# Effect of reinforcing submicron SiC particles on the wear of electrolytic NiP coatings Part 2: Bi-directional sliding

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## Abstract

As-plated and heat-treated electrodeposited NiP and composite NiP–SiC coatings were investigated in bi-directional ball-on-disc sliding tests. All tests were performed under gross slip conditions. Heat treatment decreases the wear volume loss during fretting in ambient air for all coatings investigated. Heat-treated NiP coating has a lower wear volume loss compared to composite NiP–SiC coatings for all sliding tests. The wear rate at the bi-directional sliding test was found to be lower relative to the wear rate at uni-directional sliding test.

**Keywords:** Composite coatings; Fretting; Friction; Wear; Oxides; Heat treatment



## 1. Introduction

Fretting fatigue is a phenomenon that can occur wherever vibrating machinery comes in contact and at least one of the components is subjected to bulk loading. It often results in damage that may lead to premature component failure. The knowledge of the behaviour under fretting of the materials used is of great interest to engine manufactures. The materials selection approach can help, but it is often constrained by other design requirements, so that resort is often made to the use of coatings. Reliable coatings can now be applied in a wide range of engineering situations, and the fretting wear behaviour of a number of systems has received attention in the literature [1], [2], [3], [4], [5] and [6], with some encouraging results [7], [8] and [9].

Zhang and Xue [7] found for electrodeposited CuNi multilayer that sublayers thickness < 60 nm greatly improved the fretting wear resistance. Husheng et al. [8] investigated amorphous NiP coatings. They revealed that amorphous NiP coatings remarkably increase the sliding wear resistance of chilled cast iron and the fretting fatigue resistance of low strength steel, but decrease the fretting fatigue resistance of high strength steel. Garcia and co-workers [9] reported that composite nickel coating containing approximately 5 vol.% of 0.3-μm SiC particles decreases the volumetric wear loss in bi-directional sliding tests. Consequently, for the best fretting wear resistance the nickel coatings are using [7], [8] and [9].

In the previous investigations (Part 1: Uni-directional sliding), the friction and wear behaviour of NiP and NiP–SiC coatings were shown to depend on the presence of reinforcing $Ni_3P$ and SiC particles in the coatings. Submicron SiC particles are embedded in the metal matrix during the electrodeposition of the coatings and thus the electroplating parameters determine their content. On the contrary, the $Ni_3P$ particles result from a precipitation reaction during a subsequent heat treatment of the coatings and their amount depends on the phosphorus incorporation during electroplating but also on the heat treatment parameters used. The best uni-directional sliding wear resistance against corundum balls was obtained with heat-treated particle-free NiP coatings. The electrodeposition and heat treatment of composite NiP–SiC coatings need further optimization in order to limit the crack sensitivity that results in a pull out of embedded SiC particles contributing to an enlarged abrasive wear.

This paper reports on an investigation of effect of reinforcing submicron SiC particles with size about 600 nm on the fretting wear behaviour of electrolytic NiP composite coatings. The tribological performance of the composite coatings was characterized by wear volume loss, the coefficient of friction and wear mechanism.

## 2. Experimental

NiP coatings were deposited on steel substrates from an electrolytic plating bath containing a suspension of 0, 80 or 200 g/l SiC particles with a mean diameter of 600 nm. The plating solution contained 20 g/l $H_3PO_3$. Half of the samples was heat-treated at 420 °C for 1 h in atmosphere.

Bi-directional sliding ball-on-disc wear tests were performed at a normal load of 1, 5 and 10 N, an oscillation frequency of 2 and 10 Hz, and a tangential displacement amplitude of 100 and 500 μm. The number of fretting cycles was 20 000. All the wear tests were performed against 10-mm diameter corundum balls, without lubrication, at 22 °C, and in ambient air of 50% relative humidity. The coefficient of friction was recorded continuously during the wear tests.

The surface morphology of coatings and wear tracks were examined by scanning electron microscopy (SEM-Philips 515). Chemical analysis was carried out by electron dispersive X-ray analysis (EDX) at an acceleration voltage of 10 and 20 kV. Worn samples were investigated by SEM before and after ultrasonic cleaning for 15 min in ethanol.

The volumetric material loss after the wear tests and the surface roughness were determined by white light interferometer (Wyko NT Series, Optical 3D Profiling Systems). Hereto, samples were cleaned in ethanol under ultrasonic agitation for 15 min.

## 3. Results and discussion

The microstructure of as-deposited and annealed NiP composite coatings was presented in Part 1. The as-deposited state of NiP–SiC coatings consist of dark submicron SiC particles embedded into a homogeneous nodular NiP surface of grey colour. The surface of NiP coating is very smooth with typical metallic lustre.

The mean friction values of NiP and NiP–SiC coatings in uni-directional sliding tests are almost not affected by the presence of SiC and $Ni_3P$ particles. However, typical variations in the coefficient of friction during uni-directional sliding tests revealed that different wear processes were present. In the case of NiP–SiC coatings, the oxidation of NiP matrix is apparently hindered by SiC particles as compared to particle-free NiP coatings where abrasive wear was promoted. That increased abrasion wear can result from hard SiC particles embedded in the metal matrix or on pull-out, followed by an entrapment in the contact area or a transfer of these particles to the counter body. This competition between oxidational and abrasive wear was noticed in the as-plated conditions as well as in heat-treated conditions for composite NiP–SiC coatings. To some extent, such a competition between oxidational and abrasive wear was also noticed on as-plated and heat-treated particle-free NiP coatings. A major difference was noticed, namely that the oxidation film formed in the wear track area on NiP under uni-directional sliding adheres strongly to the coating protecting it from a high wear rate.

The wear track after bi-directional sliding test with loads 5 and 10 N and amplitude 500 μm shows tribo-oxidized layer in the all as-plated coatings. Oxidational wear process is characterized by a worn surface with oxidized film. Products of wear consist of oxides. Fig. 1 shows the wear track after oxidational wear for NiP–SiC coating with concentration of SiC particles 80 g/l. EDX analyses showed the presence of O and Al in the wear track of NiP–SiC coatings (Fig. 2). This confirms the transfer of material from the corundum counter body. In NiP coatings, during sliding, oxide films formed rapidly even at low load. The formation of oxide films on the wear track is not depends on the presence of reinforcing $Ni_3P$ and SiC particles in the coatings.

Fig. 1. Microstructure of the wear tracks on as-plated NiP–SiC coatings (600 nm 80 g/l). Bi-directional wear test parameters: corundum ball as counter body, 10 N; 2 Hz, 20 000 fretting contacts, ambient air 50% RH and 23 °C.

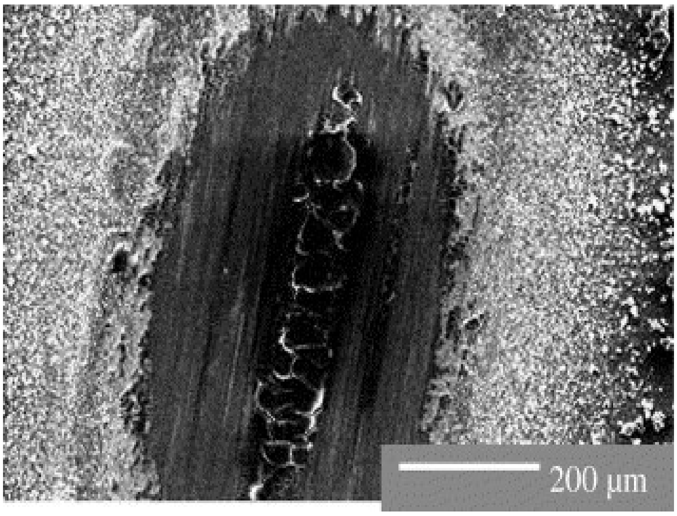
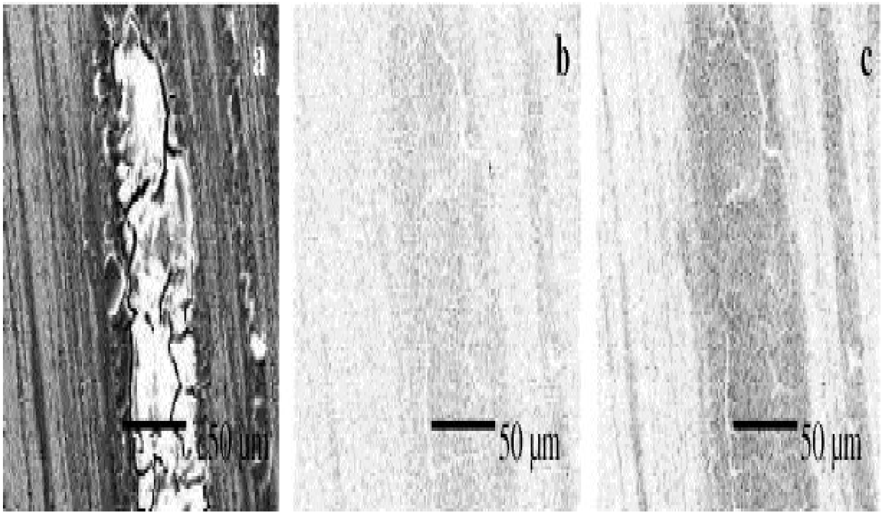


Fig. 2. Back scattered image and elemental mapping of the wear track in as-plated NiP–SiC coating, 600 nm 80 g/l: (a) backscattered image at the centre of wear track after fretting: 10 N, 2 Hz, 500 μm, 20 000 cycles; (b) elemental mapping of aluminum; (c) elemental mapping of oxygen.




The microstructure of the wear track of the heat-treated coating represents in Fig. 3, Fig. 4, Fig. 5, Fig. 6 and Fig. 7. The wear track after bi-directional sliding test with 10 N load and 500 μm amplitude shows that oxide layer forms in the NiP composite coatings in a small part because of high hardness of the surface after heat treatment (Fig. 4). These oxide films are very thin and transparent and show worn surface with removable SiC particles on the NiP–SiC coatings (Fig. 5). The wear track with 5 and 10 N loads shows cracks perpendicular to direction of the motion (Fig. 6). In the rim of the wear track, the oxide layer was noticed (Fig. 7).

Fig. 3. Microstructure of the wear tracks on heat-treated composite NiP–SiC coatings (600 nm 80 g/l). Bi-directional wear test parameters: corundum ball as counter body, 10 N; 2 Hz, 20 000 fretting contacts, ambient air 50% RH and 23 °C.

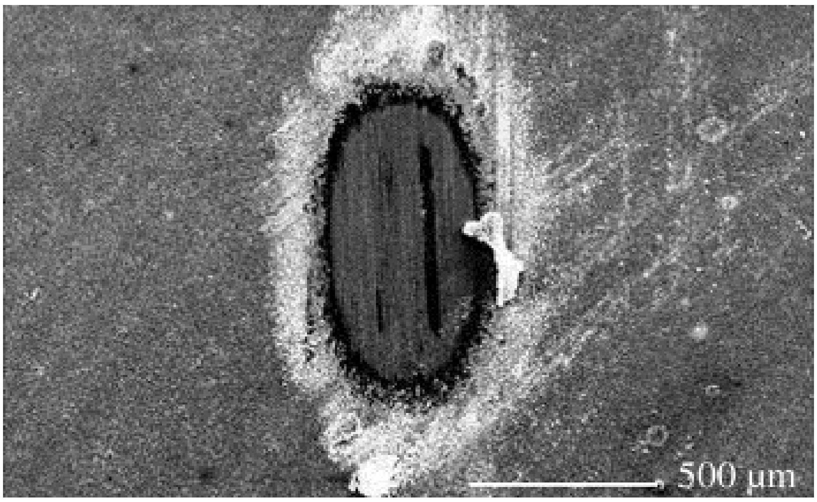


Fig. 4. Detail of Fig. 3 around black zone.

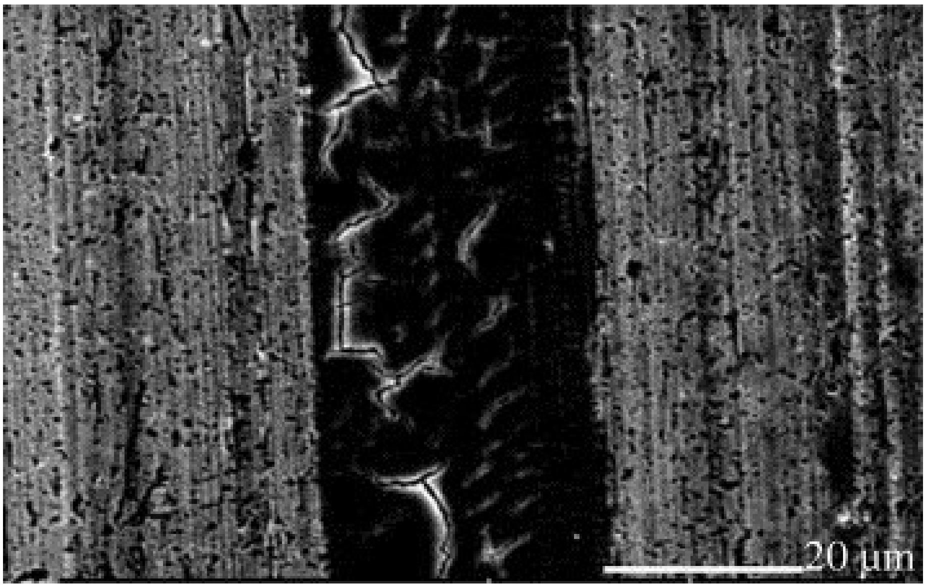



Fig. 5. Detail of Fig. 4 (a) wear track outside black zone and (b) wear track in black zone.

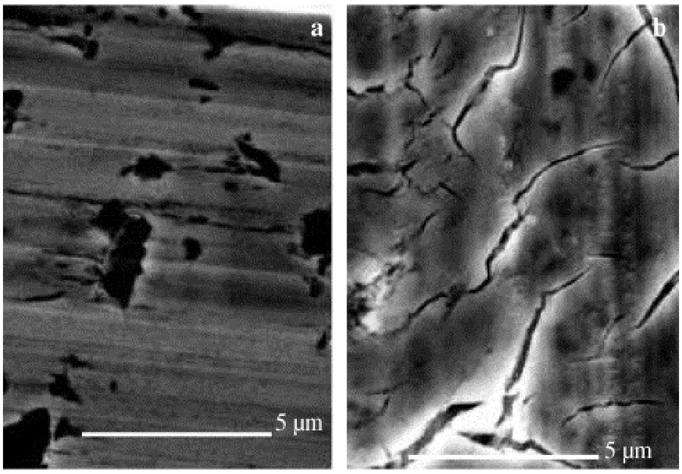


Fig. 6. Detail of Fig. 3 microcracks in the wear track near the rim.

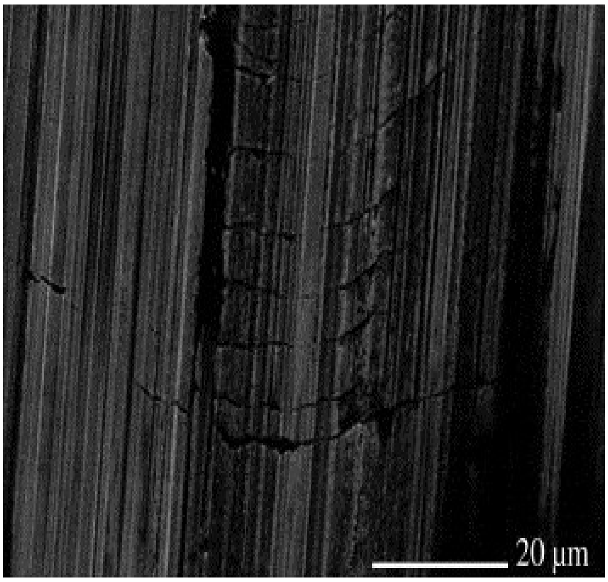
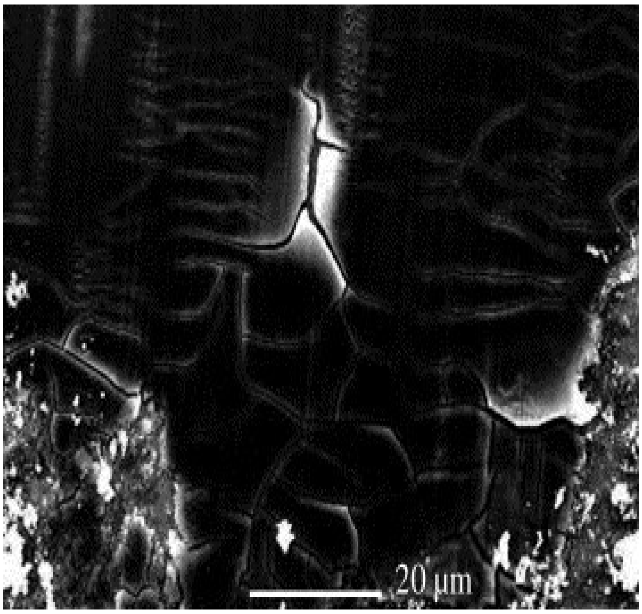



Fig. 7. Detail of the rim of the wear track in Fig. 3.




Compared with uni-directional sliding test, the formation of the oxide films in the wear track after bi-directional sliding test was noticed for all as-plated NiP composite coatings. At the uni-directional sliding test, debris and wear particles were pulled out from the wear track promoting the abrasive wear. Abrasive particles have various forms and are oriented in different directions relative to the connected surface. Ability for abrasive grain to be pressed into a surface depends from a ratio of their hardness and from the geometrical form of a grain also. For example, a cured surface or a sharp rib of the grain can be pressed without damages into a flat surface of firmer body. If between sliding surfaces there are a lot of abrasive grains one part of grains fix in sliding surfaces, another is rolled between surfaces and moving off from a contact zone settles on periphery of wear track. At the bi-directional sliding test, wear particles are not removing immediately. Wear particles are pressed by normal load to the wear track, then they melt and formed the oxide films. Consequently, the mechanism of wear at bi-directional sliding test is oxidational.

The compositional analysis of the wear track of the coatings after fretting is given in Table 1. The chemical compound of the rim in the wear track in nickel, phosphorus, oxygen and aluminum was found to be identical for all coatings investigated. Metals included in tribo-elements have various affinities to oxygen. This circumstance and also different speed of diffusion of metals in the oxidational film could cause strong segregation of metal atoms in oxidational film. During oxidation of a sliding surface, enrichment of the wear track by aluminium was noticed in the as-plated coatings.

Table 1.

EDX compositional analyses of wear tracks on as-plated and heat treated NiP and composite NiP–SiC coatings, and on debris formed during bi-directional sliding tests

| **Coatings investigated** | | **EDX analyses after bi-directional sliding tests at 10 N and 500 μm, 20 000 cycles, 2 Hz in ambient air of 50% RH and 23 °C** | | | | | |
|---|---|---|---|---|---|---|---|
| | | **Elements (wt.%)** | | | | | |
| | | **Ni** | **P** | **Si** | **C** | **O** | **Al** |
| NiP–SiC (bath 80 g/l SiC) | As-plated | 57.07 | 7.24 | 2.53 | 3.76 | 27.12 | 2.29 |
| | Heat treated | 80.34 | 10.46 | 1.32 | 4.66 | 3.22 | – |
| | Rim | 59.91 | 8.10 | 1.05 | 6.08 | 22.62 | 2.25 |
| | Debris | 51.60 | 6.89 | 0.99 | 7.15 | 30.21 | 3.16 |
| NiP–SiC (bath 200 g/l SiC) | As-plated | 59.74 | 7.90 | 3.86 | 4.42 | 21.95 | 2.13 |
| | Heat-treated | 70.52 | 9.27 | 4.53 | 5.63 | 9.09 | 0.96 |
| | Rim | 50.44 | 6.83 | 3.59 | 6.11 | 30.47 | 2.56 |
| | Debris | 42.17 | 5.36 | 3.69 | 7.38 | 37.87 | 3.52 |
| NiP | As-plated | 80.75 | 8.84 | – | – | 9.12 | 1.30 |
| | Heat-treated | 85.17 | 11.21 | – | – | 3.62 | – |
| | Rim | 62.0 | 8.96 | – | – | 26.2 | 2.76 |

| Coatings investigated | | EDX analyses after bi-directional sliding tests at 10 N and 500 µm, 20 000 cycles, 2 Hz in ambient air of 50% RH and 23 °C | | | | | |
|---|---|---|---|---|---|---|---|
| | | Elements (wt.%) | | | | | |
| | | Ni | P | Si | C | O | Al |
| | | 5 | | | | 3 | |
| | Debris | 53.73 | 8.20 | – | – | 33.23 | 4.84 |

The microstructure of the debris outside the wear tracks was the same for all coatings investigated (Fig. 8). Once formed during fretting process, the debris react with ambient atmosphere, oxidize and represent melting oxide films, whereas debris after uni-directional sliding test represent separate particles around the wear track. The chemical composition of the debris in oxygen, aluminum, nickel and phosphorus was found to be identical for all coatings investigated (Table 1). The same observation of debris was noticed on laser alloying of aluminium alloy with Ni and Cr.

Fig. 8. Microstructure of debris formed during bi-directional sliding test performed at 10 N, 2 Hz in ambient air 50% RH and 23 °C for 20 000 fretting cycles on as-plated composite NiP–SiC (600 nm, 80 g/l).

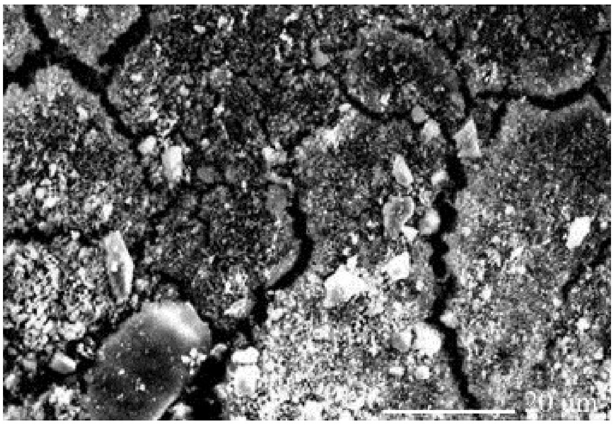


Fig. 9, Fig. 10, Fig. 11 and Fig. 12 show the evolution of the friction coefficient of NiP composite coatings when fretted against the corundum counter body in ambient air and relative humidity at different displacement amplitudes. In all the coatings, at the displacement amplitude of 100 µm, coefficient of friction was found to be 0.45 for as-plated coatings and 0.6 for heat-treated coatings. At the displacement amplitude of 500 µm, the coefficient of friction was about 0.4 for all coatings investigated. In all the coatings, at the displacement amplitude of 100 or 500 µm, coefficient of friction was found to be not varying with normal load. With the decrease of frequency from 10 to 1 Hz at the displacement amplitude of 500 µm, the protective layer – oxide films – are formed.

Fig. 9. Coefficient of friction after fretting on as-plated NiP and composite NiP–SiC coatings (100 µm, 10 Hz, 1 N, 20 000 cycles).

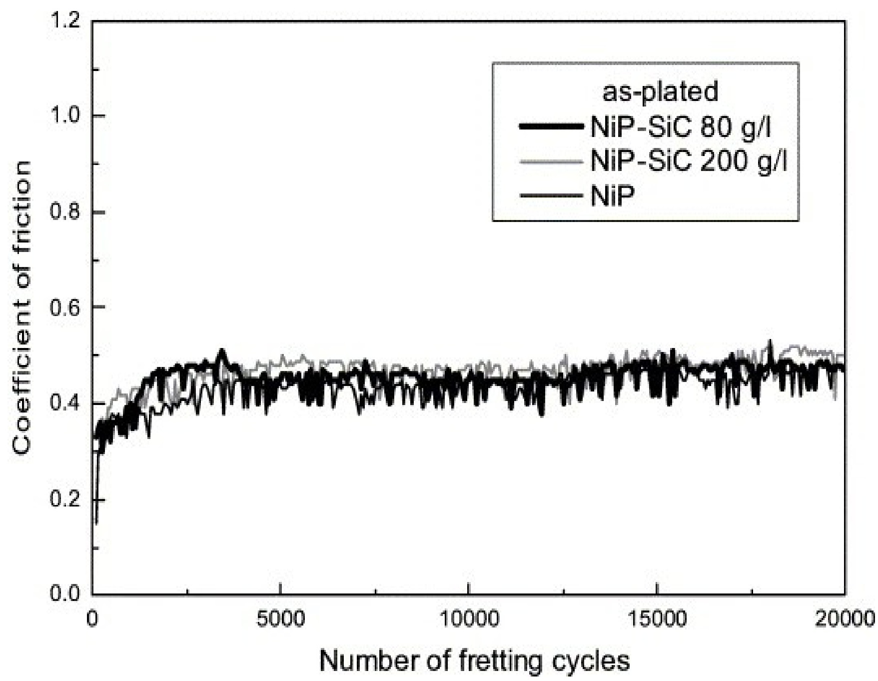


Fig. 10. Coefficient of friction after fretting on heat-treated NiP and NiP–SiC coatings (100 µm, 10 Hz, 1 N, 20 000 cycles).

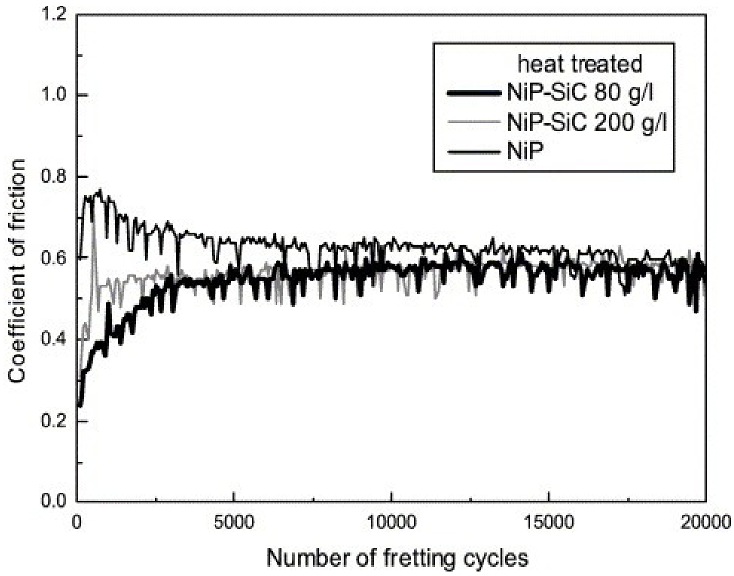



Fig. 11. Coefficient of friction after fretting on as-plated NiP and composite NiP–SiC coatings (500 µm, 2 Hz, 10 N, 20 000 cycles).

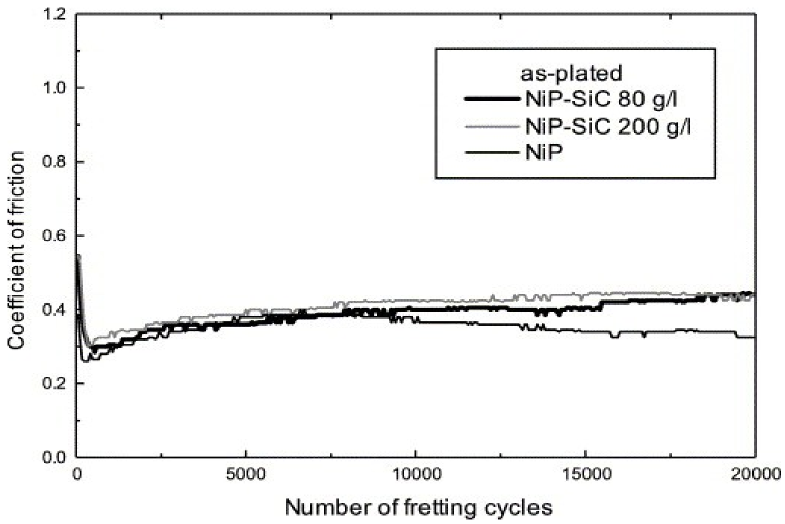


Fig. 12. Coefficient of friction after fretting on heat-treated NiP and NiP–SiC coatings (500 µm, 2 Hz, 10 N, 20 000 cycles).

At the uni-directional sliding test, the coefficient of friction in NiP composite coatings was found to be higher than the coefficient of friction at the bi-directional sliding test. At the uni-directional sliding test, the coefficient of friction in NiP composite coatings was found to be the same (0.7) for all coatings investigated, whereas at the bi-directional sliding test, the coefficient of friction in NiP composite coatings was found to be dependent on the displacement amplitude and formation of oxide film during fretting.

The comparison of the wear rate of uni- and bi-directional sliding test has some difficulties because of different schema of sliding tests and different parameters were used. For comparison of the wear rate at uni- and bi-directional sliding test, the value of the wear rate was accounted in relation between wear volume loss and product of normal load and sliding distance. The results are present in Fig. 13, Fig. 14 and Fig. 15. The slope at the bi-directional sliding test is significantly higher than the slope at uni-directional sliding test. The wear rate at the bi-directional sliding test was found to be lower than the wear rate at the uni-directional sliding test. This is because the wear particles were not removed from the wear track.

Fig. 13. Wear rate after uni- and bi-directional (40 000 fretting contacts, ambient air 50% RH and 23 °C) sliding tests vs. Vickers hardness for as-plated and heat treated composite NiP–SiC coatings with concentration of SiC particles 80 g/l. Dotted line correspond to data for uni-directional sliding test (15 000 contacts, 2 N, 0.15 m/s, 1 Hz, ambient air 50% RH and 23 °C).

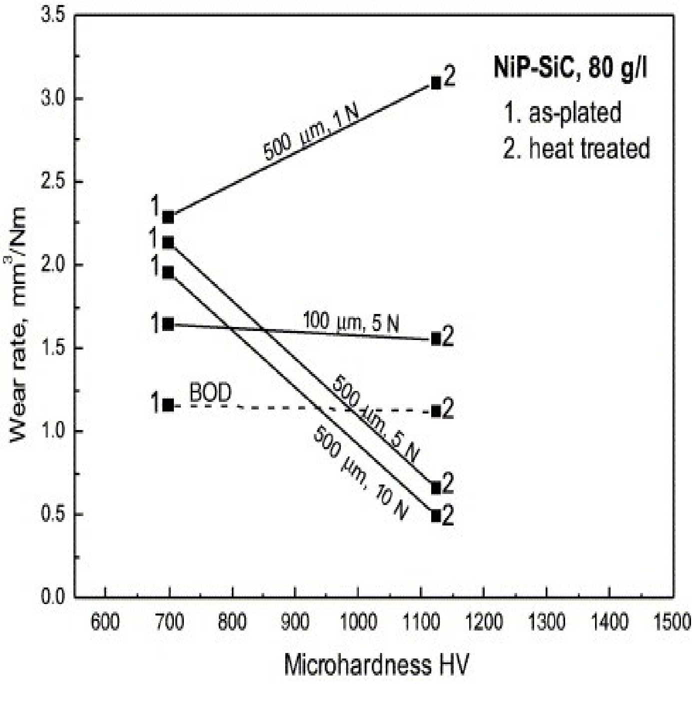


Fig. 14. Wear rate after uni- and bi-directional (40 000 fretting contacts, ambient air 50% RH and 23 °C) sliding tests versus Vickers hardness for as-plated and heat treated composite NiP–SiC coatings with concentration of SiC particles 200 g/l. Dotted line correspond to data for uni-directional sliding test (15 000 contacts, 2 N, 0.15 m/s, 1 Hz, ambient air 50% RH and 23 °C).

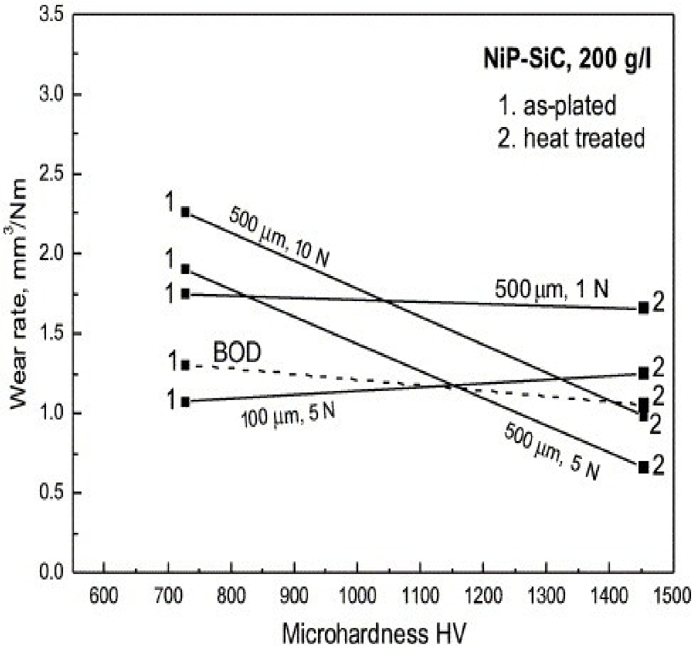
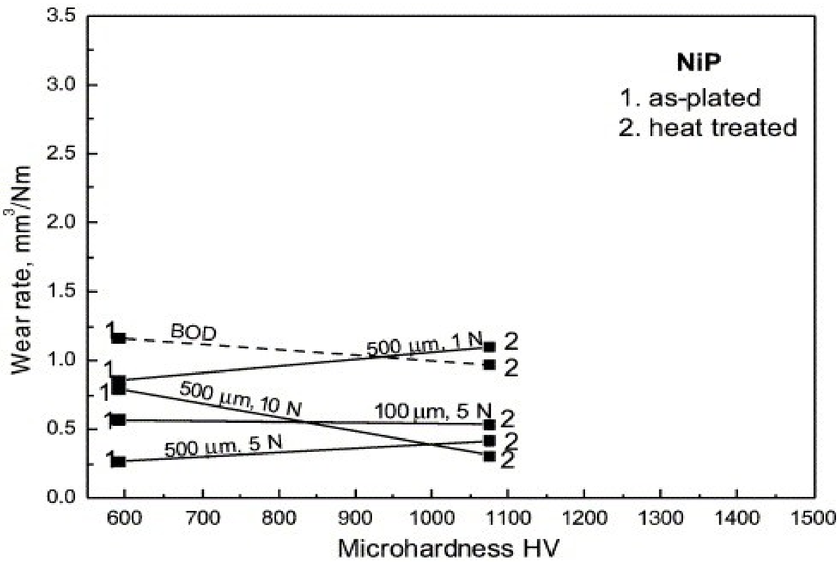



Fig. 15. Wear rate after uni- and bi-directional (40 000 fretting contacts, ambient air 50% RH and 23 °C) sliding tests vs. Vickers hardness for as-plated and heat treated pure NiP coatings. Dotted line correspond to data for uni-directional sliding test (15 000 contacts, 2 N, 0.15 m/s, 1 Hz, ambient air 50% RH and 23 °C).



Finally, for confirmation of these results, the value of the wear rate as a relation between the wear depth and the product of normal load and number of contact was accounted. The wear depth is shown in Fig. 16 and Fig. 17 for uni- and bi-directional sliding tests, respectively. The results obtained by this equation are in accordance with previous results. For instance, for the uni-directional sliding test in NiP–SiC coatings with SiC particle concentration of 200 g/l, this wear rate was $6.67 \times 10^{-9}$, whereas for bi-directional sliding test, the wear rate was lesser $4.37 \times 10^{-9}$.

Fig. 16. X-profile of the wear track of heat-treated NiP–SiC, 200 g/l at the uni-directional sliding test (15 000 contacts, 2 N, 0.1 m/s, 1 Hz, ambient air 50% RH and 23 °C).

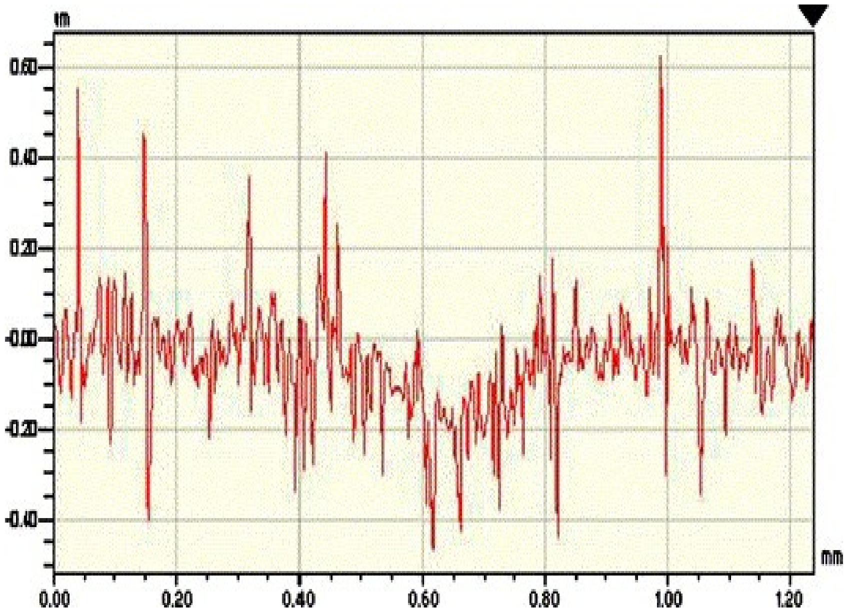
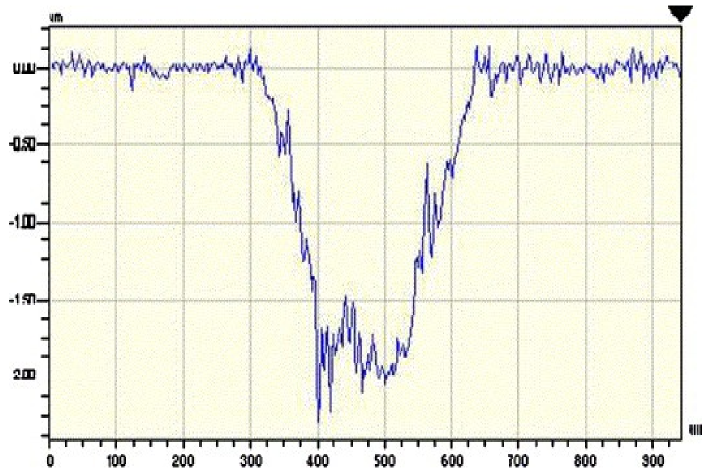


Fig. 17. X-profile of the wear track of heat-treated NiP–SiC, 200 g/l at the bi-directional sliding test (40 000 fretting contacts, 10 N, 2 Hz, ambient air 50% RH and 23 °C).



# 4. Conclusions

The wear properties of NiP–SiC composite coatings were shown to depend on the type of sliding test. At the uni-directional sliding test, abrasive wear was noticed, whereas at the bi-directional sliding test, oxidational wear was revealed.

The mean friction values of NiP and composite NiP–SiC coatings in bi-directional sliding tests are almost not affected by the presence of reinforcing SiC particles. At the uni-directional sliding test, the friction coefficient of NiP composite coatings was found to be higher than the coefficient of friction at the bi-directional sliding test. In comparison with uni-directional sliding test, formation of the oxide films in the wear track after bi-directional sliding test was noticed for as-plated NiP composite coatings. At the uni-directional sliding test, debris and wear particles remove from the wear track and mechanism of wear in this case is abrasive. At the bi-directional sliding test, once formed, the debris are trapped in the contact area, which completely modifies the interaction between the two rubbing bodies. These debris react with the ambient air, oxidize and compact into thin layer in the contact region and around the wear track. The presence of $Ni_3P$ particles in heat-treated NiP composite coatings prevents the formation of oxide films in the wear track. However, around the wear track, the oxide layer of the debris was noticed.

Heat treatment decreases the wear volume loss during fretting in ambient air for all coatings investigated. Heat-treated NiP coating has a lower wear volume loss in comparison with other coatings for all sliding tests. During fretting at the displacement amplitude of 100 µm, heat treatment has not influenced the wear volume loss, whereas at the displacement amplitude of 500 µm, heat treatment decreased the wear volume loss, twofold in NiP and threefold in NiP–SiC coatings.

The best bi-directional sliding wear resistance against corundum balls was obtained in this study with heat-treated pure NiP coatings. The wear rate at the bi-directional sliding test was found to be lesser than the wear rate at the uni-directional sliding test. This is because the wear particles are not removed from the wear track.

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