



Investigation of Dry Sliding Wear Behavior of Powder Metal (P/M) Materials Produced from Mixture of Fe-Cu-C Powders

Halil ARIK^{1,*}, Dilruba Zumer ORHUN¹

¹Gazi University, Faculty of Technology, Department of Metallurgy and Materials Engineering, 06500, Ankara, Turkey

Article Info

Received: 24/11/2018
Accepted: 20/03/2018

Keywords

Powder metallurgy
Pin-on-disk
Dry sliding wear

Abstract

In this study, dry sliding wear behavior of powder metal parts produced from ASC.100.29 iron powder with a particle size range of 0.28-40 μm and containing 2 % Cu and 0.75 % C were investigated using pin-on-disk method. In order to obtain cylindrical compacts with 10 mm in diameter and 20 mm in length the powder was compacted under 800 MPa pressure in a single action press. Then compacted powder metal parts were sintered at 1100 °C temperature for two hours in an atmosphere controlled tube furnace. After the sintering process, micro structural analyses, hardness and density measurements of powder metal parts were performed. Following the characterization of sintered samples dry sliding wear tests were performed according to ASTM-G99-05 by using pin-on-disk wear device. Wear tests were carried out under different loads (10-15-20 N), with the other test parameters being constant, to investigate the wear behavior of powder metal parts in relation to load. The test results showed that the increasing of load resulted in increase of wear values.

1. INTRODUCTION

In recent years, the powder metallurgy, as a production method has stepped forward among other production methods. With this method, it is not only possible to be able produce relatively small and complex parts in serial production and close sizes to their final dimensions but also acquire serious economic benefit due to use of maximum amount of material with low – rate waste. When compared to casting method which is only applied to the metals with low melting temperatures, powder metallurgy can be applied to almost all materials. For this reason, many different materials such as composites, high – temperature ceramics, some polymers, copper – content steels, refractory metals, cermets and mixed – phase compositions can be produced through powder metallurgy. One of the broad application areas of powder metallurgy is the production made by adding different powders at various ratios which are insoluble in each other in order to create a composite material. By controlling the dimensions, forms and quantities of the phases, the properties expected from these materials can be achieved [1-3].

Recently, whereas the performance – oriented material production activities have gained density in the iron and steel sector, the production and operation costs of materials have transformed into an important target as well. At this stage, PM steel parts are in more advantageous position in terms of production costs. For this reason, rate and share in production costs of the powder metal parts being used in too many areas headed by automotive sector are increasing rapidly. While the iron powder is consumed most in the production of powder metal parts, again the iron – based powder metal parts are produced most [4, 5].

While producing the powder metal parts from the iron powders, at a substantial scale of copper and graphite-added iron powders are being consumed, the graphite serves as the carburizing agent and the copper serves as the binding function by forming a liquid phase in sintering process [6]. In this type of composition, composite powder finds a broad application area especially in the automotive sector. The sintering of these composite powders is in form of liquid – phase sintering and there are too many variables that affect the properties of powder metal parts at the phase of use depending on the powder consumed and

*Corresponding author, e-mail: harik@gazi.edu.tr

the production parameters. Leading parameters are the chemical composition of the composite powder, shape and size of the composite powder, its mixing ratios when there is more than one composite powder, and applied parameters to sintering and compacting of the composite powders. One of the basic expected behaviours of sintered steel material is to being resistant to all kinds of wear [2, 7, 8].

The wear is the occurrence of continuous material losses resulting from different causes of friction surfaces of solid bodies. Wear causes huge economic losses in the using various machinery and equipment. Because the friction forces on the contacting surfaces cause loss of power and the wear causes the operating tolerances to be worsened. The most common types of wear are the abrasive and adhesive wears. Approximately 50% of total wear – dependent damages is caused by these kind of wear. In this respect, it is of great importance to extend the service life of machine elements in contact with another material exposed to abrasion and to increase the wear resistances of the materials in order to minimize the economic losses [9-11].

The factors that are effective on this feature of PM parts are the composition, phase ratios and distribution, the performance of getting sintered and the density of material. A great deal of performance promotion – oriented studies has been conducted up to now to improve the wear performance of such materials. In general, the studies accomplished have concentrated on the post – sintering mechanical properties and the effect of the copper on a PM part. When the pressing pressures were in the range of 300 – 650 MPa and the sintering temperature was in the range of 1120 – 1150 °C, the sintering time was taken between 30 – 60 minutes [12-17].

In this study, firstly production of PM part from the mixture powder consisted of 2% copper, 0.75% graphite, 0.8% lubricant (zinc stearate) and 99.9% pure ASC.100,29 elemental iron powder were done using powder metallurgical method. Then dry sliding wear properties of PM part were investigated using pin-on-disk method according to the increasing load.

2. MATERIAL AND METHOD

Table 1 shows the content and physical properties of the powder mixture.

Table 1. Physical properties and chemical composition of the mixture powder.

Content of mixed powder (%) by weight				Powder particle morphology	Range of powder particle Size
0.75 C	2 Cu	0.8 ZnSt	96.45 Fe	Angular	0.28-40 μm

Cylindrical wear test samples with 10 mm diameter and in 25 mm (you said 20 mm in the abstract) length were produced with single-action press under 800 MPa pressure in an axial die following the precise weighing processes (Fig. 1).

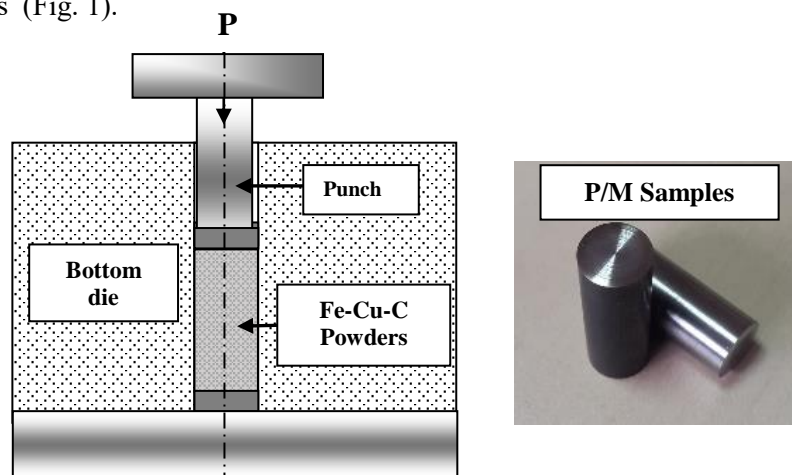


Figure 1. Mixture to be compacted inside the die

After the visual inspection of compacted powder metal samples; the raw density measurements were done. Later on, the liquid – phase sintering was performed at 1100 °C in an atmosphere – controlled tube furnace for 2 hours in flowing argon protection. Sintered density measurements were carried out according to the principle of Archimedes using a density meter equipped with an electronic balance providing the weighing in a 0.1 mg precision. Brinell hardness measurements were applied on the cross section perpendicular to the pressing direction. The hardness measurements have been performed using a ball with a diameter of 2.5 mm under a load of 31.25 kgf. The average of 5 hardness measurements was taken as the hardness value of the materials. Samples were prepared for micro-structural analysis by optic and SEM microscope after density and hardness measurements. They were gradually sanded, polished and etched by using 320-1200 mesh sandpaper, 6-3-1 μm diamond solution and phenol, respectively.

2.1. Wear Test

The dry sliding wear tests were performed on the test samples produced and prepared in accordance with ASTM G99-05 by means of pin-on-disk method under the conditions provided at Table 2 [18]. TRIBOMETER T10/20 device was used for the wear test (Fig. 2). As the opposite wear surface, a steel disc prepared from Hardox 500 steel with hardness of 52 HRC was used. In determination of wear-dependent weight losses of the sample, weights prior to test was determined with 0.1 mg precision using a SARTORIUS brand scales..

Table 2. Wear Test Parameters

Load applied (N)	Rotation speed (r/min)	Dry sliding path (m)	Trace diameter (mm)	Velocity (m/sec)
10-15-20	300-400	1000	44	0.69-0.91

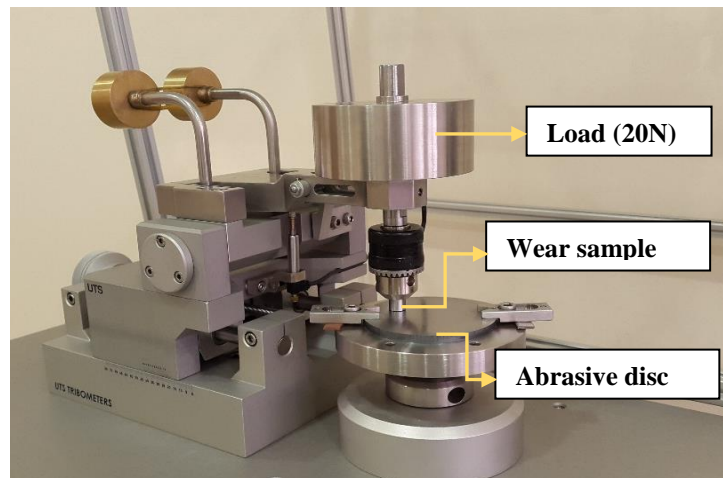


Figure 2. TRIBOMETER T10 / 20

3. RESULTS AND DISCUSSION

Green and sintered–density and hardness values were given in Table 3.

Table 3. Density and hardness of a P/M part

Green density (%)	Sintered density (%)	Hardness (HB _w)
88.77	89.75	132.75

As green density of 88.77 % of theoretical density were obtained after compaction. The green density obtained is higher than that of given in similar studies [19]. Not only higher pressure but also the graphite in the mixture powder and the zinc stearate used as the lubricant made contribution to providing higher

density. In the visual inspection of the demolded samples, no defect was seen in the form of layering or backfilling expansion on the sample surfaces.

A density increment at a rate approximately 1% has taken place in the PM parts following the sintering and the density of the sintered PM parts has been measured as 89.75%. In the literature, the maximum density values given with the PM particles produced from this type of powder mixture under similar conditions do not exceed 88% [3, 19]. The higher density values obtained in this study were due to the fact that the relatively higher pressing pressure, longer sintering durations and additives. In the studies performed by the relevant powder manufacturer, the pressing pressure was taken as more or less 500 MPa, the sintering temperature 1120°C and the sintering duration 30 minutes [20].

The sintered density value of PM part indicates that the PM part still contains approximately 10% pore. Before the etching, microstructure images taken from the surface of sintered samples were examined, we see that 2 % of copper found in the mixed powder has concentrated at the particle boundaries by creating a liquid phase in process of sintering at 1100 °C [Fig. 3]. However, we should say that the copper distribution at the particle boundaries does not give a full homogenous image. This may be attributed to the liquid phase sintering at a temperature slightly above the melting point of the copper. If the sintering temperature is raised a little more, the molten copper can spread more easily and more homogeneously in the iron powder particle boundaries. In similar studies, in general, 1120°C and above used as the ideal sintering temperature in sintering process of the iron – copper and graphite powder mixtures [21].

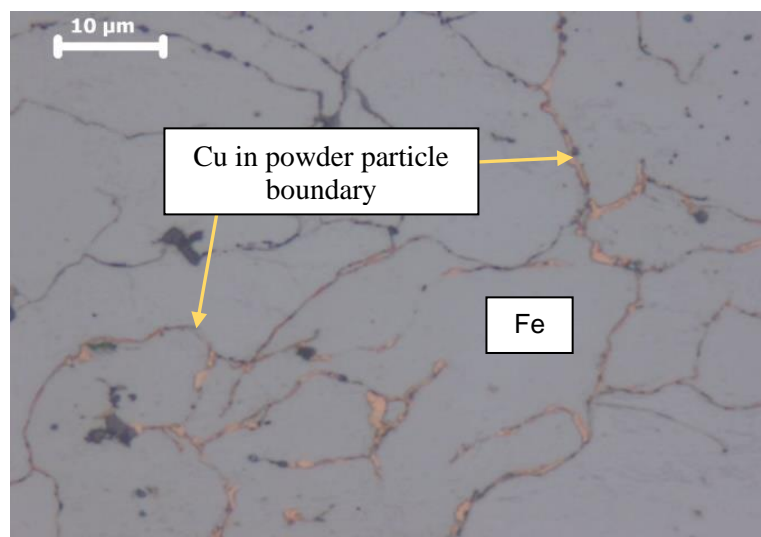


Figure 3. Liquid phase formation among powder particles

In general, while the copper existing in the content of mixture powder makes an increment in the strength of PM part and expansion effect during sintering process, so it causes an increment in the dimensions of powder metal part. The copper actualizes this effect in process of sintering in two stages. Initially, it transforms into liquid phase and then go round of the iron particles and in second step; it forms a solid solution with iron by getting spread into the ferrite structure during sintering. This situation makes the production of powder metal part with planned measurements difficult. On the other hand, addition of some carbon enables unintentional increase in the size of the powder metal part originating from the sintering process to be significantly controlled [12]. According to normally visible increased density values after sintering, appearance of only a 1% increase in these samples originates from the effect of copper in this direction.

The hardness is normally lower than a 0.75% versus only carbon steel. The main reasons for this are the pore present in the PM part, soft copper phase and perlite-ferrite phase ratios in powder particle limits. When the microstructural images of PM specimen are examined, it is seen that the ferrite and perlite phase ratios are 50% Perlite and 50% Ferrite [Fig. 4]. If the carbon content of the mixture powder is considered,

the perlite phase ratio should have been expected to be higher. However, EDS analyzes taken on the samples show that a portion of the carbon remained in form of residual carbon. The residue-like carbon appears to have concentrated especially in the current pores.

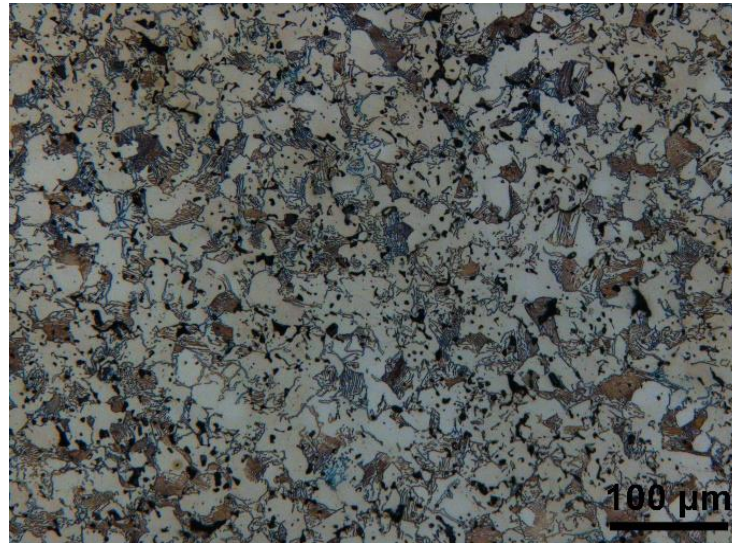


Figure 5. Phase distribution in microstructure. Light tone: Ferrite, Dark tone: Perlite, Black tone: Pore

While the pores in the powder metal material are homogeneously dispersed throughout the structure, and shapes of the pores are close to spherical and the size range is 1-6 μm. By looking the size, shape and distribution of the pores within the matrix structure, we can say that a proper sintering happened. All diffusion phases taking place in sintering process and the pores turned into independent closed pores. From the distribution of perlite and ferrite phases within overall structure, we can say that a microstructurally homogeneous PM part was obtained from the sintering and pore size, shape and distribution of the powder grain interfaces formed [Fig.5].

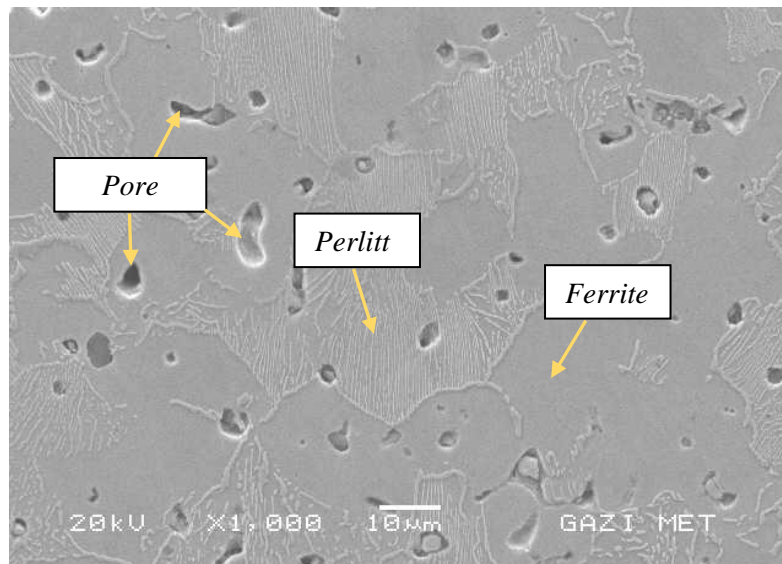


Figure. 5 SEM image of a PM part microstructure

EDS analyzes of the samples show that the carbon in the content diffused inwardly from the surface of the iron particles during the sintering and hereof has remained as residual carbon within the remaining pores while forming a pearlitic structure. According to EDS taken on the pore no. 1 in Figure – 6, while the medium appears to be 63.87% carbon in weight, as a result of analysis taken from pore no.3, a 3.76% copper phase in weight was determined. This situation generally leads to a lower hardness in PM materials in

compoarition with traditional alloyed steel containing 0.75 % carbon.by decreasing percentage of a pearlitic structure.

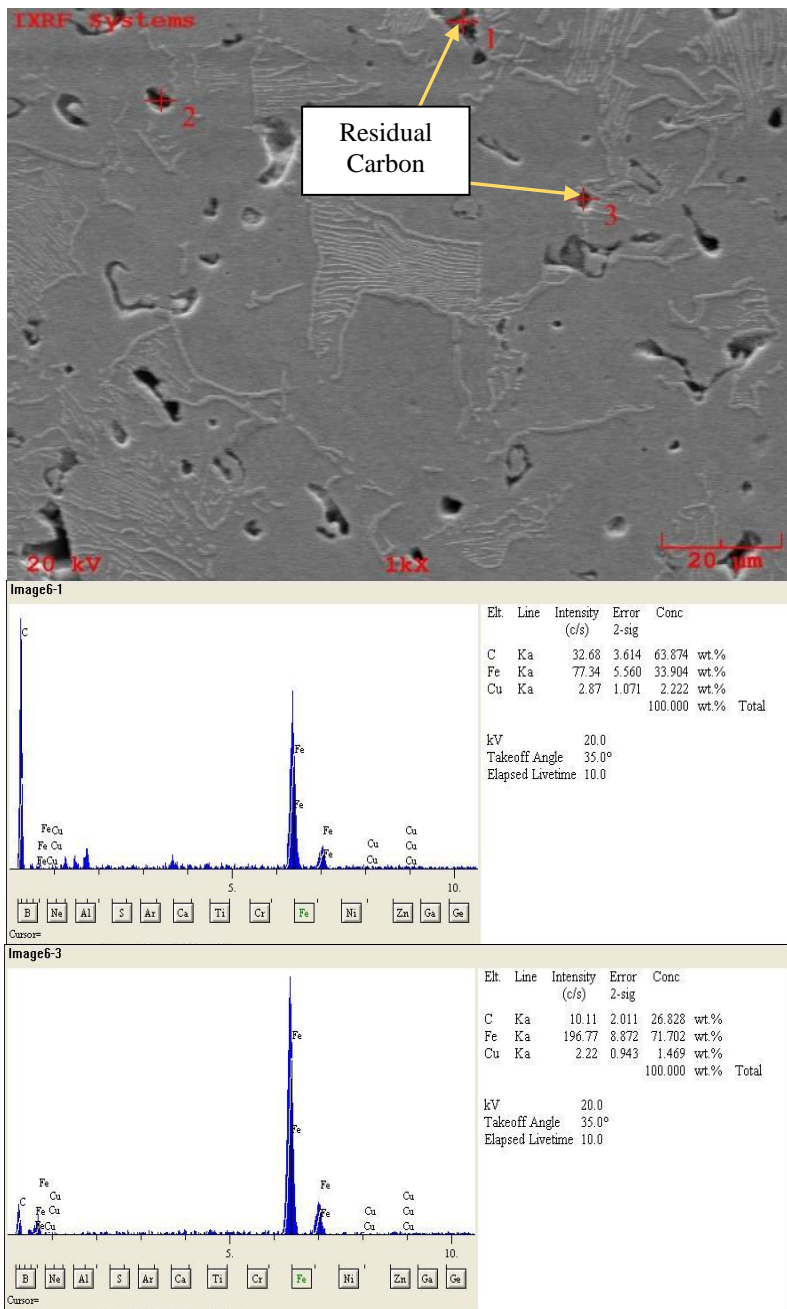


Figure 6. Printouts of EDS analyzes taken from point no.1 and 3

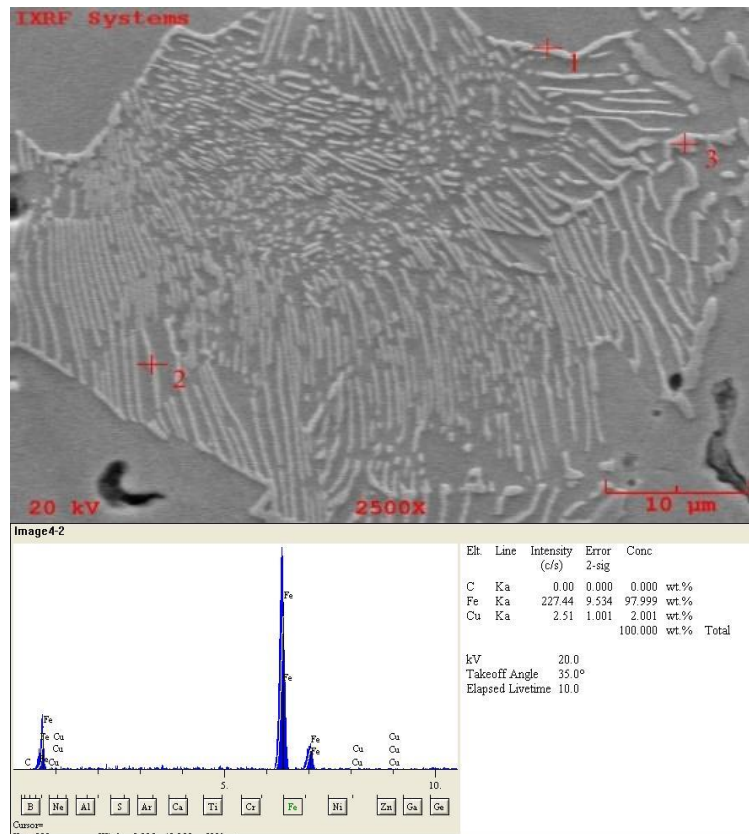


Figure. Printout of EDS analyzes taken from point no.2

In Figure – 7, no. 2 point is close to granular limit and 2 % copper in weight has been identified according to the outcomes of the EDS analysis accomplished from such point. (It seems that this EDS result is wrong. Because there should be at least 0,8 % C in the pearlite and It is not also possible that there is 2 % Cu) This indicates that the copper which has transformed into liquid phase during sintering in particle boundaries has diffused into the pearlitic structure. While this situation strengthens the bonding among the iron power particles, it generally has an effect of enhancing the strength of the powder metal part. Due to full sintering, the boundaries of particles vanished at a big ratio within these zones. From the analysis performed and the images of microstructure, it is seen that the targeted PM part was produced from the mixed powder with compacting under 800 MPa – pressure and then sintering at 1100 °C – temperature for 2 hours.

Shortly after the start of dry sliding tests, the heat and oxidization – induced and exposed oxide layer formation has caused a contamination on both the wear and abrasive surfaces [Fig. 8]. We also see that some copper has transferred on the abrasive disc during the abrasion tests.

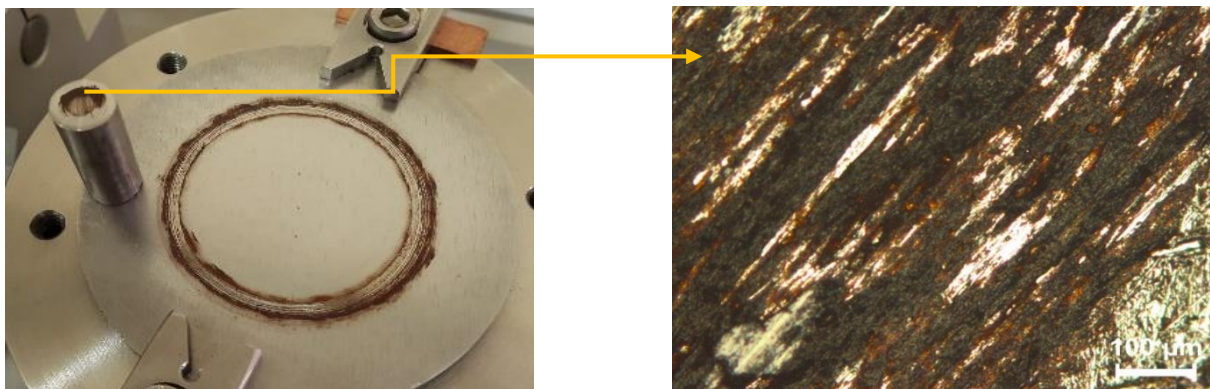


Figure 8. View of a worn sample surface

The surface condition of pins exposed to wear in the generated wear debris analyses show that predominant mechanism of wear was adhesion followed with plastic deformation (Fig. 9). The SEM micrographs of worn surfaces of pins show a presence of plastic deformation and formation of plate-like particles.

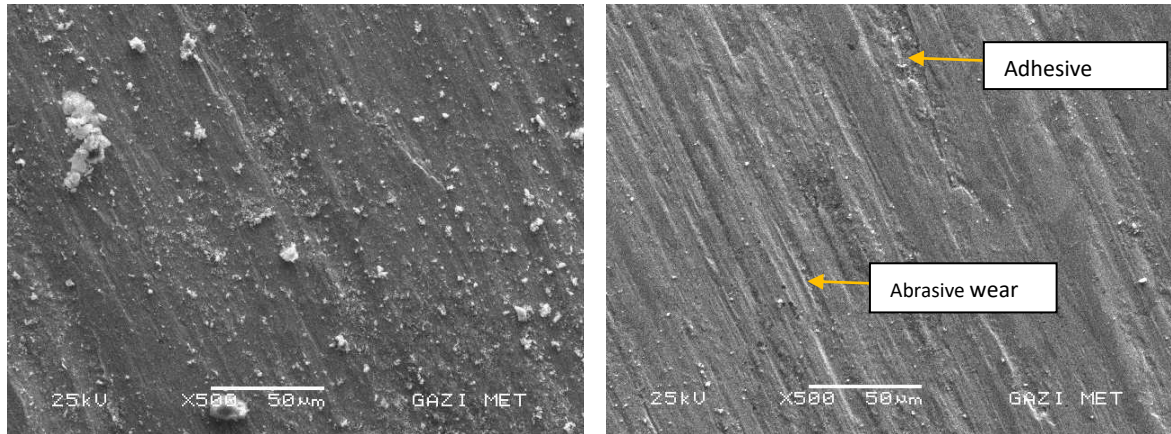


Figure 9. SEM micrographs of worn surface

Sliding speed and load have been changed in all applications of the wear tests, the friction force has varied between 2.5 and 13.5 N while the wear rate varied between 0.2 and 0.9. As the load applied during both abrasion rate increased, a very slight change has been observed in the abrasion force. Similarly, significant increase has not been in wear coefficients in general (Fig. 10 and 11). The most important reason for this is that while the abrasion continues, the residual carbon in the powder metal part has acted as a lubricant at the interface. Secondly, upon a rise in the heat at the interface, copper has partially plastered on the abrasive disc exhibiting a ductile behavior.

These elements have got worn so as to constitute an interface between PM sample and the abrasive disc thus improved the abrasion performance of PM part.

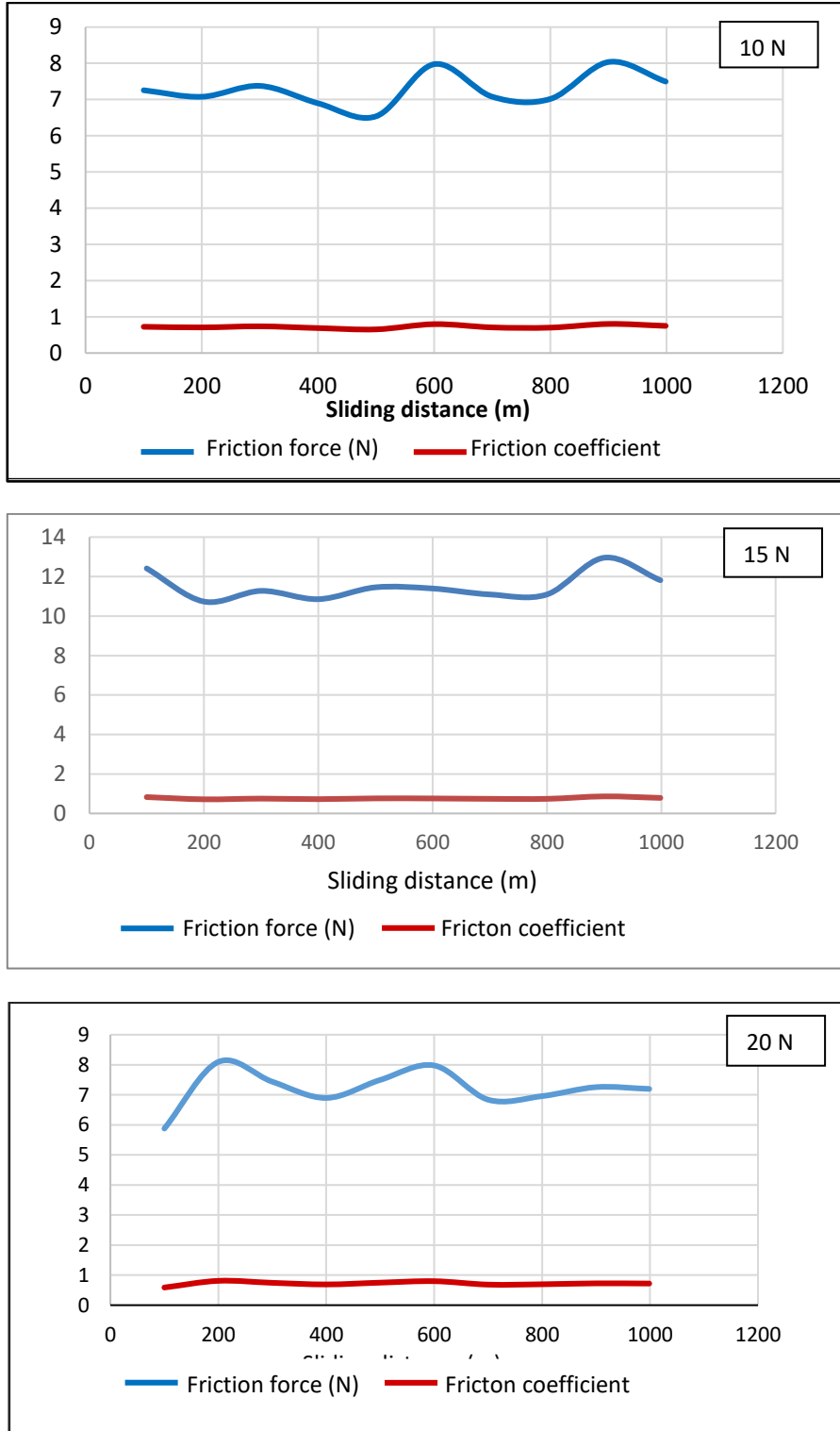


Figure 10. Changing of PM sample's friction coefficient and friction force at 0.69 m/s sliding speed and under different loads

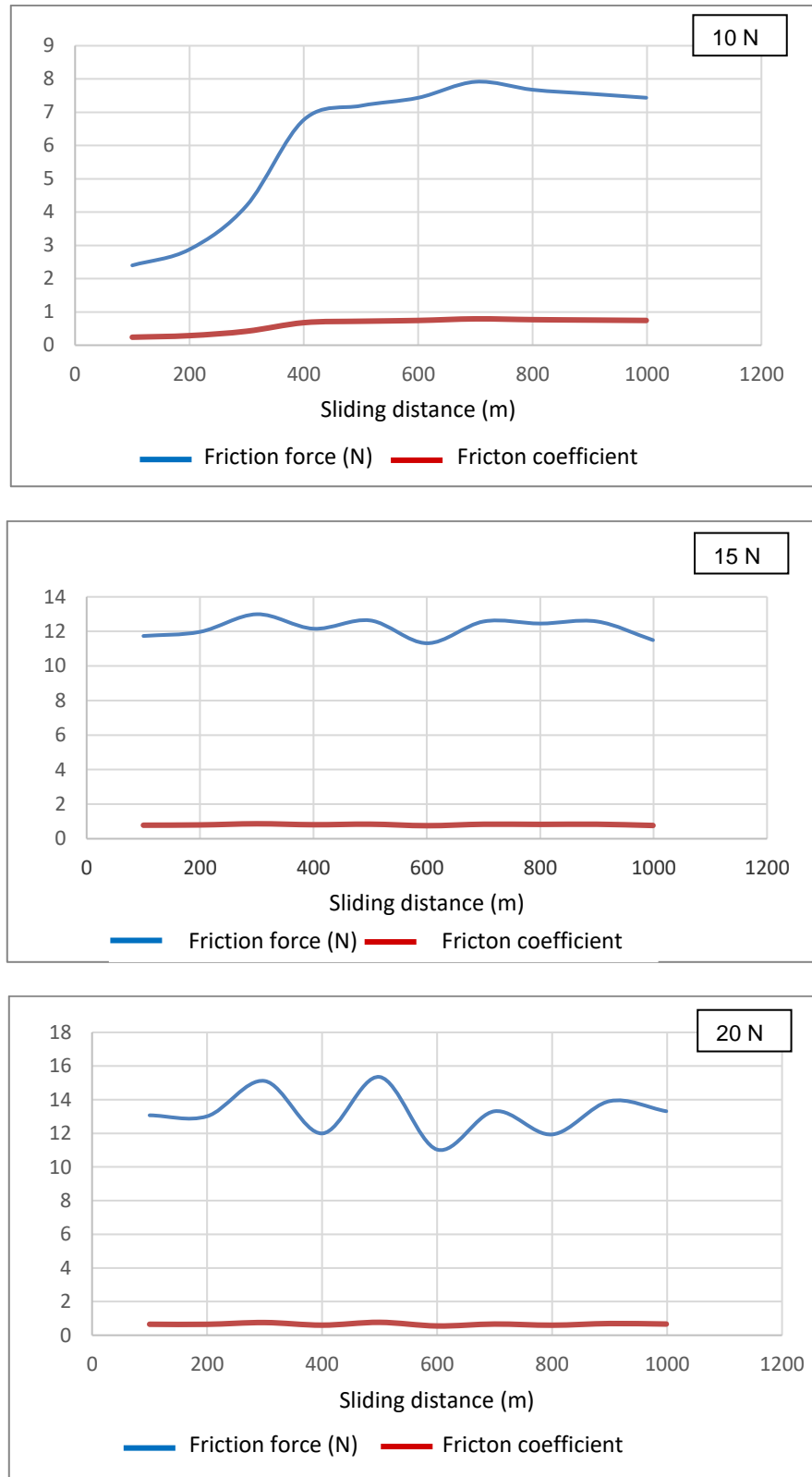


Figure 11. Changing of PM sample's friction coefficient and friction forces at 0.92 m/s sliding speed and under different loads

Naturally, as the load applied at both abrasion speed increased, naturally the amount of wear has also increased (Figure – 12). We see that the wear value increased under all applied loads when the erosion

speed increased. This can be explained by a remarkable increase in the temperature at the interface as the rate of abrasion has increased.

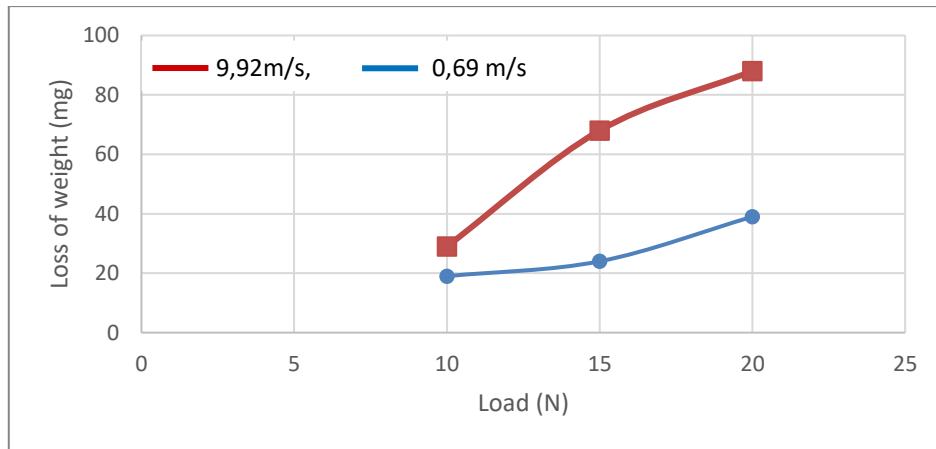


Figure 12. Weight loss due to the load and sliding speed

4. CONCLUSIONS

PM parts with projected density, hardness and microstructure from mixed powder composed of 2% copper, 0.75% graphite and 0.80% zinc stearate as the lubricant has been produced. Higher pressing pressure and the longer sintering time in comparison to similar studies in the literature enabled the production of denser and harder PM parts at lower temperature. Approximately 40% pearlitic structure formation by diffusing carbon into the iron particles during sintering. Cu provided increment in wear resistance by liquid-phase sintering and diffusing into iron particles. Increasing sliding speed and wear load led to increasing wear loss. In process of dry sliding tests realized at three different load and two different abrasion rate, as the load and abrasion speeds increased, the abrasion values have increased too. Even the residual graphite and copper remained in the PM part have acted as the lubricant at the interface in a sense and have affected the wear behavior of PM part positively.

CONFLICT OF INTEREST

The author declares that there is no conflict of interest regarding the publication of this paper.

REFERENCES

- [1] German, R.M., (Çevirenler: Sarıtaş, S., Türker, M., ve Durlu, N.), “Powder metallurgy and particulate materials processing”, 2-11, (2007). Yılmaz, R., Özyürek, D., “Wear properties of Fe-Cu-C based materials produced by powder metallurgy”, *X. International advanced technologies symposium (IATS'09)*, Karabük, 13-15 (2009).
- [2] Çavdar, U., Ünlü, B.S., Atik, E., “Effect of the copper amount in iron-based powder-metal compacts”, *Materials and technology*, 48 (6): 977-982, (2014).
- [3] Uygur, İ., “Influence of manganese addition on mechanical properties of Fe-Cu-C steels”, *J. Fac. Eng. Arch. Gazi Univ.*, 22 (3): 325-330, (2007).
- [4] Uzun, H., Ölmez, C.O., “Effect of heat treatment on hardness of Fe-Cu ve Fe-Cu-C materials”, *Sa. Ü. Journal of Science*, 4 (1-2): 91-97, (2000).
- [5] Salman, K.D., Khalifa, M.Z., Mohammd A.M, “Pore formation during sintering of two different composites materials (Fe-Cu) & (Fe-Cu-C) and its effects on wear behavior”, *J. of Engineering and Sustainable Development*, 20(05): 64-74, (2016).
- [6] Prabua, S.S., Prathibab, S., Asokan M.A., “Investigations on dry sliding wear behaviour of sintered/extruded P/M alloy steels (Fe-C-W-Ti)”, *Procedia Engineering*, 97, 2119-2126, (2014).
- [7] German, R.M., Suri, P., Park, S.J., “Review: liquid phase sintering”, *Journal of Materials Science*, 44 (1): 1-39, (2009).

- [8] Tjong, S.C., Lau, K.C., “Properties and abrasive wear of TiB₂/Al-4% Cu composites produced by hot isostatic pressing”, *Composites Science and Technology*, 59, 2005-2013, (1999).
- [9] Yıldız, T., Gür, A.K. “Wear mechanism”, *Doğu Anadolu Bölgesi Araştırmaları*, 86-91, (2006).
- [10] Gül, F. İlivan, M., “Statistical analysis of factors affecting abrasive wear behavior of Al composites reinforced by SiO₂”, *4th International Symposium on Innovative Technologies in Engineering and Science*, Alanya/Antalya, 1799-1809, (2016).
- [11] Rathore, S.S., Dabhade, V. V., “Dimensional change during sintering of Fe–Cu–C alloys: A Comparative Study”, *Trans Indian Inst Met*, 69(5): 991-998, (2016).
- [12] Morakotjinda, M., Krataitong, R., Wila, P, et al., “Sintered Fe-Cu-C materials” *Chiang Mai J. Sci.*, 35(2): 258-265, (2008).
- [13] Laurent, S.S., “Effect of powder mix formulation and sintering conditions on the dimensional stability of sinter hardening powders”, *XI. International Conference on Powder Metallurgy for Automotive and Engineering Industries*, in Pune/India in February (2011).
- [14] Bernardo, E., Oro, R., Campos, M., et. al., “New findings on the wettability and spreading of copper on iron-base substrates”, *International Journal of Powder Metallurgy*, 51(4): 29-36, (2015).
- [15] Çivi, C., Atik, E., “Effect of the sintering temperature to mechanical properties of Fe based copper and graphite containing materials”, *CBU J. of Sci.*, 13 (2): 467-472, (2017).
- [16] Narasimhan, K.S., Semel, F.J., “Sintering of powder premixes – A brief overview”, *Hoeganaes corporation 1001 Taylors lane Cinnaminson, NJ 08077*, Paper No:01-0145, (2007).
- [17] ASTM G99-05 (Reapproved 2016), Downloaded /printed by Gazi University pursuant to Licence Agreement.
- [18] Höganäs PM-school, “Sintered iron-based materials”, Page: 9-19, (2013).
- [19] Çavdar, U., Zeybek, M.S., Koç, M., et al., “Determination of the sintering temperature in the ultra high frequency induction sintering system for iron based powder metal compacts”, *7th International Powder Metallurgy Conference and Exhibition*, Ankara, 1-6, 24-28 June, (2014).
- [20] Iwaoka, T., Fujiki, A., “Microstructure and strength of Fe-2% Cu-0,8% C sintered steel using Fe-Cu alloy powder”, *J. Jpn. Soc. Powder Metallurgy*, 6 (6): 290-297, (2014).