Materials Research Express

PAPER

Fabrication and tribological behavior of Fe-Cu-Ni-Sn-Graphite porous oil-bearing self-lubricating composite layer for maintenance-free sliding components

Junde Guo, Hang Du, Geng Zhang, Yan Cao, Junwen Shi and Wei Cao

1 School of Mechatronic Engineering, Xi’an Technological University, Xi’an, 710021, People’s Republic of China
2 Light Industrial Xi’an Mechanic Design Research Institute Company Limited, Xi’an 710086, People’s Republic of China

E-mail: gjd0119@163.com

Keywords: Friction and wear, Graphite, Pore, Fe-Cu-Ni-Sn alloy, Self-lubrication

Abstract

Fe-Cu-Ni-Sn-Graphite oil-bearing self-lubricating composite was prepared using powder metallurgy method through multiple sintering-rolling processes and oil impregnation vacuum-assisted method. The tribological property of the composite were characterized by a UMT-2 tribometer. And the micro-structure and worn surface of composite were investigated by an optical microscope, SEM (scanning electron microscopy), XRD (X-Ray Diffraction) and EDS (energy disperse spectroscopy). The results show that the porous oil-bearing self-lubricating composite exhibit an excellent long-term low-friction and antiwear properties. Especially, the friction coefficient of the composite after secondary rolling and sintering decreased by 38% compared to that of the sample without sintering-rolling. After sliding for 21 h, the friction coefficient of the composite still keep relatively stable at a low value. In addition, the wear resistance of the composite has greatly improved after secondary rolling and sintering. And the wear loss of the 40Cr counterpart is almost wear-free and presents oil-graphite mixture around the wear track, which reveals that solid-liquid synergetic lubrication is the potential lubrication mechanism during the sliding process.

1. Introduction

Iron-based self-lubricating materials prepared by powder metallurgy technology were widely used on automobile, instrumentation, agricultural engineering, precision machinery and other fields due to their abundant raw materials, high strength, good shock absorption capacity and tribological properties [1–5]. The iron-based porous oil-bearing materials [3], such as cooling bed bearing, conveying track bearing, sliding guide rail of machine tool, hydraulic power steering valve sleeve and conventional engine design, are mostly prepared by powder metallurgy process, which have significant oil-bearing self-lubricating and energy-saving characteristics [6, 7]. In the course of service, the oil stored in the pores of the matrix continuously precipitates to the friction interface, keeping the friction state in the boundary lubrication condition [8]. The research on the self-lubricating characteristics is mostly focused on solid bearing materials [9]. The iron-based porous oil-bearing materials can provide more superior tribological performance on the basis of sufficient mechanical strength due to the oil-bearing lubrication effect, which is expected to improve the friction-reducing performance under boundary lubrication condition [7, 10].

As an inherent property of powder metallurgy iron-based materials, porosity can reduce the mechanical strength to some extent, which is difficult to give consideration to both the high load-bearing and excellent self-lubricating properties for powder metallurgy materials [11]. The combination of porous oil-bearing design and solid-lubrication technology can form a solid-liquid synergistic lubrication effect, which can effectively improve the tribological properties of the sliding interfaces [12]. Solid-lubrication technology is an effective method to improve the boundary lubrication performance, which is widely used in surface modification of bearings and other lubricating parts [13]. As a solid lubricant, graphite has the characteristics of low shear strength, easy sliding, loose
Table 1. Specifications of raw material powders.

| Name           | Specification | Purity/% | Appending proportion (wt%) |
|----------------|---------------|----------|----------------------------|
| Iron powder    | ≈45 μm, NC100.24, Höganas | >99.4    | 40 wt%                     |
| Nickel powder  | ≈50 μm, Shanghai, No. 2 Metal Smeltery | >99.8    | 15 wt%                     |
| Copper powder  | ≈75 μm Shanghai, No. 2 Metal Smeltery | >99.5    | 35 wt%                     |
| Tin powder     | ≈80 μm Shanghai, No. 2 Metal Smeltery | >99.5    | 6 wt%                      |
| Graphite powder| ≈20 μm Shanghai, No. 2 Metal Smeltery | >99.5    | 4 wt%                      |

2. Materials and methods

2.1. Materials

The raw materials used in the experiment include iron powder, nickel powder, copper powder, tin powder and graphite powder. The specifications of various powders are shown in table 1. The powders used in this paper are widely used in mechanical industry.

The raw materials were weighed by an electronic balance with an accuracy of 0.1mg. During the mixing process, 10 g ~ 12 g (±2 wt%) lubricating oil (room temperature viscosity: 450 CPR) was added as auxiliary material for pore forming and mixed evenly in a three-dimensional mixer for 2 ~ 3 h. The copper plated steel was used as the substrate to improve its strength and stiffness. The thickness of cladding copper was about 8 ~ 15 μm. It was pressed on a 40T pressing machine, the pressure was 600 ~ 650 MPa, which was holding for 2 min before sintering. The samples were sintered at 1080 °C for 90 min. And then cooled with the furnace. Both the sintering and cooling processes were in nitrogen atmosphere. After the above process, the samples were carried out by secondary rolling and secondary sintering. The parameters for sintering and rolling processes are shown in table 2. All the samples were treated with vacuum oil immersion (Parameter of oil: room temperature viscosity 380 cps) at 120 °C.

2.2. Characterization

The hardness of sintered samples was tested on a brinell hardness tester (HB-62.5kg, Laizhou Instrument Equipment Co., LTD., China); the mechanical properties at room temperature were measured by a universal material testing machine; the density of sintered samples was measured on electronic balance; the morphology of friction layer was observed by JSM-5600 scanning electron microscope (SEM), and the composition of friction layer was analyzed by EDS. The friction and wear properties of the material were investigated on a high temperature pin disc friction and wear tester. The test block was made of the developed material, the size of the pin was Ø5mm after wire cutting, and the test plate was 40Cr steel plate (Hardness: 30 HRC) with a size of Ø45mm. The test conditions are room temperature and atmospheric environment, and the test time is 30 min. As shown in figure 1. Test conditions: the load is 2 MPa, the friction radius is 30 mm, the sliding time was 21 h, and the test speed was 1 m s⁻¹.
3. Results and Discussions

3.1. Morphology of the frictional sintered layer
As shown in figure 2, the thickness of the sintered layer decreases after the secondary rolling process. As shown in table 3, the thickness of the composite cladding shows 16.2% decreasing after the first sintering, and 30% decreasing after the secondary rolling and sintering. The contrast of the cross profile image was enhanced by gray expanding and the binarization process based on the dynamical method. The porosity results show that porosity of the sample (graphite involved) is about 40.2% after the first sintering process, further, the porosity decreases by 17% after the first rolling, and the average diameter of the pore decreases by 5%, yet the porosity and average pore diameter of the sample decrease by about 17% and 9% after the secondary rolling and sintering. The hardness of the sample respectively can be increases by 12% and 30% after the first sintering & rolling and the secondary rolling. The above mentioned results prove that the sintering and rolling process improves alloying extent and the hardness of the composite sample.

3.2. Element analysis of the frictional sintered layer
Figure 3 shows the SEM morphologies of sintered samples and corresponding EDS results. As shown in figure 3, the matrix composition of the three kinds of sintered samples includes Fe, Cu, Ni and Sn elements, and there is no graphite-state carbon signal due to the limited precision of EDS equipment. Through the sintering and rolling processes, the content of Cu element increase and Ni decrease in proper order, which is caused by precipitation of Cu element, besides, solid solution of Ni and Fe is formed after several times of sintering. According to SEM spectrum, sintering of metal matrix phase of sample P16-6 and P16-5 are not dense. A few metal particles exist in original form, continuous phase is not formed in metal matrix, and the pores are in non-uniform distribution. Sample P16-4 shows relatively uniform pore distribution and the formation of continuous phase after secondary sintering-rolling process.

To clearly detect the element distribution in different parts of the matrix, the matrix was applied with corrosion treatment with 4wt% nitric acid solution as shown in figure 4, and it was found that the matrix has dark-grey region and light-grey region. According to EDS element test of parts of sample, the dark-grey region (A) of the sintered surface is mainly Fe and Ni solid solution, while the light-grey part (B) is mainly Cu solid solution.

3.3. Composition analysis of the frictional sintered layer
Figure 5 illustrates the XRD patterns of the sintered samples with different treatments under three different preparations. It shows that the samples are of basically the same structure but different extent of alloy solid
solution as shown in figure 5. The compositions of P16-6 and P15-5 are almost the same, contain Cu$_3$Fe$_{17}$ (Reference code: 03-065-7244), Ni$_{17}$Sn$_3$ (Reference code: 03-065-7194) and Fe-Ni-Cu solid solution. Lower peak value of Fe and Fe-Ni-Cu alloy can be detected on sample P16-4 after secondary sintering-rolling, indicating that the solid solubility of iron, copper and nickel increases. Major framework phase of Cu alloy on sample P16-4 indicates that secondary sintering-rolling process can improve alloying extent for Fe-Cu-Ni-Sn-graphite self-lubricating composite.

3.4. Friction and wear performance test

To study the tribological performance of self-lubricating samples on 40Cr steel disks, the 40Cr surface roughness was 0.043 μm. The diameter of cylinder self-lubricating pin was 3 mm, and the disks were cleaned for 15 min in ethanol absolute for ultrasonic.

Figure 6 shows the friction coefficients of three frictional sintered samples over the 21-hour sliding process. As shown in the figure 6 and figure 7, Sample P16-4, which is processed with secondary sintering-rolling, shows a stable curve of friction coefficient, with the average friction coefficient of 0.075 during the run-in period and 0.056 during the stable period. This sample has the best friction performance, with the corresponding wear loss of 2.8 mg. Friction coefficient of the sample P16-5 remains stable at average friction coefficient of 0.07 in the first 600 min of sliding test, yet continuously increases afterwards. As for sample P16-6, friction coefficient is unstable during the first 200 min and reaches 0.1 at maximum; during the sliding period of 200-900 min, the friction coefficient remains around 0.045, which is even lower than sample P16-4; and after 900 min, the friction coefficient increases rapidly to a higher value than 0.2 at least, which is unstable and fluctuates obviously. During
the sliding test, no extra lubricant was added. The friction process relies on solid-liquid lubrication generated by solid-liquid lubricant composed by lubricant and graphite flows out of the pores. Wear loss of all three kinds of materials decreases with the rolling and sintering treatment. The rolling-sintering treatment can minimize wear to a certain extent.

3.5. Wear mechanism

Figure 8 shows the worn surfaces of three kinds of frictional sintered samples. The major wear mechanisms for P16-6 sample are abrasive and fatigue wear, and the corresponding surface shows fatigue flake and furrows, the grain form of the torn surface shows potential relevance with the sintering process. However, the wear loss of the sample is not high (only 6.4 mg for 21 h sliding), the possible reason is that P16-6 can supply more lubricating oil since the larger pore size of pores. However, the amount of lubricating oil stored in the porous structure is limited and cannot be supplied for a long period, but it still has good anti-wear performance in the 21 h sliding test. All the samples show the mixture of lubricating oil and graphite overflows around the wear track after sliding for 21 h. The worn surfaces of sample P16-5 and 16-4 basically remain the initial morphology, which have only several light scratches, these results are consistent with the lower friction coefficient of the friction coefficients of two samples sliding with 40Cr steels. The wear loss of sample P16-5 and 16-4 are lower than that
of sample P16-6, due to the better lubricating oil storage capacity for sample P16-5 and sample 16-4 which has smaller pore size of pores after the secondary sintering-rolling process.

Figure 9 shows the worn morphology of 40Cr tribo-pairs sliding with Fe-based self-lubricating samples. It is observed that a large amount of black sediment transfer around the wear track on the 40Cr steel after 21 h.
sliding, which can be related to larger diameter of pores for P16-6. On the premise of constant viscosity of lubricating oil, large size of pores can weaken the storage capacity of the samples for the lubricating oil, which can be squeezed out during the process of sliding, and gather around the sliding track. Sample 16-6, which undergo the secondary sintering-rolling process, has better storage capacity for the lubricating oil and the lowest friction coefficient even in a stable period. Lubricating oil plays an important role in this period. Due to a limited amount of lubricating oil in the pores, it is unable to maintain long-term lubrication. Therefore, the friction coefficient of P16-6 rises sharply after 840 min of sliding. Worn surface of 40Cr steel plate sliding with sample P16-6 is smooth, and there are almost no scratches on the 40Cr tribo-pair, only with the remaining black mixture of oil and graphite.

To investigate the effect of lubricating oil in the friction and the oil-bearing property of the porous self-lubricating composite, the tribological property of the sample without vacuum oil immersion was tested under dry friction condition as shown in figure 10. The friction coefficient of the sample with vacuum oil immersion is about 85% higher than that of the sample without vacuum oil immersion process. Lubrication oil contained in porous materials plays a leading role in the process of friction reduction. This result shows that the oil and solid lubricant play a synergistic role in reducing friction and wear. The above results indicate the solid-liquid synergistic lubrication effect plays a leading role for the Fe-Cu-Ni-Sn-Graphite porous oil-bearing self-lubricating composite sliding with the 40Cr steel.

In summary, the hardness and tribological properties can be strengthened through the secondary sintering-rolling process, and it is easy to resist the vibration and instability under high-load sliding. The looser surface layer of the composite materials in this design, which was generated by the in situ synthesis reaction, has a better self-lubricating property. The steel substrate can effectively prevent the oil moving downward and keep the...

Figure 6. Friction coefficient curves of three frictional sintered materials.

Figure 7. Average friction coefficient and mass loss of the self-lubrication composites after sliding.
lubricant between the frictional interfaces, and the graphite can also be squeezed into the sliding interface during extrusion. The special structure design is not easy to deform under high load conditions. The friction reduction and lubrication effect of sintered oil-bearing materials is due to the storage of lubricating oil and graphite embedded in the composite. In addition, the lubricating oil expands under the action of friction heat and flows under the action of load extrusion [19]. The oil in the pores can precipitate to the friction interface and play a lubricating role [20].

4. Conclusions

1. Based on powder metallurgy technology, self-lubrication layer of multi-pore metal matrix with self-lubrication property was prepared by secondary sintering-rolling after compression molding sintering with vacuum oil immersion.

2. The macrohardness and the porosity of composite have increased using a secondary rolling-sintering treatment caused by improved alloy extent of metal mixture, the multi-pore structure with pore diameter approximately 176 μm has the best lubrication effect.

Figure 8. Worn surfaces of frictional sintered samples sliding with 40Cr steel (a), (b): P16-6; (c), (d): P16-5; (d), (e): P16-4.
3. The sample processed with secondary sintering-rolling, shows a stable curve of friction coefficient, with the average friction coefficient of 0.056 after the run-in period. The wear loss of all three kinds of materials decreases with the rolling and sintering treatment. The friction coefficient of the sample with vacuum oil immersion is about 85% higher than that of the sample without vacuum oil immersion process, which reveals that the major lubrication mechanism is solid-liquid lubrication generated by graphite and overflow lubricating oil.

Figure 9. Wear morphologies of 40Cr tribo-pairs sliding with different samples.

Figure 10. Friction coefficients of the samples with vacuum oil immersion and without vacuum oil immersion sliding with 40Cr steel.
Funding

This work was supported by Special Research Project in Shaanxi Province Department of Education (20JK0668), Innovation Capability Support Program of Shaanxi (Name: Guo Junde), Science and Technology on Diesel Engine Turbocharging Laboratory (6142212190104) and International Science and Technology Cooperation and Exchange Program of Shaanxi Province (2020KW-014).

ORCID iDs

Junde Guo @ https://orcid.org/0000-0002-9096-430X
Hang Du @ https://orcid.org/0000-0002-8806-3879
Yan Cao @ https://orcid.org/0000-0002-2959-5533
Wei Cao @ https://orcid.org/0000-0002-4545-5995

References

[1] Zhang G T et al 2019 Controllable preparation and self-lubricating mechanism analysis of bilayer porous iron-based powder metallurgy materials Acta Metall Sin 55 1448–56
[2] Hammes G et al 2017 Effect of hexagonal boron nitride and graphite on mechanical and scuffing resistance of self-lubricating iron based composite Wear 376 1084–90
[3] Zhang G T et al 2018 Tribological properties and mechanism of the bilayer iron based powder metallurgy materials Ind. Lubr. Tribol. 70 1642–8
[4] Li X W et al 2020 Tribological behaviors of vacuum hot-pressed ceramic composites with enhanced cyclic oxidation and corrosion resistance Ceram. Int. 46 12911–20
[5] Xie Z et al 2020 Theoretical and experimental investigation on the influences of misalignment on the lubrication performances and lubrication regimes transition of water lubricated bearing Mechanical Systems & Signal Processing 149 107211
[6] Shan Y et al 2020 Iron-based porous metal–organic frameworks with crop nutritional function as carriers for controlled fungicide release Journal of Colloid & Interface Ence 566 383–93
[7] de Mello J D B et al 2018 Influence of surface finishing on the tribological behavior of self-lubricating iron-based composites Tribol. T 61 569–8
[8] Campos K R et al 2015 Tribological evaluation of self-lubricating sintered steels Wear 332 932–40
[9] Iia Z N, Yan Y H and Wang W Z 2017 Preparation and tribological properties of pi oil-bearing material with controllable pore size Ind. Lubr. Tribol. 69 88–94
[10] Dimkovski Z et al 2009 Quantification of the cold worked material inside the deep honing grooves on cylinder liner surfaces and its effect on wear Wear 267 2235–42
[11] Simchi A and Danninger H 2013 Effects of porosity on delamination wear behaviour of sintered plain iron Powder Metall. 47 73–80
[12] Wu P et al 2019 Synergistic tribological behaviors of graphene oxide and nanodiamond as lubricating additives in water Tribol. Int. 132 177–84
[13] Hu T C, Zhang Y S and Hu L T 2012 Tribological investigation of mos2 coatings deposited on the laser textured surface Wear 278 77–82
[14] Gozbenko V E et al 2020 Replacement of graphite by petroleum coke in rail lubricants Coke & Chemistry 63 183–7
[15] Bender S, El Wakil S D and Chalivendra V B 2013 Fabrication and characterisation of powder metallurgy parts having porosity gradient Powder Metall. 54 599–603
[16] Mouri T et al 2015 Strength and friction-wear properties of complex layer bearing via unification powder forming and sintering process Journal of the Japan Society of Powder & Powder Metallurgy 62 371–6
[17] Guotao Z et al 2018 Tribological properties and mechanism of the bilayer iron based powder metallurgy materials Ind. Lubr. Tribol. 70 162–8
[18] Firstov S A, Pochkovsky E P and Ivanova I I 2006 High-temperature mechanical properties of powder metallurgy: porous lightweight titanium nanolaminates High Temperature Materials & Processes 25 47–58
[19] Shengyu Q et al 2019 High temperature solid-lubricating materials: A review Tribol. Int. 133 206–23
[20] Armada S et al 2013 Liquid-solid self-lubricated coatings J. Therm. Spray Techn. 22 10–7