lubricants

Communication

The Synthesis of Cu-Coated Ti_2SnC Ceramic and Its Tribological Behaviors as a Lubricant Additive

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Abstract: Lubricant additive plays an important role in reducing the friction and wear for base oil. MAX phase ceramics may have superior advantages for additive application due to their unique nanolayered structure. In this paper, Ti_2SnC ceramic is prepared by sintering the elemental mixtures at 1250 °C. In addition, Cu-coated Ti_2SnC ceramic is successfully prepared using a chemical plating method for the first time. It is confirmed that the Ti_2SnC ceramic has good self-catalytic activity, and a layer of stacking Cu nano-particles can be deposited on the Ti_2SnC surface without pretreatment. When the Cu-coated Ti_2SnC ceramic powder is doped into PAO10 base oil, the oil can exhibit excellent lubrication properties, where the friction coefficient is as low as 0.095. A layer of tribo-film can be formed during the sliding process when the Cu-coated Ti_2SnC ceramic is incorporated into PAO10 base oil, which can reduce the friction coefficient. The superior lubrication properties can be attributed to the synergistic lubrication effect of Ti_2SnC ceramic and Cu nano-particles.

Keywords: Cu coated Ti_2SnC ceramic; additive; chemical plating; microstructure; wear; tribology

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1. Introduction

MAX phases are a family of ternary layered compounds, where M is a class of early-transition metal, A is a IIIA or IVA group element, and X is a C or N element [1]. It is well known that MAX phases have combination properties of ceramic and metal, such as a superior damage tolerance, high electrical conductivity, excellent thermal shock resistance, and good machinability [2]. Therefore, MAX phases have been widely studied in high-temperature, structure, catalysis, and electrical contacts fields [3,4].

The tribological behaviors of MAX phases have also attracted increasing attention due to their unique laminate structure [5,6]. It is well known that MAX phases can exhibit better lubrication properties only under several certain conditions, such as at high speeds [7,8], under a low load [9], at a micro-scale [10], against some certain counterparts [11], in a vacuum environment [12], or under a water environment [13]. Furthermore, it has been reported that MAX phases could play lubricating roles in reducing friction and wear when doped into a NiAl or TiAl matrix, due to their tribo-oxidation effect [14,15]. It has also been confirmed that MAX and MXene phases can be used as a lubricant additive to improve the friction and wear properties of base oil [16–18]. However, the existing reports, in which MAX phases without modification are used as a lubricant additive in oil, are mostly focused on Ti_3AlC_2 and Ti_3SiC_2 ceramics [18–21]. For example, it was reported that HVI500 base oil with Ti_3SiC_2 crystals had a good anti-wear capability in comparison to pure HVI500 base oil [19,20]. Xue et al. [21] also found that 100SN base oil containing Ti_3AlC_2 samples exhibited good tribological behaviors with a load of 15 N. However, the tribological properties of MAX phases used as additives for base oil still need more investigation.

It has been reported that some additives modified by Cu nano-particles could exhibit an excellent anti-friction ability when incorporated into base oil [22,23]. In the present study, different from the other tribological applications of MAX phases, the tribological properties of Cu-coated MAX phases used as an additive are studied. For the first time, ternary layered Ti_2SnC ceramic powder is selected as a lubricant additive for poly-alpha-olefin (PAO10) base oil. In order to improve the lubrication properties of the Ti_2SnC ceramic particles, novel nano-Cu-coated Ti_2SnC ceramic particles are synthesized via a chemical plating route, in which the chemical plating method is highly efficient and controllable [24,25]. The tribological behaviors of Ti_2SnC and nano-Cu-coated Ti_2SnC ceramic used as a lubricant additive for PAO10 base oil are studied as well. It is worth noting that the Cu-coated Ti_2SnC ceramic can evidently reduce the friction coefficient of PAO10 base oil. The lubricating mechanism is also proposed.

2. Materials and Methods

An experiment schema of Cu-coated Ti_2SnC ceramic is presented in Figure 1. Here, Ti (400 mesh, 99.5 wt.% pure), Sn (300 mesh, 99.8 wt.% pure), and graphite (400 mesh, 99.5 wt.% pure) powders were chosen as the starting materials to prepare the Ti_2SnC ceramic. Briefly, the above three powders were uniformly mixed with a molar ratio of 2:1.1:1. Then, the mixed powders were first heated at a heating rate of 10 °C/min from room temperature to 1250 °C, and then sintered at 1250 °C for 1 h under an argon atmosphere in a tube furnace. At last, the as-sintered Ti_2SnC specimen was ground into powders in a milling machine and sieved using a sifter (800 mesh).

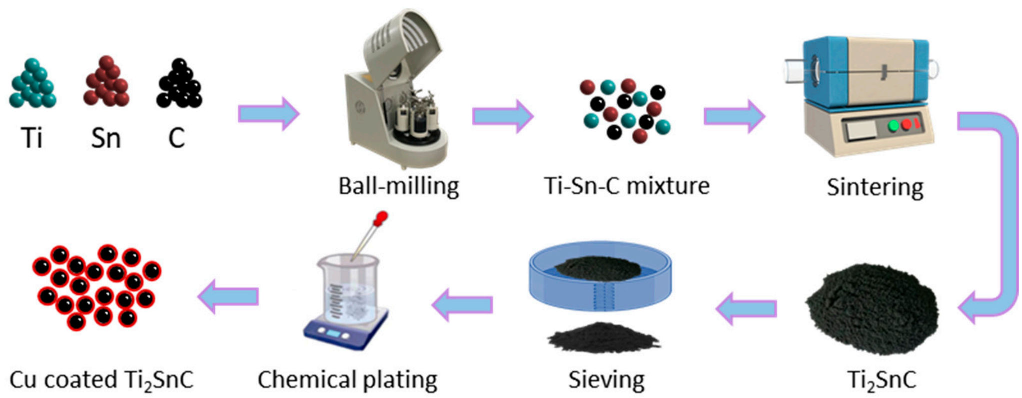


Figure 1. Schematic illustration of synthesis process of Cu coated Ti_2SnC ceramic.

The Cu-coated Ti_2SnC ceramic was prepared using a chemical plating method, where the chemical compositions of the plating solution can be found elsewhere [25]. Briefly, the chemical plating solution was magnetically stirred and heated to 60 °C in a water bath. During the stirring process, 10 g of Ti_2SnC powder was poured into the chemical plating solution (1 L). The pH of the chemical plating solution was controlled at 13 by dropping NaOH solution. The chemical compositions of the plating solution are listed in Table 1. The plating mechanism can be described as follows:

$$2HCHO + Cu^{2+} + 4OH^- \rightarrow 2HCOO^- + Cu\downarrow + H_2\uparrow + 2H_2O. \quad (1)$$

Table 1. Chemical compositions of the plating solution.

Chemical Composition	Content
Deionized water	1 L
$CuSO_4 \cdot 5H_2O$	8 g/L
EDTA-2Na	31 g/L
NaOH	10 g/L
HCHO	15 mL/L

The plating process was finished as the solution became clear. Then, the as-prepared Cu-coated Ti_2SnC ceramic powder was washed repeatedly using deionized water until the pH of the filtrate reached around 7. At last, the obtained wet powder was dried in a vacuum freeze-drying machine.

The tribological behavior tests were conducted on a UMT-2 rational tribometer. The tribometer that shows the different parts and location of the different PAO oils is illustrated in Figure 2. For the sliding tests, 5 wt.% as-prepared Cu-coated Ti_2SnC powder and Ti_2SnC powder were added into PAO10 base oil, respectively, and the mixtures were ultrasonically stirred for 1 h before the sliding tests. The mixed oils were dropped onto the GCr15 (AISI 52100) steel disk surfaces before the sliding tests, in which the GCr15 steel ball (Φ 6.35 mm) was selected as the counterpart. The applied load, rational speed, and rational radius were set as 10 N, 300 r/min, and 5 mm, respectively. Based on the Hertz model, the applied normal pressure can be determined to be approximately 875 MPa. The friction coefficient versus the sliding time was recorded by the computer automatically. Moreover, the three-dimension (3D) wear track morphologies were obtained by a Nano Focus µsurf-expert 3D profilometer.

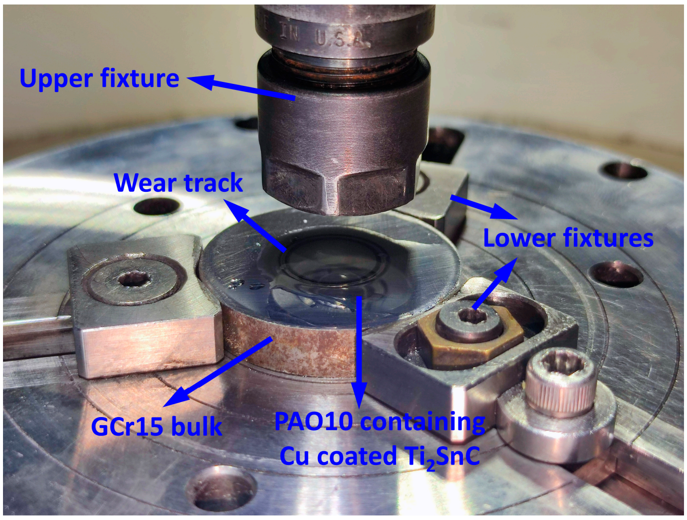


Figure 2. Schematic diagram of UMT-2 ball-on-disk tribometer.

The phase compositions of the as-prepared Ti_2SnC and Cu-coated Ti_2SnC were examined using a Bruker D8 Advance type X-ray diffractometer (XRD) with Cu Kα radiation. Both a JSM-7800F type field-emission scanning electron microscope (FESEM) and FEI Tecnai F30 type transmission electron microscope (TEM) were taken to observe the microstructures of the as-prepared Ti_2SnC and Cu-coated Ti_2SnC powders. Energy dispersive spectroscopy was also taken to obtain the content of the Cu in the Cu-coated Ti_2SnC powders. In order to retain the original wear morphologies, an optical microscope was employed to observe the wear tracks of the GCr15 disks after the sliding tests. The wear scars of the GCr15 balls were examined using the optical microscope as well.

3. Results and Discussion

Figure 3 shows the XRD patterns of the as-prepared Ti_2SnC and Cu-coated Ti_2SnC powders. As shown in Figure 3, the diffraction peaks at 43.4°, 50.6°, and 74.2° can be indexed as Cu (PDF # 99-0034) and the peaks at 36.0° and 41.8° can be assigned to TiC_x (PDF # 65-8807). Other diffraction peaks, such as 13.05°, 26.1°, 32.8°, 33.5°, 35.4°, 38.4°, 39.6°, 47.0°, and 52.3° et al., can be associated with the Ti_2SnC phase, according to the Ti_2SnC standard peaks (PDF # 89-5590). The diffraction peaks of the Cu can be examined after the chemical plating process, apart from Ti_2SnC and minor TiC_x. For the Cu-coated Ti_2SnC ceramic, obviously, the phase constituents mainly consisted of Cu and Ti_2SnC

phases. Based on the XRD results, it is confirmed that the Cu phase can be generated successfully according to the chemical plating process.

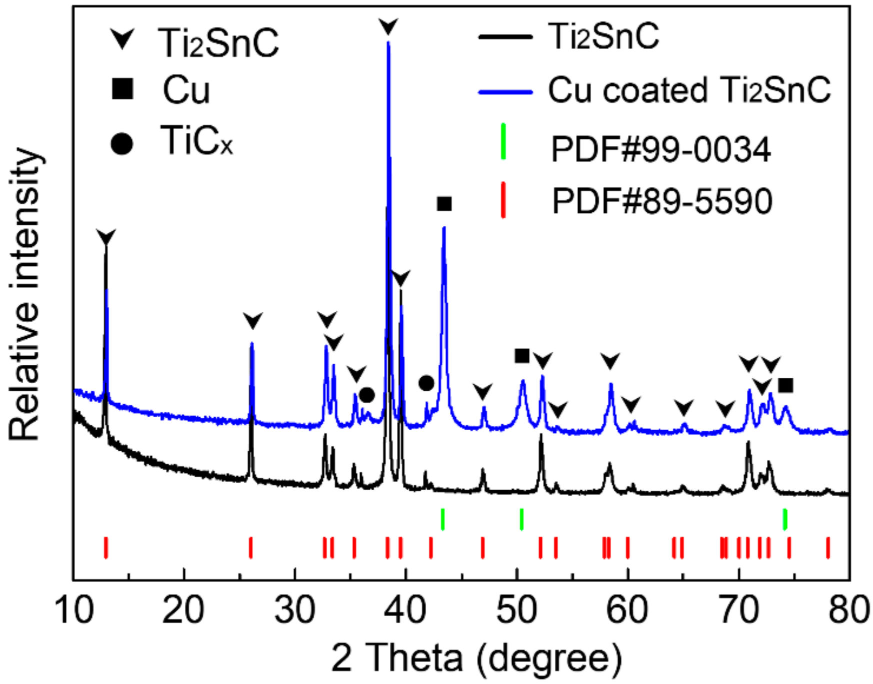


Figure 3. XRD patterns of as-prepared Ti_2SnC and Cu-coated Ti_2SnC.

The SEM images of the as-prepared Ti_2SnC and Cu-coated Ti_2SnC are shown in Figure 4. Clearly, the sizes of the as-prepared Ti_2SnC particles are smaller than 10 μm in length, as shown in Figure 4a,b. Like some other MAX phases, the as-prepared Ti_2SnC ceramic particle exhibits a laminate structure feature as well. For the as-prepared Cu-coated Ti_2SnC ceramic, large stacking Cu particles that are nano-scale cover the Ti_2SnC ceramic particle, as shown in Figure 4c,d. The laminate structure feature cannot be observed on account of this covering of Cu nano-particles. Some reports have argued that MAX phases need to be pretreated before Cu deposition [24]. For the most part, ceramic particles should be treated to increase the micro-roughness of the surface before the plating treatment. The increment in the micro-roughness of the surface contributes to the plating of the Cu nano-particles. Herein, we found that the Ti_2SnC ceramic particles had good self-catalytic activity, and the stacking Cu nano-particles layer could be deposited onto the Ti_2SnC ceramic surface without pretreatment. The Cu content was approximately calculated to be 10 wt.%. An EDS analysis was also conducted to evolute the Cu content in the Cu-coated Ti_2SnC particles. As shown in the inserts, the atomic ratio of the Ti and Sn in the Ti_2SnC particles was approximately 2:1, suggesting that the Ti_2SnC ceramic was successfully prepared. For the Cu-coated Ti_2SnC particles, the atomic ratio of Ti and Sn still retained about 2:1, and the atomic content of Cu was 7.8 wt.%, which was in agreement with the calculation result. It should be mentioned that it is not possible to quantify carbon using an EDS analysis.

TEM characterizations were also carried out to examine the structures of the as-prepared Cu-coated Ti_2SnC particles. In order to expose the Ti_2SnC matrix, the particles were ground in a mortar. As shown in Figure 5a, the dot-like Cu nano-particles were distributed on the Ti_2SnC surface. Based on the HRTEM image of a Cu-coated Ti_2SnC particle, the plane (103) of Ti_2SnC can be indexed, as shown in Figure 5b. Furthermore, the planes (110) and (101) of the Cu nano-particles can be identified as well. The XRD, SEM, and TEM evidence suggests that the Cu-coated Ti_2SnC particles were successfully prepared.

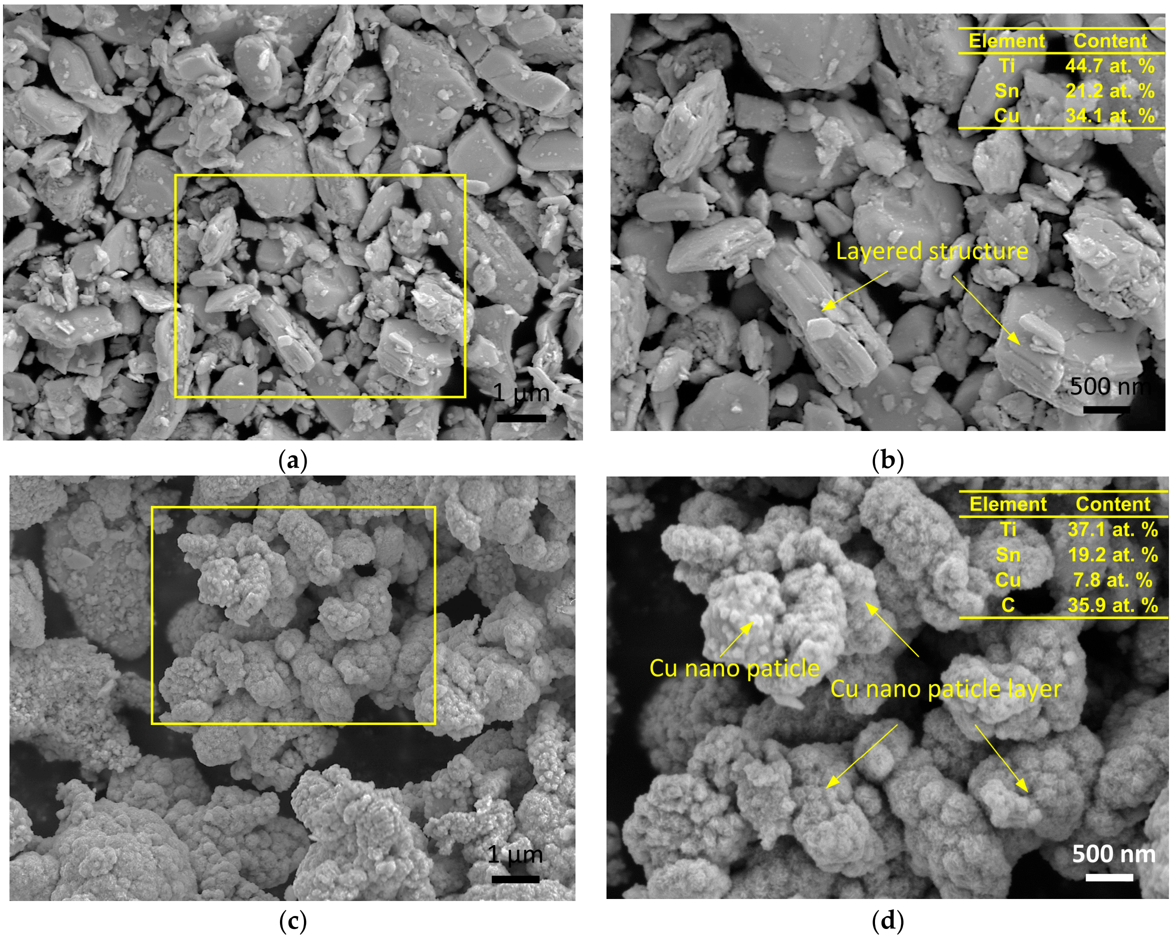


Figure 4. (**a**) Low-magnification and (**b**) high-magnification SEM images of as-prepared Ti_2SnC particles, and (**c**) low-magnification and (**d**) high-magnification SEM images of as-prepared Cu-coated Ti_2SnC ceramic particles. (The high-magnification SEM images shows the corresponding yellow rectangle parts in low-magnification SEM images.)

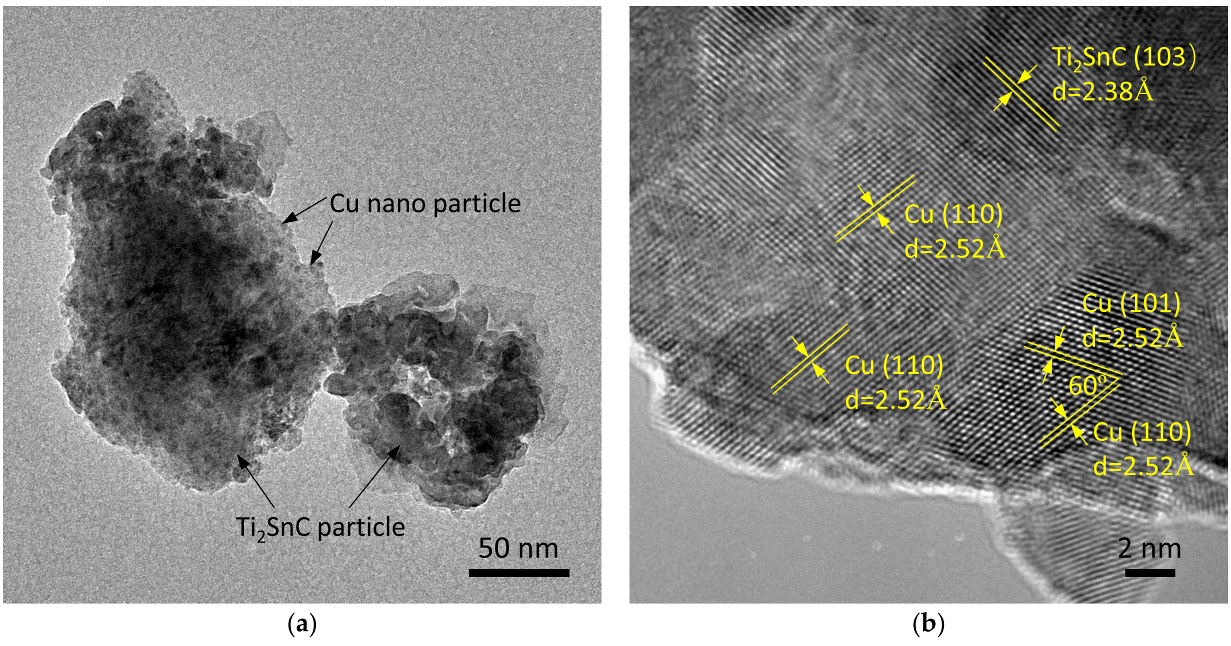


Figure 5. (**a**) Bright field TEM and (**b**) HRTEM images of Cu-coated Ti_2SnC particles.

Figure 6a shows the friction coefficient curves versus the sliding time of the PAO10 base oil containing the Ti_2SnC and Cu-coated Ti_2SnC. Figure 6b shows the wear volumes of the GCr15 disks after the sliding tests. As shown in Figure 6a, the friction coefficient of the PAO10 base oil was around 0.122. When the Ti_2SnC powder was incorporated into the PAO10 base oil, the friction coefficient decreased down to 0.114. Furthermore, when the Cu-coated Ti_2SnC powder was added into the PAO10 base oil, it showed the best lubrication behavior among the three oils, in which the friction coefficient was as low as 0.095. It can be seen that the GCr15 tribo-pair showed the best lubricating behavior as the Cu-coated Ti_2SnC powder was incorporated into the PAO10 base oil. It also can be found that the fluctuation of the blue curve was higher than that of the others. As the PAO10 base oil was employed as lubricant, the wear volume of GCr15 was as high as 0.28 mm^3. When the Ti_2SnC or Cu-coated Ti_2SnC additives were added, the wear volume of the GCr15 disk evidently came down. When the Cu-coated Ti_2SnC was doped into the PAO10 base oil, the GCr15 disk had the smallest wear volume (0.13 mm^3). The result shows that Cu-coated Ti_2SnC powder coupled with PAO10 base oil can reduce friction and wear obviously.

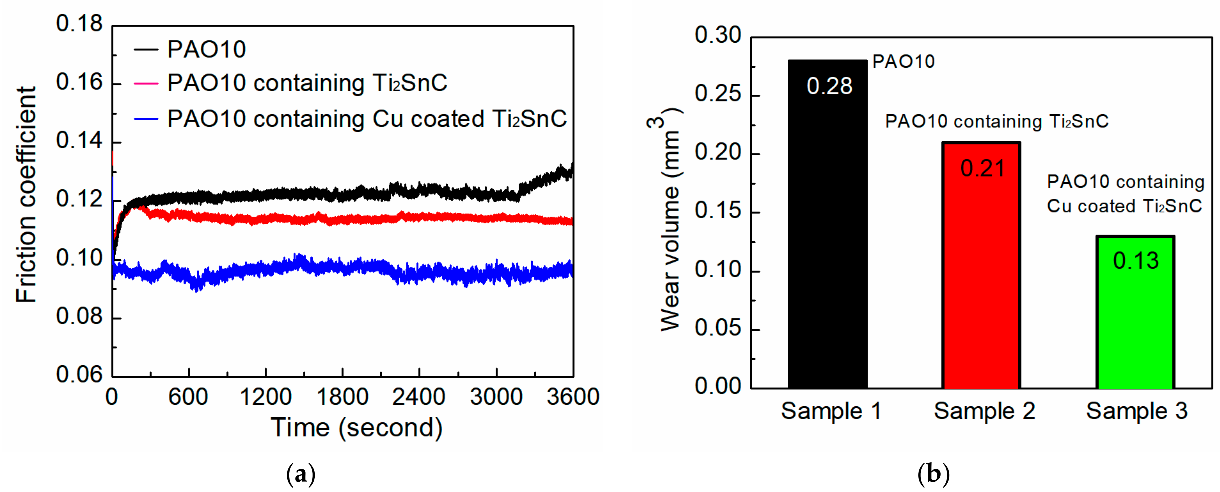
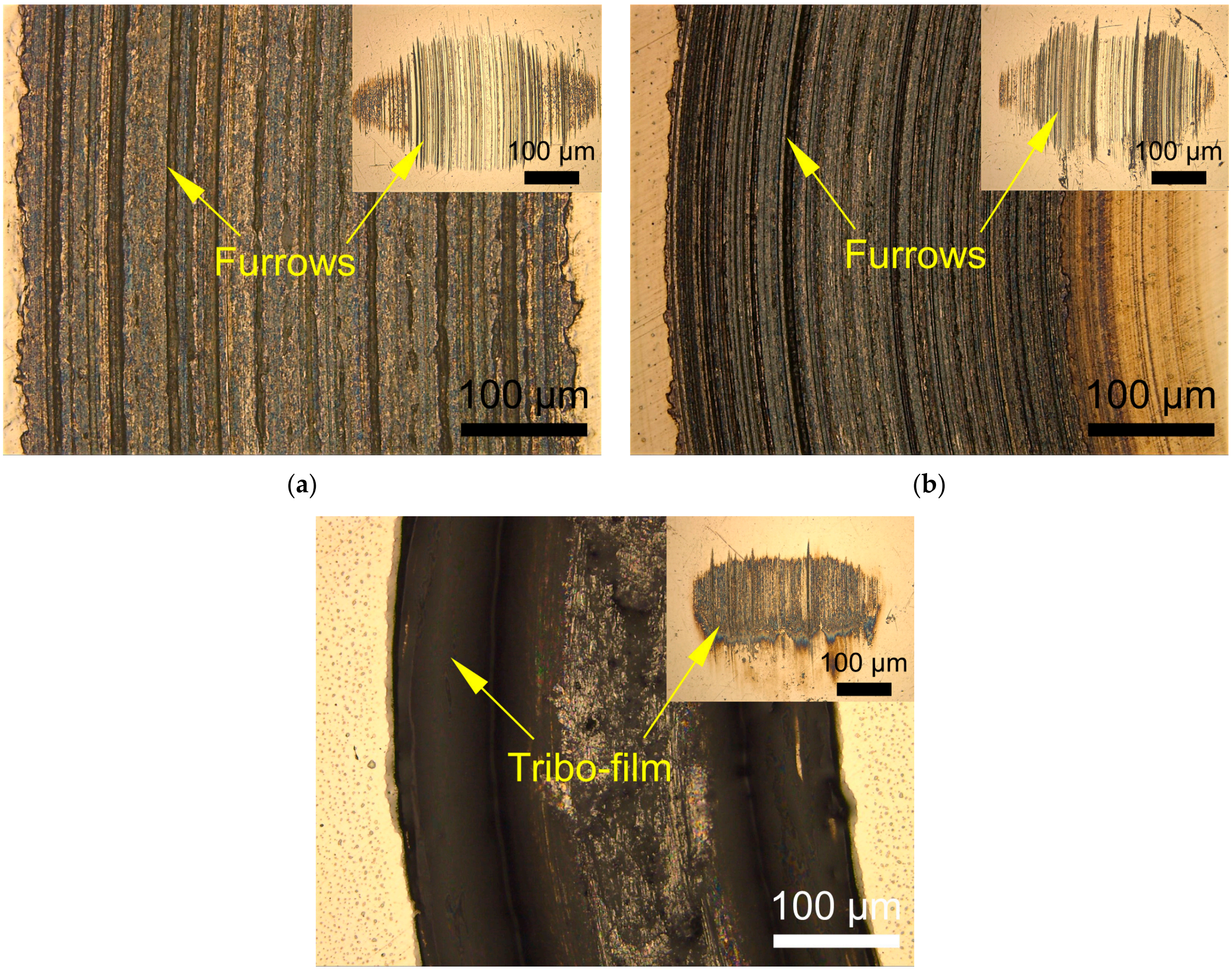


Figure 6. (**a**) Friction coefficient curves versus sliding time and (**b**) wear volume of GCr15 disks lubricated by PAO10 base oil containing Ti_2SnC and Cu-coated Ti_2SnC.

Figure 7 shows the optical wear images of the GCr15 disks after the sliding tests that were lubricated by PAO10 base oil, PAO10 base oil containing 5 wt.% Ti_2SnC, and PAO10 base oil containing 5 wt.% Cu-coated Ti_2SnC. As show in Figure 7b,c, some remnant Ti_2SnC and Cu-coated Ti_2SnC particles could be detected on the unworn surfaces of the GCr15 disks. For the PAO10 base oil, large continuous furrows could be observed, which could be attributed to two-body abrasive wear between the GCr15 tribo-pairs, as shown in Figure 7a. When 5 wt.% Ti_2SnC was added into the PAO10 base oil, the furrows were diminished, as shown in Figure 7b. The wear scars of the GCr15 balls had similar wear morphologies, as shown in the inserts of Figure 7a,b. The addition of PAO base oil between the sliding interface could result in a low friction coefficient, where the friction coefficient was as low as 0.122. With the incorporation of Ti_2SnC into the PAO base oil, the friction coefficient slightly came down (0.114). Wu and Xue et al. reported that, when Ti_3SiC_2 powders are doped into base oil, the friction coefficient could also be reduced, in which the friction coefficient could come further down with an increasing sliding speed [19,21]. This indicates that a base oil containing MAX phase powders can improve the lubricating behaviors of the base oil. However, in this study, it appeared that the Ti_2SnC ceramic showed a superior lubricating effect in the base oil in comparison to the Ti_3SiC_2 MAX phase. In particular, for the PAO10 base oil containing 5 wt.% Cu-coated Ti_2SnC, the friction coefficient was decreased by 22% compared the pure PAO10 base oil, in which the friction coefficient was as low as 0.095. As shown in Figure 7c, the furrows that were caused by two-body abrasive almost disappeared. Instead, a layer of tribo-film could be found on the wear track. The tribo-film, which was traced to the tribo-chemical reaction of the Cu-coated Ti_2SnC, could further reduce the friction coefficient. Furthermore, the corresponding wear scar of the GCr15 ball was covered by a layer of tribo-film as well. The most likely explanation could be attributed to the modification of the layer of Cu nano-particles. On the one hand, the layer of Cu nano-particles might have been destroyed and distributed into the PAO10 base oil during the sliding process, which could result in a slight fluctuation of the friction coefficient. On the other hand, a layer of tribo-film induced by a tribo-chemical reaction could be generated due to the incorporation of Cu nano-particles, which can reduce friction and wear.



(**a**) (**b**) (**c**)

Figure 7. Optical wear images of GCr15 after sliding tests that lubricated by (**a**) PAO10, (**b**) PAO10 + 5 wt.% Ti_2SnC, and (**c**) PAO10 + 5 wt.% Cu-coated Ti_2SnC; inserts show the corresponding wear morphologies of GCr15 balls.

The 3D wear tracks of GCr15 after the sliding tests that were lubricated by PAO10 base oil, PAO10 base oil containing 5 wt.% Ti_2SnC, and PAO10 base oil containing 5 wt.% Cu-coated Ti_2SnC are shown in Figure 8. For the PAO10 base oil, the width of the wear track was as high as 413 μm, as shown in Figure 8a. When the Ti_2SnC and Cu-coated Ti_2SnC were incorporated into the PAO10 base oil, the width of the GCr15 wear tracks came down to 392 μm and 350 μm, respectively, as shown in Figure 8b,c. Furthermore, the depth of the wear tracks gradually became shallow when the Ti_2SnC and Cu-coated Ti_2SnC were doped into the PAO10 base oil. The variation in the GCr15 wear tracks also confirmed that the PAO10 base oil containing 5 wt.% Cu-coated Ti_2SnC could display a better wear-resistant property. On account of the addition of Ti_2SnC powder, the oil film strength could be notably elevated, which could reduce the friction coefficient during the sliding [26]. It is well known that Cu nano-particles can contribute to generating a stable tribo-film to diminish the destruction of lubricating films during sliding [27]. Moreover, Cu nano-particles can also increase the intensity of an oil film because of the adsorption effect of these Cu nano-particles, in which a higher intensity of tribo-film could lead to a better lubrication effect. Therefore, due to the synergistic lubrication effect of the Ti_2SnC and Cu nano-particles, the PAO10 base oil within the Cu-coated Ti_2SnC powder could exhibit superior lubrication behaviors.

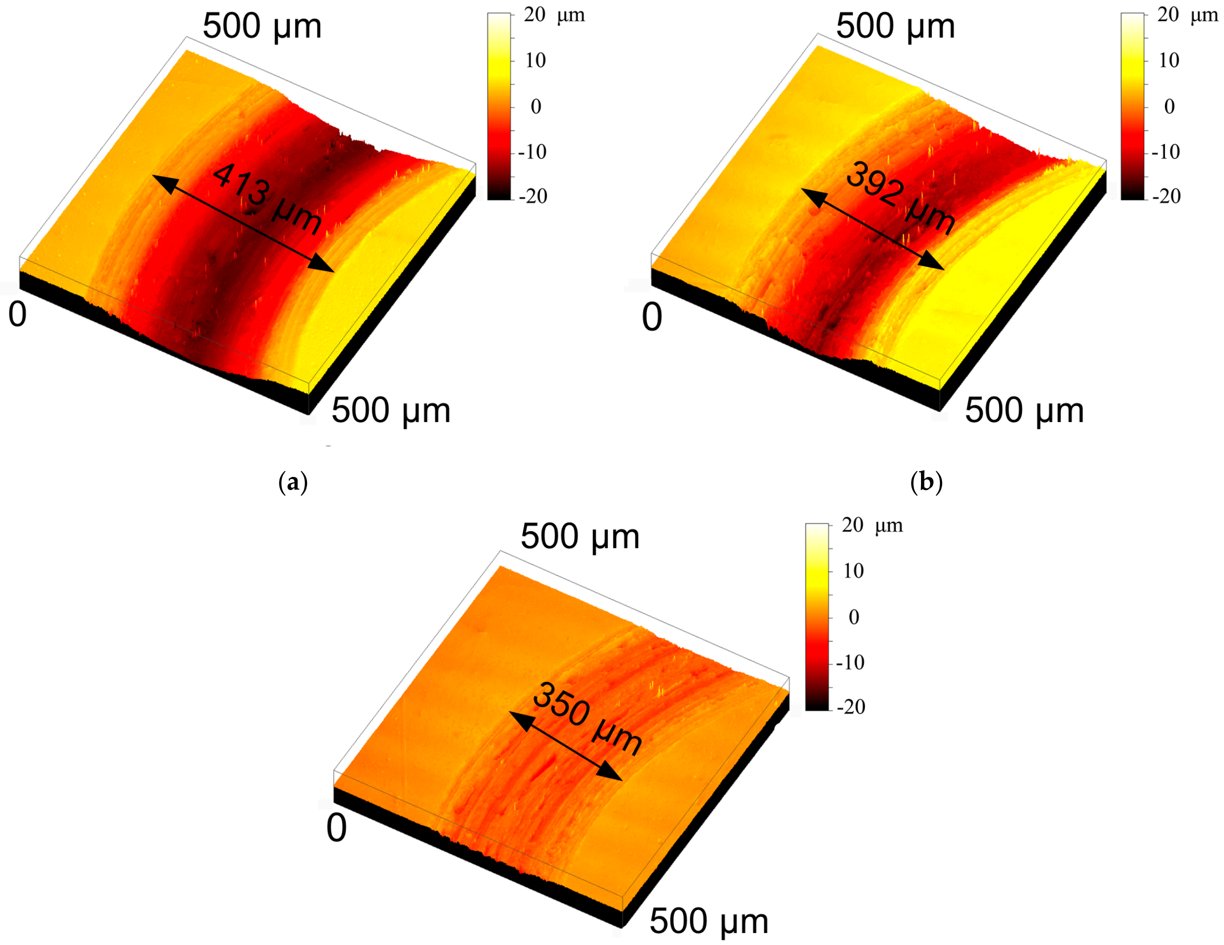


Figure 8. 3D wear tracks of GCr15 after sliding tests that lubricated by (**a**) PAO10, (**b**) PAO10 + 5 wt.% Ti_2SnC, and (**c**) PAO10 + 5 wt.% Cu-coated Ti_2SnC.

4. Conclusions

In this paper, a Cu-coated Ti_2SnC ceramic that was employed as a lubricant additive for PAO10 base oil was prepared and its tribological behaviors were studied. Herein, Cu-coated Ti_2SnC ceramic was prepared for the first time using a chemical plating method. It was established that the Ti_2SnC ceramic had favorable self-catalytic activity. The Cu nano-particles could be stacked onto the surface of the Ti_2SnC particles without pretreatment. When Ti_2SnC ceramic particles were incorporated into the PAO10 base oil, the friction and wear of the GCr15 tribo-pair was slightly improved. The main wear mechanism was two-body abrasive wear. It was established that the friction coefficient of PAO10 base oil can be greatly improved by incorporating Cu-coated Ti_2SnC ceramic powder, in which the friction coefficient can be as low as 0.095. The two-body abrasive wear can be greatly inhibited when Cu-coated Ti_2SnC ceramic is doped into PAO10 base oil. The better lubrication behaviors can be attributed to the synergistic lubrication effect of Ti_2SnC and Cu nano-particles. It can be concluded that Cu-coated Ti_2SnC ceramic can be employed as an additive for base oil in tribological fields.

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