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PORE FORMATION DURING SINTERING OF TWO DIFFERENT COMPOSITES MATERIALS(Fe-Cu)&(Fe-Cu-C) AND ITS EFFECTS ON WEAR BEHAVIOR

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Abstract: The effect of pores in Fe- Cu and Fe-Cu-C generated by sintering with solid state has been studied. The additive alloying elements Cu & C and the size of the pores were affected on wear rate, there for the carbon reduces wear rate, and larger pore lowing wear rate. Geometrical distributions of the secondary pores were independent on Cu and C fraction used. Structural materials sintered with solid state and Argon atmosphere (Ar), it seems therefore desirable to use as fine alloying powders as possible to attain optimum wear resistance. This work was done by two stages: First stage, pore formation during sintering with two different additive alloying elements Cu & C. While the second stage wear test was performed by using pin-on- disc machine for different loads (2, 4, 6 and 8 N) to each composite material used in this work under dry sliding conditions. The results showed that obviously improvement in wear resistance for C additive alloying element more than Cu.

Keywords: *Powder Metallurgy, Composite materials, Porosity, Wear.*

تكوين المسامات أثناء التلبيد لمادتين مركبتين مختلفتين(Fe-Cu-C, Fe-Cu) وتأثيرها على سلوك البلى

الخلاصة:تمت دراسة تأثير المسامات الناتجة عن التلبيد بالحالة الصلبة في Fe-Cu و Fe-Cu-C.أن عناصر السبك المضافة C & Cu وحجم المساماتالمتكونة توثر على معدل البلى, لذا فان وجود الكاربون يقلل من معدل البلى ,وكذلك كلما كبر حجم المسامات قل معدل البلى . ان توزيع المسامات الثانوية لا يعتمد على الكسر ألحجمي لدقائق C & Cu المستخدمة وقد تبين أن التلبيد بالحالة الصلبة في جومن الاركون(Ar) يفضل عند استخدام مساحيق سبائكية ناعمة قدر الإمكان للحصول على مقاومة البلى المثلى .تم إجراء البحث على مرحلتين: المرحلة الأولى : تكوين المسامية أثناء التلبيد لعناصر السبك المختلفة المضافة. Cu & Cبينما المرحلة الثانية : تم إجراء اختبار البلى باستخدام ماكنة المسمار – على – القرصعند أحمال مختلفة (8,6,4,2 نيوتن)لكل مادة مركبة مستخدمة في البحث تحت تأثير ظروف الانزلاق الجاف. أظهرت النتائج تحسن واضح في مقاومة البلى باستخدام C كعنصر سبك مضاف أكثر من Cu .

1. Introduction

One of advantages of powder metallurgy method is to combine at least two material components in powdered forms to produce a composite material with a wider range of desirable properties .The sintered composites produced by solid lubricant addition

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usually provide promising tribological properties [1].

In view of economic and technical reasons sintered Irons find extensive applications reasons in bearing and gears. Criteria for these gears and bearings are that materials used for these purposes should have high strength, good wear resistance and low coefficient of friction [2]. The problem encountered in these types of sintered products for making gears and bearings is pores developed in powder metallurgy routes .In these parts pores effecton the strength, fatigue properties and wear resistance [3].

For the production of high strength sintered alloys elemental starting powders are frequently used to attain maximum compatibility and minimum tool wear. One of the most widely used materials in powder metallurgy is certainly Fe-Cu and Fe-Cu-C.
One of the most common alloying elements used in ferrous powder metallurgy is copper. The use of copper in ingot metallurgy is restricted to weathering iron only. While in powder metallurgy, because of its low melting point, copper is known to activate sintering at low temperature, which ensures homogeneous mixing of the powder. Presence of copper during liquid phase sintering results in compact swelling. It has been observed that compact swelling can be compensated for by the addition of carbon in ferrous alloys [4]. Dilator studies of Fe- Cu-C found that the large expansion associated with the copper growth phenomenon decreases with increasing carbon content. Originally this beneficial effect of carbon was attributed to the reduction of Cu solubility in iron and development of a liquid phase. [5].

There are many previous studies were done in this field, S. Dhanasekaran and R. Gnanmoorthy [6], were studied the development of self-lubricating sintered steels for tribological applications. The friction and wear characteristics of sintered steels containing of solid lubricant molybdenum mdisulphide (MoS_2) was studied using a pin, which arereported in this paper. While NeeravVerma and SaurabhAnand [7], were focused in their study on the effect of carbon addition and Cu in SH737 alloys systems via microstructural studies .SH 737 -2 Cu alloys were compacted ,sintered and characterized. According to this study the materials were characterized according to their density, densification، shape factor, and pore size distribution.

The aim of this work is to study the effect of pore formation during sintering and its effects on wear behavior of two different composites material Fe- Cu and Fe-Cu-C.

2. Experimental Work

Once the process (mixing, handling, compacting, sintering) has been perfected, the cause of scattering lies with the raw materials .In this work, Fe-Cu and Fe-Cu-C composites were produced by powder metallurgy process .Test samples were prepared from the elemental powders, the nominal composition of the samples developed is 94% iron, 6% copper for first alloy, while the second alloy for the same dimension contains 94% iron, 5% copper, 1% carbon. As basic materials have been used in this study are the iron powder (manufactured by water atomized method) and the copper powder (manufactured by electrolytic. method). Ammonium bicarbonate (NH_4HCO_3) powder was used as space holder. The ammonium bicarbonate fraction of 125-250 µm was used in all experiments. Table 1 shows the component of the mixture.

Table 1. Mixture compositions of all specimens.

Sample code	Components of mixtures %			
	Fe (wt %)	Cu (wt %)	C (wt %)	NH_4HCO_3 (vole)
Fe	100	-	-	50
Fe - Cu	94	6	-	50
Fe - Cu - C	94	5	1	50

2.1 *Stages of Preparing Models*

A. The mixing was performed manually into two stages. Initially iron and copper powders were mixed together during 15 minutes. A small amount of gasoline was also added (1% by weight) to avoid powders segregation. Then after the ammonium bicarbonate powder was added in the mixture. The duration of mixing for the second stage was 5 minutes. The same stages were applied for the second Composite material containing copper and carbon powders as additives.

B.The powder mixture was compacted in a hydraulic press: cylindrical test specimens of dimensions 10 mm diameter and 10 mm lengths. The compaction pressure varying from 100 to 400 Mpa with the step of 100 Mpa was used.

C. The relative densities of obtained compact $\boldsymbol{\rho}_{rell}$ were calculated using the following relation [8].

$$\rho_{rel} = \frac{m}{0.785}.d^2.h.\rho_{mix} \tag{1}$$

Where:
m: weight of sample (gm).
d , h : diameter and height of the sample .
ρ_{mix} : theoretical density of powder mixture ,determined by the following equation:

$$\rho_{mix.} = 0.5.\ \rho_{Fe-Cu} + 0.5.\rho_{pf}\ (for\quad Fe-Cu\ alloy) \tag{2}$$

$$\rho_{mix.} = 0.5.\ \rho_{Fe-Cu-C} + 0.5.\rho_{pf}\ (for\quad Fe-Cu-C\ alloy) \tag{3}$$

Where:
ρ_{pf}: 1.586 g /cm^3 theoretical density of ammonium bicarbonate [9].
$\rho_{\mathbf{Fe-Cu}}$: Theoretical density of iron and copper mixture calculated according to the equation:

$$\rho_{Fe-Cu} = \frac{1}{(\frac{xFe}{\rho_{Fe}}) + (\frac{xCu}{\rho_{Cu}})}. \tag{4}$$

$$\rho_{Fe-Cu-C} = \frac{1}{(\frac{xFe}{\rho_{Fe}}) + (\frac{xCu}{\rho_{Cu}}) + (\frac{xC}{\rho_{C}})} \tag{5}$$

X_{Fe}: is iron content (by volume fraction).
X_{cu}: is copper content (by volume fraction).
ρ_{Fe}: 7.874 gm/cm^3 is theoretical density of iron.
ρ_{cu}: 8.93 gm/cm^3 is theoretical density of copper
ρ_c: 3.514 gm/cm^3 is theoretical density of carbon.

D. These samples were sintered at temperature of 1120 ^{0}C during 30 min in Ar atmosphere [10] . Heating rate was maintained at 5 ^{0}C/min.

Thereat ammonium bicarbonate particles have been decomposed to ammoniac, carbon dioxide and water leaving large pores. The sintered samples were mounted with the help of epoxy, and wet grinded by using a series of Sic emery papers (500,800, 1000μ_m) followed by cloth polishing using a suspension of 1 μ_n and 0.03 μ_n alumina. "Fig. 1", shows the microstructure of the two different composites materials used in this work.

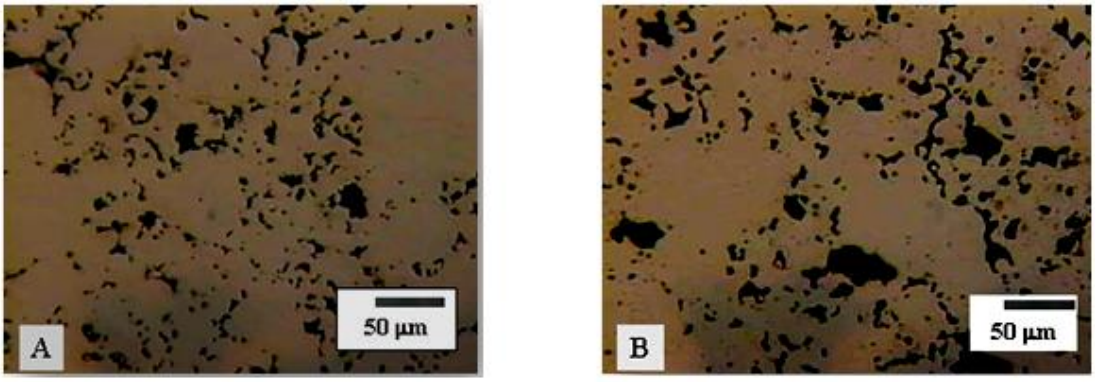

Figure 1. Optical microstructure of samples for: (A):Fe –Cu Composite material and (B): Fe –Cu– C Composite material.

3. Porosity and Hardness

A Pore acts as a stress concentrator and a plays an important role in material failure. The porosity affects on mechanical and physical properties of sintered materials which has been studied frequently. There are two types of pores forming:

a. Primary pores formed at the time of compaction process.
b. Secondary pores formed at the time of sintering process due to transient liquid phase sintering.

Primary pores are large pores that result from geometric packing of the particles or from binder burn out. While the secondary porosity is typically smaller in nature and may be attributed to residual porosity from liquid phase formation and diffusion of alloying addition, such as Cu. Due to secondary pore formation, the sintered density is lowered than the green density. Porosity of the specimens is measured according to the following equations [11].

$$\text{Porosity for compacted specimen} = \left(\frac{Theoretical\ density - Compact\ dencity}{Theoretical\ density}\right) \times 100$$

$$\text{Porosity for sintered specimen} = \left(\frac{Theoretical\ density - Sintered\ dencity}{Theoretical\ density}\right) \times 100$$

While the hardness measurements are carried out through the use of way of Rockwell hardness test (HRB).

4. Wear test procedure

Dry sliding wear have been carried out using a pin-on- disc machine. The mating surface of the disc is ground; the disc is clean in acetone before the commencement of wear test. Sintered cylindrical pins are tested by wear machine changing the load by 2, 4, 6 and 8N, with constant sliding velocity equal to 300cm/s ,and then weighing the specimens by using electronic weighing balance of 0.01 mg accuracy. The tested specimen is measured in the direction perpendicular to the wear track. The volume of the worn out material is calculated from the weight loss of material divided by the density of the material. The wear rate is evaluated from the volume of worn out material divided by sliding distance. The wear mechanism is studied by observing surface using optic a microscope. The initial surface roughness of all the samples is measured by using (Talysurf– 4 apparatus product by English Taylor- Hobson Company) before and after wear test for all the specimens.Table 2 shows the results of Roughness test.

Table 2. Roughness values.

No.	Specimen	Roughness (µm)
1	Fe	0.١٧
2	Fe- Cu	0.051
3	Fe-Cu -C	0.٠٢٥

5. Results and Discussion

Common liquid phase sintering systems of Fe-Cu and Fe-Cu-C alloys are used in the automotive industry, in which the inherently low costs are important. Adding copper to iron in mixed powder compacts causes swelling, called copper growth. Introducing element such as graphite to prevent dimensional instability has widely been reported. Two dominant methods have been proposed to restrain volumetric expansion: (a) carbon addition prevents the grain boundary penetration of a liquid Cu rich phase, and (b) carbon addition reduces the grain boundary energy of iron and induces grain boundary pining. The results of this investigation are agreed with [12].

5.1. *Effect of Cu and C on porosity*

It is shown from the table. 2 that the green density, sintered density and the percentage of porosity decreases with addition of C. While the hardness (HRB) increases with addition of C. This is attributed to that the addition of carbon decreases the liquid temperature of the Fe – Cu melt (1120°C), and delays the dissolution of copper into the iron, thus allowing a prolonged time with liquid phase sintering. Fortunately, the suitable copper and carbon levels fall within the span of improving wear resistance. These phenomena for this work are agreed with [13, 14]. Powder packing in the green bodies as well as grain shapes can be seen in "Fig. 2", for two compacted specimens (Fe-Cu, Fe-Cu-C).

The measurements of density, porosity and hardness (HRB) for the specimens of this work are shown in Table 3.

Table 3. The values of density, porosity % and hardness for Fe –Cu and Fe-Cu –C composites.

Alloy	*Green density gm/cm^3*	*Sintered density gm/cm^3*	*Porosity %*	*Hardness HRB*
Fe - Cu	7.09	6.71	14.7	80.2
Fe – Cu - C	6.91	5.83	11.3	85.3

5.2. *Pores formation*

The porosity which is investigated has mainly primary porosity or secondary porosity, the primary porosity has already been present in the green compacts. While the secondary porosity formed during the sintering process.

It is due to the formation of transient liquid phase during sintering, the liquid phase penetrating the matrix grain boundaries, at least in case of satisfactory wetting of the matrix by the melt, in the addition of the alloying element particles, pores are left, and the homogenization may thus lead to an overall expansion of the material during sintering, this can be clearly seen in "Fig. 2", which is agreed with [7].

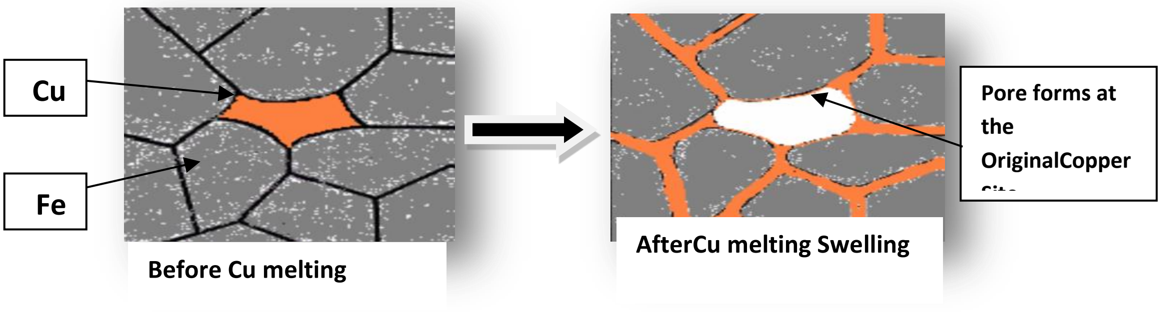

Figure 2. Process of transient liquid phase sintering resulting in the diffusion of copper, porosity, and swelling in sintered hardened sample [7].

In the absence of carbon, we obtain slight swelling due to the diffusion of copper. As the amount of carbon is added, the sintered density is reduced as shown in Table 3, also the swelling is reduced, and then the carbon hinders "copper growth". This can be seen in "Fig. 2", [7]. Preventing excessive penetration of copper rich liquid along grain boundaries or an inter particle boundary. Because of the increase in the dihedral angle between iron and copper, there was a reduction in swelling due to the carbon addition.

5.3. *Effect of Compacting Pressure and Sintering Temperature on the Porosity*

Increasing compaction pressure lead to increasing in green density as shown in "Fig. 3", it is due to that during compaction under pressure are the bulk movements or the rearrangement of particles and the deformation of particles. Movement of particles' depends upon the particle to particle contact and free space into which the particle may move. This movement is limited by the frictional forces developed between neighboring particles and between particles and the die surfaces. Therefore, any increasing in motion leads to decreasing the porosity.

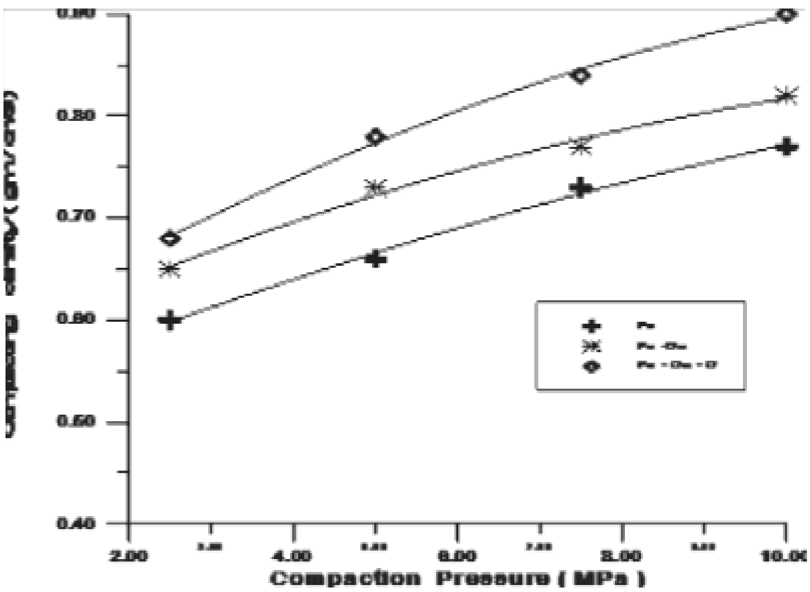

Figure 3. Relationship between compacting pressure and compacting density for all specimens (Fe, Fe-Cu, Fe-Cu-C).

Also increasing in sintering temperature lead to decrease in porosity, because it is observed that the rate of change of sintered density is higher than that for the compacting. The reason of behind this kind of behavior is associated with the driving force required for sintering. In the earlier stages of sintering, the driving force is produced by the surface energy, which is associated of the driving force. In general, sintering involves initial bonding among particles, neck growth, pore channel closure, pore rounding and densification or pore shrinkage, which in turn leads to decrease the porosity. "Fig. 4", shows that obviously and it is agreed with [11].

As stated above, further decrease of the secondary pore size can be expected to result in a significant decreasing of the wear rate.

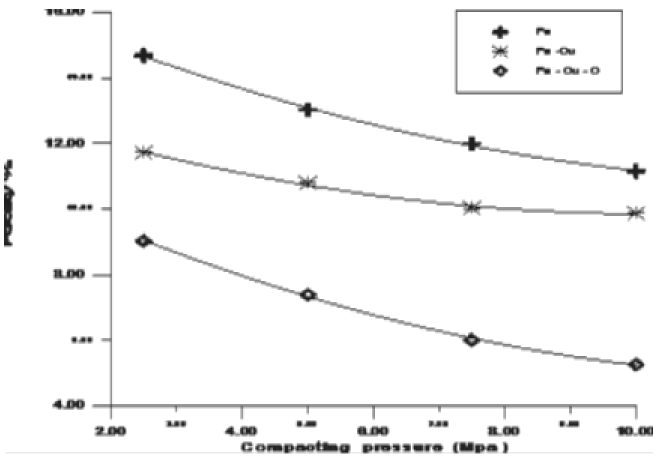

Figure 4. Relationship between compacting pressure and porosity % for all specimens (Fe, Fe-Cu, Fe-Cu-C).

5.4.*Wear behavior*

The mass loss quantified during the wear test for the Fe –Cu and Fe –Cu- C are shown in "Fig. 5".The Fe-Cu –C samples exhibit a low mass loss compared with Fe –Cu pins indicating a higher wear resistance. The wear rate measured at different applied normal loads for the two compositions. The Fe –Cu composition showed an increase in wear rate with increasing applied normal loads. The wear rate of Fe –Cu –C marginally increases with applied normal loads. For materials containing carbon a decreased wear rate can be observed. This is due to severe action of shearing and small amount of plastic deformation. In Fe –Cu –C, the wear occurs by the deformation mode that only results in the formation of groove.

Keeping the sliding velocity constant at 300 cm/s ,a family of wear curves was produced over a loads about 2, 4, 6, and 8 N. Irrespective of load ,there basic types of wear curve were obtained "Fig. 5". The relevant features are

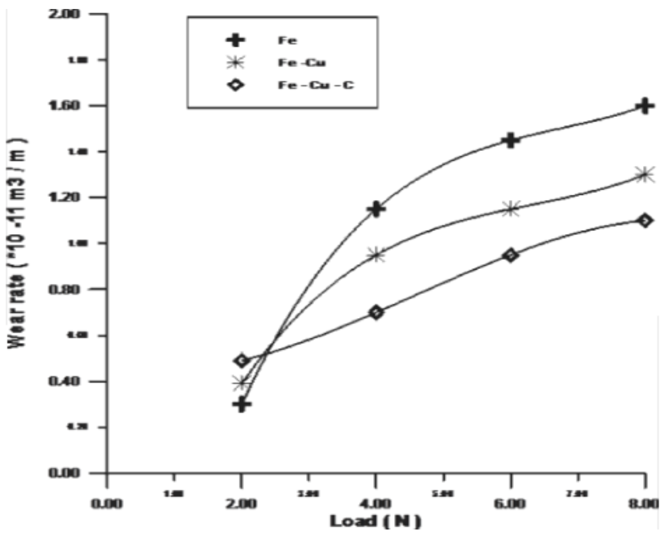

Figure (5): Relationship between loads and Wear rate of Specimens (Fe, Fe-Cu, Fe-Cu-C).

First: At low loads, running in wear was negligible and an oxide was quickly produced on both wear surfaces.

Second: Increasing the loads a more complex behavior occurred. Dunning running in metallic debris was produced, metal particles adhered to the disc wear track and the wear rate was high.

Third: At a higher load metallic wear occurred which not sub -sequentially reduced to a lower level. In Fe-Cu-C composite alloys the wear rate is low than in Fe-Cu and Fe, it is due to the C as alloying element which in turn increasing the hardness leading to decreasing in wear rate.

Figure .6 shows the worn surfaces of sintered iron pins before and after addition of C. The worn surfaces also show the smeared pores. A deeper wear track can be seen in the Fe –Cu alloy indicating severe wear due to combined action of shearing and abrasion.

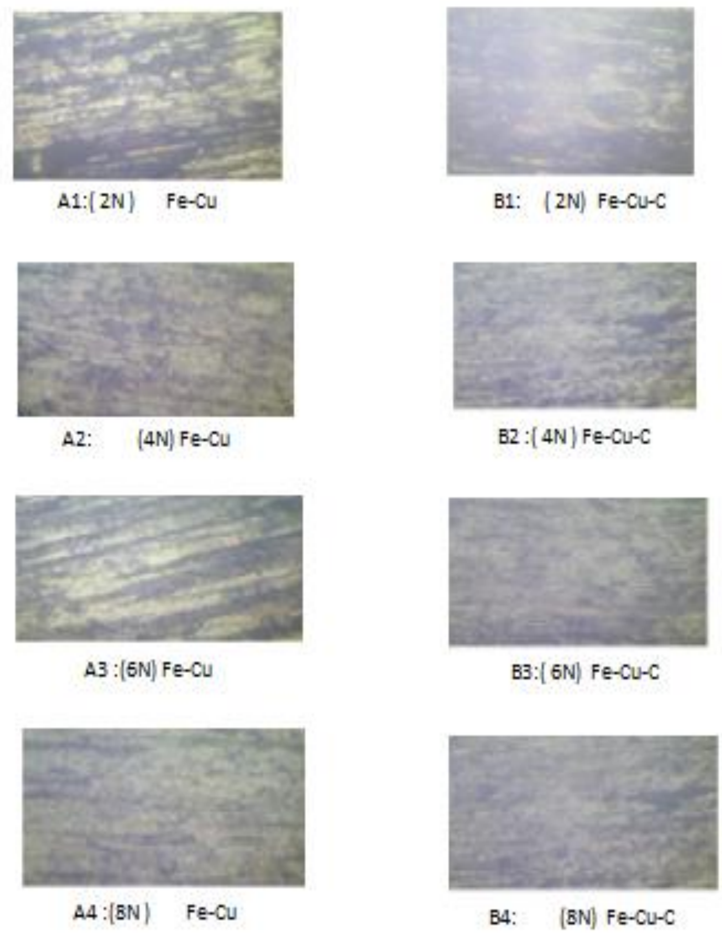

Figure 6.Photomicrographs With magnification (100X) of Wear behavior of the pins for:
A: Fe - Cu . B: Fe-Cu-C

The deeper grooves were formed in the composition Fe –Cu. This groove indicates a sever wear condition. While for the composition Fe –Cu –C and at all loads exhibits decreasing in wear rate because of the smaller grooves formed. Also this is due to increasing in hardness which to make the groove finer than for Fe – Cu composites. Surface roughness measurements carried out on the worn out surfaces. The average centre line surface roughness is about 0.051 μm as shown in Table 2, whereas a rough wear track indicating deeper grooves and irregularities was observed in Fe –Cu alloys. The average center line surface roughness for Fe – Cu – C is about 0.025 μm significantly lower compared with the C addition material tested at similar conditions.

The copper particles adhere temporarily to one of the sliding surface and plows out as a groove .In the two different compositions tested, the worn surface appearance looks like a serrate edge and this is higher in the Fe –Cu because of the copper wear debris formed in between the contact zone. This harder wear debris gets trapped in between two sliding surfaces, this is agreed with [15, 16]. The hard particles impinge on the contact surface and causes more wear. But in the Fe –Cu –C, this effect was very low due to the presences of the C.

6. Conclusions

The following conclusions can be made based on the results obtained from this study:

1. An addition of C (1 %) improves the compacting density and sintering density with less porosity and high hardness than Fe - -Cu composite.
2. The alloy based on Fe –Cu- C system exhibits high wear resistance at normal loads.
3. A smoother wear tracks were observed in Fe – Cu – C specimens.

7. References

1. S Mahathanabodee ,T. Palathui , S . Raadnai , R.TongsriandSombatsompop (2013), "Comparative studies on wear behavior of sintered 316L stainless steels loaded with h – BN and MoS_2 , Advanced Materials Research . Vol .747, pp (307-310) .
2. P. Siva kumar ,N .G . Renganathan (2011), “studies on mechanical properties and wear characteristics of Fe-C-Cu alloy with and without Molybdenum di sulfide” ,Int .J.Chem-Res. Vol .5, NO. 12-21, pp. (2249 - 0329).
3. Kevin Dchristian , Randall M German (1995), “ Relation between pore structure and fatigue behavior in sintered iron - copper- carbon”, Int .J. powder Metal, 31, pp. (51-61).
4. K. Dannin Ger (1987), “Pore formation during sintering of Fe –Cu and its effects on mechanical properties”, powder metallurgy international .Vol.19, No.1, pp (19-23).
5. Klas Boivie, Woxencentrum , “SLS application of the Fe-Cu-C system for liquid phase sintering ” Woxenecentrum , KTH, The Royal Institute of Technology, pp(141- 148) .
6. S. Dhanasekarn and R .Gnanamoortny (2004), “ Develapment of self lubricating sintered steels for triological applications , Internetionalsymposiam of Research students on Materials Science and Engineering”, chermai –India. Vol. 20, PP (20-22).

7. NeeravVerma and Saurabh Anand (2006), “Effect of carbon Addition and Sintering Temperatures on Densification and Micro structural Evolution of Sinter – Hardening Alloy Steels” ,Research articles , pp.53-59.
8. Natalia Rudenko, Alexander Laptev , Leonid popivnenko (2012), “Influence of Copper additive on pressing and bending strength of Iron based highly porous powder materials”, The 3 rdInterational conference on Dignosis and production in mechanical engineering systems ,2012 Galati, Romania .Vol.3, pp. 1- 4.
9. German, R.M (1996), “Sintering Theory and Practice”. Ed. John willey & Sons ,Inc.
10. Anil Kumar Sinha (1987), “Powder Metallurgy” ,Second Revised Edition published by J.C. Kapur, for DHANPAT RAI and SONS, Jullundur. Delhi.
11. Siddhartha Tiwari, PriyankaRaiput and Sanjay Srivastava (2012),” Densifcation Behaviour in the Fabrication of Al-Fe Metal Matrix Composite using powder .Metallurgy Route ", ISRN Metallurgy, Vol.7 , pp. 1-5.
12. PO- Liang Liu and Shun – Tian Lin (2002), "Relation between widths of the Grain Boundary Energy cusps at special Disorientations and the Micro structural characteristics in the sintering of a Fe-Cu-C Alloy", Material Transactions . Vol. 43, No.3, pp 544-550.
13. Runfors .u (1987), "Powder Metallurg”, Dep ,of physical Metallurgy , KTH, Sweden.
14. German R.M (1985)," Liquid phase sintering, plenum press.
15. R.M German, K.A.d Angelo (1984); Int .Metal Reviews,Vol. 29, pp 249-260.
16. D.Berner , H.E. Exner, G.petzow: Modern Dev. In powder Met.6 , H.H Hausner ,G. D . Smith ed (1974), “MPIF –APMI” , Princeton N.J, pp. 237-245.