

Tribological properties of spherical and mesoporous NiAl particles as ionic liquid additives

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Abstract: In this study, spherical and mesoporous NiAl particles (abbreviated as sNiAl and mNiAl) were introduced as lubricant additives into two alkyl-imidazolium ionic liquids (ILs) (1-butyl-3-methylimidazolium tetrafluoroborate (LB104) and 1-butyl-3-methyl imidazolium hexafluorophosphate (LP104)) to explore their tribological properties. The sNiAl and mNiAl particles were modified in-situ by anion and cation moieties of ILs through chemical interaction, thereby enhancing their dispersibility and stability in ILs. The mNiAl particles have better dispersibility than the sNiAl ones in ILs because of high specific surface area. LP104-modified sNiAl particles show better friction reduction and wear resistance, mainly relying on the synergy of the hybrid lubricant. These particles form a protective layer that prevents friction pairs from straight asperity contact and improves the tribological behaviors.

Keywords: ionic liquids; alloy particles; spherical and mesoporous structure; tribological properties

1 Introduction

With the ever-increasing performance requirements of moveable mechanical components for long-term reliability, accuracy, and environmental friendliness, it is of great significance to look for high-performance green lubricants for resolving the friction and wear issues [1, 2]. Although intensive efforts have been made to synthesize new liquid lubricants including refined mineral oils, silicone oils, polyalphaolephines, perfluoropolyethers, etc. [3, 4], and evaluate their tribological properties, there still exist difficulties with the requirements of modern or future technology under certain rigorous conditions. Liquid lubricants can reduce friction and wear in the running-in stage and mild test conditions by the formation of a thin oil-film on the contact interfaces [5]. With the increase of temperature and load, the oil-film can reach a

limiting thickness and be damaged. In such cases, lubricant additives or lubricants with active elements can provide friction reduction and increase wear resistance by using tribo-reaction products [6–8]. The physicochemical properties and composition of contact surfaces affect the wettability, corrosion rates, adhesive properties, and failure mechanisms [9]. Therefore, the synergy of oil-film and tribo-film surfaces by using high-performance lubricants or additives is an optimal strategy for significantly improving tribological properties.

Room-temperature ionic liquids (ILs) as high-performance lubricants began to emerge in 2001 [10]. Since then, various ILs have been synthesized by changing the anion-cation combination and their tribological behaviors have been evaluated under different environment and work conditions [11, 12]. ILs have proved to be good alternative lubricants for

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tribological problems or good candidates for traditional volatile lubricants because of their unique structure and properties including high polarity, negligible vapor pressure, thermal stability, non-flammability, high ionic conductivity, and a wide electrochemical stability window [13, 14]. More importantly, IL-films can be decomposed to form anti-wear components by the interaction of active elements with metal elements on the friction pairs under severe friction conditions [15]. These performance characteristics have attracted much attention in the field of lubrication, and previous literatures have reported the tribological behaviors of ILs for the contact of steel/steel [16, 17], steel/aluminium [18, 19], steel/copper, and steel/modified surfaces [20–23], ensuring that ILs have a good lubrication function for various friction pairs.

Moreover, ILs with active halogen elements (such as tetrafluoroborates and hexafluorophosphates) can form anti-scratch compounds. However, the alloy matrix can be corroded by halogen elements, thereby causing a more severe wear and reduction of service life [24]. To avoid the possible corrosion in the presence of ILs, inert carbon nanomaterials were introduced as lubricating additives and corrosion inhibitors. For example, the IL gels made up of multiwall carbon nanotubes (MWCNTs) have good tribological properties and corrosion resistance because of the synergy of ILs and MWCNTs [25]. IL-modified graphene oxide (GO) and graphene with good dispersity and stability, as additives, improve the physicochemical properties of ILs and significantly reduce friction and enhance anti-wear performance [26]. When adding ILs to traditional grease, boundary-film lubrication formed by physical absorption and chemical reaction plays a decisive role in friction reduction and wear resistance [27, 28]. In addition, some metal and alloy nanoparticles, (such as oil-soluble Cu, Ni, NiO, MgB₂, ZnB₂, etc.) as lubricant additives, possess excellent friction-reducing, anti-wear, and high load carrying capacities due to the formation of a tribo-chemical reaction film [29–31]. However, the research on NiAl particles as IL additives is still pending. Given that the combination of NiAl particles with ILs forms a protective layer, it will enhance the wear and corrosion resistance.

In this study, hybrid lubricants were prepared by adding spherical and mesoporous NiAl particles (abbreviated as sNiAl and mNiAl) into 1-butyl-3-

methylimidazolium tetrafluoroborate (LB104) and 1-butyl-3-methyl imidazolium hexafluorophosphate (LP104), respectively, and their stability and tribological behaviors were investigated in detail. The structure and composition of NiAl particles before and after friction were explored by scanning electron microscopy (SEM) and X-ray photoelectron spectroscopy (XPS). Friction mechanism was explored by the microtopography and chemical composition of wear tracks and wear debris.

2 Experimental details

2.1 Materials

The sNiAl and mNiAl particles of size less than 18 μm were prepared by an atomization method and a pore forming technology, respectively [32]. The raw Ni and Al powders were first fully smelted, then impinged into metal droplets under the action of a high-speed air stream, and finally completely solidified under the surface tension of the droplets. mNiAl particles were prepared by a similar procedure with the addition of a pore former. The preparation method of LB104 and LP104 is reported in [33]. All reagents in this research were purchased from CHRON CHEMICALS (Chengdu, China), and were of analytical reagent grade.

2.2 Preparation of the lubricant

The hybrid lubricants were prepared as follows: the sNiAl and mNiAl particles with addition amount of 2.0 wt% were introduced into LB104 and LP104, respectively. Subsequently, the hybrid lubricants with good disperse stability were obtained by ultrasonic dispersion method with 50 % power for 1 h.

2.3 Characterization of the IL-modified NiAl alloy particles

While investigating the dispersity of sNiAl and mNiAl particles in ILs, it was found that the functional groups of ILs could interact with NiAl particles to form IL-modified NiAl particles. After the NiAl hybrid lubricants remained stationary for a week, some NiAl particle precipitates were collected and washed ultrasonically with ethyl acetate and ethanol to analyze their structure and chemical composition.

SEM (JSM-6610, Japan) and XPS (ESCALAB 250Xi, America) were used for investigating the microstructures and chemical components of IL-modified NiAl particles.

2.4 Tribological properties

The tribological behaviors of lubricants were evaluated on the reciprocating ball-on-disk UMT-3 multi-specimen test system at room temperature. The stationary upper sample is AISI 52100 steel ball with a diameter of 10 mm, and the lower sample is AISI 52100 steel block with 24 mm × 7.9 mm. The sliding friction experiments were operated at 2 Hz frequency, 5 mm amplitude, and applied loads of 20 N, 40 N, and 60 N, respectively. The surface of the lower sample was coated with 50 μ l of lubricants. After the sliding friction tests, the wear debris on the worn surface were collected to explore the friction and wear mechanisms.

2.5 Surface analyses

SEM, Bruker Contour GT white light interferometer, and optical microscope (OM) were used for analyzing the microstructures of the worn surfaces and wear debris; XPS was applied to investigate the worn surfaces.

3 Results and discussion

3.1 Analysis of modified NiAl alloy powders

Improvement in the tribological properties of lubricants by adding solid additives must consider their dispersity and compatibility. The designation given to IL-modified sNiAl and mNiAl particles in our experiment is shown in Table 1. Figure 1 shows the dispersity of the NiAl particles in ILs. From these images we can observe the change of dispersity and stability with increase in time. The mNiAl particles could easily disperse in the two ILs, because their sedimentation rate was relatively low. The sNiAl particles almost totally precipitated after three days, whereas the mNiAl particles precipitated completely after seven days. The mNiAl particles have better dispersity and stability than sNiAl particles in both the ILs, which could be attributed to the mesoporous structure with greater specific surface area and relatively low weight.

Table 1 IL-modified sNiAl and mNiAl particles and their designations.

Sample	LB104-mNiAl	LP104-mNiAl	LB104-sNiAl	LB104-sNiAl
Designation	A	B	C	D

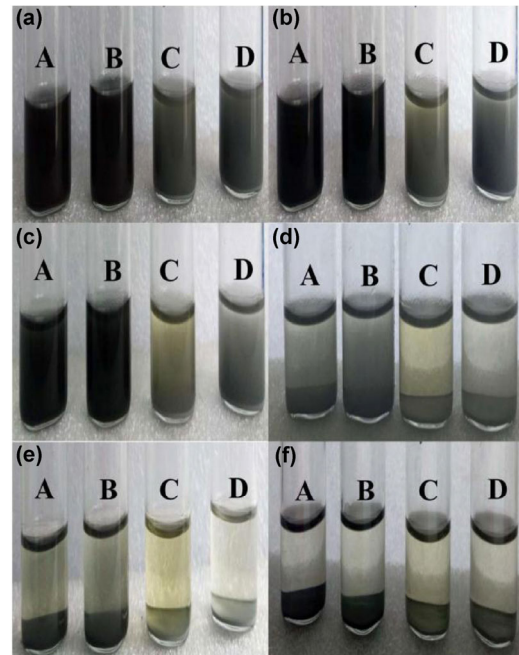


Fig. 1 Dispersity images of NiAl particles in ILs with the extension of the settling time (a) just when prepared, (b) 30 min, (c) 160 min, (d) 1 day, (e) 3 days, and (f) 7 days.

SEM is an essential method to observe the micro-morphology of a sample. Figure 2 shows the images of the original and modified NiAl particles. sNiAl particles were modified by LB104 and LP104 ILs. These modified particles presented a relatively intact cover layer, which could be due to the physical adsorption of ILs on the surface of the particles and a chemical reaction between them. However, this phenomenon was not visible in mNiAl particles possibly because ILs were absorbed into the mesoporous structure and interacted with the alloy.

To explore the modification mechanism of NiAl particles by ILs, the chemical states of typical elements were investigated by XPS. Figure 3 shows the XPS spectra of C1s, F1s, N1s, Ni2p, and Al2p of IL-modified NiAl particles. The distinct C peak at 284.8 eV is identified as C in air. The binding energy of N1s peak is at 399.5 eV, indicating the presence of C–N bonds in LB104 and LP104 [26]. The F1s peak of mNiAl particles, at 686.4 eV, is identified as F in AlF₃ [34].

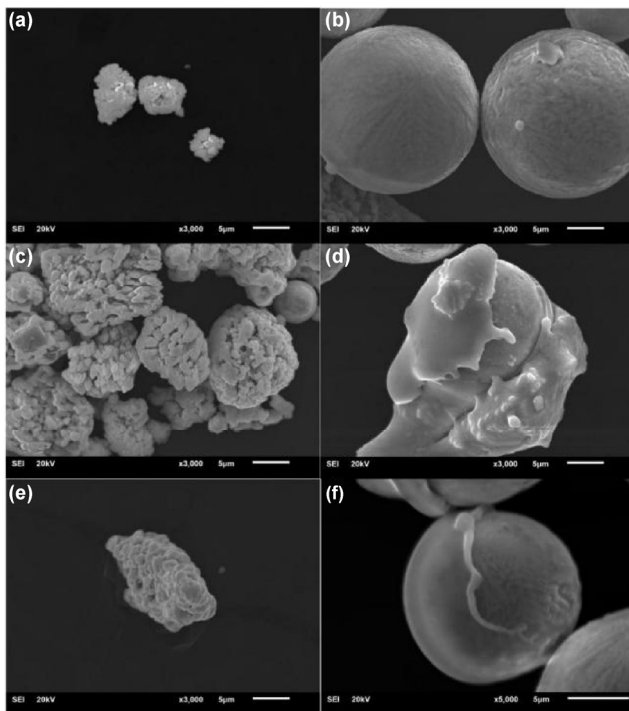


Fig. 2 SEM images of (a) mNiAl, (b) sNiAl, (c) LB104-modified mNiAl, (d) LB104-modified sNiAl, (e) LP-104-modified mNiAl, and (f) LP104-modified sNiAl particles.

F1s peak of IL-modified sNiAl powders is at 685.4 eV, indicating the formation of Ni–F bonds in NiF_2 , which can be further inferred by the Ni2p peaks [35]. Original NiAl particles generate Ni2p peak at 853.3 eV owing to the metal nickel. However, the binding energy of Ni2p shifts to 857.9 eV after being modified by ILs, ensuring that NiF_2 is formed on the surface of NiAl particles. XPS results illustrate that the chemical interaction of ILs and NiAl particles occurred and formed a chemical modification layer, which is consistent with the SEM results.

Combining the results of SEM morphology and XPS analysis, we conclude that the hybrid lubricants are comprised of ILs as base oil and IL-modified mNiAl and sNiAl particles as additives. NiAl particles were effectively modified by ILs by physical absorption and chemical reaction, making these particles useful on a large scale. The modification of metal particles by ILs is easy to achieve, and the modified particles have better dispersity in lubricants, which can improve the tribological behaviors of lubricants.

3.2 Tribological test

ILs are attractive as lubricants because of their double

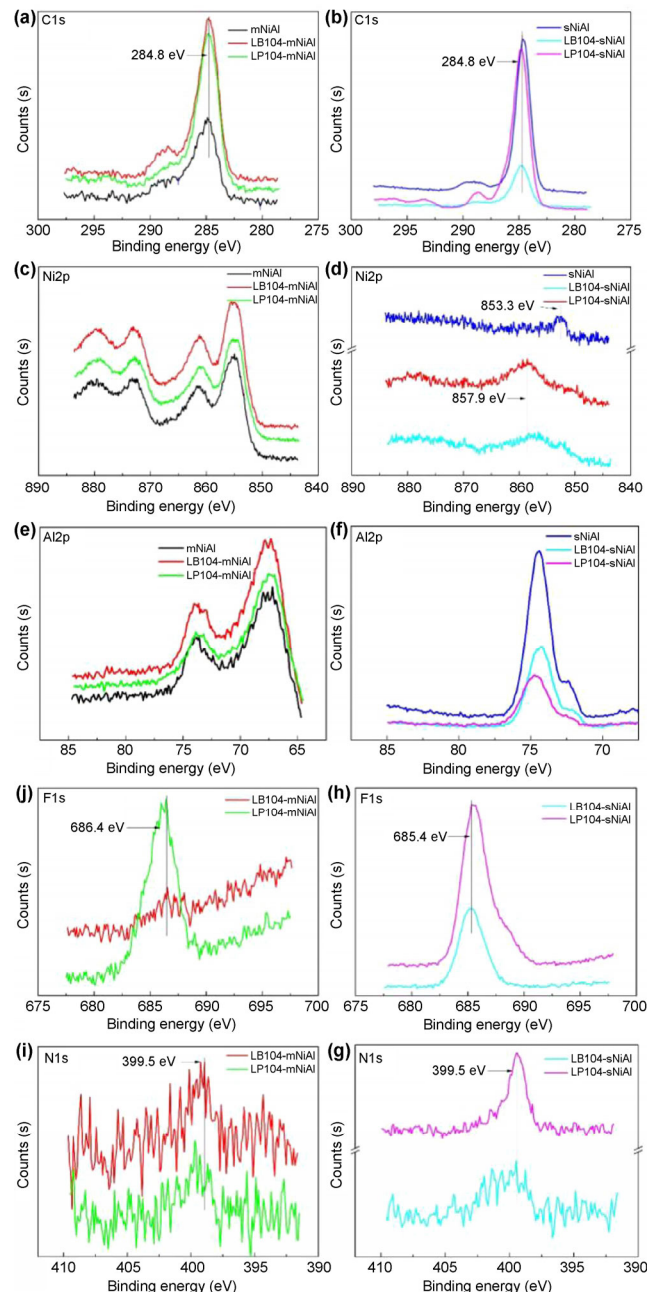


Fig. 3 XPS spectra of original and IL-modified NiAl particles.

bilayer structure, in the solid state, such as that of graphite and molybdenum disulfide [19]. To explore the tribological properties of the NiAl hybrid IL lubricants, the UMT-3 tester was operated according to the testing parameters. Figure 4 shows the sliding friction curves and average friction coefficient of the lubricants under different loads (20 N, 40 N, and 60 N). Observing the friction curves in Figs. 4(a) to 4(c), LB104-modified NiAl hybrid lubricants (including mNiAl and sNiAl particles) display the distinct

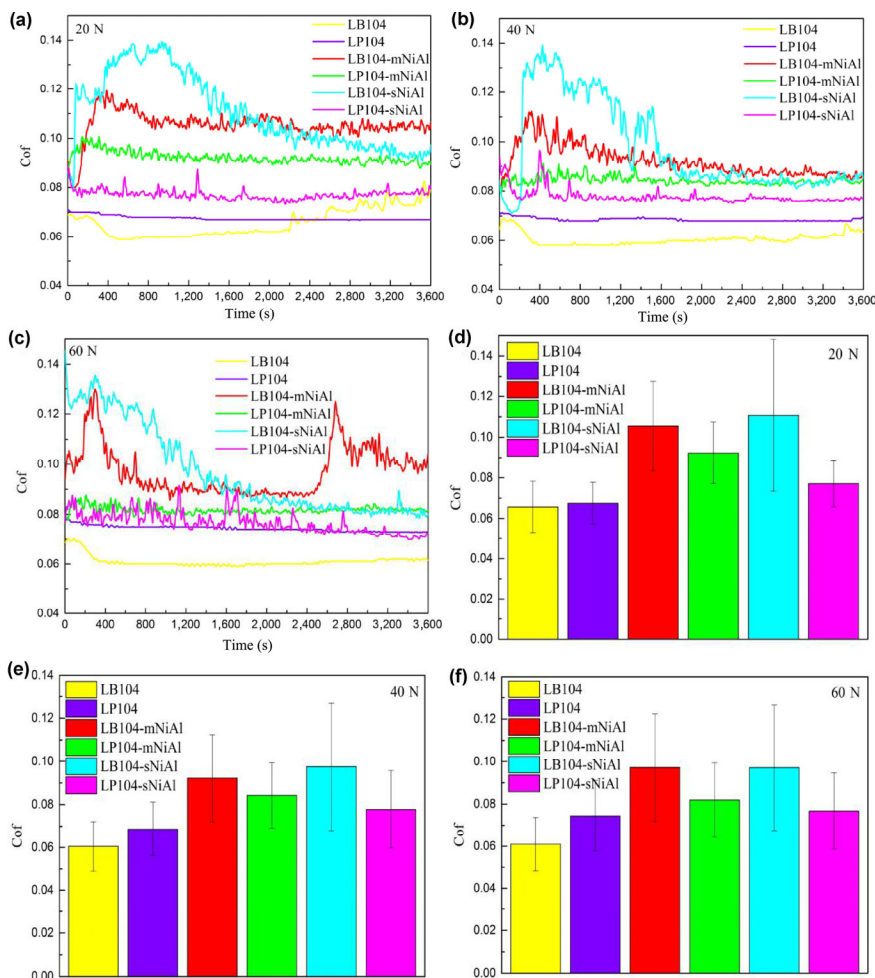


Fig. 4 Friction curves and average friction coefficient of NiAl hybrid lubricants at applied loads of 20 N, 40 N, and 60 N.

running-in stage with a high and fluctuating friction coefficient. However, LP104-modified NiAl hybrid lubricants show smoother friction curves with lower friction coefficients. Pure ILs provide stable friction curves and a low friction coefficient.

For the contact of steel/steel, the difference of tribological behaviors for these lubricants mainly stems from the chemical composition and performance features of the base oil and additives. Figure 5 shows the contact angles. The contact angle of LP104 ILs on a surface-smooth stainless-steel disk is lower than that of LB104 ILs, indicating that LP104 ILs have better wettability to steel to form an effective boundary film. Furthermore, the anion moiety of LP104 (PF_6^-) easily forms phosphates and fluoride on the wear surfaces owing to the mechanical force and friction heat. IL-film and tribochemical reaction film with good extreme pressure properties can improve the load-bearing

capacity, reduce friction, and enhance wear resistance [15, 16]. Figures 4(d) to 4(f) show that the addition of NiAl particles increases the friction coefficient of the lubricants owing to the participation of these large sized particles on the sliding interfaces. These particles can act as abrasives during the friction process and lead to abrasive wear.

Table 2 The contact angles of LB104 and LP104.

Sample	LB104	LP104
Contact angle	66.5°	56.7°

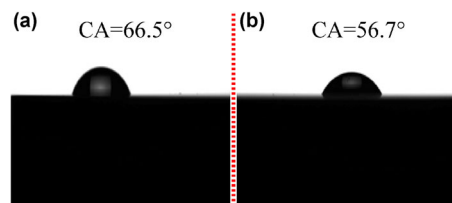


Fig. 5 Contact angle (CA) of (a) LB104 and (b) LP104.

The service life of mechanical components mainly depends on their abrasion resistance, which is usually elucidated by wear volume and wear rate. In addition, the friction coefficient responds directly to easy shearing. The wear volume of substrates under the NiAl ILs and ILs lubrication is shown in Fig. 6. The wear volume at 20 N generally increases with the addition of NiAl particles because these particles, as abrasives, increase the width and depth of wear tracks. Moreover, NiAl particles cannot effectively fill the wear scars or wear tracks under low applied loads. Compared to pure ILs, LB104-mNiAl and LP104-mNiAl hybrid lubricants provide higher wear volume at 40 N because the mNiAl particles are easy to be crushed to smaller irregular particles. Moreover, NiAl particles can destroy the IL-film, and cause difficulty in forming the “third body” on the mating interfaces. The wear volume of LB104-mNiAl lubricant is higher than that of LP104-mNiAl lubricant. This difference in wear resistance depends on the modification of particles, their dispersion in ILs, and the properties of ILs. Table 3 shows that the kinetic viscosity of LB104 ILs is lower than that of LP104 ILs, which allows LB104 ILs to flow and easily carry away the wear debris from the sliding interfaces. LB104-sNiAl lubricant provides the highest wear volume at relatively high applied loads, whereas the wear volume of the substrate

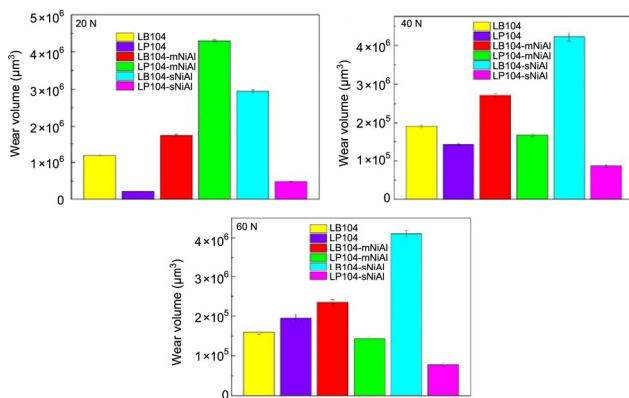


Fig. 6 The wear volume of the substrates under lubrication conditions at the applied loads of 20 N, 40 N, and 60 N.

Table 3 Kinematic viscosity of LB104 and LP104.

Sample	Kinematic viscosity (mm^2s^{-1})	
	40 °C	100 °C
LB104	27.9	5.7
LP104	72.7	10.2

lubricated by LP104-sNiAl lubricant is minimal. Some sNiAl alloy particles can easily roll between the friction pairs to prevent a direct contact and reduce the trend of the longitudinal development of abrasion. More importantly, the IL lubricants can form effective boundary and chemical reaction films in the rolling process and the abrasion can be filled by the crushed metal particles to overcome the increase of wear volume [11, 21]. It is worth noting that the wear volume of the substrates lubricated by NiAl hybrid LP104 ILs decreases with the increase in applied loads, especially for LP104-sNiAl lubricant. The increase of applied load is beneficial to crush the particles that fill the wear tracks and accelerate the decomposition of ILs to form tribo-chemical reaction products.

3.3 3D morphologies and SEM of worn surface

The worn surfaces of substrates lubricated by pure and NiAl hybrid ILs at an applied load of 40 N are shown in Fig. 7. These SEM images and 3D

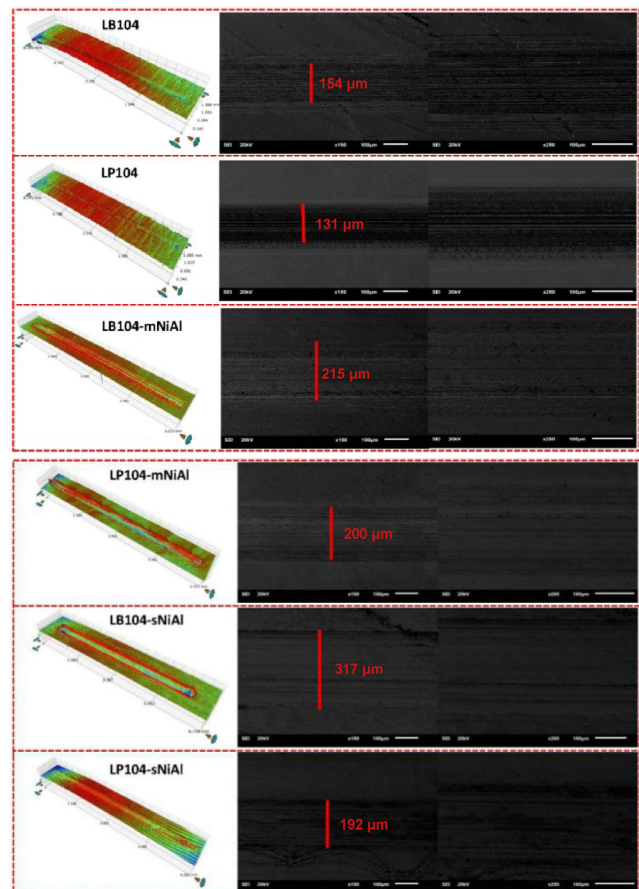


Fig. 7 SEM and 3D morphology images of worn surfaces at an applied load of 40 N.

morphologies were measured in the same condition. The worn surface of the substrate lubricated by pure ILs shows small and narrow wear tracks with a shallow depth, whereas the worn surface of the substrate lubricated by IL-NiAl lubricants shows wide wear tracks. In addition, the wear resistance of LP104-NiAl hybrid ILs is superior to that of LB104-NiAl hybrid lubricant. Some wear debris were deposited on the edge of the worn surfaces under NiAl hybrid IL lubrication.

3.4 OM images of steel ball

Figure 8 shows the OM images of a steel ball. The wear scar on the steel ball lubricated by ILs has a regular round shape and the wear track is relatively smooth and narrow, which is the embodiment of favorable lubrication of ILs. However, the wear scar of the steel ball lubricated by the NiAl hybrid lubricants is irregular in shape. In particular, NiAl hybrid lubricants show a relatively large wear scar with a large amount of wear debris deposited on the wear scar and its edges. Moreover, these wear scars are covered by a gray layer, indicating that the NiAl particles were involved in the friction process. The result is consistent with the previous speculation.

3.5 Analysis of the worn surface

XPS is very sensitive to the chemical state of an element. XPS spectra of typical elements on the worn surfaces are generated to verify the tribochemical products and explore the lubrication mechanism of IL-NiAl

additives. Figure 9 shows the XPS spectra of typical elements on the worn surfaces of substrates.

The distinct C peak is located at 284.8 eV, identified as C in air. The O peaks appear at 532.3 eV and 530.1 eV, indicating that the worn surface has oxides. The relatively weak N1s peaks appear in the binding energy range from 398.8 eV to 401.3 eV, which possibly include a C–N bond from the imidazole ring of ILs and friction-induced nitrogen or nitric oxide [36, 37]. The peaks of F1s XPS spectra are at 685.0 eV, ensuring that there are F⁻ ions in the worn surface owing to the formation of fluorine compounds like FeF₂ and FeF₃ [38]. The relatively distinct peaks of Fe2p XPS spectra appear at binding energies of 724.8 eV and 711.3 eV, indicating the existence of iron oxides (like Fe(OH)O), iron fluoride (FeF₂, FeF₃), and FePO₄ on the wear tracks [25, 39]. The lower binding energy of 707.4 eV could be attributed to FeB and FeP because the BF₄⁻ and PF₆⁻ of ILs were decomposed into active atoms, which chemically reacted with iron [40]. The weak P2p peaks are in the binding energy range from 132.5 eV to 134.7 eV, further confirming the existence of FeP and FePO₄. This result corresponds to the binding energy of Fe2p at 711.3 eV [41]. The Ni2p and Al2p XPS spectra indicate that NiAl alloy particles were transferred to or deposited on the mating surface during the friction process.

3.6 SEM images and EDS spectra of wear debris

To further explore the role of the particles in friction reduction and wear resistance, SEM micrographs and

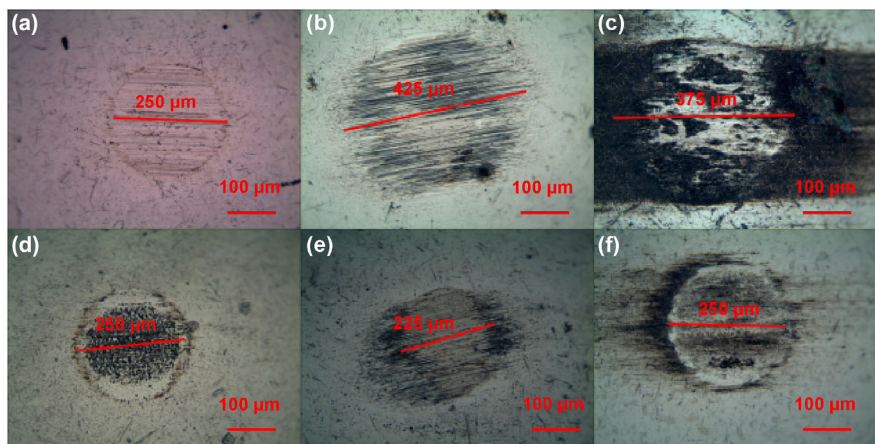


Fig. 8 OM images of steel ball lubricated by (a) LB104, (b) LB104-mNiAl, (c) LB104-sNiAl, (d) LP104, (e) LP104-mNiAl, and (f) LP104-sNiAl.

EDS spectra of NiAl wear debris are shown in Fig. 10. These wear debris originated from friction-induced NiAl particles. Compared to the original NiAl particles, the mNiAl particles became visibly smaller than the spherical ones because they were crushed by the upper and lower specimens during the friction. However, the sNiAl particles were changed only by extrusion and a few particles were crushed to pieces owing to the rolling effect of the spherical ones. The EDS

spectrum of mNiAl wear debris gives the iron peak, indicating that the iron was transferred from the friction pairs to the particles by crushing and scratching the contact surfaces. However, iron was not detected on the sNiAl wear debris possibly because the transfer of iron was difficult to achieve owing to the rolling effect.

The results indicate that the lubrication function of IL-NiAl hybrid lubricants mainly depends on the

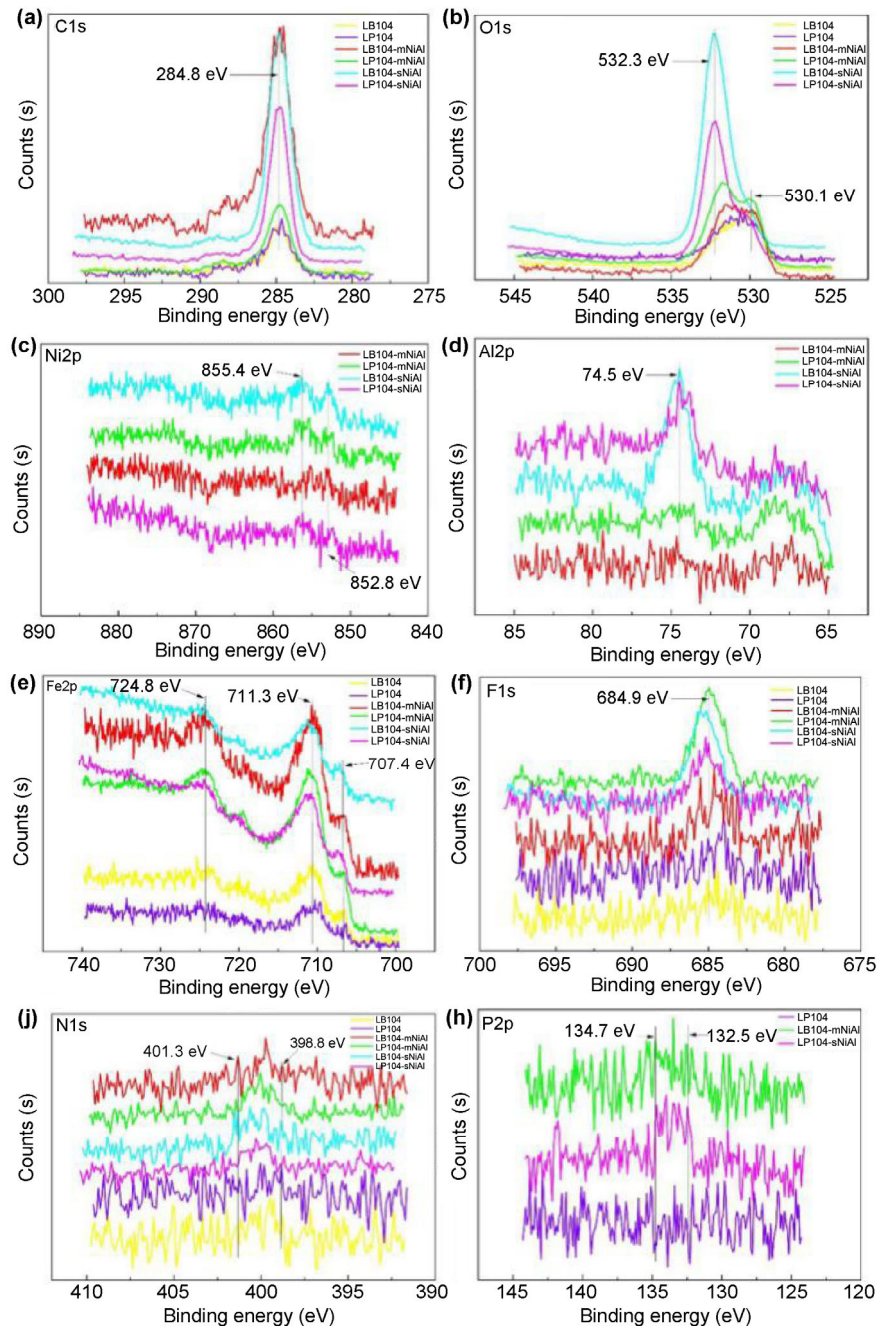


Fig. 9 XPS spectra of worn surfaces on the substrates lubricated by IL-NiAl hybrid lubricants.

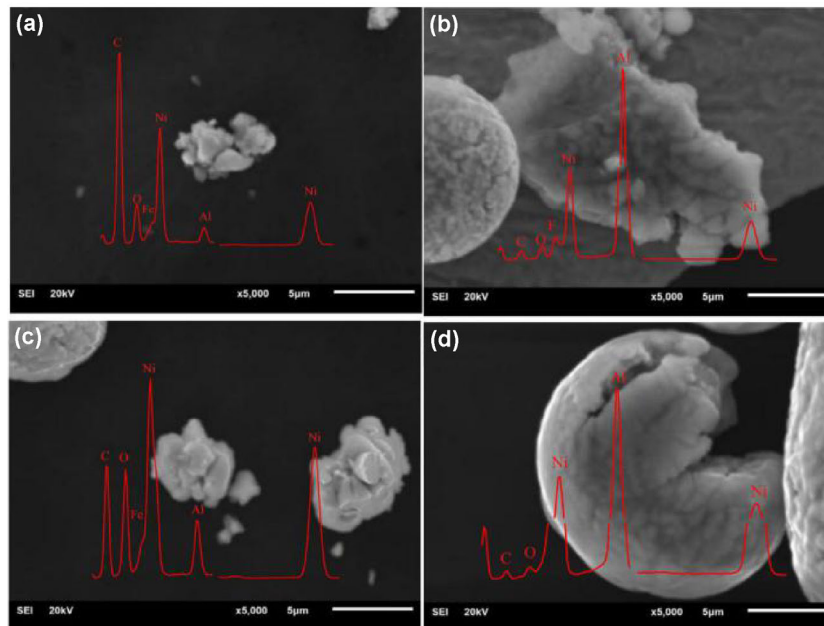


Fig. 10 SEM images and EDS spectra of wear debris of (a) LB104-mNiAl, (b) LB104-sNiAl, (c) LP104-mNiAl, and (d) LP104-sNiAl.

synergistic effect of NiAl particles and ILs. The excellent tribological properties of ILs are derived from the boundary lubrication film formed by physicochemical adsorption and tribo-chemical reaction films of ILs. The NiAl particles involved in friction can prevent the friction pairs from coming into direct contact and the wear tracks are continuously filled by these particles to reduce the wear. In addition, the rolling action of spherical particles can prevent the longitudinal development of wear, and ILs adhered on the particles can be transferred to the friction pairs for improving friction and wear. The XPS analysis reveals that the protective film consists of iron oxide, ferric fluoride, iron phosphide, iron boride, and phosphate, which further enhance the mechanical and tribological performance.

4 Conclusions

In this study, the tribological properties of micron-sized sNiAl and mNiAl particles as additives into ILs (LB104 and LP104) were investigated in detail. The conclusions drawn are as follows:

(1) mNiAl and sNiAl particles show good dispersion and stability in ILs because they were modified in-situ by anion and cation moieties of ILs through chemical interaction, especially mesoporous particles.

(2) LB104-modified mNiAl particles provide better tribological behaviors than the spherical ones, whereas the opposite is true for LP104-modified particles.

(3) LP104-modified sNiAl particles show the best wear resistance owing to the synergistic effect between LP104 IL and sNiAl particles, i.e., the formation of a tribo-chemical reaction film by the decomposition and further reaction of LP104 IL adsorption film, and the rolling and mending effect of the NiAl particles and their wear debris.

(4) The protective film on the wear surfaces are made up of iron oxide, ferric fluoride, iron phosphide, iron boride, and phosphate, which prevent direct contact between the friction pairs.

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