Tribology Letters (2018) 66:135
https://doi.org/10.1007/s11249-018-1091-1

ORIGINAL PAPER

Exploring Lubrication Function of MACs Greases for TC4 Alloy Under Sliding and Fretting Conditions

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Received: 25 July 2018 / Accepted: 17 September 2018

Abstract

Lubrication function of multialkylated cyclopentanes (MACs) greases for the contact TC4/Steel was investigated in detail under the sliding and fretting conditions, and the worn tracks were measured by surface/interface analysis techniques to explore the friction mechanisms. The results demonstrate that these lubricating greases have good friction-reducing function under fretting and sliding conditions, and their friction reduction is superior to wear resistance, especially calcium sulfonate complex grease. Good lubrication function of MACs greases under sliding friction mainly depends on synergies of grease-film and friction-induced chemical reaction film. Fretting behaviors depend on not only grease-film and chemical reaction film, but also fretting wear mechanisms including abrasion, oxidation, and delamination.

Keywords Fretting · Sliding · Lubricating grease · Boundary lubrication

1 Introduction

Titanium alloys with high strength-to-weight ratio, good mechanical property, excellent corrosion resistance, good bio-compatibility, have been applied broadly to aerospace, biomedical, energy and various industries, and so forth [1–5]. Unfortunately, their poor friction-reducing and anti-wear properties have hindered the wide application in sliding and fretting conditions due to their low anti-shearing intensity, severe adhesive wear, low thermal conductivity, high chemical activity, low hardness, and elastic modulus. Intensive efforts have been made to enhance the tribological performance and to realize the long-term mechanical stability [6]. To reduce or eliminate the severe adhesive wear of titanium alloys, surface techniques have been applied to enhance the mechanical characteristics [7]. Li et al. prepared the modified layer consisted of TiO_2, Ti_2O_3, and TiO on the titanium alloys via by plasma based ion implantation [8], Nie et al. prepared the layered hydroxyapatite/TiO_2 coatings on the titanium alloys by using a combination of micro-arc oxidation and electrophoresis [9], their goals are to overcome the drawback of poor wear resistance. Deposition techniques (physical vapor deposition and chemical vapor deposition) have been introduced to enhance the load-carrying capacity and improve the tribological properties [10–12]. Sharma and Sehgal achieved the considerable reduction in friction and wear of titanium alloy (Ti6Al4V) by using lubricative media under sliding condition, and the analysis of wear mechanism indicated the co-existence of cutting, fatigue, and sliding wear [13]. Wang et al. investigated the tribological behaviors of micro-arc oxidation coatings under unlubricated conditions and the lubrication of oil, indicating that the coatings with oil lubrication for the contact of Ti6Al4V/Steel can reduce the shear stress and adhesive wear at the fretting conditions [14]. Considering the costs of surface modification techniques and the limitation of liquid lubricants, the use of lubricating grease may be a relatively simple approach to improve the wear resistance and broaden the application of titanium alloys.

Grease is a two-component colloid disperse system consisting of base oil (mineral, vegetable or synthetic oil) and thickener (metal soap, polyurea, polytetrafluoroethylene, and so on) [15, 16]. Greases have been widely applied for lubrication and sealing protection of mechanical

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Published online: 21 September 2018

components around the world, dependence on their corrosion resistance, semi-permanent lubrication, adhesion and seal, good friction-reducing and anti-wear abilities [17, 18], thereby enhancing the long-term safety and dependability of mechanical components. Synthetic oils are a better choice in some respects than mineral base oils, in case where mineral oils have difficulty in meeting service requirements under the coupling conditions of operating modes and environmental factors. Given that colloid structure, lubrication function, physicochemical properties of grease are mainly determined by base oil and thickener, multiply-alkylated cyclopentanes (MACs) as a class of novel lubricant are expected as an alternative to traditional base oils due to its ultra-low vaper pressure, viscosity–temperature characteristics, good durability, load-carrying capacity chemical and thermal stability, and so forth [19–22]. In our previous study, the lubrication function of MACs and its greases for Steel/Steel and Coatings/Steel contact have been studied, and found that MACs greases with different colloid structure showed significant differences in friction reduction and wear resistance [23, 24]. But the research efforts in lubrication function of MACs greases for the contact of Ti–6Al–4V/Steel have not been made public, so it is of great importance to evaluate the tribologial behaviors of Ti–6Al–4V under lubrication of MACs greases for achieving the high-performance requirements of modern/future machines and widening the engineering applications of titanium alloys and MACs greases.

Here, four MACs greases were prepared using lithium complex soap, polyurea, calcium sulfonate complex soap, and polytetrafluoroethylene (PTFE). Their lubrication function for Ti–6Al–4V/Steel contact were systematically investigated under sliding and fretting conditions, and the friction mechanism was also explored by scanning electron microscope (SEM), X-ray photoelectron spectroscopy (XPS), and electron-prohe microanalyzer (EPMA).

2 Experimental Procedure

2.1 Materials

Commercial titanium alloys (Ti6Al4V, TC4) disks with hardness of HRC35 and surface roughness of 30 nm were used as substrate for the contact of TC4/Steel. AISI 52,100 steel balls as upper specimen with 10 mm diameter, hardness of 710 HV and surface roughness of 30 nm run against the substrates. Four MACs greases were prepared according to our previous report [23], and all the MACs greases are composed of 75 wt% MACs base oil and 25 wt% thickener. Four MACs greases are named according to the thickener, including lithium complex grease (LCG), polyurea grease (PUG), calcium sulfonate complex grease (CSCG), and polytetrafluoroethylene (PTFE) grease (PFG). Their physical properties are listed in Table 1.

2.2 Tribological Behaviors of MACs Greases

2.2.1 Fretting Wear Experiment

Fretting wear tests of TC4 under four MACs greases lubrication were conducted on self-made reciprocation fretting wear tester with ball-on-block contact configuration. The tangential fretting friction experiment was performed at 10 N (contact pressure of 0.784 GPa) and 20 N (contact pressure of 0.987 GPa), at a frequency of 2 Hz with constant amplitude displacement of 50 μm for 60 min at room temperature (25 °C). After friction tests, the friction pairs were ultrasonically cleaned using petroleum ether and ethanol for surface analysis. To promise the repeatability and accuracy of friction test, it was repeated three times under the same conditions.

2.2.2 Sliding Friction Experiment

Sliding friction tests of TC4 under greases lubrication were investigated on an UMT-3MT ball-on-disk reciprocation friction tester at 5, 10, and 20 N at 25 °C, respectively. All friction tests were operated at an amplitude of 4 mm and the frequency of 2 Hz for an hour. To ensure the repeatability and accuracy of friction tests, it was repeated three times under the same condition.

2.3 Characterization and Analysis of Worn Surface

Three-dimensional (3D) morphology of wear tracks was observed by a Bruker Contour GT white-light interferometer, and the wear volume of lower specimen was calculated. The morphology of worn surfaces with energy dispersive X-ray spectroscopy (EDS) was investigated by JSM-6610LV scanning electron microscope (SEM, JEOL, Japan). The elements' chemical valence states on the wear tracks were obtained by Thermo Scientific ESCALAB 250Xi X-ray photoelectron spectroscopy (XPS, America). The binding energies of the target elements were calibrated by referencing the C 1s peak at 284.8 eV with a resolution of about ± 0.3 eV, using a pass energy of 20 eV. In addition, the worn surface

Table 1 Physical properties of four MACs greases

MACs grease	LCG	PUG	CSCG	PFG
Dropping point (°C)	260	274	290	253
Penetration (1/4 mm)	92.4	85.5	80.4	72.2
Colloid stability (w/w%)	1.7	0.8	1.8	1.1

of TC4 substrates was also analyzed by electron-probe microanalyzer (EPMA, JXA-8230, Japan).

3 Results and Discussion

3.1 Fretting Wear Properties

3.1.1 F_t–D Curves

The friction force versus displacement loops (F_t–D) gives the most important signal for analyzing the fretting behavior and deformation behavior, which mainly depends on the contact condition, normal force and displacement amplitude [25, 26]. Generally, the fretting wear (FW) regime can be divided into the partial slip regime (PSR), mixed fretting regime (MFR) and the slip regime (SR) according to three kinds of F_t–D curves including linear, elliptical, and parallelogram shapes, respectively [27].

Figure 1a shows the F_t–D curve with an approximate parallelogram shape, the horizontal line segments AA_1 and BB_1 are assigned to the sliding mode of the TC4/Steel contact, and the slanting segments AB and A_1B_1 correspond to the static friction force. All F_t–D curves (as shown in Fig. 1) exhibit the shape of parallelograms-like shape during the entire fretting process at 10 N. The fretting running modes at the contact interface were gross slip and ran in the SR. Besides, for the maximum kinetic friction force under fretting conditions, in the early stage it was often greater than that in the later stage. Compared with unlubricated TC4/Steel, TC4/Steel under MACs greases lubrication (Fig. 1a–e), the CSCG allows the friction pairs to keep the more stable and smaller friction force value (Fig. 1d). Observing the Fig. 1f, the maximum kinetic friction force emerges at about 178 cycles, and the tendency of friction force with the increase in cycles is to first increase, then decrease, and finally remain steady at a constant value. When the applied load was increased to 20 N, however, the F_t–D curves of unlubricated TC4/Steel (Fig. 2a) and PUG-lubricated TC4/Steel (Fig. 2c) markedly differ from the other three greases, showing a parallelogram-like shape at the first several cycles and then similar quasi-elliptical shape after about 400 cycles (as shown in Fig. 2f). This indicates that the fretting running states of TC4/Steel and under PUG lubrication were unstable and shifted between PSR and SR, and were defined as MFR according to fretting map theory [25]. These results demonstrate that the introduction of MACs greases for TC4/Steel contact could change the fretting running states and the tribological behaviors via the effect of grease-film. Figure 2f illustrates that the first maximum kinetic friction force emerged at about 70 cycles, whose F_t–D curve was parallelogram-like shape

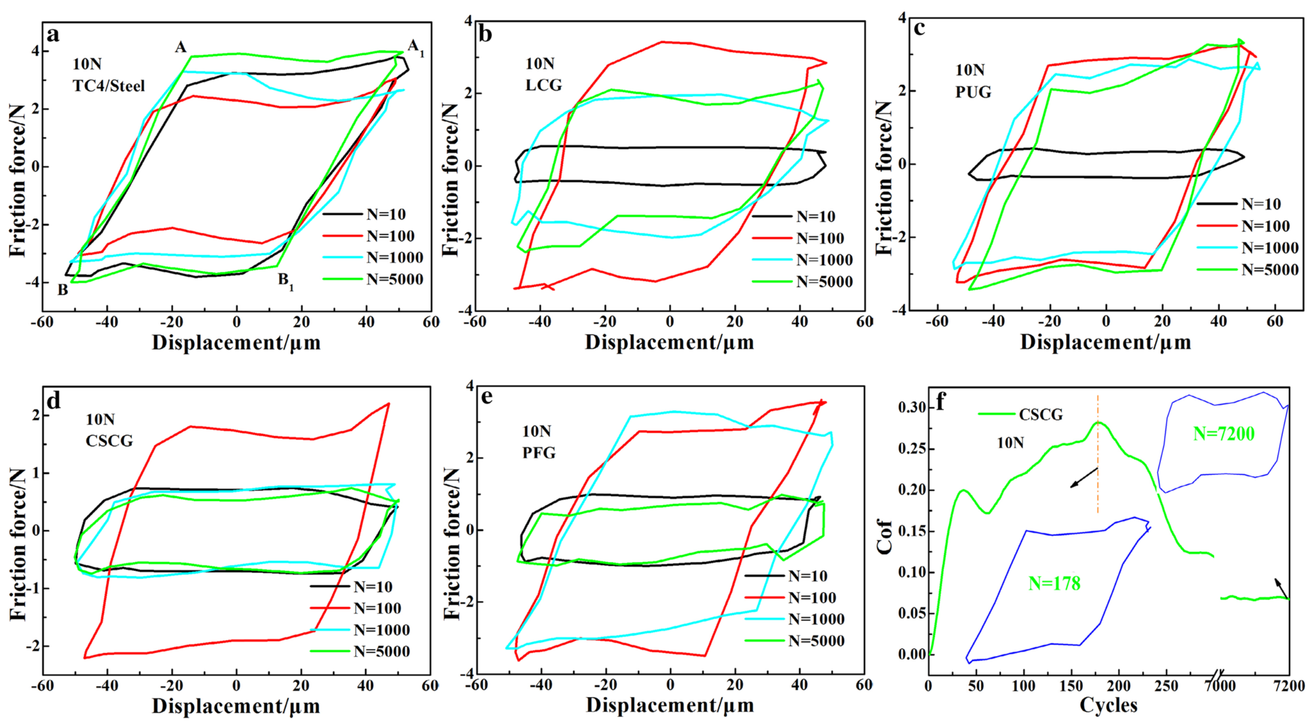

Fig. 1 F_t–D curves as a function of the number of cycles for TC4/Steel contact at applied load of 10 N with different MACs greases lubrication: **a** lubricant–free one, **b** LCG lubrication, **c** PUG lubrication, **d** CSCG lubrication, **e** PFG lubrication and **f** the relationship between friction coefficient and F_t–D curves in SR

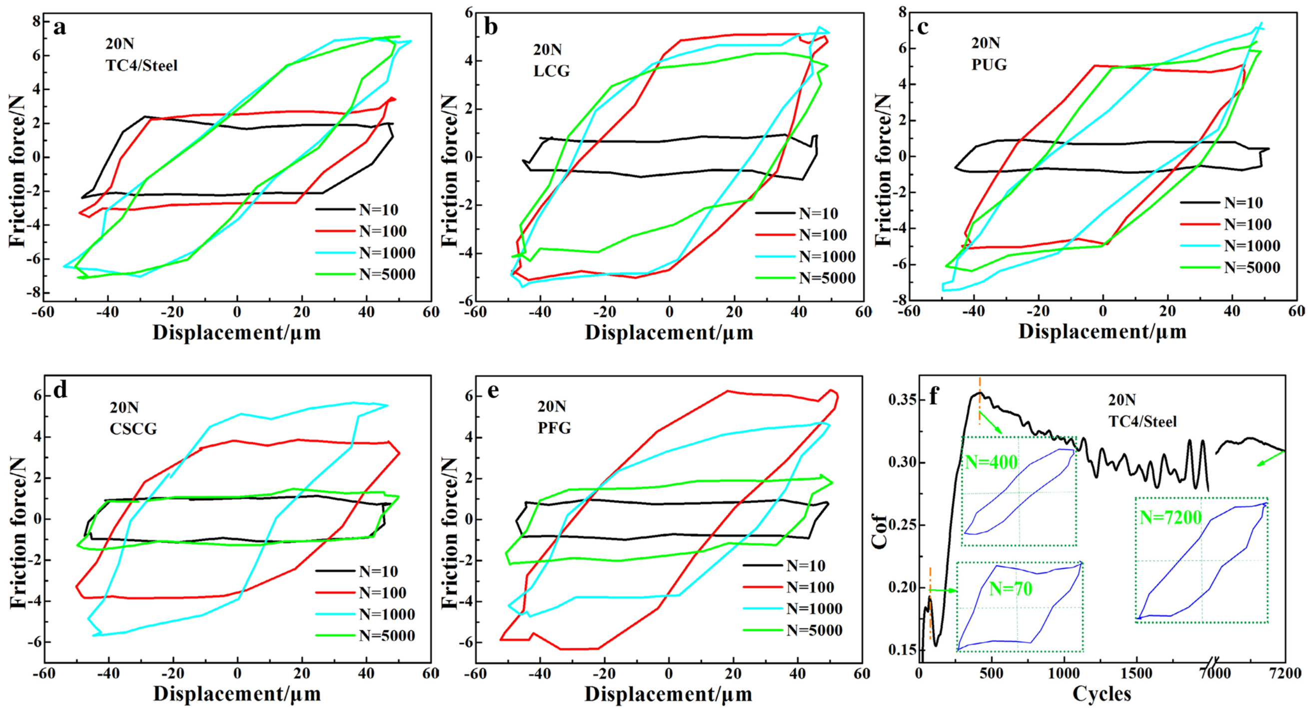

Fig. 2 F_t–D curves as a function of the number of cycles for TC4/Steel contact under different MACs greases lubrication at 20 N: **a** unlubricated one, **b** LCG, **c** PUG, **d** CSCG, **e** PFG and **f** the relationship between friction coefficient and F_t–D curves in MFR

and the F_t–D curve was changed to quasi-elliptical shape at the second maximum kinetic friction force about 400 cycles.

3.1.2 COF and Wear Volume

To investigate the lubrication function of MACs greases for TC4/Steel contact, Fig. 3 shows the real-time friction curves under MACs greases lubrication (Fig. 3a) at the applied load of 10 N, their average coefficient values (Fig. 3b) and the wear volume of the TC4 substrates (Fig. 3c) as well as cross-section profiles of the wear tracks at 20 N (Fig. 3d). Figure 3a shows the unstable friction curves for TC4/Steel contact under LCG and PUG lubrication at the low applied load of 10 N. Friction coefficient values of unlubricated TC4/Steel are greater than that under lubrication with four MACs greases at 10 N and at 20 *N*, especially CSCG, which reduces friction coefficient to about 0.07, indicating that CSCG has played an important role for lubrication and realizing the lowest friction. CSCG with stable and low friction could greatly reduce the wear (as shown in Fig. 3c), whereas the friction curves of LCG and PUG (Fig. 3a) show high fluctuation, which causes the increase in wear loss even higher than that of unlubricated TC4/Steel (as shown in Fig. 3c). Figure 3d exhibits the profile micrographs of the wear scars on TC4 substrates. Along with the running direction, the maximum wear depth occurred at the center of contact zone, and LCG gave the maximum depth and width of wear scar.

Surprisingly, the minimum wear scar originated from lubricant-free TC4/Steel, it might be because the adhesive layer and the wear debris made up the fretting wear. Generally, the efficient lubrication of grease is due to a grease-film on the mating surfaces. Compared with lubrication function of four MACs greases under fretting modes, the results indicate that MACs greases with different thickeners affect the friction reduction and wear resistance at different level because of the difference in the physicochemical characteristics of four greases, the structure and tribo-chemical reaction of four thickeners.

3.1.3 Wear Surface Analysis

Figure 4 shows SEM images with EDS of the wear tracks on the TC4 substrates at 20 N. CSCG plays an efficient role in lubrication function, which provides the smallest worn area than others and unlubricated one. Some typical elements [oxygen, iron, calcium originating from thickener of CSCG (point h), fluorine from PTFE (point i and j)] were measured on the worn surface, indicating that some complex oxygen species and tribo-chemical products could be formed on the worn surfaces. Observing LCG and PFG, it represents some grooves on the relatively dark area paralleled to fretting direction. Through corresponding EDS point scanning analysis (point *c* and *i*), it was speculated that these area might form a transfer layer or reaction layer, and wear

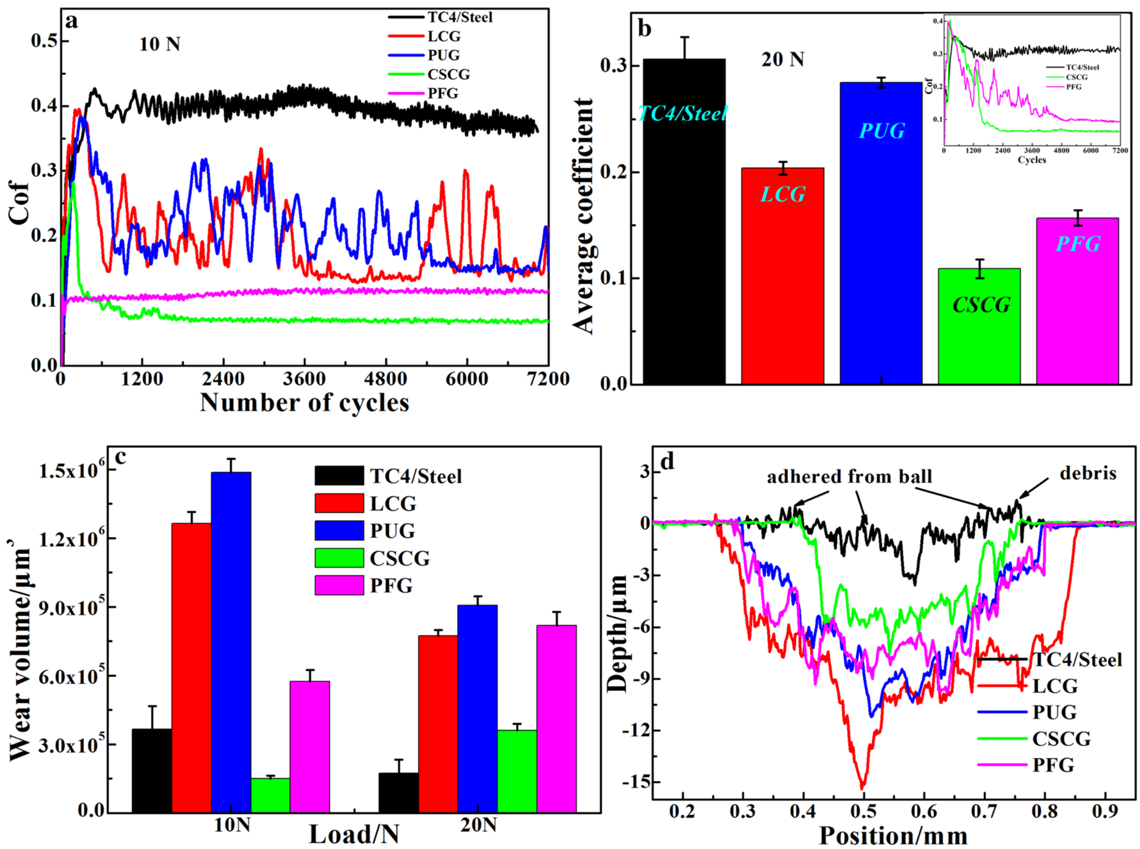

Fig. 3 The friction curves as a function of number of cycles (**a**), the average coefficient with the friction-cycles curves (**b**),wear volume (**c**) of TC4/Steel and four MACs greases at room temperature (25 °C) and cross-section profiles of the wear tracks with the applied load of 20 N (**d**)

is mainly attributed to adhesion and plastic deformation, in addition, it was also found that the wear debris and some fragments have adhered to the wear scars.

Figure 5 exhibits the distribution of typical elements' content on the worn surfaces including chromium, iron, titanium, and calcium by EPMA. For unlubricated TC4/Steel, a greater number of iron and chromium elements were adhered to the whole worn surface of TC4, illustrating that the worn debris of steel balls can be stuck between the contact interfaces and formed a transfer layer [28]. The iron and typical elements of thickeners were detected on the wear tracks by lubrication, illustrating that tribo-chemical reaction products and transfer layer have been formed on the friction interfaces. The difference is that these typical signals were detected on the edges of worn tracks and little iron on the center of worn surfaces lubricated by LCG, PUG, and PFG, implying that the wear debris composed of thickener and tribo-reaction products were squeezed out of the contact interfaces and could lead to poor lubrication. CSCG shows the excellent friction reduction and wear resistance for the TC4/Steel contact possibly because it has good physico-chemical and mechanical performance characteristics, and lubricating films made up of MACs-film and tribo-chemical reaction film play a role in lubrication function as well [23]. Transfering film of TC4/Steel could be easily broken with the increase in applied load, thereby leading to the high friction coefficient [29, 30].

To further confirm the possible tribo-chemical products and explore the fretting wear mechanism, the XPS spectra of typical elements on the worn surfaces were obtained. Figure 6 depicts the spectra of C 1s, O 1s, Ti 2p, Fe 2p, Ca 2p, N 1s, S 2p, and F 1s on the wear tracks lubricated by MACs greases and unlubricated TC4 alloy at 20 N. The C 1s spectra give the relatively weak peaks at 286.7 and 288.6 eV, illustrating the generation of carbon oxide including C–O and C=O bonds [31]. The C 1s peaks around at 291.6 and 293.1 eV can be identified as C–C bond, carbon–oxygen and carbon–fluoride compounds, respectively, which could arise from the breakdown products of thickener and tribo-chemical products. The O 1s peaks at 531.9 and 530.2 eV are assigned to the appearance of complex oxide products, and another peak at 536.0 eV belongs to hydroxide or oxide products. The Ti 2p peaks at 458.9 and 464.9 eV are attributed to titanium oxidation (Ti–O bond) because titanium with high chemical activity can easily react with oxygen to form an oxidation layer [32, 33]. XPS spectrum of Fe

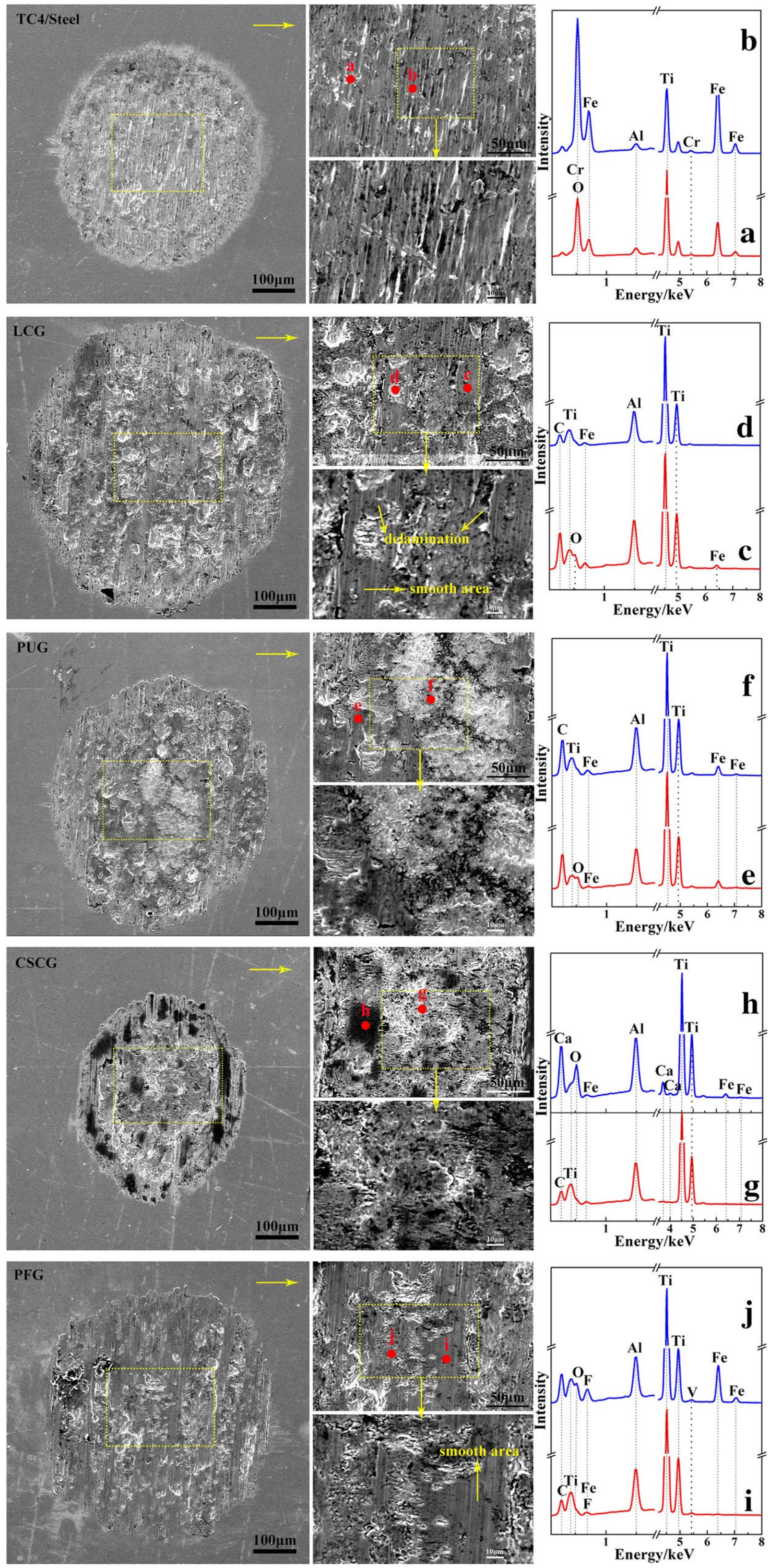

Fig. 4 SEM micrographs with low and high magnification and corresponding EDS point scanning analysis of the wear surfaces on TC4 substrates with dry friction and grease lubrication (LCG, PUG, CSCG and PFG) at the applied load of 20 N

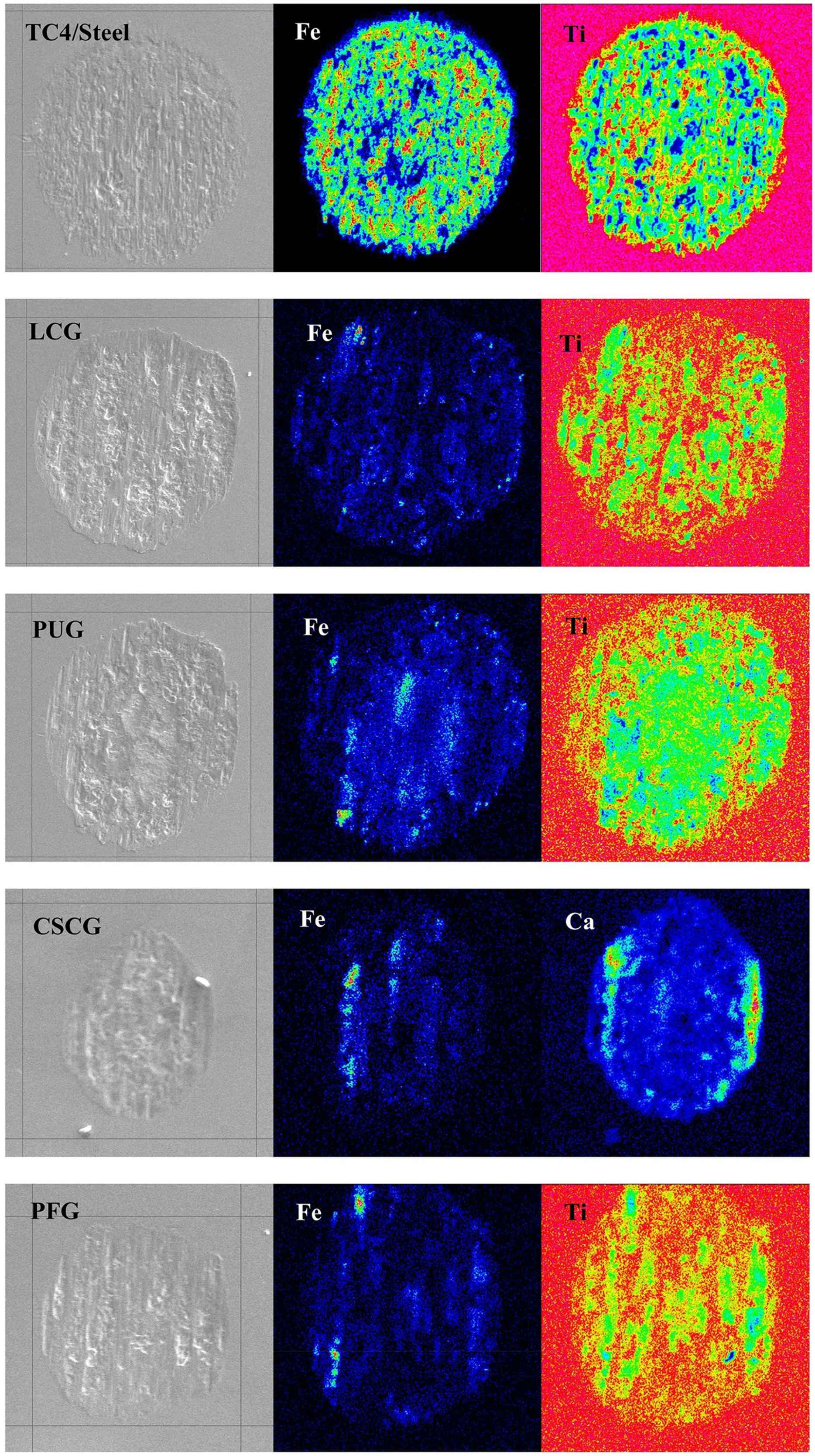

Fig. 5 The EPMA analysis of TC4/Steel and four MACs greases on the worn TC4 alloy surface at the applied of 20 N

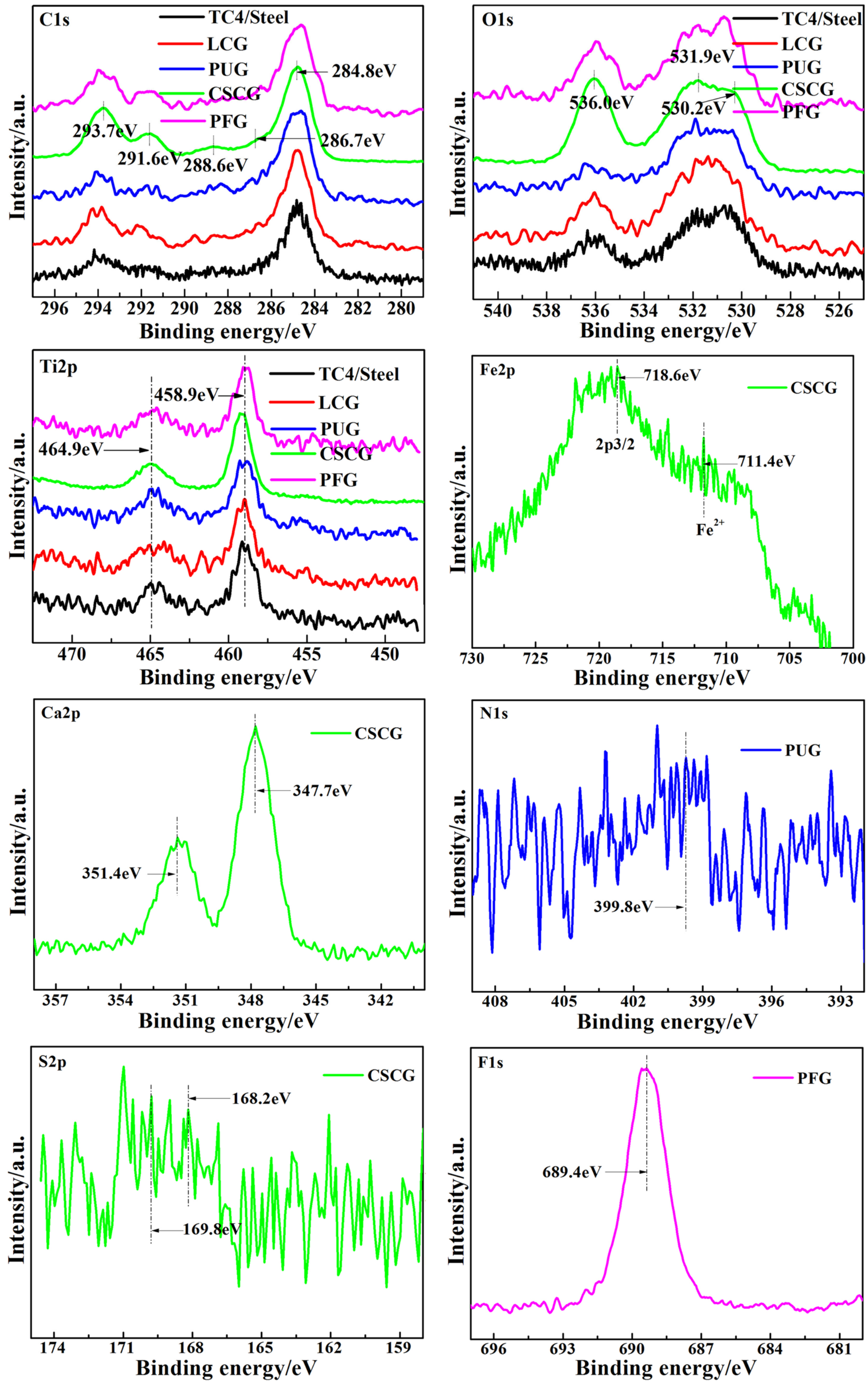

Fig. 6 X-ray photoelectron spectra of the typical elements (C 1s, O 1s, Ti 2p, Fe 2p, Ca 2p, N 1s, S 2p, and F 1s) on the worn surfaces under TC4/Steel and lubrication by four MACs greases at applied load of 20 N under room temperature

element at around 718.6 and 722.4 eV corresponds to Fe $2p_{3/2}$ and could be assigned to the iron compounds [34]. Two distinct Ca 2p peaks at 347.7 and 351.4 eV were observed under CSCG lubrication, which was assigned to the calcium oxide and calcium carbonate [35], respectively. The N 1s peak located at 399.8 eV could be assigned to the nitrogen oxide and/or carbonitride from the polyurea [36]. The S 2p spectrum was not clearly detected on the worn surface of CSCG, whereas by certain signs in S 2p peak it can be speculated the formation of the sulfide and sulfate, probably because the production was removed by ultrasonic cleaning. The strong F 1s peak at 689.9 eV is due to C–F bond from the PTFE thickener of PFG [37].

These analysis results of worn surfaces including SEM with EDS, EPMA, and XPS illustrate that the physical-absorbing film, transfer film and oxygen species (including carbon, titanium, iron, calcium, nitrogen compounds) were generated on the worn surfaces at the fretting conditions. What's more, the tribo-chemical reaction film on the fretting interfaces also plays an important role in friction reduction and wear resistance under grease lubrication, which was formed via the interaction with active elements of thickener and metal elements of friction pairs under high applied load or other harsh conditions. For the contact of TC4/Steel under LCG, PUG, and PFG lubrication, these films as the third body can reduce the friction, but accelerate the wear due to the behavior of squeezing wear debris out of the contact interface. For CSCG, its tribo-chemical reaction film composed of calcium oxide and calcium carbonate and absorbing grease-film have better load capacity so that CSCG has excellent friction-reducing and anti-wear abilities with the increase of applied loads. Furthermore, the wear mechanisms of TC4/Steel were dominated by abrasion and oxidation. Under LCG, PUG, CSCG, and PFG, the wear mechanisms were mainly abrasion, oxidation, delamination, and plastic deformation.

3.2 Sliding Wear Properties

3.2.1 Tribological Properties

The tribological properties of TC4/Steel contact with lubrication by four MACs greases under sliding conditions were evaluated at 5 N, 10 N, and 20 N, respectively, with unlubricated one as a comparison. Figure 7 shows the real-time friction curves of TC4/Steel contact under greases lubrication and wear volume of the TC4 substrates. The friction curves of unlubricated TC4/Steel are unstable and appear to fluctuate throughout the sliding process, as shown in Fig. 7a, and the friction coefficient values are the highest (~0.5),

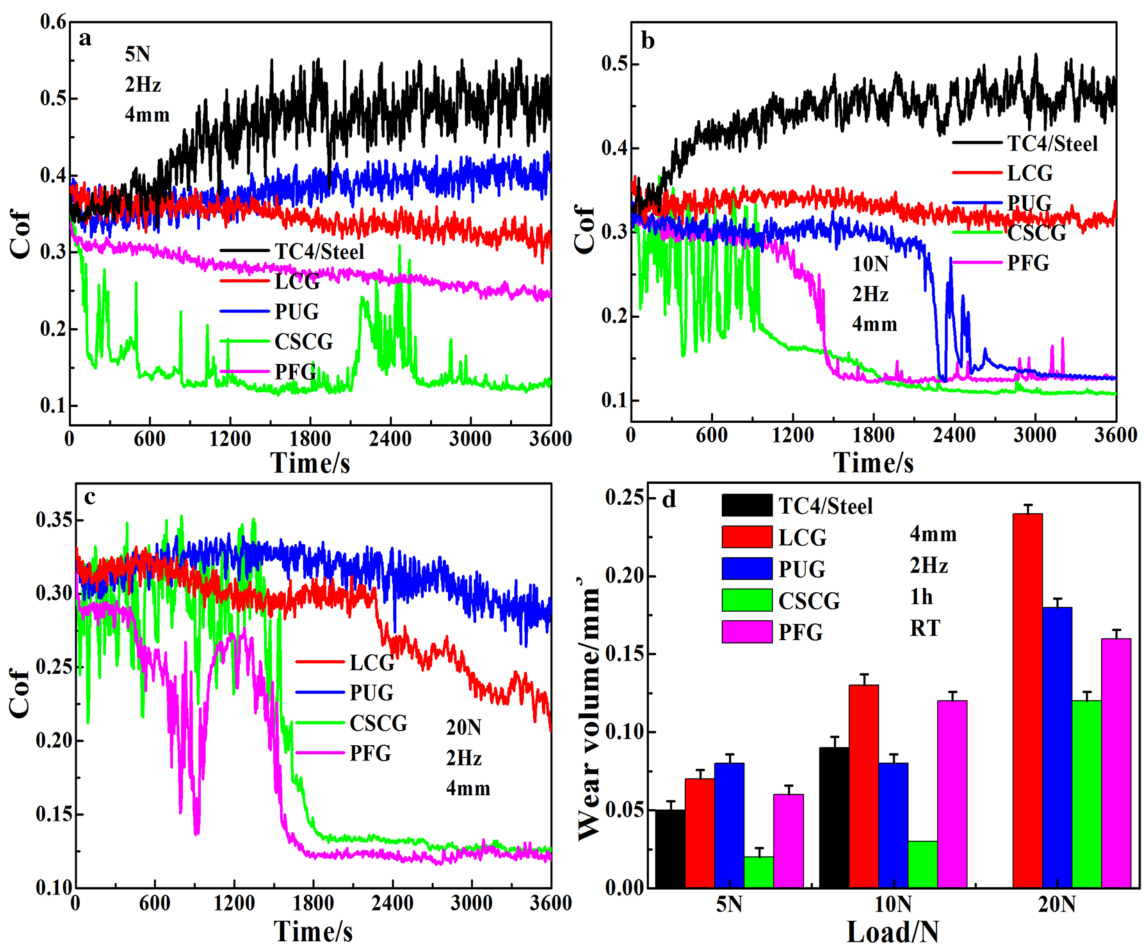

Fig. 7 The friction curves as function of time (**a, b, c**) and wear volume (**d**) of TC4/Steel and four MACs greases with the applied loads of 5 N, 10 N and 20 N

which differs from the tribological behaviors under greases condition (friction curves with relatively steady stage) and CSCG can give the lowest friction coefficient about 0.12 at the normal loads during steady stage. For CSCG and PFG, their friction curves with relatively high fluctuation appear in the starting-up stage and relatively lower friction coefficient at steady stage. The results illustrate that friction of TC4/Steel under greases lubrication was divided into initial stage, descending stage and steady stage, and greases have an effective effect on friction reduction, especially CSCG showing the most stable and smallest friction coefficient at the steady stage. Figure 7d gives the wear volume of TC4 substrates under LCG, PUG, CSCG, and PFG lubrication at normal load of 5 N, 10 N, and 20 N. CSCG has the smallest wear volume than others, and exhibits the excellent anti-wear property. Figure 8 gives the morphology, the cross-section profiles and 3D topography of tracks on the TC4 alloy with non-lubrication and MACs greases lubrication at 10 N. As is shown in Fig. 8, the worn surface without lubrication presents relatively wide and deep tracks with

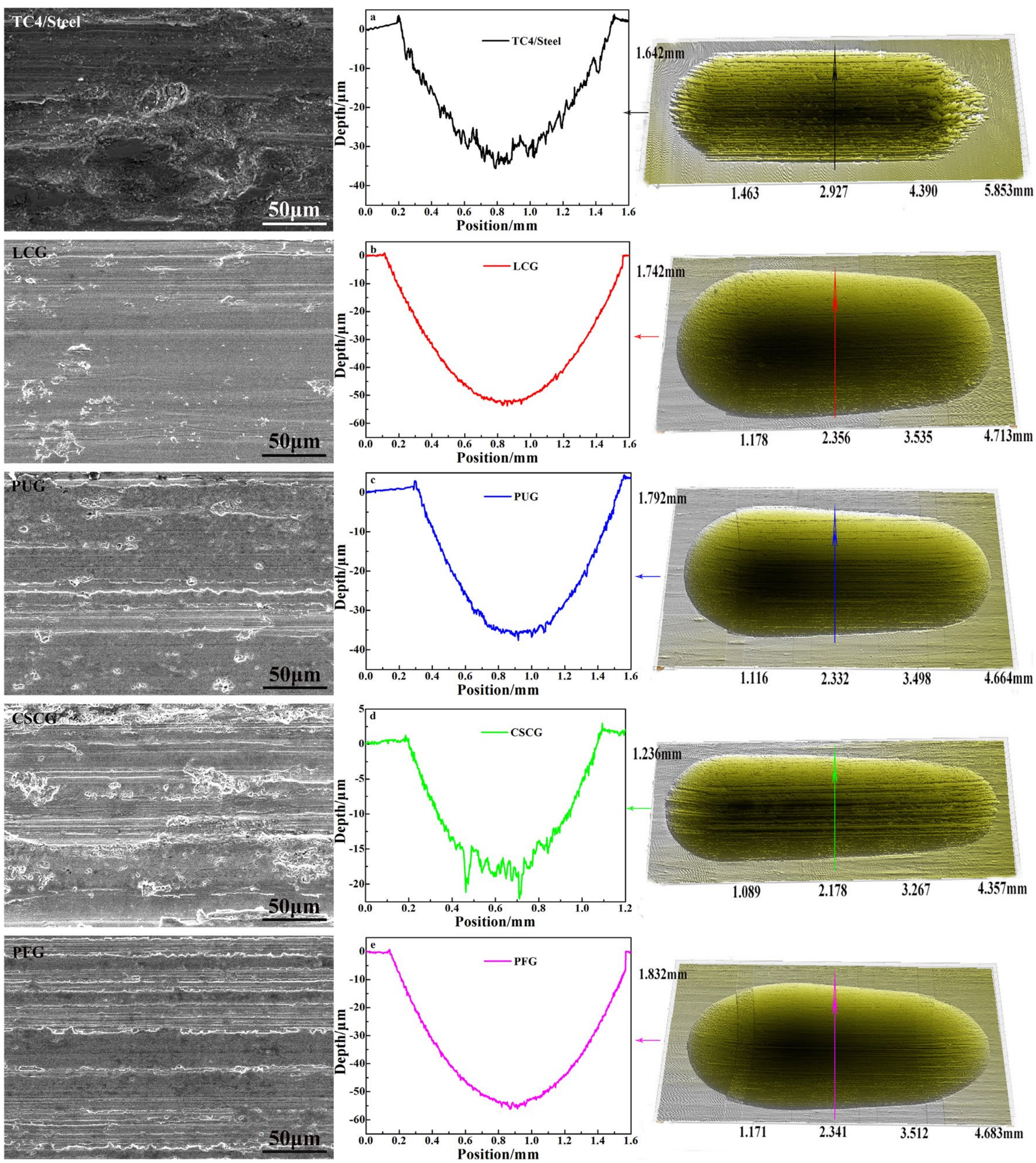

Fig. 8 The SEM micrographs, cross-section profiles, and 3D topography of the worn surfaces under non-lubrication and four MACs greases lubrication at the applied load of 10 N

numerous grooves and some large-area wear scars along the sliding direction, it also shows server adhesion and delamination. The large worn surface of TC4 substrate under LCG lubrication exhibits the deepest wear tracks mainly due to fatigue wear and plastic deformation. As being lubricated by PUG and PFG, it can be seen that the relatively deep wear tracks on the contact surfaces. The small worn surface of the substrate with CSCG lubrication presents the shallow wear tracks with a little grooves parallel to the sliding direction.

3.2.2 Analysis of Friction Mechanism

Figure 9 displays the XPS spectra of the typical elements on the wear tracks under sliding conditions at 10 N. The C 1s peak appears at 288.6 eV, ensuring the formation of some carbon oxide on the worn surfaces of TC4 alloy. Two O 1s peaks at 531.8 and 530.2 eV correspond to the complicated oxide. The peaks at binding energy of 458.5 and 464.2 eV are assigned to Ti $2p_{3/2}$ and Ti $2p_{1/2}$ components, which is titanium oxidation. Fe 2p peaks locate at ~711.2 eV, which is assigned to iron compounds. The Ca 2p spectrum could be fitted into two peaks at 347.5 and 351.0 eV, which originates from calcium oxide and calcium carbonate, respectively. The N 1s peak at 400.4 eV is due to the nitrogen oxide and/or carbonitride originating from PUG. Similarly, the obvious S 2p spectrum was not detected on the worn surface of CSCG. F 1s spectra give the lower binding energy at 684.6 eV because of fluoride,and the binding energy at 689.2 eV due to C–F bond [38]. Hence, the XPS spectra confirm the generation of friction-reduced reaction film on the sliding surfaces, which can reduce friction and wear of the sliding pairs. CSCG exhibits the best friction reduction and wear resistance dependence on the synergistic effect of the outstanding characteristics of CSCG and the chemical reaction film comprised of ferric fluoride, calcium carbonate, and oxide species.

In view of the continuous-increasing performance requirements of mechanical components with long-term reliability and accuracy, it is of great significance for the design of lubrication technologies and choice of lubricants. The operation of mechanical equipment often gets into trouble with friction and wear under sliding and fretting conditions, especially soft titanium alloys, so the research on tribological properties of soft alloy under lubrication is very important. The lubrication function of greases under sliding and fretting conditions will play a vital role in friction reduction and wear resistance. Hence, the as-prepared four MACs greases were introduced into the contact interfaces of TC4/Steel, and their tribological behaviors were evaluated in detail. The friction reduction of four greases is superior to their wear resistance under sliding and fretting conditions, such lubrication function for TC4/Steel contact is mainly attributed to synergies of grease-film and tribochemical reaction film formed by reaction of active elements in thickener and metal elements. Under fretting conditions, lubrication can induce the transformation of fretting running regime via reducing friction and enhancing mechanical properties of the mating surfaces, thereby showing the difference in friction behaviors from sliding conditions. This work offers a significant step for achieving the high-performance requirements of modern/future machines using titanium alloys under greases lubrication and widening their engineering applications.

4 Conclusion

The tribological behaviors of TC4/Steel contact with four MACs greases lubrication under the fretting and sliding conditions were investigated in detail, and friction mechanisms were also explored systematically. The conclusions can be summarized as follows:

(a) High-performance MACs greases improve the tribological properties under fretting condition, which is attributed to the lubrication-induced change of fretting running regime via synergy of grease-film and tribo-film.
(b) MACs greases show relatively poor anti-wear ability under fretting conditions due to the squeezing wear debris out of the contact interface, except CSCG with excellent physicochemical and tribological properties. The wear of unlubricated TC4/Steel was dominated by abrasion and oxidation under fretting conditions, whereas the wear with greases lubrication was mainly ascribed to the abrasion, oxidation, delamination, and plastic deformation.
(c) MACs greases provide the excellent friction-reducing abilities under the sliding conditions, mainly depending on the tribo-chemical reaction film such as complex oxides, fluoride and iron compound, whereas their poor anti-wear properties are due to the replacement of easily-broken film.
(d) CSCG as a novel multipurpose grease with excellent physicochemical and tribological properties can greatly reduce friction and wear, relying on the synergistic effect of the physical-absorbing film and tribo-chemical reaction film.

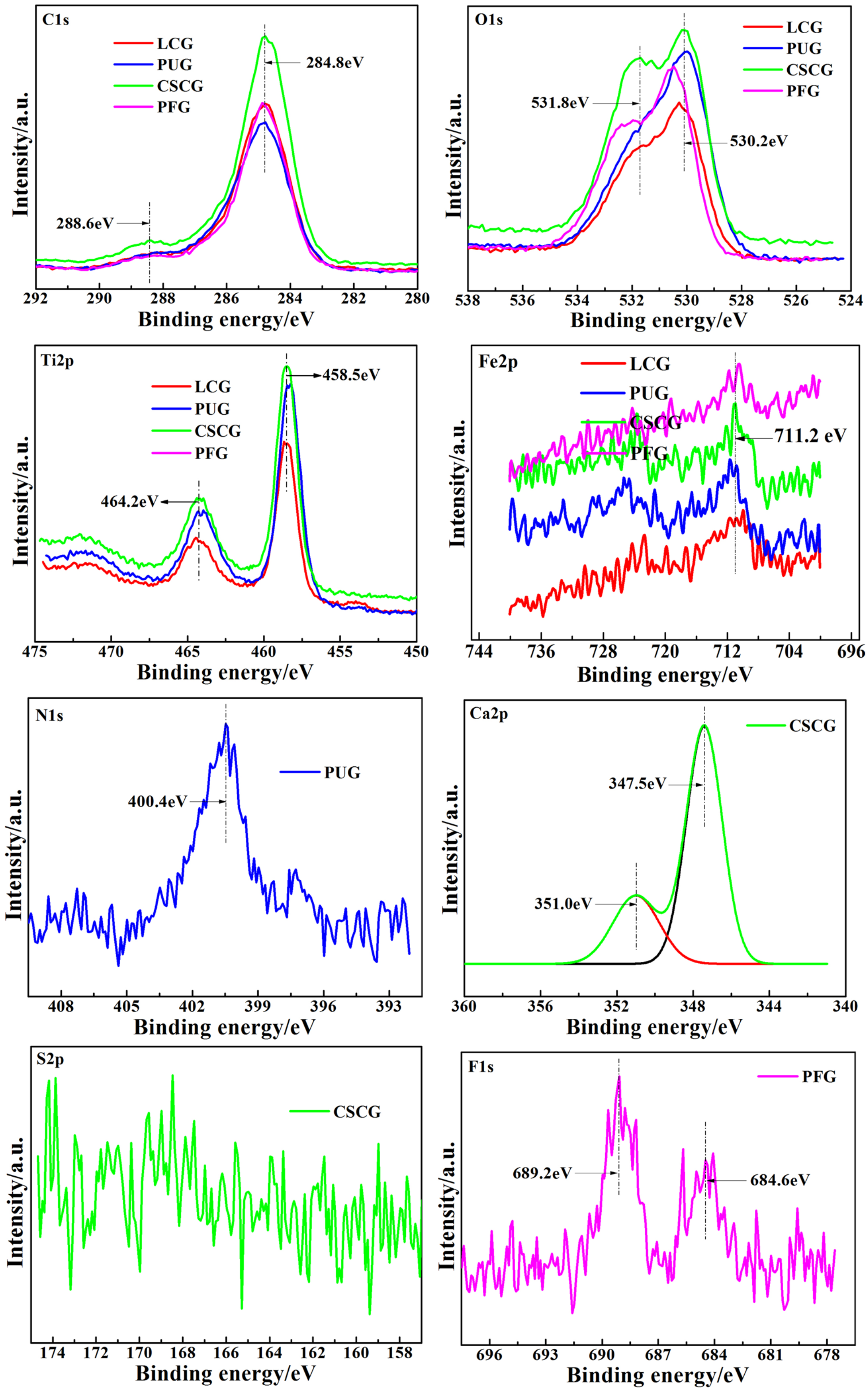

Fig. 9 X–ray photoelectron spectra of the elements (C 1s, O 1s, Ti 2p, Fe 2p, N 1s, Ca 2p, S 2p, and F 1s) on the worn surfaces under lubrication by four MACs greases at applied load of 10 N

Acknowledgements The authors gratefully acknowledge the financial support provided by the National Natural Science Foundation of China (Nos. 51705435, 51627806 and 51505050), the Key Cooperation Project of Sichuan Department of Science and Technology (No. 2018JZ0048) and Fundamental Research Funds for the Central Universities (No. 2018GF05).

References

1. Zhou, Y., Shen, M.X., Cai, Z.B., Peng, J.F., Zhu, M.H.: Study on dual rotary fretting wear behavior of Ti6Al4V titanium alloy. Wear **376**, 670–679 (2017)

2. Zhang, B.R., Cai, Z.B., Gan, X.Q., Zhu, M.H., Yu, H.Y.: Dual motion fretting wear behaviors of titanium and its alloy in artificial saliva. Trans. Nonferr. Met. Soc. **24**, 100–107 (2014)
3. Mo, J.L., Zhu, M.H., Zheng, J.F., Luo, J., Zhou, Z.R.: Study on rotational fretting wear of 7075 aluminum alloy. Tribol. Int. **43**, 912–917 (2010)
4. Long, M., Rack, H.J.: Titanium alloys in total joint replacement: a materials science perspective. Biomaterials **19**, 1621–1639 (1998)
5. Rack, H.J., Qazi, J.I.: Titanium alloys for biomedical applications. Mater. Sci. Eng. C. **26**, 1269–1277 (2006)
6. Yetim, A.F., Celik, A., Alsaran, A.: Improving tribological properties of Ti6Al4V alloy with duplex surface treatment. Surf. Coatings. Technol. **205**, 320–324 (2010)
7. Liu, X., Chu, P.K., Ding, C.: Surface modification of titanium, titanium alloys, and related materials for biomedical applications. Mater. Sci. Eng. R. **47**, 49–121 (2004)
8. Li, J.L., Sun, M.G., Ma, X.X.: Structural characterization of titanium oxide layers prepared by plasma based ion implantation with oxygen on Ti6Al4V alloy. Appl. Surf. Sci. **252**, 7503–7508 (2006)
9. Nie, X., Leyland, A., Matthews, A.: Deposition of layered bioceramic hydroxyapatite/TiO_2 coatings on titanium alloys using a hybrid technique of micro-arc oxidation and electrophoresis. Surf. Coatings. Technol. **125**, 407–414 (2000)
10. Thorwarth, G., Hammerl, C., Kuhn, M., Assmann, W., Schey, B., Stritzker, B.: Investigation of DLC synthesized by plasma immersion ion implantation and deposition. Surf. Coatings. Technol. **193**, 206–212 (2005)
11. Cassar, G., Banfield, S., Wilson, J.A., Housden, J., Matthews, A., Leyland, A.: Tribological properties of duplex plasma oxidised, nitrided and PVD coated Ti–6Al–4V. Surf. Coatings. Technol. **206**, 395–404 (2011)
12. Cassar, G., Wilson, J.A., Banfield, S., Housden, J., Matthews, A., Leyland, A.: Surface modification of Ti–6Al–4V alloys using triode plasma oxidation treatments. Surf. Coatings Technol. **206**, 4553–4561 (2012)
13. Sharma, M.D., Sehgal, R.: Experimental study of friction and wear characteristics of titanium alloy (Ti–6Al–4V) under lubricated sliding condition. Ind. Lubr. Tribol. **66**, 174–183 (2014)
14. Wang, Y.M., Lei, T.Q., Guo, L.X., Jiang, B.L.: Fretting wear behaviour of microarc oxidation coatings formed on titanium alloy against steel in unlubrication and oil lubrication. Appl. Surf. Sci. **252**, 8113–8120 (2006)
15. Yoshii, Y., Hattori, H.: Sliding friction characteristics of MACs grease under vacuum condition. Tribol. Online. **1**, 14–18 (2006)
16. Peterangelo, S., Gschwender, L.J., Snyder, C.E., Jones, W.R., Nguyen, Q., Jansen, M.J.: Improved additives for multiply alkylated cyclopentane-based lubricants. J. Synth. Lubr. **20**, 31–41 (2008)
17. Lugt, P.M.: A review on grease lubrication in rolling bearings. Tribol. T. **52**, 470–480 (2009)
18. Lugt, P.M.: Modern advancements in lubricating grease technology. Tribo. Int. **97**, 467–477 (2016)
19. Jones, W.R., Shogrin, B.A., Jansen, M.J.: Research on liquid lubricants for space mechanisms. J. Synth. Lubr. **17**, 109–122 (2000)
20. Qi, J., Liu, H., Luo, Y., Zhang, D., Wang, Y.: Influences of added sand–dust particles on the tribological performance of graphite-like coating under solid–liquid lubrication. Tribol. Int. **71**, 69–81 (2014)
21. Gschwender, L.J., Snyder, C.E., Massey, M., Peterangelo, S.: Improved liquid/grease lubricants for space mechanisms. Lubr. Eng. **56**, 26–31 (1998)
22. Leong, J.Y., Satyanarayana, N., Sinha, S.K.: A tribological study of multiply-alkylated cyclopentanes and perfluoropolyether lubricants for application to Si-MEMS devices. Tribol. Lett. **50**, 195–206 (2013)
23. Fan, X.Q., Li, W., Li, H., Zhu, M.H., Xia, Y.Q., Wang, J.J.: Probing the effect of thickener on tribological properties of lubricating greases. Tribol. Int. **118**, 128–139 (2018)
24. Zhuang, W.H., Fan, X.Q., Li, W., Li, H., Zhang, L., Peng, J.F., Cai, Z.B., Mo, J.L., Zhang, G.A., Zhu, M.H.: Comparing space adaptability of diamond-like carbon and molybdenum disulfide films toward synergistic lubrication. Carbon **134**, 163–173 (2018)
25. Zhou, Z.R., Nakazaw, K., Zhu, M.H., Maruyama, N., Kapsa, P., Vincent, L.: Progress in fretting maps. Tribol. Int. **39**, 1068–1073 (2006)
26. Vingsbo, O., Soderberg, S.: On fretting maps. Wear **126**, 131–147 (1988)
27. Kalin, M.: Influence of flash temperatures on the tribological behaviour in lowspeed sliding: a review. Mater. Sci. Eng. A. **374**, 390–397 (2004)
28. Zhang, P., Lu, W.L., Liu, X.J., Jiang, X.Q.: A comparative study on torsional fretting and torsional sliding wear of CuNiAl under different lubricated conditions. Tribol. Int. **117**, 78–86 (2018)
29. Cui, G.J., Bi, Q.L., Zhu, S.Y., Yang, J., Liu, W.M.: Tribological behavior of Cu–6Sn–6Zn–3Pb under sea water, distilled water and dry–sliding conditions. Tribol. Int. **55**, 126–134 (2012)
30. Jia, J.H., Chen, J.M., Zhou, H.D., Hu, L.T.: Comparative study on tribological behaviors of polyetheretherketone composite reinforced with carbon fiber and polytetrafluoroethylene under water-lubricated and dry-sliding against stainless steel. Tribol. Lett. **17**, 231–238 (2004)
31. Fan, X.Q., Wang, L.P.: High-performance lubricant additives based on modified graphene oxide by ionic liquids. J. Colloid Interface Sci. **452**, 98–108 (2015)
32. Yang, Y., Zhang, C.H., Wang, Y., Dai, Y.J., Luo, J.B.: Friction and wear performance of titanium alloy against tungsten carbide lubricated with phosphate ester. Tribol. Int. **95**, 27–34 (2016)
33. Luo, Y., Ge, S.: Fretting wear behavior of nitrogen ion implanted titanium alloys in bovine serum lubrication. Tribol. Int. **42**, 1373–1379 (2009)
34. Cai, M.R., Liang, Y.M., Zhou, F., Liu, W.M.: Tribological properties of novel imidazolium ionic liquids bearing benzotriazole group as the antiwear/anticorrosion additive in poly (ethylene glycol) and polyurea grease for steel/steel contacts. ACS. Appl. Mater. Interfaces. **3**, 4580–4592 (2011)
35. Ji, X.B., Chen, Y.X., Zhao, G.Q., Wang, X.B., Liu, W.M.: Tribological properties of $CaCO_3$ nanoparticles as an additive in lithium grease. Tribol. Lett. **41**, 113–119 (2010)
36. NIST X-ray Photoelectron Spectroscopy Database, National Institute of Standards and Technology: Gaithersburg. http://srdata.nist.gov/xps/
37. Phillips, B.S., John, G., Zabinski, J.S.: Surface chemistry of fluorine containing ionic liquids on steel substrates at elevated temperature using Mössbauer spectroscopy. Tribol. Lett. **26**, 85–91 (2007)
38. Sanders, J.H., Cutler, J.N., John, G.: Characterization of surface layers on M-50 steel exposed to perfluoropolyalkyethers at elevated temperatures. Appl. Surf. Sci. **135**, 169–177 (1998)