

Friction and Wear Behavior of Nanocrystalline Nickel in Air and Vacuum

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Abstract A bulk and dense nanocrystalline (NC) Ni with a mean grain size of 26 nm and a microhardness of 370–382 HV were fabricated by an electro-deposition technique. The microstructure and microhardness of the NC Ni samples were examined. The friction and wear behavior of the NC Ni samples under dry condition both in air and in vacuum were investigated in comparison with a conventional coarse-grained (CG) Ni with a mean grain size of 2.2 μm and a microhardness of 80–90 HV using a pin-on-disk type tribometer. The results show that the NC Ni sample possesses lower friction and higher wear resistance both in air and in vacuum, compared with the CG Ni sample. An important and interesting result is that for the both samples tested, the friction coefficients in vacuum are higher than those in air, while the wear loss occurs reverse case. In the air atmosphere, the NC Ni exhibits a mild wear, and avoids the severe wear that happens on the CG Ni, which is mainly attributed to which is mainly attributed to formation of a mechanical mixed layer on the worn surface of the harder NC Ni. In the vacuum atmosphere, compared with CG Ni, NC Ni shows a significant decrease in the plastic deformation and adhesive wear.

Keywords Nanocrystalline · Nickel · Vacuum ·
Wear mechanisms · Adhesive wear

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1 Introduction

Nanocrystalline (NC) materials, as a result of the considerable reduction of grain size and their significant volume fraction of grain boundaries inhibiting the mobility of dislocations, usually have very high hardness and strength compared with their conventional coarse-grained (CG) counterparts [1–4]. The increase in strength and hardness in NC metals may provide an opportunity for designing new materials as structural parts with improved wear resistance [5]. Also, they are also expected to find structural applications in the aerospace field. During the last decade, friction and wear behavior of NC metals have been paid more attention and many experimental results have shown a significant enhancement in friction and wear properties of NC metals [6–8]. For example, Farhat et al. [9] investigated effect of grain size on friction and wear properties of NC Al, using a miniature pin-on-disk type tribometer in air condition. Their results showed that when the grain size of Al was reduced from 1 to 16 nm, the peak of friction coefficient decreased by 57 %, and the wear resistance of those materials increased with decreasing grain size.

On the other hand, several studies also had been undertook to investigate the friction and wear behavior of NC Ni [10–12]. Guidry et al. [10] reported significant reductions in the friction coefficients and wear rate of electrodeposited Ni films with nanocrystalline grain structures compared with their microcrystalline counterparts. Mishra et al. [12] investigated effect of grain size on the tribological behavior of NC Ni, and showed that the friction coefficient for NC Ni was almost half that of polycrystalline Ni. It should be mentioned that most investigations on friction and wear behavior of NC metals, especially NC Ni, had been performed in air condition. Previous studies [9, 13] showed that testing environment as important factor affects friction and

wear behavior of materials including NC metals. Farhat et al. [9] showed that there was almost 40 % difference between the friction coefficients measured in the air and vacuum for all NC Al samples investigated. However, for NC Ni, only references [14, 15] studied the role of the testing environments involving in ambient air and argon environments on the initial and steady-state wear behavior of an NC Ni with a grain size of 15 nm using pin-on-disk wear tests. Unfortunately, little research gave detail analyses effect of vacuum environment on friction and wear behavior of NC Ni. Therefore, in order to meet the requirement of space applications for the NC Ni, it is necessary and important to investigate the effect of vacuum environment on friction and wear behavior of NC Ni.

In this study, a bulk and dense NC Ni was achieved by means of an electro-deposition technique. The microstructure and microhardness of the NC Ni were examined and analyzed to reveal the tribological characteristics and wear mechanism of the NC Ni in air and vacuum conditions. The results would be benefit to selection of nanocrystalline materials for the friction pairs used in spacecraft.

2 Experimental Methods

The substrates used as cathode for electrodeposited nanocrystalline nickel were 08Al steel with a size of 150 mm × 60 mm × 1 mm. They were ground to 2,000 grit, ultrasonically cleaned in acetone for 5 min, and rinsed using de-ionized water. A nickel plate (180 mm × 80 mm × 10 mm in size) with a purity of 99.97 wt% was used as anode material. NC Ni was electrodeposited using Watt's bath (Ni(SO₃NH₂)₂·4H₂O 300 g/l, NiCl₂·6H₂O 15 g/l, H₃BO₃ 30 g/l, C₇H₅NO₃S 1 g/l) was also added to the bath in the electro-deposition experiments for reducing the grain size. Electro-deposition experiments were performed at 50 °C using a current density of 2 A/dm² with direct current. The pH of the bath was constant at a value of 3, selected for each individual bath by adding diluted sulfuric acid (to decrease pH) or NaOH (to increase pH). All the deposition experiments were duplicated and good reproducibility was obtained. Bulk and dense NC Ni sheet with a thickness of about 3.5 mm was synthesized, as microstructural studies indicated uniform deposition.

The microhardness was measured on a HVS-5 type vickers microhardness tester under a load of 1 kg in a dwelling time of 10 s. An average of five measurements was performed on the as-deposited NC Ni. An X-ray diffraction spectrometer (Model D/max-rB, made in Japan Science Co.) was employed to determine the phase constituents of the as-deposited NC Ni, using a Cu K α (1.5418 Å) radiation source with an energy of 40 keV. Microstructure and grain size of the as-deposited NC Ni

were investigated by means of a TECNAI-G² type transmission electron microscope operated at 200 kV. Thin foil specimens for TEM observation were prepared by ion milling.

The friction and wear tests were performed both in air and vacuum (10⁻⁴ Pa) using a pin-on-disk type tribometer. To make comparison, the friction and wear behavior of CG Ni in air and vacuum were also investigated. The CG Ni was attained by annealing one electrodeposited Ni plate at 300 °C for 3 h. The circular sheet specimens with dimension of Φ 9 mm × 3.5 mm were cut from the as-deposited NC Ni and CG Ni sheet by using an electrodischarging machine and then polished to a mirror-like finish surface prior to friction and wear tests, since the circular sheet samples of NC Ni and CG Ni used as pin of friction couple is too thin to not be fixed, so the circular sheet samples of NC Ni and CG Ni were adhered to Al alloy cylinder with the dimension of Φ 9 mm × 15 mm by using a resin adhesive. The counterface material was GCr15 steel with the dimension of Φ 70 mm × 10 mm, surface roughness of 0.35 μ m (Ra) and hardness of 60 HRC. The GCr15 steel disk sample was rotated against a fixed pin sample (the circular sheet sample of NC Ni and CG Ni adhered to Al alloy cylinder, respectively, used as pin) to form friction pair. Contact geometry of pin and disk was as shown in Fig. 1. A pin sample was held with its axis perpendicular to the surface of a disk, and one end (with flat wear face, 9 mm in diameter) sliding against the disk in a dry friction condition, under a constant axial load, was applied with a dead weight. The pins stayed over the disks with a freedom degree of their axial direction, which allows normal load application by direct contact with the surface of the disk. A normal force of 10 N and a sliding velocity of 0.2 ms⁻¹ were chosen as testing parameters. All the disk and pin samples were cleaned with acetone in an ultrasonic cleaner prior to tests. Total sliding time was chosen as 80 min to

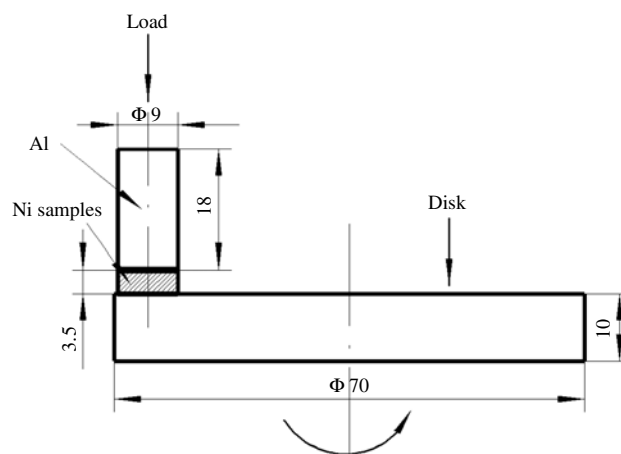


Fig. 1 Schematic diagram of the pin-on-disk test configuration

obtain stable friction and wear for the friction tests. The friction coefficient was recorded continuously during the friction and wear tests. For each specific sample, the friction and wear test were carried out three times in order to assure repeatability.

After friction and wear tests, the worn surfaces of pin and disk samples were examined using a scanning electron microscope (SEM) equipped with an energy dispersive spectrometer (EDS). The wear loss of NC Ni and CG Ni was determined using a microbalance with a precision of 10^{-5} g.

3 Results and Discussion

3.1 Microstructure and Microhardness

Figure 2 shows the X-ray diffraction patterns of the deposited bulk NC Ni and the CG Ni samples. The both samples show single face centered cubic (fcc) nickel phase. For the CG Ni, the strongest diffraction peak is (220) plane, while for the NC Ni, the strongest diffraction peak is (200) plane. The reason for the transition of the preferred texture can be attributed to the change of the surface energy of the different growth planes induced by saccharine during electro-deposition [16]. Compared with the CG Ni, the broadening of the diffraction peaks of the NC Ni is clearly observed in Fig. 2. According to the Scherrer's formula [17], the broadening of the diffraction peaks confirm that the mean grain size of the NC Ni is far smaller than that of the CG Ni.

Figure 3a shows TEM micrographs and the selected area electron diffraction rings of the as-deposited bulk NC Ni and the CG Ni. In the plan view image, almost all NC

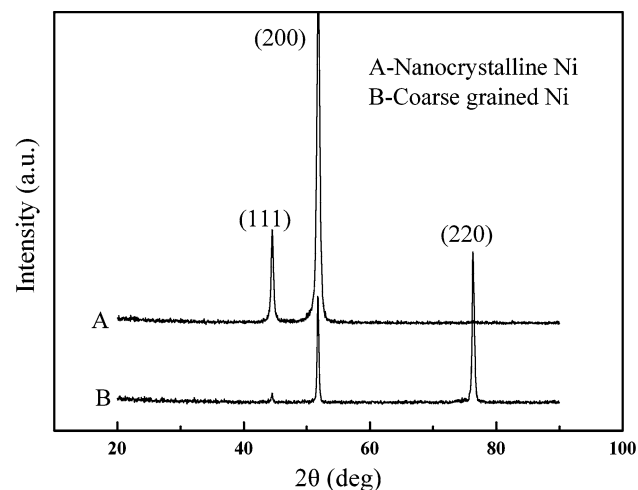


Fig. 2 X-ray diffraction patterns of the as-deposited bulk NC Ni and CG Ni samples

grains exhibit an equiaxed shape and a very narrow grain size distribution from 5 to 55 nm. A statistical analysis of about 500 grains from several TEM images indicate that the NC Ni has a mean grain size of 26 nm for volume fraction, shown by the grain size distribution plot of Fig. 3b. The inset selected area diffraction pattern in Fig. 3a shows the uniform diffraction rings, and no some large diffraction spots enmeshed in the diffraction rings, which is in agreement with the observed narrow grain size distribution of the NC Ni. The CG Ni has a mean grain size of 2.2 μm and some growth twins were obviously observed

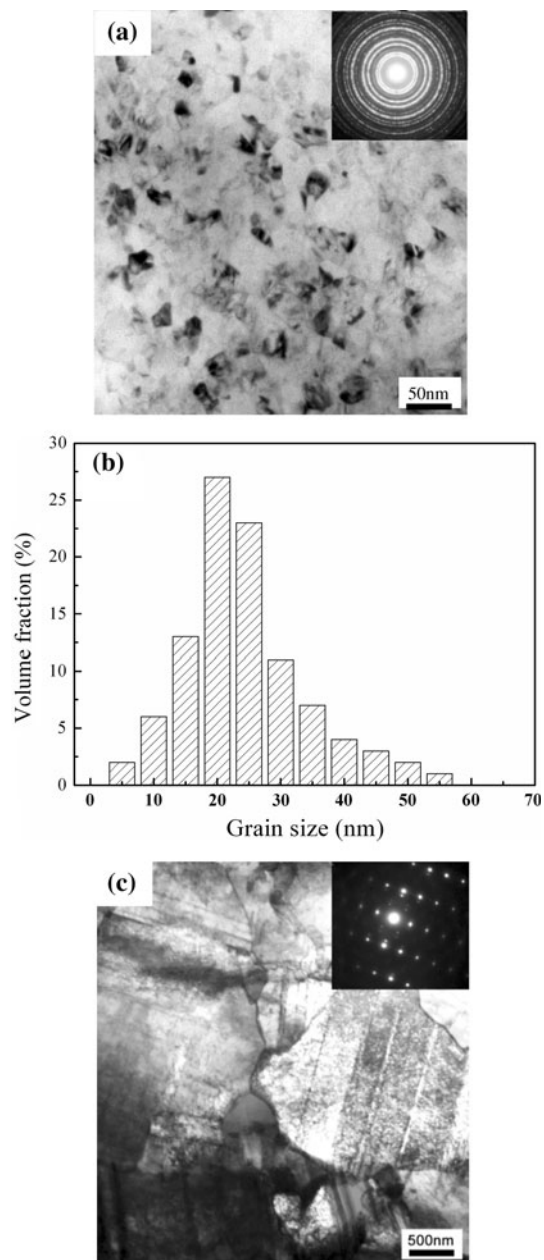


Fig. 3 a The TEM image of the as-deposited bulk NC Ni, b the grain size distribution plot of the NC Ni, and c the TEM image of the CG Ni

in the annealed Ni, which is agreement with the twin diffraction spots in the inset of Fig. 3c. The growth twin induced by annealing was also observed in the literature

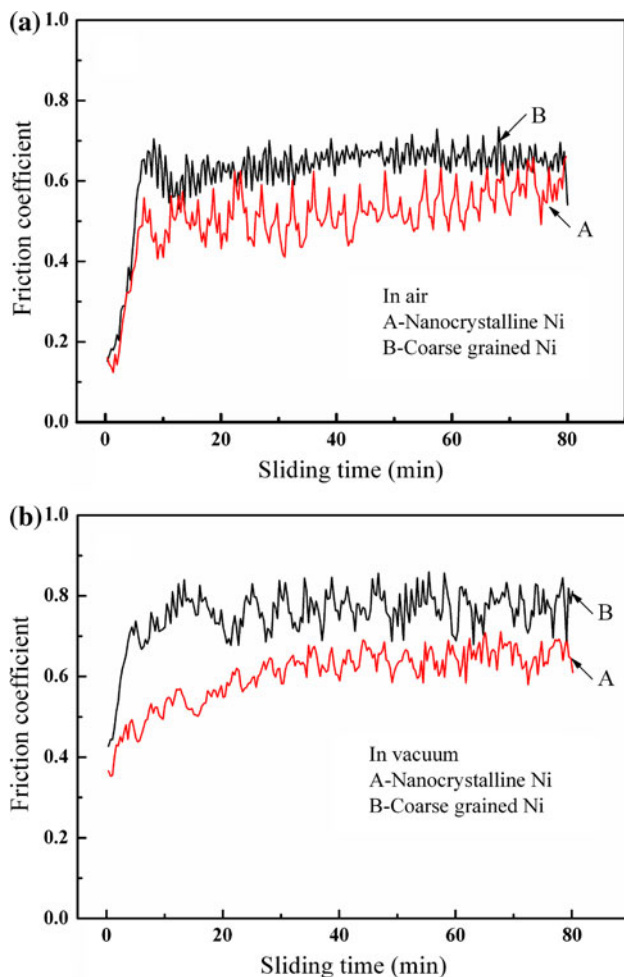


Fig. 4 Friction coefficients as a function of sliding time for NC Ni and CG Ni against GCr15 **a** in air and **b** in vacuum

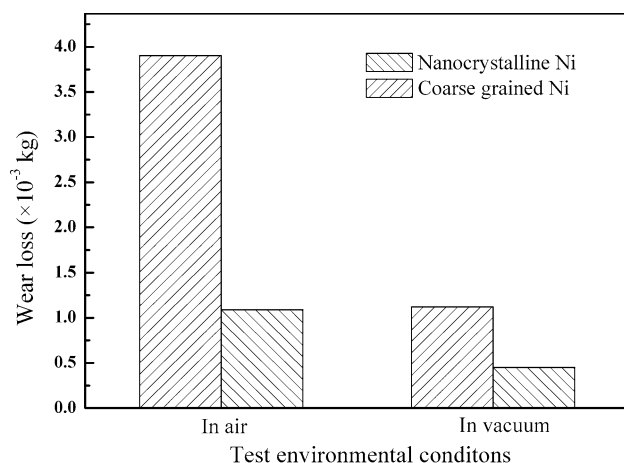


Fig. 5 The wear loss of the NC Ni and the CG Ni samples in air and in vacuum

[18]. Microhardness analysis shows that the microhardness along the cross-section of as-deposited bulk NC Ni and CG Ni is little change, varying at a level of 370–382 and 80–90 HV, respectively.

3.2 Friction and Wear Behavior

Figure 4 shows the variation of the friction coefficients with sliding time for NC Ni and CG Ni against GCr15 in air and vacuum, respectively. It is clearly seen from Fig. 4 that the friction coefficients of the NC Ni at a given sliding time are always lower than those of the CG Ni both in air and in vacuum. It is worthy to note that the friction coefficient of the NC Ni has stronger fluctuation than that of the CG Ni during the steady friction stage in air (see Fig. 4a), the case is opposite in vacuum (see Fig. 4b), which suggests that the friction behavior of the NC Ni and the CG Ni is different in air and vacuum. The steady-state friction coefficients are ranged from 0.41 to 0.65 for the NC Ni

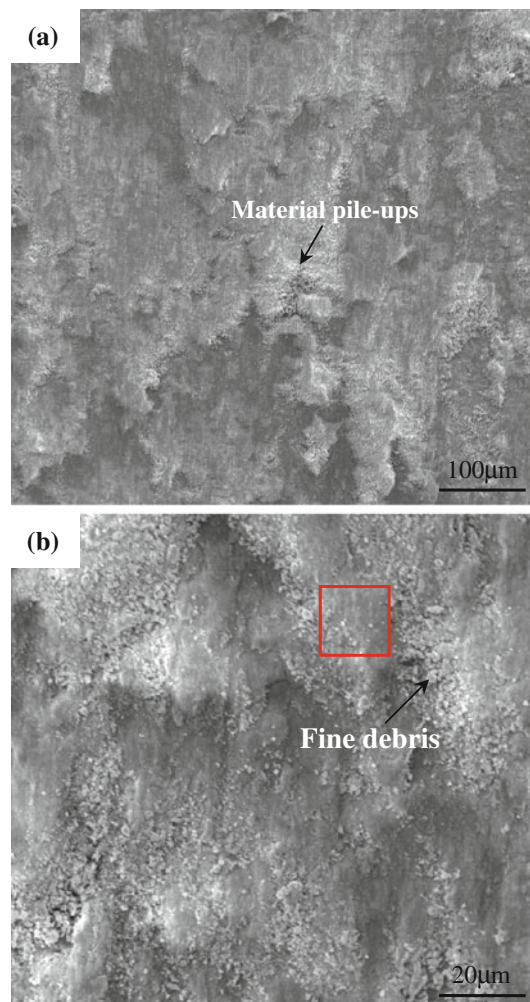


Fig. 6 SEM photographs showing the worn surface of the CG Ni sample in air under dry sliding

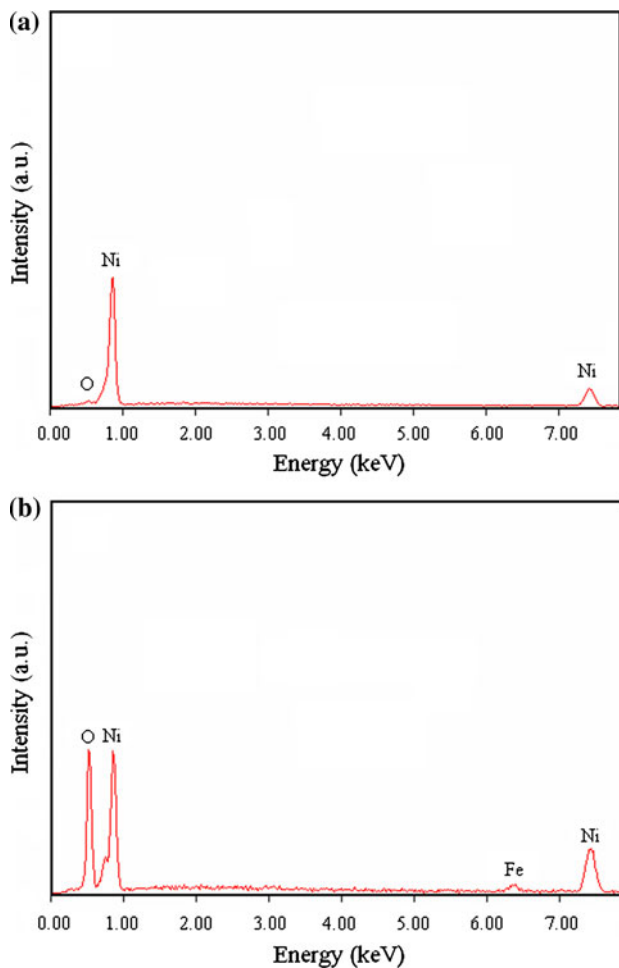


Fig. 7 The EDS spectra taken from **a** random area of unworn surface and **b** worn surface marked in *red rectangularity* in Fig. 6b for CG Ni in air (Color figure online)

samples and from 0.57 to 0.73 for the CG Ni ones in air. Whereas the steady-state friction coefficients are ranged from 0.55 to 0.71 for the NC Ni samples and from 0.67 to 0.86 for the CG Ni ones in vacuum. In addition, for both samples tested, the steady-state friction coefficients in vacuum are distinctly higher than those at a given sliding time in air, which may be attributed to different wear mechanisms in air and vacuum.

The wear loss of the NC Ni and the CG Ni samples in air and in vacuum is presented in Fig. 5. Compared with the CG Ni sample, the NC Ni sample exhibits lower wear loss both in air and in vacuum. Detailed calculation indicates that the wear loss of the NC Ni samples in air and in vacuum is higher than those of the CG Ni samples by 3.58 and 2.49 times, respectively. Notably, the wear loss of both samples is higher in air than those in vacuum by 3.48 and 2.42 times, respectively. Based on the above results and analyses, it can be concluded that compared with CG Ni sample, the NC Ni sample possesses lower friction and relatively higher wear resistance both in air and in vacuum.

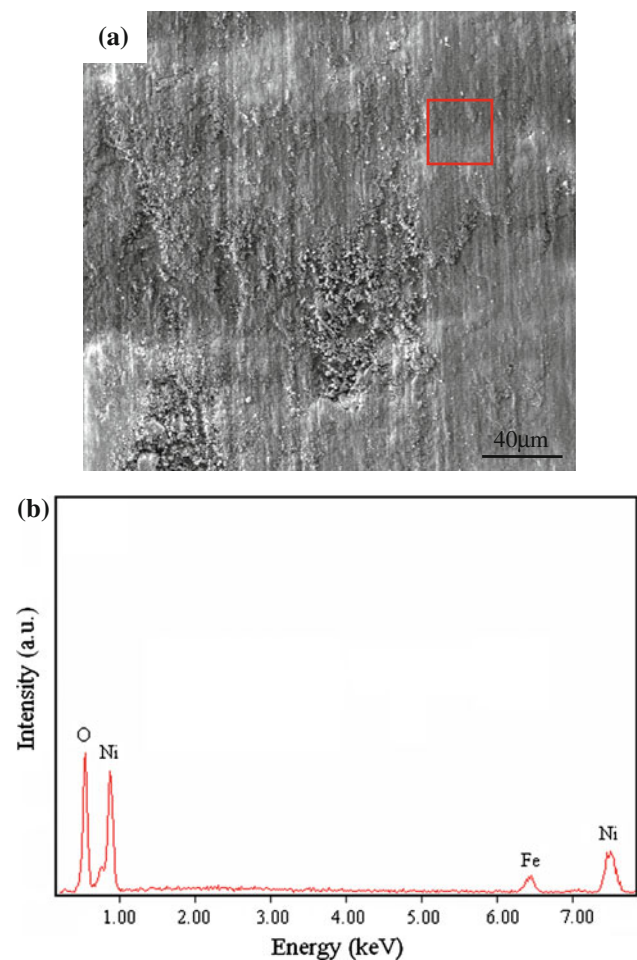


Fig. 8 **a** SEM photographs of the worn surface of GCr15 against the CG Ni sample in air and **b** the EDS spectra acquired from a selected area indicated by *red rectangularity* in **a** (Color figure online)

3.3 Analyses of Worn Surfaces

Figure 6 shows the worn surface morphologies of the CG Ni sample in air. It is observed that the worn surface of the CG Ni exhibits rougher and large numbers of fine black debris, as shown in Fig. 6a, b. Obvious material pile-ups are observed in some local regions on the worn surface of CG Cu (see Fig. 6b), which is attributed to plastic deformation on the contact surface of the CG Cu induced by friction. The results of EDS analysis of the unworn/worn surfaces for the CG Ni samples are shown in Fig. 7. The oxygen concentration of the worn surface is obviously higher than that of the unworn surface, implying that the worn surface is oxidized, forming brittle oxide layer during the wear in air. The brittle oxide layer is easy to be fractured, producing fine black wear debris. It can be speculated that an oxidative wear mechanism dominates the wear process and accelerates the wear of CG Ni sample. Moreover, SEM micrographs and EDS spectra acquired from a random area (indicated by the cross) on the worn surface of

the GCr15 disks confirm the existence of transferred materials containing nickel during wear in air, and the transferred materials almost cover entire worn surface of GCr15 disks, indicating that a severe adhesive wear occurs, as shown in Fig. 8a, b.

Morphologies of the worn surfaces on the NC Ni samples in air are shown in Fig. 9a, b. For the NC Ni samples, the worn surface is characterized by a discontinuous smooth polished surface. EDS spectra of the smooth polished surface indicate that the smooth polished surface is oxidized, as shown in Fig. 9c, forming a mechanical mixed surface layer [19]. For a nanocrystalline metal, it is easy to form the mechanical mixed layer because of the presence of more grain boundaries which act as nucleation sites for the oxides and diffusion paths of oxygen during the mixing process caused by sliding [16, 20, 21]. In addition, if the substrate is soft and can be plastically deformed easily it might not sufficiently support the mechanical mixed layer, which is the reason why a continuous and effective

mechanical mixed layer cannot be formed for CG Ni samples. The mechanical mixed layer formed during friction of NC Ni in air helps to give rise to lower friction coefficient and provides a surface protection, which leads to lower wear loss, compared with CG Ni. Notably, extensive microcracks due to fatigue under the action of circular load and sliding velocity are observed on the mixed surface layer of the NC Ni. Their propagation and coalescence result in local delamination of the mechanical mixed layer [22], forming larger-flaky shape debris, and leaving behind fresh surface (labeled as 2 in Fig. 9b) which is confirmed by EDS spectra analysis (see Fig. 9d). Therefore, it could be concluded that the wear of the NC Ni in air is characterized by delamination of the mechanical mixed layer formed by oxidation of the worn surface. The mechanical mixed layer is continually formed and subsequently peeled off, which induces the evident fluctuation of the friction coefficients during sliding of NC Ni sample against GCr15 disk in air. In addition, no plastic

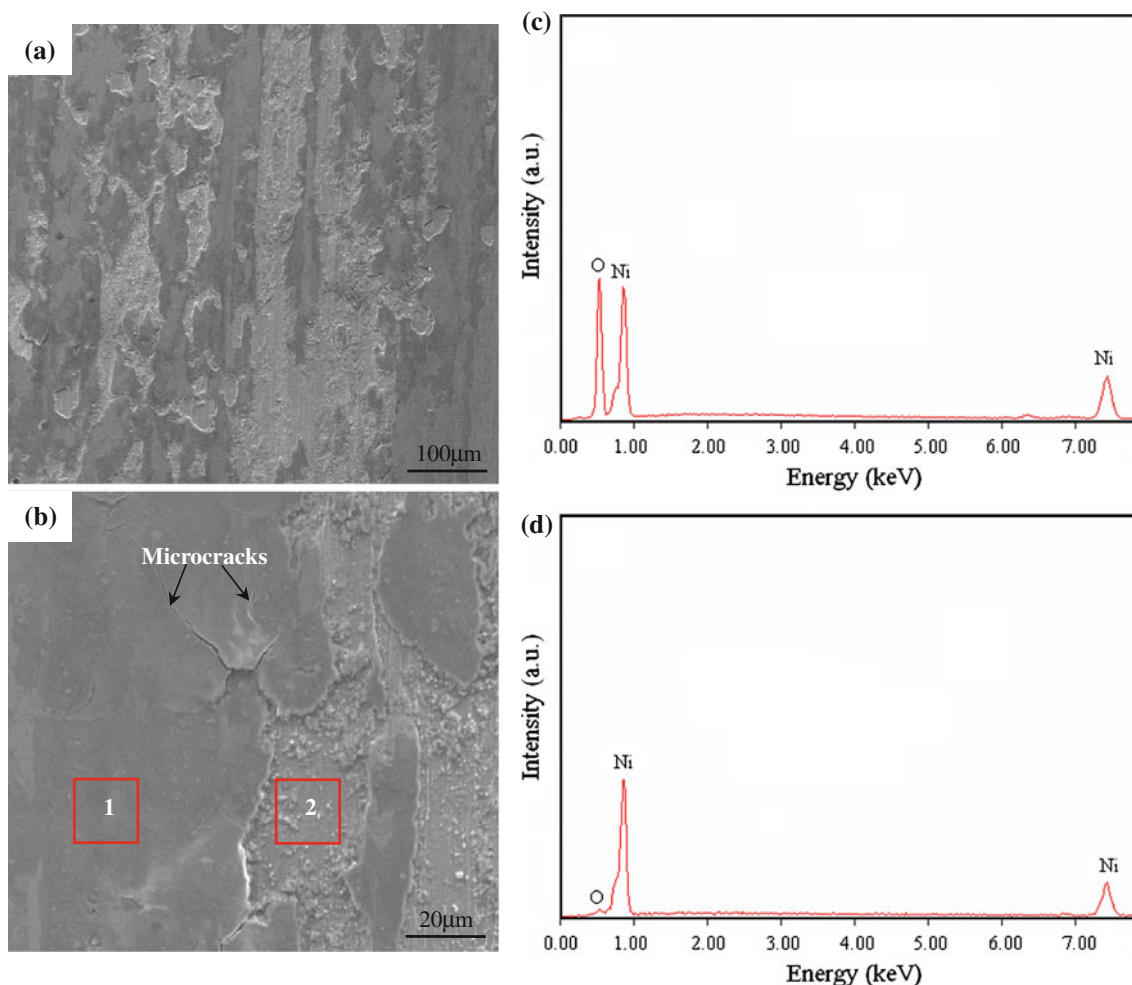


Fig. 9 a, b SEM photographs showing the worn surface of the NC Ni sample in air under dry sliding. c EDS spectra of the area labeled 1 in b. d EDS spectra of the area labeled 2 in b

deformation is found on the worn surface of the NC Ni, implying that compared with soft CG Ni, the hard NC Ni could prevent plastic deformation.

On the other hand, it is found that transferred materials exist on the worn surface of the GCr15 against NC Ni sample in air, as shown in Fig. 10a. EDS analysis confirms that the transferred materials contain nickel element from the NC Ni sample, as shown in Fig. 10b. Moreover, original morphology of the GCr15 disks still can be observed. The above analyses suggest that a mild adhesive wear occurs during sliding of the NC Ni against GCr15 in air.

In vacuum, the wear mechanisms of GC Ni and NC Ni samples are different from those in air. The most distinct difference between air and vacuum is the existences of oxygen that may lead to the changes in wear mechanism. The worn surfaces of CG Ni in vacuum are characterized by a number of parallel deep plowing grooves and a typical material delamination, as shown in Fig. 11a, b. The deep plowing grooves can be attributed to as a result of plastic deformation. In addition, microcracks caused by fatigue

under repeating loads exist on the worn surface, as shown in Fig. 11c. Figure 12a shows the worn surface morphologies of the GCr15 disks against the CG Ni samples in vacuum. It can be seen from Fig. 12a that transferred

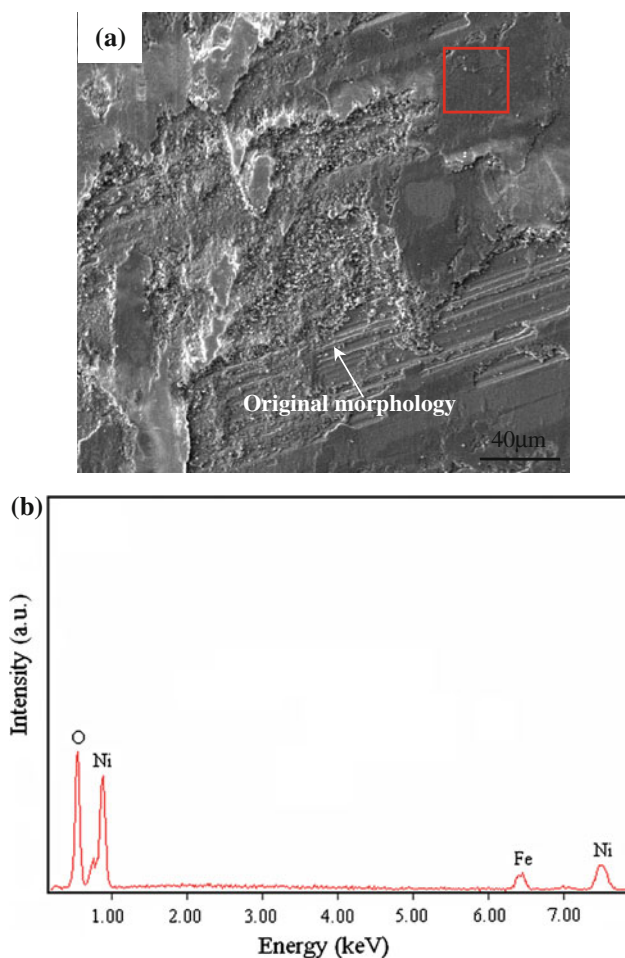


Fig. 10 **a** SEM photographs of the worn surface of GCr15 against the NC Ni sample in air and **b** the EDS spectra acquired from a selected area indicated by red rectangularity in **a** (Color figure online)

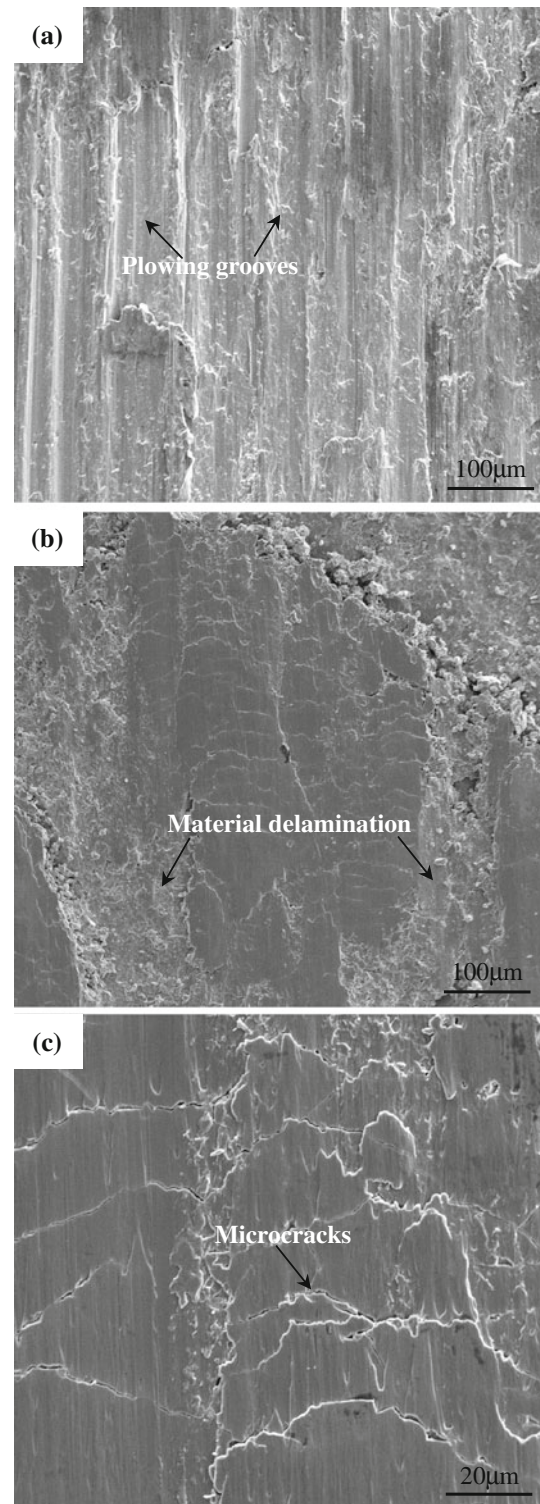


Fig. 11 SEM photographs showing the worn surface of the CG Ni sample in vacuum under dry sliding

materials containing nickel element are observed on the worn surface. EDS spectra in Fig. 12b indicate that the material transfer occurs from the CG Ni sample to the GCr15 disk during friction in vacuum. This result suggests that CG Ni experiences adhesive wear in vacuum. This could explain why the material delamination and the scale-like morphology occur on the worn surface of CG Ni sample.

In vacuum, the worn surface morphologies of the NC Ni are similar to those of the CG Ni. The worn surface of the NC Ni also exist plowing grooves, a typical material delamination and microcracks, as shown in Fig. 13. However, compared with the CG Ni, the plowing grooves are much fewer, narrower and shallower on the worn surface of the NC Ni, which the NC Ni resists damage caused by plastic deformation more effectively. On the other hand, SEM and EDS analysis on the worn surface of GCr15 disk against NC Ni confirm the existence of transferred materials from the NC Ni pin during wear in vacuum, as shown

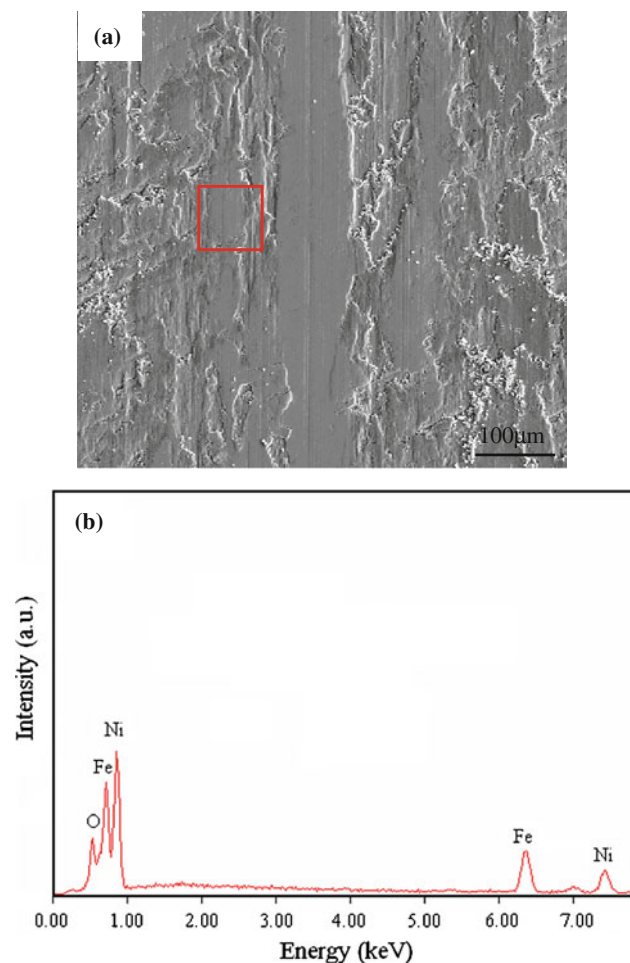


Fig. 12 **a** SEM photographs of the worn surface of GCr15 against the CG Ni sample in vacuum and **b** the EDS spectra acquired from a selected area indicated by red rectangularity in **a** (Color figure online)

in Fig. 14. Moreover, original morphology of the GCr15 disks can be observed, which indicates that a mild adhesive wear occurs on worn surface of NC Ni samples in vacuum.

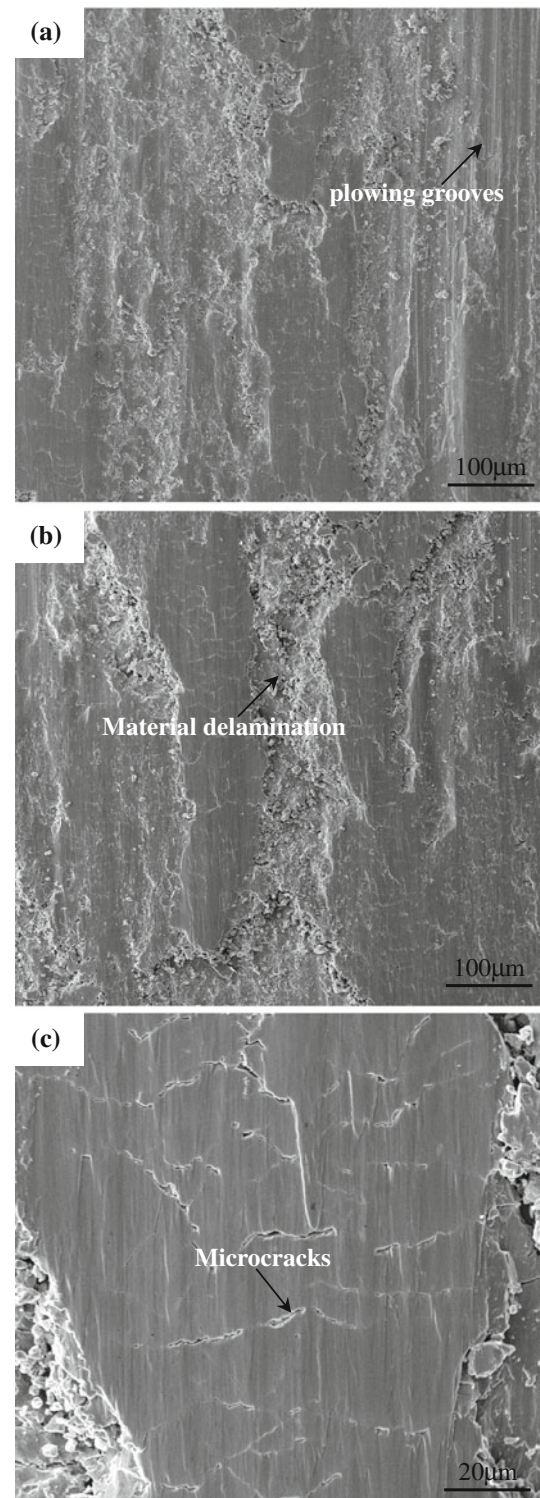


Fig. 13 SEM photographs showing the worn surface of the NC Ni sample in vacuum

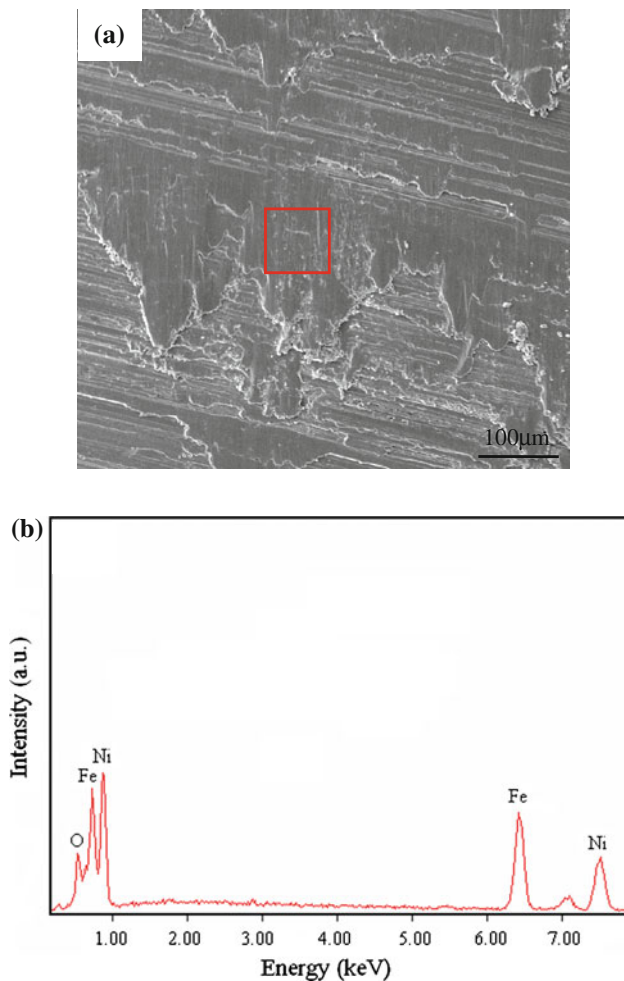


Fig. 14 **a** SEM photographs of the worn surface of GCr15 against the NC Ni sample in vacuum and **b** the EDS spectra acquired from a selected area indicated by red rectangularity in **a** (Color figure online)

Based on the above results and analyses, it is believed that compared with CG Ni, the plastic deformation and adhesion wear on the worn surface of NC Ni during friction both in air and in vacuum decrease a lot, which can contribute to significantly increase the hardness and strength to nanocrystalline induced by reduction of grain size. Therefore, the wear loss of the NC Ni samples in both environmental conditions is lower than that of the CG Ni samples. Since friction coefficient (u) consists of an adhesion component (u_a) and a deformation component (u_b) which result from the adhesion force and deformation force [23], respectively, during sliding. The adhesion component (u_a) plays an important role in sliding wear [24]. Therefore, it can be suggested that the decrease in plastic deformation and adhesion of NC Ni both in air and in vacuum confirmed by SEM analysis, results in lower friction coefficient compared with the CG Ni, which should in turn improve the wear property. The above results are in accordance with the observation of Stolyarov et al. [25]. So

far, the mechanism on how the nanocrystalline structures reduced the u_a and u_b , and is not clear and needs further systematic study. The NC Ni had higher strength, higher hardness, and lower ductility than the CG Ni. It is difficult to single out which mechanical property affected the u_a and u_b , or how much each property affected u_a and u_b .

4 Conclusions

From the above, the following main conclusions can be drawn:

- A bulk and dense NC Ni with a mean grain size of 26 nm and a thickness of 3.5 mm were produced by an electro-deposition technique.
- Friction coefficients of the NC Ni are always lower than those of the CG Ni both in air and in vacuum. The friction coefficient of the NC Ni has stronger fluctuation than that of the CG Ni during the steady friction stage in air, the case is opposite in vacuum. For both samples tested, friction coefficients become higher when the sliding test is performed in vacuum.
- Compared with the CG Ni sample, the NC Ni sample exhibits lower wear loss both in air and in vacuum. Moreover, the wear loss of both samples tested in air is higher than those in vacuum.
- In air atmosphere, the NC Ni exhibits a mild wear, and avoids the severe wear that happens on the CG Ni. The enhancement of the wear properties of the NC Ni is associated with the stability of a mechanical mixed layer formed by oxidation and high hardness of the nanocrystalline structure.
- In vacuum atmosphere, plastic deformation and the adhesion dominates the wear process of both samples tested. Compared with CG Ni, the plastic deformation and adhesive wear on the worn surface of NC Ni decrease a lot, which can contribute to significantly increase the hardness and strength to nanocrystalline induced by reduction of grain size.

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