

# High Temperature Tribological Performance of Steel/Copper Friction Pairs Lubricated With a Modified C-WS<sub>2</sub>-(Fe<sub>3</sub>O<sub>4</sub>+TiN) Nanoadditives in Non-Copper Coated Solid Wires

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## Original Article

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# Abstract

In this study, four kinds of nanoparticles: graphite,  $WS_2$ ,  $Fe_3O_4$  and TiN were used as lubricating additives for steel/copper friction pairs to solve the problem of welding contact tube wear with non-copper-coated solid wire at high temperature. The single and composite nanoparticles have excellent dispersion stability in absolute ethanol under the action of the compound surfactant NaSTA+OA+PVP. The tribological test results showed that the maximum decrement with reference to the average coefficient of friction and wear volumes were measured with nanoparticle concentration in 1:1:1 ratio at 300°C. Compared with dry friction, the average friction coefficient and wear volume are reduced by 74.3% and 84.8%, respectively, which may be attributed to the formation of a stable tribo-film mainly composed of C-O,  $Fe_2O_3$ ,  $WO_3$ ,  $TiO_2$ ,  $TiN_xO_y$  composite on the worn surface. Therefore, it is considered that the combined lubrication effects of the ball-bearing effect, repairing of worn surfaces and the tribo-film resulted in the lowest friction and wear.

## 1. Introduction

Environmentally-friendly non-copper-coated solid wires is an inevitable trend in the development of robotic automatic arc welding[1]. Compared with traditional copper plating solid wires, non-copper-coated solid wires has many advantages such as less environmental pollution, beautiful shape, stable arc, less splash and smoke, good rust resistance, stable wire feeding, etc[2–4]. However, there are still problems such as serious wear of the contact tube, poor wire feedability and arc stability[5, 6]. Currently, non-copper-coated solid wires mainly uses nanomaterials as surface coating. Wesling et al[7] studied the influence of the surface coating elements of the welding wire on the mechanical properties of the arc and weld metal. Cr coating caused of arc length shortening and the tensile strength increased by 44%, and the fatigue strength of the weld metal with Cr and Ti coatings also significantly increased. In addition, the temperature is an important factor that affects the performance of the surface coating of the welding wire and the wear of the contact tube[8, 9]. Li et al[10] found that using the mixture of nano-graphite,  $MoS_2$  and  $Fe_3O_4$  as the surface coating of the non-copper coated solid wires can significantly reduce the wear of the contact tube at room temperature, and the mass loss rate of the contact tube is as low as 0.35%. Therefore, the study of the friction and wear of nanoparticles in steel/copper friction pair at high temperature has important theoretical significance for reducing the wear of copper contact tube and improving the welding performance of steel non-copper coated solid wires.

Due to the physical and chemical properties of nanoparticles (large specific surface area, high diffusibility, easy sintering, low melting point, high hardness, etc[11, 12].), it not only forms a low-shear-stress film on the surface of the friction pair which contributes to reduce the friction coefficient, but also have repairing effect on the worn surface[13, 14]. Therefore, it has excellent anti-wear and anti-friction properties, and has great application potential as a lubricating additive in many fields. The effect of anti-wear and anti-friction performance depends on the type of additive and the number of additives. Furthermore, the optimal ratio of nanoadditives is also a key factor. A high concentration of

nanoadditives can cause surface damage due to extensive wear and higher frictional heat. At present, the content of nanoadditives is generally in the range of 0.1 ~ 3% [15, 16]. However, one of the defects of nanoadditives is their poor dispersibility in the lubricating medium. Therefore, the particle size, proportion and dispersibility of nanoparticles would be the key focus of further research, to increase the dispersion stability of lubricating nano-additives.

Most of the new lubricants have different properties enhanced by adding a specific proportion of special compound additives at room temperature. But there are few studies on the effect of temperature on composite nano-lubricating additives. Dan et al [17] prepared WS<sub>2</sub>/GP nanocomposites and illustrated that the friction coefficient and wear rate of WS<sub>2</sub>/GP at 100°C was reduced by 70.2% and 65.8% at 0.02 ~ 0.04 wt.% compared with the base oil. The additives form a layer-by-layer mixed structure, forming a synergistic lubrication mechanism at high temperatures. An et al [18] reported the tribological properties of ZnO doped with WS<sub>2</sub> nanoparticles at room temperature and high temperature. ZnO nanoparticles have little effect on the friction coefficient of WS<sub>2</sub> at 25°C. At 400°C, the ZnO-WS<sub>2</sub> component produces a strong conversion of tribochemical reaction at high temperature and exhibits an unstable friction coefficient, while pure WS<sub>2</sub> shows a lower and more stable trend.

Several studies have been conducted on the friction and wear of steel/copper and steel/steel friction pairs under lubrication or dry friction conditions [19, 20]. Generally, steel wears relatively little due to its higher hardness during friction. The wear of copper is relatively high, and plastic deformation is serious [21]. However, in the search for the most effective nanoadditive ratio and its tribological performance and lubrication mechanism of steel/copper under high temperature, only a few have been verified and characterized.

This work aims to investigate the high temperature tribological performance of modified C-WS<sub>2</sub>-(Fe<sub>3</sub>O<sub>4</sub> + TiN) as a lubricant additive for low-carbon steel and copper alloy Cu-Cr-Zr friction pair. High temperature tribological tests are conducted under boundary lubrication to simulate the face contact between non-copper coated solid wires and contact tube. The effects of individual and hybrid nanoadditive components on the tribological properties of steel/copper friction pairs at 300°C were studied. Finally, the lubrication mechanism of nanoparticles at the steel/copper friction interface was analyzed.

## 2. Experiments

### 2.1 Materials

The C-WS<sub>2</sub>-(Fe<sub>3</sub>O<sub>4</sub> + TiN) nanoadditives with an average size of 30nm were synthesized by surface modification method. The final prepared C-WS<sub>2</sub>-(Fe<sub>3</sub>O<sub>4</sub> + TiN) nanocomposite lubricants were added into absolute ethanol solution, and is stirred with the addition of surfactants dispersion to achieve stable dispersions. The surfactants are sodium stearate (NaSTA), oleic acid (OA) and polyvinylpyrrolidone (PVP). In order to fully mix the nanoparticles, surfactants and absolute ethanol, the experiment uses a combination of ultrasonic vibration dispersion and constant temperature water bath heating for

dispersion. The 1wt.% designed nanoparticles of different components and contents and 2wt.% surfactants were added to absolute ethanol, and ultrasonic treatment was performed for 20 minutes at 50°C. Among them, the compound surfactants was prepared according to NaSTA: OA: PVP = 1:1:1, and an appropriate amount of cyclohexane was added to promote mutual dissolution of the surface modifier. The nanoadditives designed in the experiment was shown in Table 1.

Table 1  
Chemical compositions of employed lubricants wt.%

Lubrication type	Composition		
	C	WS <sub>2</sub>	Fe <sub>3</sub> O <sub>4</sub> + TiN
1	Dry condition		
2	1	0	0
3	0	1	0
4	0	0	1
5	0.5	0.5	0
6	0.5	0	0.5
7	0	0.5	0.5
8	0.333	0.333	0.333

## 2.2 Tribological tests

The high temperature tribological property was tested using a pin-on-plate in a reciprocation model (UMT-3, Bruker Nano Inc, USA). Schematic diagram of the high temperature tribological tests was shown in Fig. 1. The experiments were performed for 30 min at high temperature 300°C. The applied load was 2.5N, the sliding speed was 2Hz, and the sliding travel was 10mm. The temperature and friction coefficient were automatically recorded by computer. The chemical composition of the friction pair material is shown in Table 2. The test steel pin was Q235 ( $\Phi 8\text{mm} \times 16\text{mm}$ , surface roughness was 0.02–0.10 $\mu\text{m}$ ), copper plate was Cu-Cr-Zr (40mm $\times$ 30mm $\times$ 6mm, surface roughness was 0.05–0.20 $\mu\text{m}$ ). The samples were cleaned with ultrasonic before and after the test.

**Table 2** Chemical compositions of the pin and plate materials wt.%

Materials	Chemical composition							
	Pin - Q235	C	Mn	Si	S	P	Fe	
≤ 0.17		≤ 1.4	≤ 0.35	≤ 0.035	≤ 0.035	Bal.		
Plate - Cu-Cr-Zr	Cr	Zr	Fe	Al	Mg	Si	P	Cu
	0.1-	0.3-	0.5	0.1-	0.1-	0.5	0.1	Bal.
	0.8	0.6		0.25	0.25			

## 2.3 Characterization methods

When the nanoadditives was fully dispersed, the absorbance of the stable supernatant of the ethanol mixture was measured after standing for 1 hour, and the dispersion stability of nanoparticles in absolute ethanol was investigated. The absorbance of the supernatant was measured with a ultraviolet-visible-near-infrared spectrophotometer (UH-4150), and the scanning wavelength was 400 ~ 850nm. Fourier infrared spectrometer (FT-IR, VERTEX 70) was used to measure the infrared spectra of different nanoparticles before and after modification, and the scanning range was 500 ~ 4000  $\text{cm}^{-1}$ . The position and intensity of the absorption peak of the molecular structure of the characterizing substance determine the chemical group of the analyte.

The worn surfaces images of copper plate were obtained by Scanning Electron Microscopy (SEM, Hitachi S4800, Japan). The wear volumes were calculated by using a three-dimensional non-contact surface profiler (ST400, NANOVEA, USA). The Three-dimensional(3D) profiles and roughness of worn surface were characterized by a laser scanning confocal microscope (CLSM, Olympus LEXTOLS4100, Japan). X-ray photoelectron spectroscopy (XPS, Thermo escalab 250Xi, USA) was used to analyze the structure of oxide friction film.

## 3. Results And Discussions

### 3.1 Dispersion analysis of nanoparticles

Different additives are more likely to agglomerate when they are compounded, which affects their performance. This effect is obvious between different elements, but is not evident between the same elements. Figure 2 shows the effect of different surfactants on four kinds of nanoparticles. The larger the spectrophotometer, the better and stable the dispersion of modified nanoparticles in absolute ethanol. Different modifiers have different effects on these four kinds of nanoparticles. PVP has a significant effect on graphite.  $\text{WS}_2$  is modified by NaSTA and OA, has excellent dispersion stability. NaSTA and PVP have poor effect on  $\text{Fe}_3\text{O}_4$  modification. The overall order of dispersion effect in absolute ethanol from high to low is: PVP, OA, NaSTA. It shows that the compound surfactants has a more excellent dispersion effect on the composite nanoparticles.

For the most suitable surfactants for the four kinds of nanoparticles, qualitative research before and after modification was carried out using infrared spectroscopy. Figure 3a) and d) shows the position around  $3325\text{cm}^{-1}$  is the typical absorption peak of O-H stretching vibration in water, the strong absorption broad peak at  $2972\text{cm}^{-1}$  is C-H stretching vibration, and the strong peak at  $1450 \sim 1420 \text{cm}^{-1}$  is caused by methylene  $-\text{CH}_2$  bending vibration absorption peak. Around  $1290 \text{cm}^{-1}$  is the characteristic peak of the amide group ( $-\text{CONH}-$ ) of the PVP molecule. Comparing the two curves before and after modification of  $\text{WS}_2$  and  $\text{Fe}_3\text{O}_4$ (Fig. 3b) and c)), it can be seen that the peak shape of the infrared spectrum of the modified nanoparticles has changed significantly. The absorption peak at  $2848 \text{cm}^{-1}$  indicates the presence of methylene  $-\text{CH}_2$ ,  $1469 \text{cm}^{-1}$  is the stretching vibration of  $-\text{CH}_2$ . It can be seen that the characteristic absorption peaks of long-chain functional groups similar to the surfactant appear on the four kinds of nanoparticles after modification, indicating that the surface modifier has successfully modified the nanoparticles. The main chain of the three surfactants is the hydrophobic segment of the C-C bond and the presence of non-polar methylene  $-\text{CH}_2$  has lipophilicity. The hydrophilic group allows it to be well adsorbed on the surface of the nanoparticles, while the lipophilic group allows the nanoparticles to be well suspended in absolute ethanol.

## 3.2 Tribological behavior of individual and hybrid nanoadditives

The results show that graphite and  $\text{WS}_2$  have excellent anti-friction and anti-wear performance, and the wear process is stable at  $300^\circ\text{C}$ , while the friction coefficient under the separate action of  $\text{Fe}_3\text{O}_4$  and TiN is abnormal and the wear extent increases, see Fig. 4. It shows that the anti-wear and anti-friction effect of single nano-graphite is more significant, and the friction coefficient under the action of graphite is as low as 0.1682. The anti-wear performance under nano-TiN lubrication is the worst, and the wear volume is as high as  $17.2059 \times 10^{-2} \text{mm}^3$ . Therefore, the individual component action of nano-modified graphite and  $\text{WS}_2$  at  $300^\circ\text{C}$  can significantly improve the anti-friction and anti-wear performance of steel/copper.

According to Fig. 5, it shows that under the same experimental conditions, the anti-friction performance of the hybrid nanoadditives is better than that of the individual nanoadditives. Compared with dry friction, the average friction coefficient of the nanoadditive component of C: $\text{WS}_2 = 1:1$ , C:( $\text{Fe}_3\text{O}_4 + \text{TiN}$ ) = 1:1,  $\text{WS}_2$ :( $\text{Fe}_3\text{O}_4 + \text{TiN}$ ) = 1:1, C: $\text{WS}_2$ :( $\text{Fe}_3\text{O}_4 + \text{TiN}$ ) = 1:1:1 is reduced by 46.7%, 65.2%, 46.3%, and 84.8%, respectively, and the wear volume is reduced by 32.6%, 67.3%, 40.1%, and 74.3%. Particularly, when the modified hybrid nanoadditives is C: $\text{WS}_2$ :( $\text{Fe}_3\text{O}_4 + \text{TiN}$ ) = 1:1:1, the tribological performance is the best excellent at  $300^\circ\text{C}$ . This may be related to the interaction between nanoparticles and the distribution and thickness of the protective film formed on the surface of the steel/copper friction pair. The fluctuation of friction coefficient can be explained by more intense tribochemical reactions between four kinds of nanoparticles at  $300^\circ\text{C}$ .

## 3.3 Surface morphology and chemical composition analysis of the copper worn surfaces

The Fig. 6 shows the three-dimensional surface morphology and roughness curves of Cu-Cr-Zr under different composite lubricants at 300°C. Compared with dry condition, the addition of hybrid nanoadditives has a significant impact on roughness of wear surface. Under the action of C:(Fe<sub>3</sub>O<sub>4</sub> + TiN) = 1:1, the wear surface of Cu-Cr-Zr is obviously covered with a layer of flake material and its thickness has exceeded the depth of the wear scar, and the surface roughness is relatively large. When the modified hybrid nanoadditives is C:WS<sub>2</sub>:(Fe<sub>3</sub>O<sub>4</sub> + TiN) = 1:1:1, the surface profile is smooth, but there is still micro-furrowing, and the corresponding surface roughness is 0.179µm. Compared with dry friction, it is reduced by 83.8%, which shows that the modified hybrid nanoadditives exhibits a good anti-friction and anti-wear effect, avoids direct metal contact, and can inhibit obvious wear on the surface of Cu-Cr-Zr. And through the cross-sectional profile curve comparison, it is shown that the surface of Cu-Cr-Zr has been improved to different degrees after adding the hybrid nanoadditives. The reason is that the deposition of nanoparticles produces a soft layer on the friction surface. The direct contact between copper repairs the micro-damage of the worn surface during the friction process, and makes the worn surface smooth without obvious scratches, cracks and other defects, so the surface roughness and cross-sectional profile curve are smoother.

To further investigate the wear mechanism, Fig. 7 shows the SEM images of the lubricant samples obtained for the Cu-Cr-Zr worn surface. The surface is found smoother and there is no delamination of the layers. The addition of the nanoparticles results in less wear as they provide a defensive film between the surfaces during steel and copper blocks moving. However, a further increase in nanoparticles results in more amount of wear volumes. Figure 7a) shows that the worn surface is severely damaged and accompanied by a large number of lamellar flaking. The flaked hard particles are embedded in the wear scar under dry friction. As shown in Fig. 7b), c) and d), under the action of different composite lubricants, there are a small amount of wear debris and massive adhesion materials on the wear surface of Cu-Cr-Zr, and there are obviously a large number of flake oxides and a small amount of block oxides, indicating the existence of friction film. When the content of nanoparticles added separately is optimal C:WS<sub>2</sub>:(Fe<sub>3</sub>O<sub>4</sub> + TiN) = 1:1:1, the average friction coefficient and wear scar diameter of the friction pair can be minimized, the friction contact surface is smoothest, the shallowest grooves and scratches, and solid lubrication is significantly weakened, inducing decreased wear (Fig. 7e). It mainly shows that when the ratio of the four kinds of nanoparticles in the friction process at 300°C is an average ratio, a tribochemical reaction is generated to form a more uniformly distributed friction film, which reduces the direct contact of the friction pair.

It can be seen from the surface element distribution (Fig. 8) that C: WS<sub>2</sub>: (Fe<sub>3</sub>O<sub>4</sub> + TiN) = 1:1:1 modified hybrid nanoadditives have a uniform distribution of C, Fe, Ti and W elements on the wear surface, indicating that the graphite, WS<sub>2</sub>, Fe<sub>3</sub>O<sub>4</sub> and TiN nanoparticles added at 300°C reacted with the matrix material of the friction pair to form a uniform repairing film containing four elements. The friction film separates the friction pair, avoids direct contact between steel and copper, effectively inhibits the further occurrence of surface wear, and also explains the reason for different distribution, size and roughness of the friction film in the scanning electron microscope image. In addition, the reduction of Cu element

indicates that the nanoparticles did not react with the material of friction pair when the friction surface moved relative to each other. As shown in Fig. 9, the anti-friction and anti-wear effect is due to the small size of the nanoparticles, which can play the role of micro-bearing. The nanoparticles and oxides are embedded in the pits and damaged parts of the friction interface during the friction process, which play a role in repairing the rough surface, making the worn surface smooth and reducing the surface roughness.

The XPS fine spectrum analysis results of the wear surface elements under the action of C:WS<sub>2</sub>:(Fe<sub>3</sub>O<sub>4</sub> + TiN) = 1:1:1 modified hybrid nanoadditive are shown in Fig. 10. The full spectrum mainly detected elements such as Cu, Fe, O, Ti and C, which were consistent with the results of EDS. O1s mainly produces copper oxide and carbon oxide during the friction process. It can be seen from Fig. 10b) and d) that the Fe2p peak with a binding energy of 710.9eV and the O1s peak with a binding energy of 530.2eV are attributed to Fe<sub>2</sub>O<sub>3</sub>, and the Fe2p peak with a binding energy of 708.1eV and O1s with a binding energy of 529.3eV The peak of the spectrum is attributed to FeO, indicating that a chemical reaction film containing Fe<sub>2</sub>O<sub>3</sub> and FeO is formed on the surface of the friction pair through tribochemical action. Analyzing Ti2p, two peaks of 456.79 eV and 458.85 eV are fitted at the Ti2p<sub>3/2</sub> peak(Fig. 10f)), which are the corresponding peaks of Ti-containing oxides, representing TiN<sub>x</sub>O<sub>y</sub> and TiO<sub>2</sub>, respectively.

By analyzing the above mentioned elements, it can be seen that a substance containing multiple oxides such as Cu, C, Fe, W and Ti, etc. It shows that the four kinds of nanoparticles added chemically react during the friction process of the steel/copper friction pair, forming an extremely thin repairing film composed of CO, Fe<sub>2</sub>O<sub>3</sub>, WO<sub>3</sub>, TiO<sub>2</sub> and TiN<sub>x</sub>O<sub>y</sub> on the worn surface, which plays a role in repairing the worn surface of Cu-Cr-Zr.

### 3.4 Lubrication mechanisms

Figure 11 shows the lubrication mechanism of the steel/copper friction interface. During the friction process at 300°C, the nanoparticles and their decomposition products aggregate, adsorb, and react on the surface, forming a friction film, which plays a role of lubrication and protection, and is the mechanism of decomposition and film formation. Nanoparticles are extremely small in size, and they are embedded in the pits of the friction interface during the friction process to repair the rough surface and make the worn surface smooth. When the low carbon steel and Cu-Cr-Zr slide relatively, the nanoparticles have a ball-bearing effect. Hence, the modified hybrid nanoadditives generates a tribochemical reaction at the friction interface at high temperature to form a repairing film, which shows good anti-wear and anti-friction and repairing properties.

## 4. Conclusions

(1) The deaggregation and prolonged dispersion of four kinds of nanoparticles are modified by surface modification with NaSTA, OA, PVP in absolute ethanol. The modified nanoparticles exhibit very stable dispersion at a high absorbance value, compared with the untreated nanoparticles. The dispersion stability of the compound surfactant NaSTA + OA + PVP is obviously better than that of the single

surfactant. Stable dispersion of nanoparticles in a lubricating medium can significantly improve its anti-friction and anti-wear performance.

(2) Compared with dry friction, individual and hybrid nanoadditives can reduce the amount of wear and friction coefficient, improve the anti-friction and anti-wear properties of steel/copper friction pair. Particularly, hybrid nanoadditives C:WS<sub>2</sub>:(Fe<sub>3</sub>O<sub>4</sub> + TiN) = 1:1:1 is superior to others individual and hybrid nanoadditives in improving the anti-wear and anti-friction performance Cu-Cr-Zr. Its average friction coefficient and wear volume are reduced by 84.8% and 74.3% respectively, and the worn surface is the smoothest and the surface roughness value is the lowest.

(3) The lubrication mechanism for steel/copper friction pairs with modified C-WS<sub>2</sub>-(Fe<sub>3</sub>O<sub>4</sub> + TiN) hybrid lubricants at high temperature may be identified as that, when low-carbon steel and Cu-Cr-Zr are relatively sliding, the four kinds of nanoparticles at the friction interface interact with tribochemical reactions at 300°C to form a friction film composed of CO, Fe<sub>2</sub>O<sub>3</sub>, WO<sub>3</sub>, TiO<sub>2</sub>, TiN<sub>x</sub>O<sub>y</sub> etc. It shows excellent anti-friction and anti-wear performance and repairing effect. Different nanoparticles effectively play the role of synergistic lubrication of ball-bearing lubrication, repairing of worn surfaces and film formation by tribochemical reactions. The results are promising for reducing the wear of copper contact tube and improving welding performance of steel non-copper coated solid wires.

## Declarations

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### Availability of data and materials

The datasets supporting the conclusions of this article are included within the article.

### Authors' contributions

The author' contributions are as follows: Hong Li was in charge of the whole trial; Jing Zhu wrote the manuscript; Yu Zhang, Zhuoxin Li and Bo Meng assisted with sampling and laboratory analyses.

### Competing interests

The authors declare no competing financial interests.

### Consent for publication

Not applicable

## Ethics approval and consent to participate

Not applicable

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## Figures

### Figure 1

Schematic diagram of the high temperature tribological tests

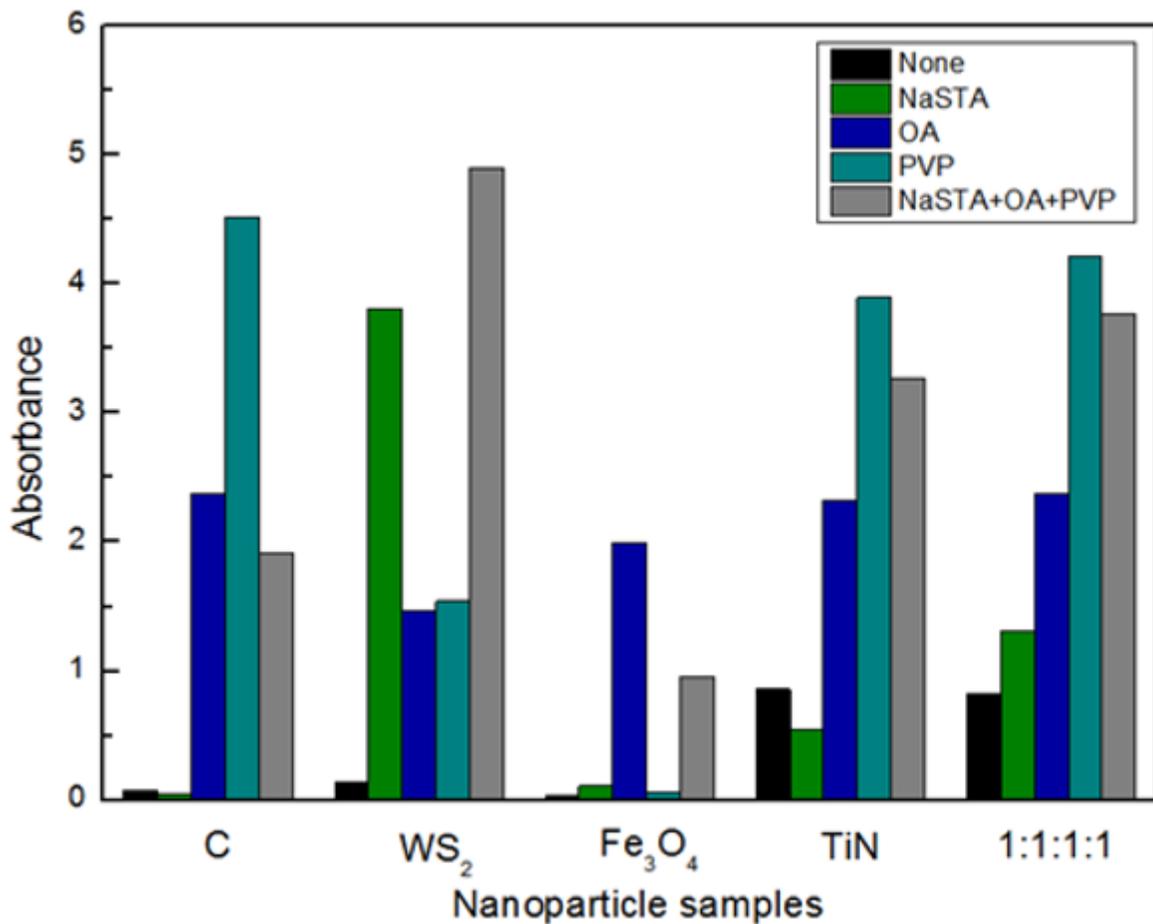
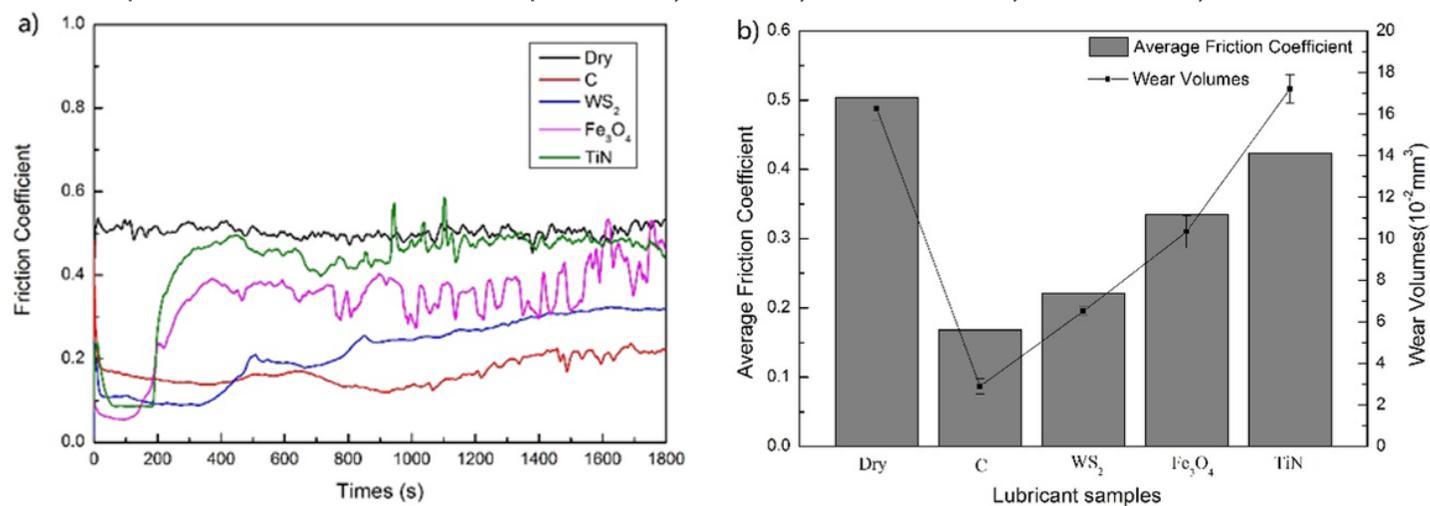


Figure 2

Effect of modifier types on the dispersion stability of different nanoparticles

Figure 3

Infrared spectra of four modified nanoparticles a) C-PVP, b) WS<sub>2</sub>-NaSTA, c) Fe<sub>3</sub>O<sub>4</sub>-OA, d) TiN-PVP



#### **Figure 4**

a) Compare the variation of coefficient of friction with time for single nano-lubricants, b) Average friction coefficient and wear volumes of different single nano-lubricants

#### **Figure 5**

a) Variation of coefficient of friction with time for nanocomposite lubricants, b) Average friction coefficient and wear volumes of different nano composite lubricants

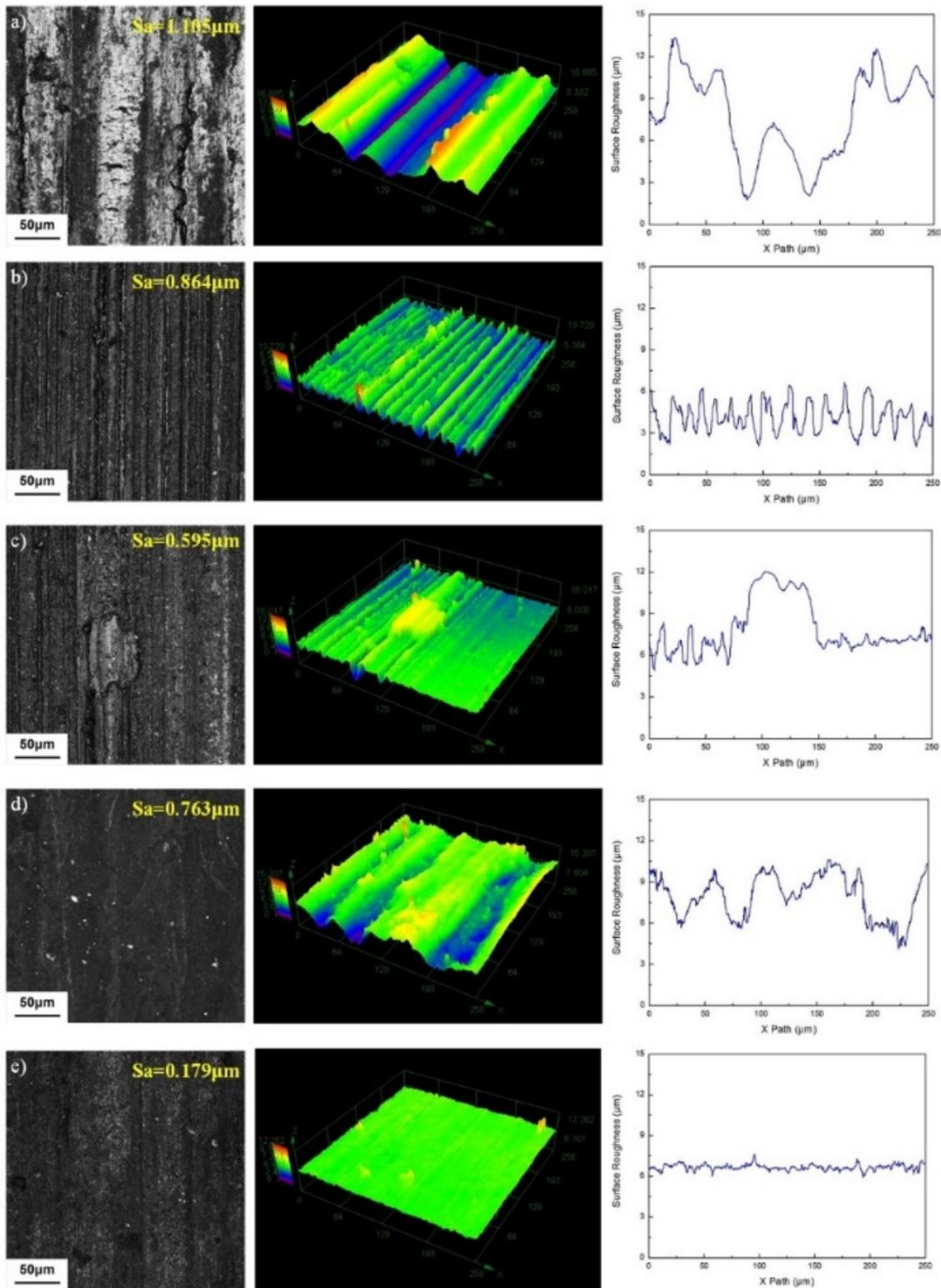


Figure 6

Surface topographies and roughness curves of worn surface under different lubricating conditions a) Dry, b) 1-1-0, c) 1-0-1, d) 0-1-1, e) 1-1-1

Figure 7

SEM morphologies of worn surfaces and EDS spectrum of the tribofilm for the copper lubricated by different modified hybrid nanoadditives at 300°C a) Dry, b) 1-1-0, c) 1-0-1, d) 0-1-1, e) 1-1-1

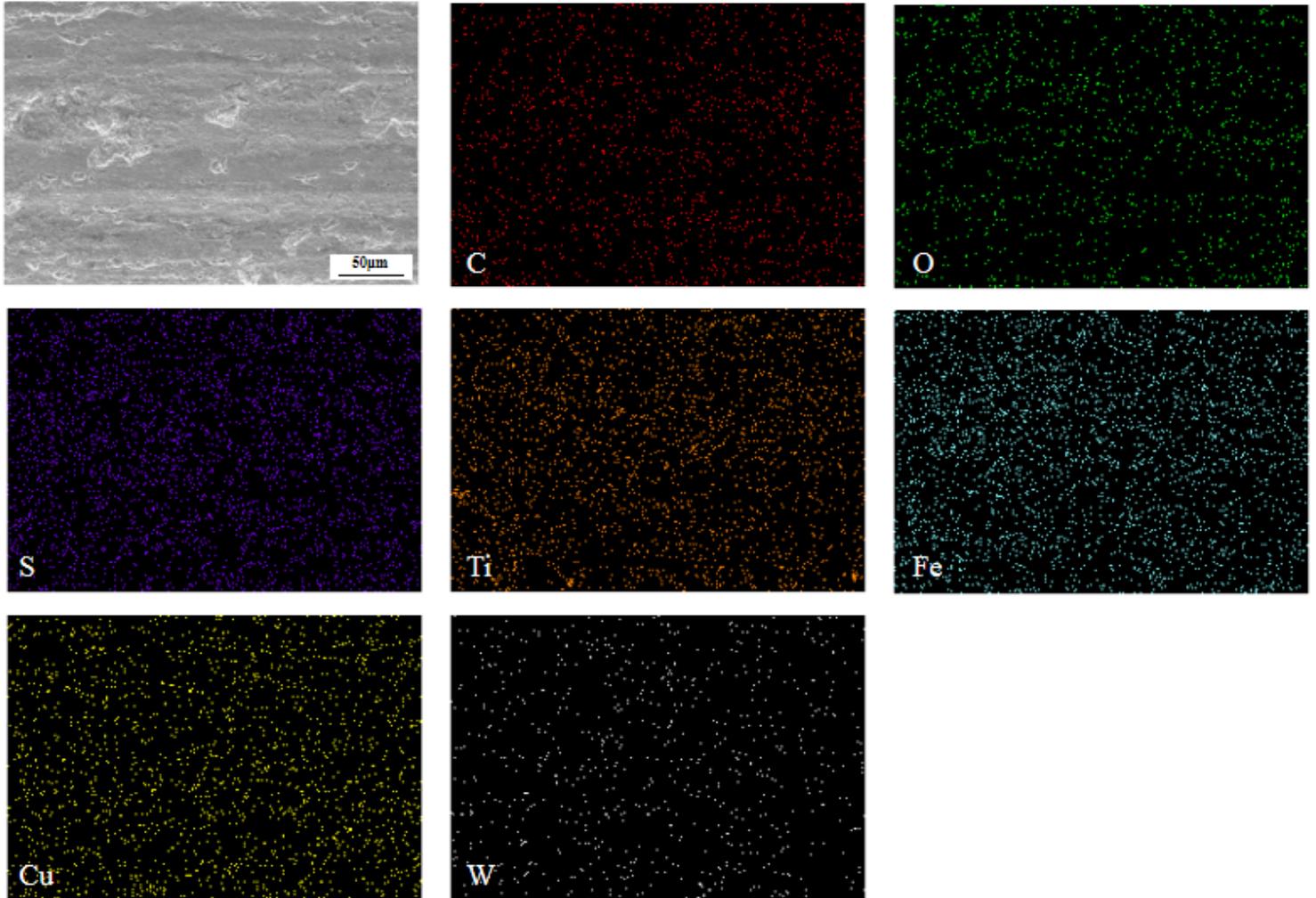
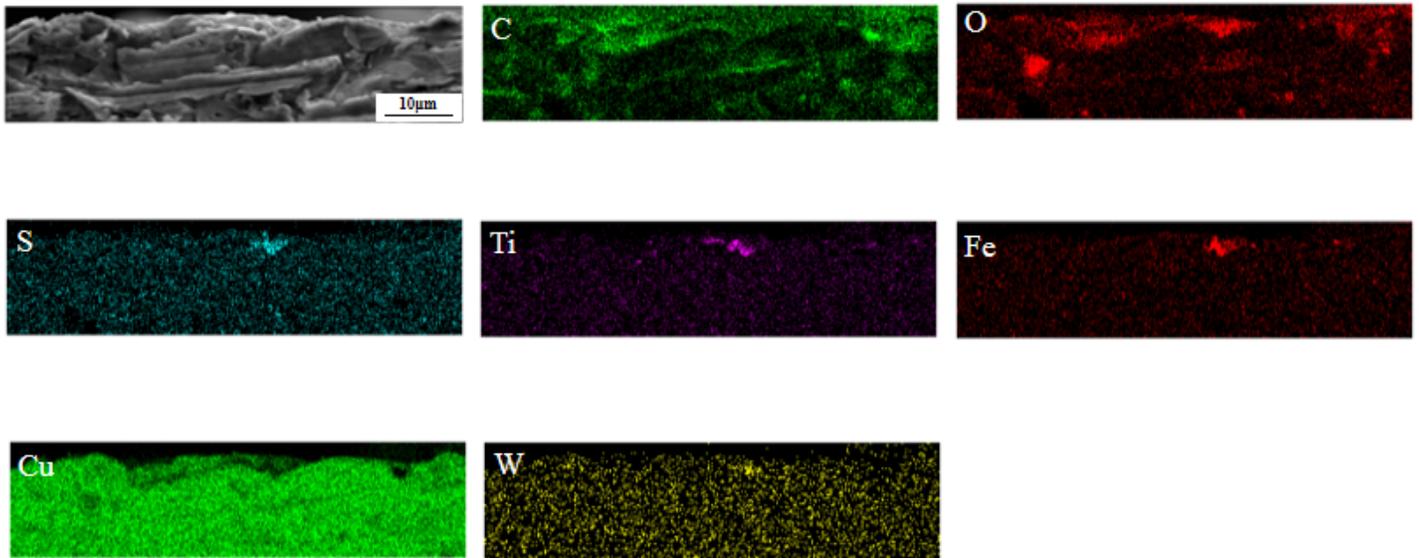


Figure 8

Elemental mapping analysis of the tribofilm identified in Figure 7e)



**Figure 9**

EDS surface scanning of C: WS<sub>2</sub>: (Fe<sub>3</sub>O<sub>4</sub>+TiN) = 1:1:1 on the Cu-Cr-Zr wear section

**Figure 10**

XPS spectrum of the worn surface lubricated with C:WS<sub>2</sub>:(Fe<sub>3</sub>O<sub>4</sub>+TiN)=1:1:1

**Figure 11**

Model of lubrication mechanism of steel-copper friction interface with nanoadditives as lubricants