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FRICTION AND WEAR PROPERTIES OF SINTERED SULFUR-CONTAINING HIGH-SPEED STEELS AT ELEVATED TEMPERATURE

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Abstract

Ferrous alloys developed on the basis of P/M high speed steels have shown enormous potential in addressing friction, wear, and lubrication problems faced by many military and civil industries. Sulfur in the alloys can react with certain elements to form some sorts of solid lubricants, which provides a promising solution to lubrication problems in many high-temperature engineering fields. Sulfur-containing high speed steels were prepared using powder metallurgy techniques here. High temperature friction properties of the samples were studied using a self-matching friction couple on a pin-on-disk tribo-meter. Worn surfaces were analyzed and the possible friction mechanism was discussed. It was found that the friction coefficient curve became more stable but the values decreased gradually with increasing temperature. The contained sulfides help to reduce the friction coefficient. Oxides formed on the worn surfaces can be considered as a major contribution to the tribological mechanism.

Introduction

Ferrous alloys developed on the basis of P/M high speed steels have shown enormous potential in addressing friction, wear, and lubrication problems faced by many military and civil industries [1,2]. Some studies have shown that the tribological properties of iron-based alloys (and also nickel-based superalloys) can be effectively improved by introducing an appropriate amount of elemental sulfur into the alloys. It is believed that sulfur in the alloys can react with certain elements to form some sorts of solid lubricants, either during alloy fabrication or during the process of friction, which provides a promising solution to lubrication problems in many high-temperature engineering fields, such as aerospace, power machinery, and metal processing [3,4]. In this study, we propose to examine the high-temperature friction performance of sulfur-containing high-speed steels mating with self-match counter materials. A high speed steel fabricated by powder metallurgy (P/M) techniques was chosen as the matrix alloy. Friction performance and mechanism at high temperature will be further discussed, which is of great significance to the research and development of new, improved iron-based friction materials [5,6].

1. Experiment details

1.1 Fabricating process

Raw materials used here are M3/2 high speed steel powders of -100 mesh in size made by Höganäs and MoS_2 powders of -250 mesh in size. The chemical composition of high speed steel powders is provided in Table I. M3/2 is a fully pre-alloyed powder that was annealed after water atomization. 97.5 wt% M3/2 and 2.5 wt% MoS_2 were mixed and blended in a -V-shape blender for 120 min, and then pressed in a steel mould under a pressure of 7 ton/cm^2. The green parts pressed were sintered at 1250°C in the DL-VF-30 vacuum furnace for 60 min and then cooled down to room temperature by furnace cooling.

Table I. Chemical composition of M3/2 high-speed steel(wt%)

M3class2	C	Cr	Co	Mn	Mo	Si	V	W	O
Min	0.90	3.75	-	-	4.75	-	2.75	5.00	-
Max	1.10	4.50	1.00	0.40	6.50	0.45	3.35	6.75	1000ppm
Tolerance±	0.05	0.25	-	0.12	0.30	0.12	0.20	0.30	250

1.2 Test and analysis

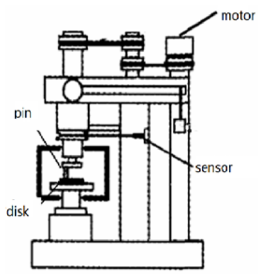


Figure 1. High-temp. face wear tester (MMU-10G)

Hardness was measured with Rockwell hardness tester. Friction coefficient was determined using a MMU-10G high-temperature tribo-meter, which performs pin-on-disk friction testing with a fixed disk in contact with a rotating pin (Figure 1). Sizes of the pin and the disc were measured as Φ4 mm × 18 mm and Φ50 mm × 4 mm, respectively, and the rotating radius was 12mm. Self-match (a high speed steel containing 2.5 wt% MoS_2) was chosen to be the mate materials under tests. The bend strength and the hardness of the tested material were measured to be 1380MPa and 37 HRC, respectively. Samples' surfaces were polished with No. 500 sandpaper, and after being degreased and cleaned, the samples were presented for friction testing under conditions of a load of 100 N, a rotational speed of 400 rpm（0.46m/s）, a time period of 30 min, and a variety of temperatures (400°C, 550°C, and 700°C). The data was collected and recorded, and the average friction coefficient was calculated in a fully automatic way. Wear rate was determined by weight loss with considering the effects of high temperature oxidation. Microstructures were examined using a 6532-01 MeF3A microscope and crystalline phases were identified using a Japan D/max-γ X-ray diffractometer. Worn surfaces were also examined under a JSM-6360LV scanning electron microscope (SEM) with energy dispersive spectroscopy (EDS) and a K-Alpha 1063 X-ray Photoelectron Spectroscopy(XPS).

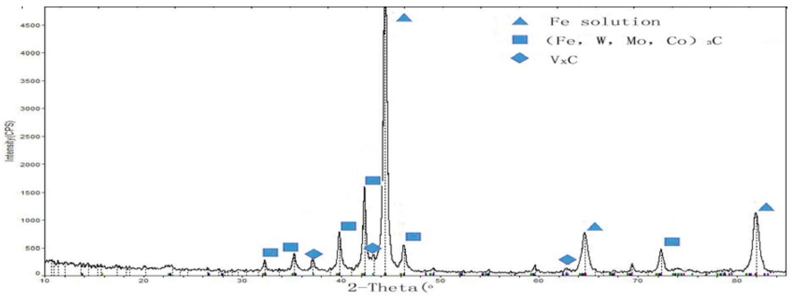
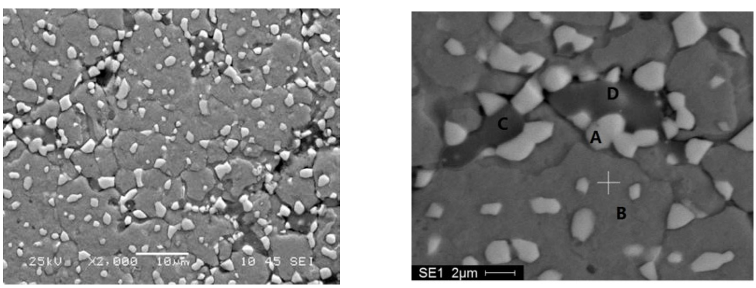


Figure 2. XRD of materials

2. Microstructures

Two major phases, vanadium carbide and the ferrous solid solution, (Fe,W,Mo,Co) $_3$C , were identified by X-ray diffraction (XRD) (Figure 2). The crystalline phase of MoS_2 was not detected in the XRD pattern which was expected due to its decomposition as a result of sintering at temperatures above 1100 °C. Further metallographic and EDS analyses (Figure 3 and Table II) reveal that at least other four different phases exist in the alloy: at area A (a dispersed particle) or B (the iron-based matrix), the chemical composition is dominated by Fe. The contributions of W, Mo, Cr and V are also detected, while sulfur is not present. Area C is enriched in S, followed by Mn, Cr, Fe and V. The composition in area D is somewhat similar as that in area A, but with the presence of 13% S. It is suggested for area C and D that sulfur has been decomposed from MoS_2 and might have formed some sorts of sulfides with the alloying elements. The presence of a large number of fine dispersed carbide phases in the iron-based matrix is favorable to improve the high temperature performance of the material.



(a) SEI (b) BEI

Figure 3. SEM of M3/2-2.5% MoS_2

Table II. Element distribution in different zones(wt%)

Tissue	W	Mo	S	V	Cr	Mn	Fe
A	26.74	23.04	0	3.07	3.21	0	43.93
B	4.94	3.37	0	1.44	4.00	0	86.25
C	3.94	7.10	27.93	12.40	15.01	19.82	13.79
D	12.75	12.97	13.06	7.93	8.61	0.57	44.11

3. Results and discussions

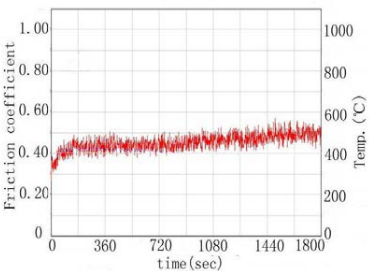
3.1 Friction coefficient and wear rate

Friction coefficient and wear rate of the material sensitively depend on the ambient temperature. Table III compares the average measurement of friction coefficients and wear rates under three different temperatures (400°C, 550°C and 700°C). It is seen that the friction coefficient decreases with the increasing temperature, from 0.47 at 400°C to 0.34 at 700°C, while the wear rate is even eight times higher at 700°C than at 400°C.

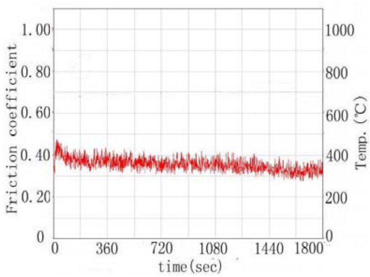
Table III. Average friction coefficient and wear rate

Temperature(°C)	400	550	700
Friction coefficient	0.47	0.38	0.34
wear rate($\times 10^{-14} m^3/N.m$)	0.92	1.47	7.5

Figure 4 presents the curves of the friction coefficients varying with time measured at 400°C and 700°C, respectively. It is noticed that at lower temperatures the friction coefficients are rather volatile over time. At 700°C, the friction coefficients are slightly reduced, but also somewhat stabilized with time.



(a) 400°C

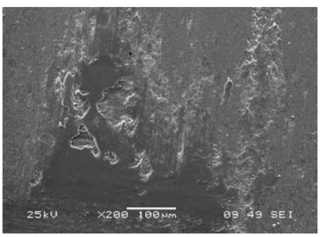


(b) 700°C

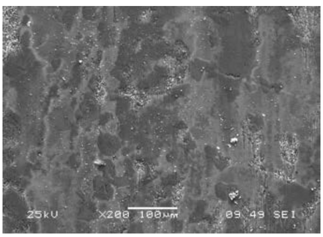
Figure 4. Curves of friction coefficient vs. time at different temperatures

3.2 Tribological mechanism analysis

Figure 5 shows the SEM secondary electron images of the worn surfaces after the friction tests. The corresponding EDS results are shown in Table IV.



(a) 400°C



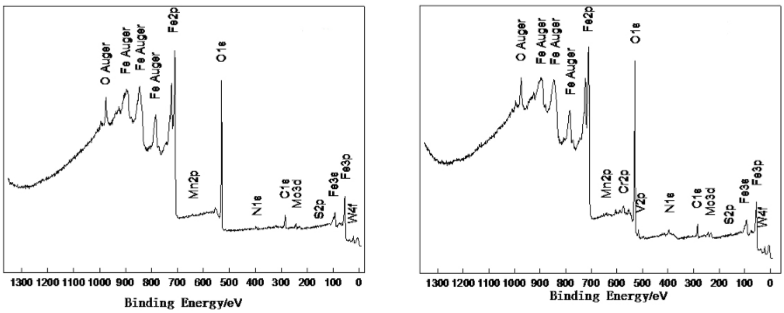
(b)700°C

Figure 5. SEM of worn surface

Table IV. EDS of worn surface at different temp. (wt%)

Element	O	W	Mo	S	V	Cr	Mn	Fe
400°C	0.22	9.45	8.32	0.98	4.41	4.26	0.42	71.92
700°C	0.71	5.03	3.95	0.0	2.36	2.08	1.15	84.72

It is evident from the Table IV that there exists a small concentration of sulfur on the worn surfaces at 400°C, which is a sign of solid lubrication effects due to sulfide softening [7-9]. As the temperature rose, the sulfides began to decompose while oxides began to form on the worn surfaces. At 700°C, sulfides have been reduced to a nearly undetectable level, and instead, more oxides have been produced.



(a)400°C （b）700°C

Figure 6. XPS of worn surface

Table V. XPS results of worn surface at different temp. (at%)

Name	400°C		700°C	
	Peak BE	At. %	Peak BE	At. %
W4f	35.28	0.19	35.39	0.42
Mo3d	231.29	0.44	232.07	0.72
C1s	284.78	17.22	284.82	18.91
V2p	514.84	0.19	515.25	0.75
O1s	529.9	43.86	529.86	43.87
Cr2p	587.94	0.2	573.02	1.46
Fe2p	709.57	37.09	709.48	31.72
S2p	161.52	0.51	161.18	1.63
Mn2p	641.9	0.28	641.01	0.53

Figure 6 presents the XPS spectra of the worn surfaces. The surface elemental chemistry differs little from each other, suggesting that the responsible friction mechanisms were basically the same. The existence of sulfur on worn surface at 700°C, which is not detected in EDS(in Table IV), is mainly because of the differences for these two analyzing manners. The atomic concentrations of sulfur at 700°C are higher than 400°C, possibly because the sulfides became soft and easier to disperse out on the surface at high temperatures. This provides self-lubrication of the material and also helps reduce and stabilize the friction coefficient. From Table V, the formation of oxides on the worn surfaces is also evident. As the temperature rises, the compositions of the oxides also change accordingly: the amounts of W, Mo, V, Cr and Mn oxides have increased while, in contrast, Fe-containing oxides have decreased. It is known that oxides on the worn surfaces can form a glazing layer that acts as solid lubricant film [10-12]. Therefore, the increasing amount of the oxides, together with the presence of softened sulfides, should be responsible for the reduced friction coefficients at high temperatures. They can also explain for the resulting smoother curves of friction coefficient vs. time as observed in Figure 4.

4. Conclusions

a. The average friction coefficients of the material follow a common general trend of decreasing continuously with increasing temperature, while the wear rates exhibit a reversed tendency. Meantime, the curve of friction coefficients vs. time is found to become smoother as temperature rises.

b. The increasing amount of the oxides, together with the presence of softened sulfides, should be responsible for the reduced friction coefficients at high temperatures. They can also explain for the resulting smoother curves of friction coefficient vs. time.

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