auxiliary liquid used was water (density = 1 g/cm^3**).**

$$\text{Sp.Gr for sample} = \frac{W1}{W1-W2} \times Sp.Gr - - - - - (1)$$

$$P = \frac{W3-W1}{w3-w2} \times Sp.Gr - - - - - - (2)$$

where:-

P = porosity of sample

w_1 = weight of sample in air (g)

w_2 = weight of sample suspended in liquid (g).

w_3= weight of wet sample i.e. weight of soaked sample air(g).

$Sp._{Grfor\ sample}$= the specific gravity of material

3.2 Microstructure and Microhardness

Sintered samples were prepared for microstructure examination by sequence processes. A grinding process was done by using SiC paper of 320, 500,800 and 1000 with using water as a coolant. Polishing process was carried out with using special cloth and (1 µm) diamond paste and lubricant. Etching process was carried with using etchant which consisted of 1% hydrofluoric acid and 99% distilled water. Then the samples were washed by water and alcohol and dried in hot air . Optical microscope type (MT9000 MEIJI) ,made in Japan was used to study the microstructure of prepared samples at different sintering temperatures. Micro hardness Vickers tester type (Leco Micro Hardness Tester LM248AT) was used to measure microhardness of all samples. The applied load was 1.96 N for loading time of 15 sec. Three – five indentations were made on each specimen and the average reading was taken to find the Vickers hardness**.**

3.3 Electrochemical Corrosion Test

In this work, to evaluate the corrosion parameters of the samples of(Al-Cu-Mg) alloy at different sintering temperatures (520, 540, 560, 580 and 600)C° in 3.5%NaCl solution. The sample with surface area of (1cm2) put in electrochemical cell of 1000ml capacity which consists the three electrodes. The working electrode (W.E) is sample of the corroding metal with 1cm2 exposed to solution. The counter or auxiliary electrode (A.E) is generally an inert conductor, platinum rod, with (100x10)mm dimensions . The reference electrode or standard hydrogen electrode (S.H.E)is used in measuring the working electrode potential . The range of the cyclic polarization test was (-250) ,(+1000mV,+1500)below and above the open circuit potential respectively**.**

During cyclic polarization test scan rate was 5mV/sec. The electrochemical corrosion tests by Tafel extrapolation method and cyclic polarization tests were carried out by using a WENKING MLabmulti channels and SCF-MLab .Corrosion measuring system from Bank Electronics-Intelligent controls GmbH, Germany 2007.The electrochemical cell is present in the Ministry of Science and Technology –Bagdad as shown in Figure (2). Corrosion parameters are determine such as Ecorr and Icorr in order to calculate the Corrosion rate(C.R) from equation as follows[Hintze &,Calle ,2006].

$$CR(mpy) = 0.13 \times Icorr \times (EW/\rho) \quad (3)$$

where

CR(mpy) = corrosion rate by units (mils per year)

Icorr= The corrosion current in (µA/cm2)

EW = The equivalent weight in (grams/equivalent)

ρ = Density of the corroding specimen (g/cm3)

4- RESULTS AND DISCUSSION :-

4.1 Density and Porosity

Table(2) shows the results of density and porosity% for all sintered samples of (Al-4.5%Cu-1.5%Mg)alloy at different sintering temperatures. It was seen that the higher sintering temperature(560ºC) gives higher sintered density due to more driving force for sintering which has resulted from residual stress introduced during mechanical mixing. During sintering ,the diffusion between particles occurs. It is generally agreed with researches that the primary driving force for sintering is reduction of surface energy ,it own individual surface so the total surface area confined in the compact is very high. Under the influence of heat the surface area is reduced through the formation and growth of bonds between the particles with associated reduction in surface energy. The finer the initial powder size, the higher the total surface and the greater the driving force behind the process **[Hao Yu, 2010]**. Sintering involves mass transport to create the necks and transform them into grain boundaries. The principle mechanism by which this occurs is diffusion and other possible mechanisms include plastic flow. Shrinkage occurs during sintering as a result of pore size reduction. This depends to large extent on the density of the green compact which depends on the pressure during compaction**[Singer,1963]**. It was found that the better density and less porosity were at 560ºC while decrease density and increase porosity were at higher temperatures at 580ºC and 600ºCis due to grain growth. It was shown the result of X-ray diffraction analysis(XRD) of sintered sample at (560ºC)indicates the successfully the sintering process through appeared phases (Al_2CuMg) and ($Mg_2Cu_6Al_5$) ,as shown in **Figure(3) .**

4.2 Microstructure and Microhardness

Figure (4-a&b) shows the optical and SEM micrographs of sintered sample of (Al-4.5wt%Cu- 1.5%Mg) alloy at the sintering temperature of (560ºC) respectively. The best or consistent density and microhardness were achieved for compact sintered samples at (560 ºC). It is believed that has been sufficient time for appreciable densification through conventional mechanisms such as particles rearrangement and enable Cu atoms and Mg atoms to diffuse into Al matrix so as to form solid solution of (α-Al phase) and harden the alloy . There is different grain sizes and clear grain boundaries of α-Al phase.

Figure (5) shows the variation in the microhardness with sintering temperatures .It was seen that the compact sample of (Al-4.5wt%Cu-1.5%Mg) alloy has the highest microhardness (89HV) at 560ºC. This is due to that the increasing sintering temperature gives higher microhardness due to improved sinterability as a result of inherent residual stresses which acts as driving force for sintering . The gradual increase of microhardness with increase in sintering temperature up to560ºC because of it enhances the diffusion of atoms across the particle interface at sintering temperature of 560 ºC and then the microhardness hardness decreases at higher sintering temperatures at 580 and 600ºC. This is due to reducing the density, whereas increase in density leads to increasing the microhardness. These results are in agreement with **[Muna &Mohammed**, **2015].**

5 - CORROSION RESULTS :-

5.1 - Open circuit potential-time measurements

Figure (6)shows the open circuit potential (Eocp)values for all sintered samples of base alloy(Al-4.5%Cu-1.5%Mg) at different temperatures (520, 540, 560, 580 and 600ºC).This Figure indicates that the potential began at (-650 mV) for sintered sample of at (520ºC) and then it reaches to stability at (-667mV). While the sample sintered at (540ºC) reaches to stable potential at (-720mV) and then increases to(-630mV) at (560ºC) and gradually increases to stable at (-812mV) at (580ºC). The sample sintering temperature of (600ºC) gave Eoc more negative than sintered alloy which reaches stable value at (-820 mV). During anodic or cathodic polarization, or at open circuit, ion transfer reaction of metal ions and oxygen ions will take place. The potential reaches to steady state due to formation of passive oxide film of Al_2O_3on surface. The rate of passive film dissolution depends on the local potential drop at the interface, the pH and the activity of the metal ions at the oxide surface .These are confirmed by **[Rana et al.,2014].**
In the present work ,the presence of Cu, Mg in Al- α phase of alloy and also intermetallic phase Al_2CuMg ,$Mg_2Cu_6Al_5$(as indicated by XRD) as cathodic sites which encourage the dissolution of protective oxide film and enhance the pitting corrosion .These results are confirmed by **[Blanc et al.,2006]**who indicates that reactively of Cu ,Mg Intermetallic compounds play in homogenous dissolution of these compounds and copper re-deposition followed by local dissolution of the surrounding Al matrix according to the following equations :

$$\mathbf{Al \rightarrow Al^{+3} + 3e^-} \quad (4)$$

$$\mathbf{Mg \rightarrow Mg^{+2} + 2e^-} \quad (5)$$

$$\mathbf{Al_2Cu \rightarrow Cu^{\bullet} + 2Al^{+3} +} \quad (6)$$

$$\mathbf{Al_2Cu \rightarrow 2Al^{+3} + Cu^{+2} + 8e ,} \quad (7)$$

Many other researchers **[Suhalya Noori,2006],[Hintze &,Calle ,2006]** discussed the role of intermetallic particles such as Al_2CuMg and Al_2Cu as initiation sites for pitting corrosion.

5.2-Potentiodynamic Polarization

Table (3) show the corrosion parameters results of (Al-Cu-Mg) alloy at different sintering temperatures. It was seen that the corrosion rate increases with increasing sintering temperature. The sintering temperature elevation leads to an increase in corrosion rate up to 560ºC and then the corrosion rate decreases again at higher sintering temperatures (580 and600ºC). This is due to combined effect including the dependence of reaction kinetics on temperature and the higher diffusion rate which increase the porosity and voids with increasing temperatures. It was noted that there is relationship between porosity% and corrosion rate .The sample sintered at the temperature of 560ºC contains less porosity(16%) and higher density(2.7 g/cm^3) than the samples sintered at other temperatures (520 ,540, 580 and 600 ºC). While the sintered sample at 600ºCcontains more porosity(19.3%) and less density (2.62g/cm^3) as compared to other sintering temperatures, therefore the corrosion rate of this sample was greater than the samples sintered at other temperatures. It was found that whenever increases porosity increases corrosion rate. This is due to the penetration of corrosion solution in these pores which act as pits or pits initiation leading to corrosion rate increased more rapidly at higher temperatures.

5.3 Cyclic Polarization Results

Figures (7 to 11) show the cyclic polarization curves of compact alloy(Al-4.5%Cu-1.5%Mg) at different sintering temperatures (520 ,540 ,560 ,580 and 600ºC)respectively. These figures indicates that the potential for the reverse scan curves are more positive than that for the forward scan .This is due to the stable oxide film formed on the surface during the forward scan of alloy and hysteresis loop(energy for pitting)was decreased. It was observed that **Epit** of sample sintered at 560Cº shifts toward less negative potential (more noble) than the samples sintered at other temperatures while the $\mathbf{E_{break}}$ shifts to more positive potential (+1250 mV) than other samples. That means large passive region in anodic polarization curve.

5. 4 Micrographs after Corrosion

Figure (12-a and b) shows the micrographs of sintered alloy before and after electrochemical corrosion test respectively. The microstructure contains a homogeneous grains of α-Al phase. While this sample after corrosion test contains large pits. This effect is attributed to the preferential dissolution of aluminum from the pit region leading to the formation of Cu – rich surface inside pits. Also the surface of this alloy displays black marks after the first few hours of corrosion test according to the chemical reaction:

$Cu + ½ O_2 \rightarrow CuO$

6 - CONCLUSIONS :-

1 - Powder technology method was successfully applied to prepare (Al-4.5%Cu-1.5%Mg) alloy.

2 - XRD analysis indicates the existence of precipitation of second phases such as ($Mg_2Cu_6Al_5$) and (Al_2CuMg) in aluminum matrix of sintered alloy at temperature 560°C.

3 - It was shown that the best sintering temperature was at 560C°for the compact(Al-4.5%Cu-1.5%Mg) alloy which gave the highest density and microhardness as compared with other sintering temperatures .

4 - It was shown that corrosion rate of sintered sample at (560°C) was the lower than other samples which sintered at temperatures of 540, 540 ,580 and 600°C.

5 - It was found that the sample sintered at 560°C has the best pitting corrosion resistance (or less pitting energy) as compared with the samples sintered at other temperatures.

6 - During reverse scan in anodic polarization curve shows the hysteresis loop decreases as sintering temperature increases up to 560°C and then it increases at higher temperatures .

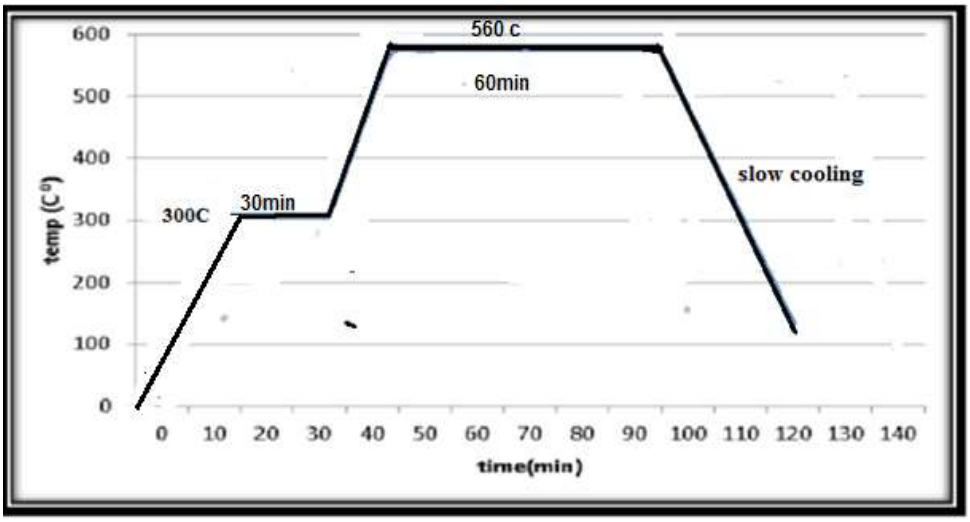
Table (1) : The materials properties used as different powders

Powder	Purity%	Average particle size(μm)
Aluminum	99.98	34.56
Copper	98.50	21.28
Magnesium	99.98	44.04

Table (2): Density and porosity results of compact alloy (Al-4.5%Cu-1.5%Mg) after sintering at different temperatures

Sintering Temperature(C°)	Density g/cm^3	Porosity%
520	2.63	17.5%
540	2.65	17.2%
560	2.70	16%
580	2.64	18.2%
600	2.62	19.3%

Table(3): Corrosion parameters results of base alloy at different sintering temperatures



Condition / Parameter	520°C	540°C	560°C	580°C	600°C
Ecor mV	-704.5	-711.5	-700.2	-758.9	-823
Icor $\mu A.cm^{-2}$	181.14	195.29	177.18	215.53	279.67
C. R(mpy)	91.7	99.0	89.7	109.0	141.4
Eo	-666.9	-720	-630	-812	-820
io	$1.2x10^{-2}$	$1.3x10^{-2}$	$1.3x10^{-2}$	$1.2x10^{-2}$	$1.3x10^{-2}$
Epass	-625	-650	-600	-1000	-600
ipass	$6x10^{-1}$	$8x10^{-2}$	$2.5x10^{-2}$	$1x10^{-2}$	$3x10^{-1}$
Ebreak	1400	1200	1250	1000	1200
ibrek	$1.5x10^{0}$	$4x10^{-1}$	$5x10^{0}$	$1.1x10^{-1}$	$1.7x10^{-1}$
Erep(Epit)	-550	-590	-500	-950	-600
irep(ipit)	$3.5x10^{-1}$	$1.2x10^{-1}$	$1.2x10^{-1}$	$1.5x10^{-2}$	$4x10^{-1}$
Pitting Energy	3.1	2.2	1.4	6.8	8.5

Fig.(1): Heating and cooling cycle used in sintering process

Fig.(2) : Electrochemical corrosion cell used in this study

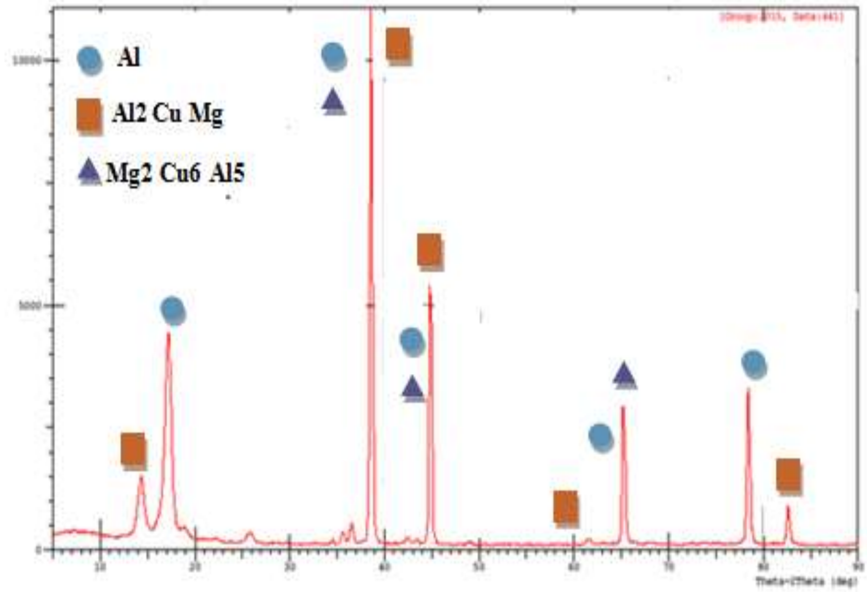


Fig. (3): XRD analysis results of sintered sample of (Al-4.5%Cu-1.5%Mg) at 560ºC

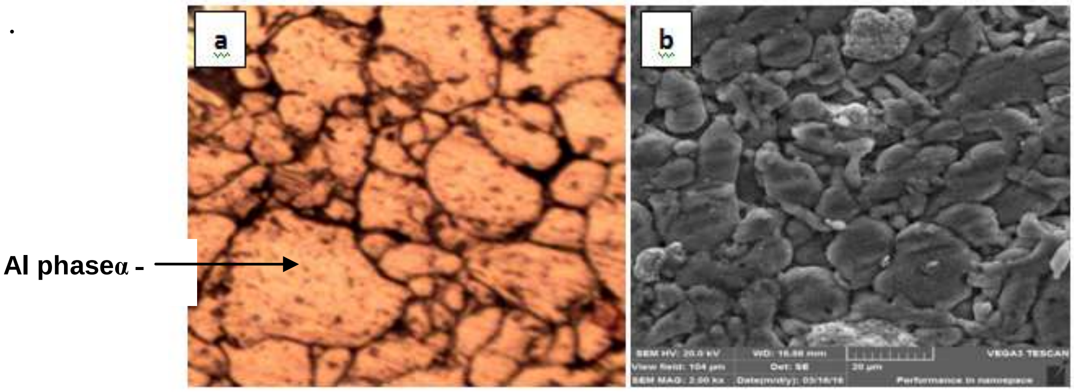


Fig.(4): Optical and SEM micrographs for sintered sample of (Al-4.5wt%Cu- 1.5%Mg) alloy at 560C°, a)Optical micrograph at 100x , b) SEM micrograph

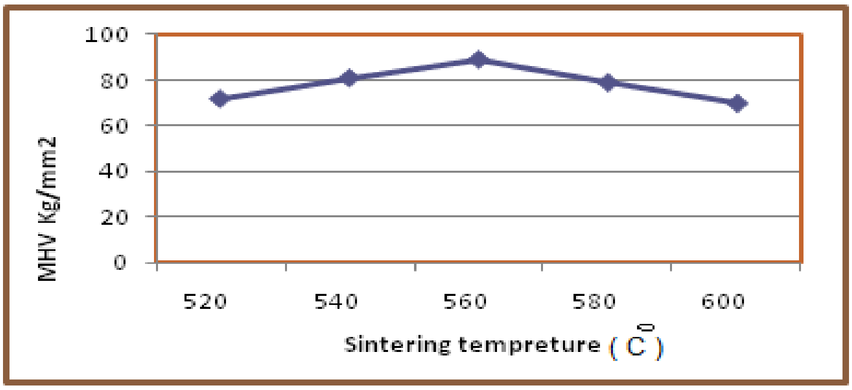


Fig.(5): Relationship between sintering temperature and microhardness

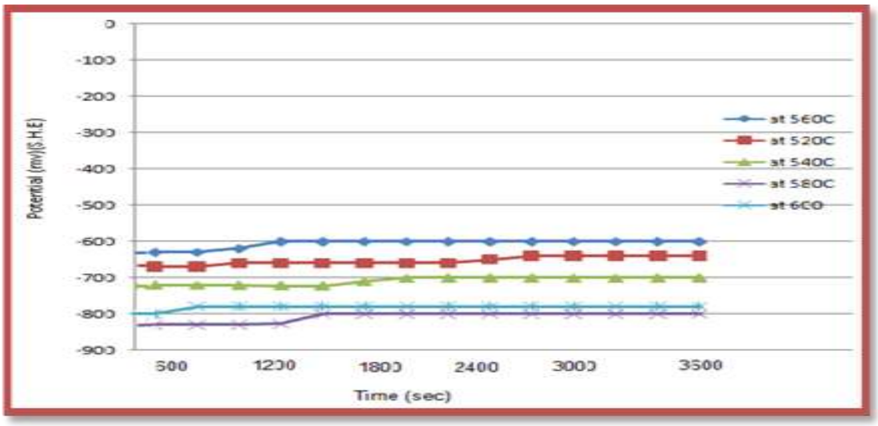


Fig. (6): Variation of potential - time of Al-Cu-Mg alloy at different sintering temperature

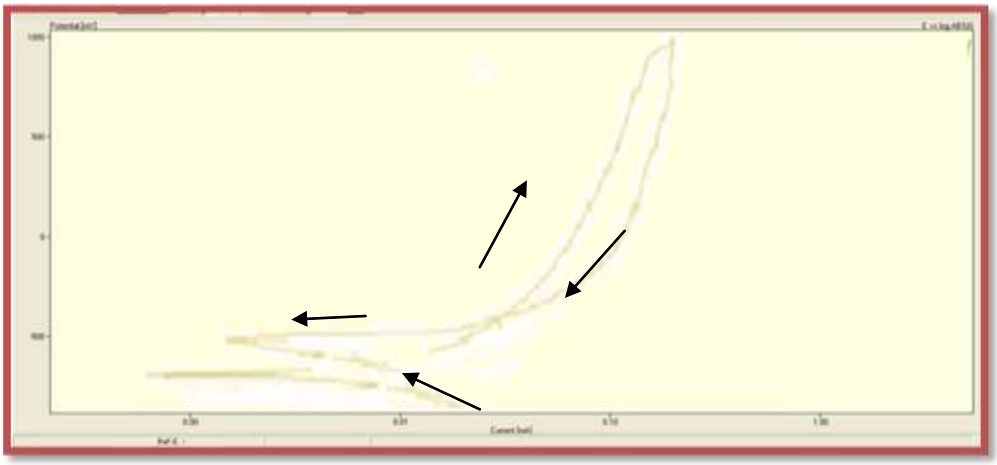


Fig. (7): Cyclic polarization curve of sintered alloy(Al-4.5%Cu-1.5%Mg) at 520C°

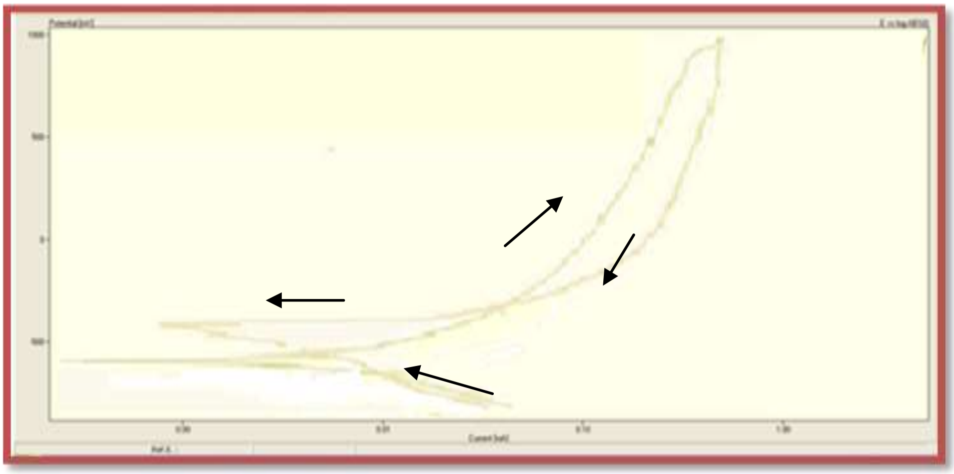


Fig. (8): Cyclic polarization curve of sintered alloy(Al-4.5%Cu-1.5%Mg) at 540C°

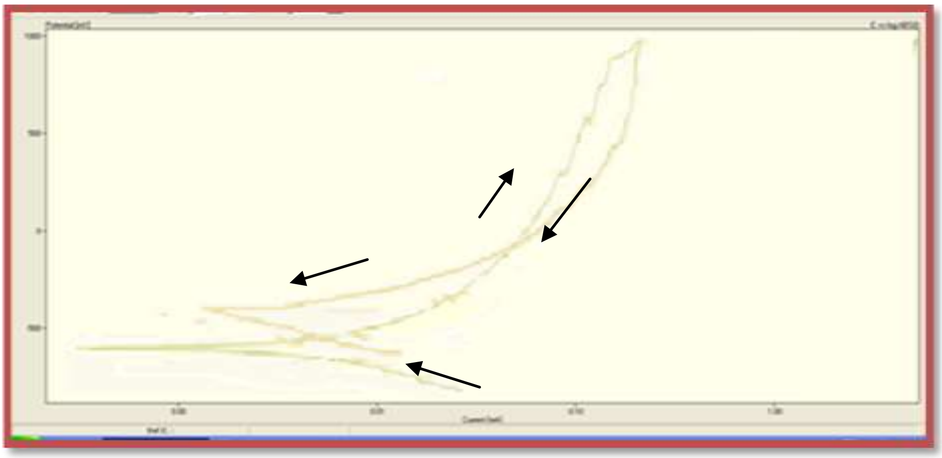


Fig. (9): Cyclic polarization curve of sintered alloy(Al-4.5%Cu-1.5%Mg) at 560C°

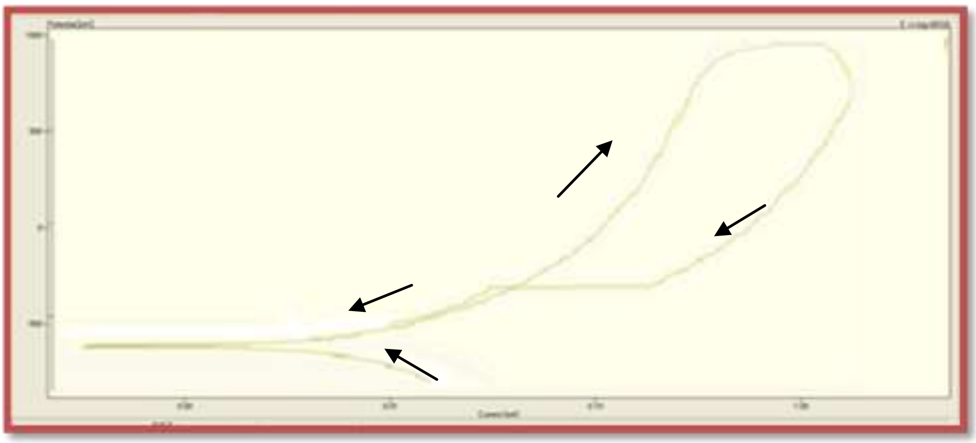


Fig. (10): Cyclic polarization curve of sintered alloy(Al-4.5%Cu-1.5%Mg) at 580C°

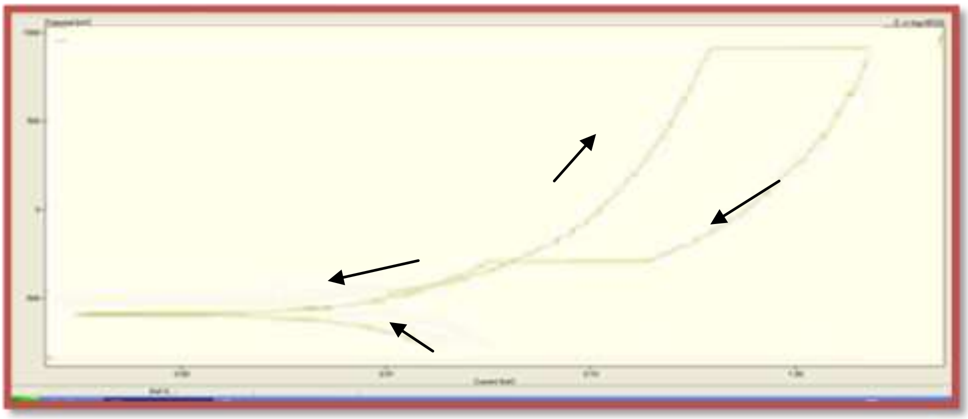
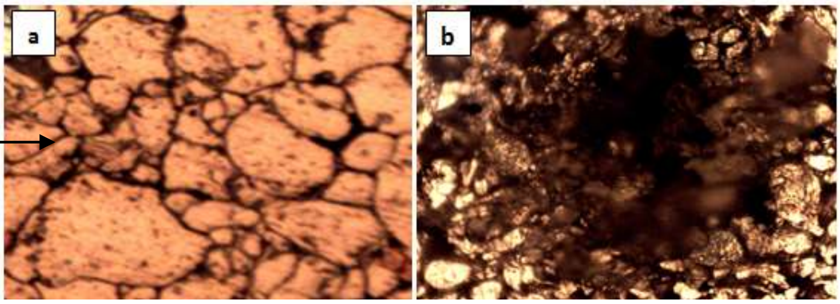


Fig. (11): Cyclic polarization curve of sintered alloy(Al-4.5%Cu-1.5%Mg) at

Al- α phase



(a)before and **(b)**after corrosion test, 100x

Fig. (12): Micrographs by optical microscope for sintered sample of (Al-4.5wt%Cu- 1.5%Mg) alloy

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