https://doi.org/10.1007/s41230-023-2104-8

Friction and wear behavior and mechanism of low carbon microalloyed steel containing Nb

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Abstract: Dry sliding friction and wear test of Nb containing low carbon microalloyed steel was carried out at room temperature, and the effect of Nb on the wear behavior of the steel, as welll as the mechanism was studied. Scanning electron microscopy (SEM) and energy dispersive spectrometry (EDS) were employed to analyze the morphology and composition of the worn surface, and the structure evolution of the plastic deformation layer. The carbide content and type in the steel were analyzed by the electrolytic extraction device and X-ray diffraction (XRD). The experimental results demonstrate that the addition of 0.2% Nb can refine the grain and generate NbC to improve the wear resistance of the steel. By enhancing the load and speed of wear experiment, the wear mechanism of the test steel with 0.2% Nb changes from slight oxidation wear to severe adhesion wear and oxidation wear. Compared with the load, the increase in the rotation speed exerts a greater influence on the wear of the test steel.

Keywords: Nb content; low carbon microalloyed steel; friction and wear; carbide

CLC numbers: TG142.31 **Document code:** A **Article ID:** 1672-6421(2023)03-263-08

1 Introduction

Low-carbon steel with carbon content lower than 0.25% is also called mild steel because of its low strength, low hardness and high softness. Carbon as the most common interstitial solution element can significantly improve the strength of steel, but at the same time, the increase of C content will reduce the elongation, impact toughness and weldability [1]. In steel, whether C exists in solid solution state or combined state, the toughness of steel decreases with the increase of C content, and the ductile-brittle transition temperature increases. Completely solid solution C can weaken the embrittlement tendency of steel [1-3]. In practical application, there are high requirements for strength, plasticity, toughness and wear resistance. It is a feasible method to reduce the C content and add alloying elements into steel to improve the mechanical properties of steel [4-6]. Micro-alloying element Nb can significantly increase the coarsening temperature and recrystallization temperature of austenite in steel, demonstrating the functions of refining austenite grains and dispersion strengthening. It is one of the most effective alloying elements to improve the strength and toughness of materials [7-9]. Owing to the large load-bearing tonnage of heavy-duty vehicles, the cast steel universal joint of the chassis transmission system requires high toughness and excellent wear resistance. With the purpose of reinforcing the wear resistance and prolonging the service life of the system, the micro-alloying of Nb was utilized to adjust the type and distribution of carbide, so as to regulate the strong-plastic fit and improve the wear resistance of steel. Additionally, the low-carbon microalloyed steel involved in this study adopted normalizing heat treatment and was compared with the quenched and tempered state to avoid the phenomenon that large workpieces are prone to deformation and cracking during quenching. The changes of niobium carbide (NbC) in the matrix and friction-affected layer, as well as its influence on wear resistance of low-carbon microalloyed steel, were investigated.

2 Experimental method

The experimental steel was a button-type ingot prepared using a DHL-1250 vacuum arc furnace. The mass of the ingot was about 80 g, and the addition amount (mass fraction) of Nb was: 0% (#1), 0.2% (#2), and 0.4% (#3). The PMI-MASTER PRO mobile direct reading spectrometer was employed to analyze the chemical composition of the test steel, and the

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Received: 2022-07-21; Accepted: 2023-02-03

experimental results are listed in Table 1. The normalizing heat treatment process was: (a) Homogenization annealing: kept at 1,100 °C for 2 h and cooled in the furnace. (b) Isothermal normalizing: heated at 930 °C for 2 h, cooled with the furnace to 620 °C and held for 2 h, and then air cooling. The quenching and tempering process used in the comparison experiment was: heated to 860 °C and held for 160 min, then cooled by water for 12 s, and subsequently held at 580 °C for 4 h, then air cooling.

Table 1: Experimental low alloy steel chemical composition (wt.%)

Steel	C	Si	Mn	Cr	Mo	Ni	Nb
#1	0.200	0.240	0.751	0.539	0.195	0.508	0.003
#2	0.203	0.245	0.760	0.538	0.191	0.520	0.192
#3	0.187	0.236	0.774	0.544	0.193	0.511	0.380

The dry sliding friction test was carried out on the friction and wear tester model MFT5000 produced by RTEC Corporation in the United States, and the pin-on-disc contact method was used, as shown in Fig. 1. The button-type ingot was wire-cut and processed into a cylindrical pin with a diameter of 6 mm and a height of 7.9 mm after normalizing heat treatment. Since the universal joint mostly contacts with 45 steel in the service environment, a 5 mm thick normalized 45 steel disc (hardness 150 HBW) was selected as the friction pair. The pair of grinding discs was fixed on the base, and the pins performed circular motion on the disc. The experimental conditions were: atmospheric pressures 30 N, 50 N; rotating speeds 100 rpm, 150 rpm; and experimental time 30 min. The friction and wear tests of the steels with 0%, 0.2%, and 0.4% Nb after normalizing heat treatment and the steel with 0.2% Nb after quenching and tempering were performed for 30 min at 30 N-100 rpm, 30 N-150 rpm and 50 N-100 rpm, respectively. The test pins and discs were polished by 400–1200 mesh sandpaper. The test pins before and after wear were ultrasonically cleaned in an acetone solution and then dried. The 0.1 mg precision balance was utilized to weigh the mass change of the pin before and after wear, and the mass loss after wear was counted. Three parallel weights were carried out for each test, and the average value of the data was taken.

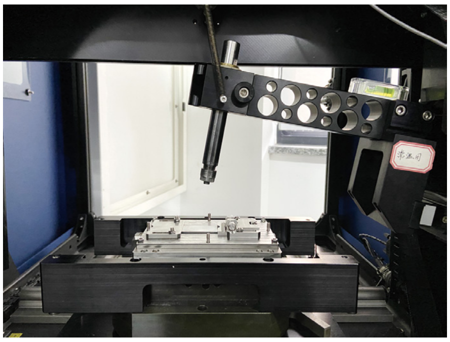

Fig. 1: Schematic diagram of MFT5000 friction and wear testing machine and pin-on-disc matching

The microstructures were characterized by a scanning electron microscope (SEM). Local chemical composition determination and the distinction of carbides were performed with energy-dispersive X-ray spectroscopy (EDS). The effects of carbide type and content and grain size on the wear resistance of the test steels were analyzed by the electrolytic extraction method and original austenite grain detection method (GB/T6394-2017). The MH-3L microvickers hardness tester was adopted to perform stepped micro-hardness measurement on the prepared wear section samples to obtain hardness data at different depths from the wear surface. The experimental load of the microscopic Webster's hardness measurement was 500 g·F, and the pressure holding time was 10 s. The electrolytic extraction test was performed on the experimental steels. The electrolyte was a mixed solution of 375 mL of water, 125 mL of concentrated hydrochloric acid and 16 g of citric acid particles. The extraction voltage and time were 15 V and 12 h, respectively. After the extract was stood, centrifuging-dried, and weighed, the carbide content in the steel was counted. Furthermore, the phase constitution of the extracted precipitate powder was explored by a A Rigaku18kW D/MAX2500V+/PC X-ray diffractometer (XRD) (a Cu target) with a scanning angle of 10°–90° and a scanning speed of 4°·min^{-1}.

3 Results and analysis

3.1 Effect of Nb content on wear resistance of test steel

Friction and wear tests were performed on test steels with different Nb contents and heat treatment processes (normalizing was indicated by "N", quenching and tempering was indicated by "Q and T"). The mass loss of the test steels under different wear conditions is illustrated in Fig. 2. It can be found that the mass loss of the normalized sample increases with the increase of load and speed. When the load is 30 N and the speed increases from 100 rpm to 150 rpm, the increase of mass loss is significant, and the average increase rate of weightlessness is 155.4%. The average increase rate of weightlessness is 44.1% when the rotating speed is 100 rpm and the load is increased from 30 N to 50 N. Under the same experimental parameters, the mass loss firstly decreases and then increases with the increase in the Nb content, and the wear loss is the smallest when the Nb content is 0.2%. In other words, the samples with 0.2% Nb has the better abrasion resistance in the experimental range. Under the conditions of 30 N-100 rpm, 30 N-150 rpm, and 50 N-100 rpm, the wear loss of the sample containing 0.2% Nb is reduced by 56.2%, 19.3%, and 28.8%, respectively, compared with the sample without Nb. The wear resistance of the quenched and tempered 0.2% Nb samples is better than that of the normalized one. Under the same wear conditions, the wear loss of the quenched and tempered steel with 0.2% Nb is 92.9%, 83.7%, and 83.6% of the normalized state, and the average weight loss is 86.7% of the normalized state, indicating that the abrasion resistance of 0.2% Nb normalized sample is relatively close to the quenched and tempered sample commonly used in industry.

Figure 3 displays the wear surface morphology of the three kinds of normalized steels with different Nb contents under a load of 30 N and a rotation speed of 100 rpm for 30 min. It can be observed from Fig. 3 that the presence of high hardness micro-convex and wear debris on the friction pair has a "ploughing" effect on the test steel. Under the action of contact pressure and cutting stress, delamination and cracks along the wear direction on the surface appear, as shown in Fig. 3(a). Obvious embossing [Fig. 3(c)] and peeling pits on the surface of the sample containing 0.4% Nb appear, as revealed in the rectangular box of Fig. 3(d). When the sample is rotated at high speed, the temperature of the pin-on-disc contact surface increases, and cementite becomes coarse and precipitates along the original austenite grain boundary, leading to the decreased grain boundary embrittlement and the weakened hardness of the matrix. After a certain period of wear, the substrate around the carbide is ground flat and the embossing begins to appear. When the amount of wear continues to increase, the raised carbides will peel off, causing more severe abrasive wear. However, the wear surface of the sample with Nb content of 0.2% exhibits only a few oxidative peeling and shallow scratches [Fig. 3(b)], and the plastic deformation is small, indicating that its wear resistance is better compared with other samples.

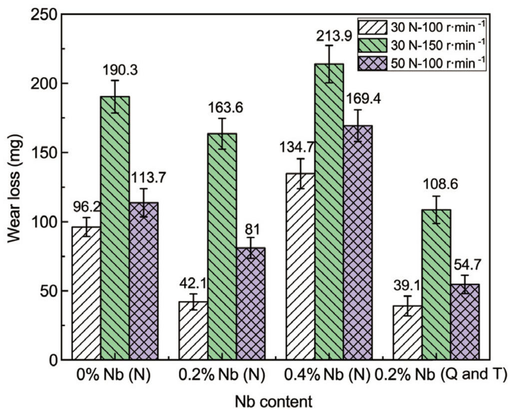

Fig. 2: Wear loss of experimental steels with different Nb contents

3.2 Microstructure analysis

There are many factors that affect the wear resistance of materials. Not only the matrix structure, grain size, and hardness of the material will affect the wear resistance, but also the size and distribution of carbides in the structure are closely related to the wear resistance [8]. As observed in the SEM morphology of the steels with different Nb contents shown in Fig. 4, it is visible that the grain size is significantly reduced when Nb is added, and more virgulate and spherical white bright structures are precipitated on the grain boundaries. Figure 5 shows the EDS analysis for 0% Nb and 0.2% Nb steels. The precipitate of the sample without Nb contains

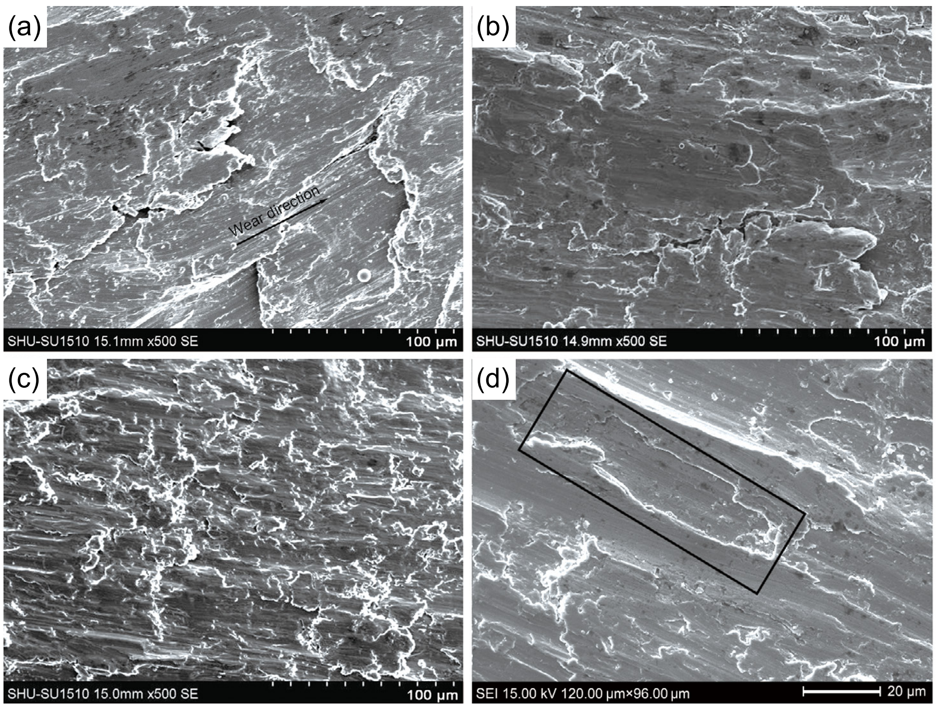

Fig. 3: Worn surface of normalized samples under condition of 30 N and 100 rpm for 30 min: (a) 0 Nb; (b) 0.2% Nb; (c, d) 0.4% Nb

carbides of Cr and Fe. Nonetheless, the precipitates were transformed into NbC with higher hardness after the addition of 0.2% Nb [12].

The carbide content of normalized test steel with different Nb contents was measured by carbide extraction experiment and the carbide type was analyzed by XRD (carbide content was measured by weightlessness method). The results are presented in Figs. 6 and 7. The grain diameter and hardness were measured, and the statistical results are illustrated in Table 2. It can be discovered that the total amount of carbides in the steel significantly increases with the addition of the Nb element. The NbC phase appears in the steel after addition of Nb and its content increases with the increase of the Nb content. The content of the $(Fe, Cr)_3C$ phase increases firstly and then decreases with the increase of Nb content. This is because Nb is a strong carbide forming element, $(Fe, Cr)_3C$ will be converted to NbC when the content of Nb exceeds 0.2% [11]. The larger size NbC has high hardness, which can better resist the "ploughing" effect caused by abrasive wear and improve the wear resistance of steel. The hardness of the test steel also shows a trend of firstly increase and then decrease when the Nb content changes from 0% to 0.4%. The average grain diameter of the steel containing 0.2% and 0.4% Nb is decreased by 41.3% and 38.8%, respectively compared with the sample without Nb, which indicates that the addition of 0.2% Nb can refine the grain obviously better resist the plastic deformation caused by the wear process, and greatly help to improve the wear resistance of the material. The grain size of 0.4% Nb test steel is slightly larger than 0.2% Nb due to the segregation of carbides [15]. High Nb content allows Nb-containing carbides to grow up and coarsen on the grain boundary. Some carbon-poor areas that are difficult to form carbides, reduce the pinning of carbides on the grain boundary and inhibit the migration of grain boundary and heterogeneous nucleation. These factors lead to the increase of the inhomogeneity of the grain size (Fig. 4). In the 0.4% Nb steel, there are some scattered and abnormally grown grains, whose diameters reach 40–50 μm. The alloying element segregation increases the inhomogeneity of the surface properties, and then weakened the wear resistance of the steel.

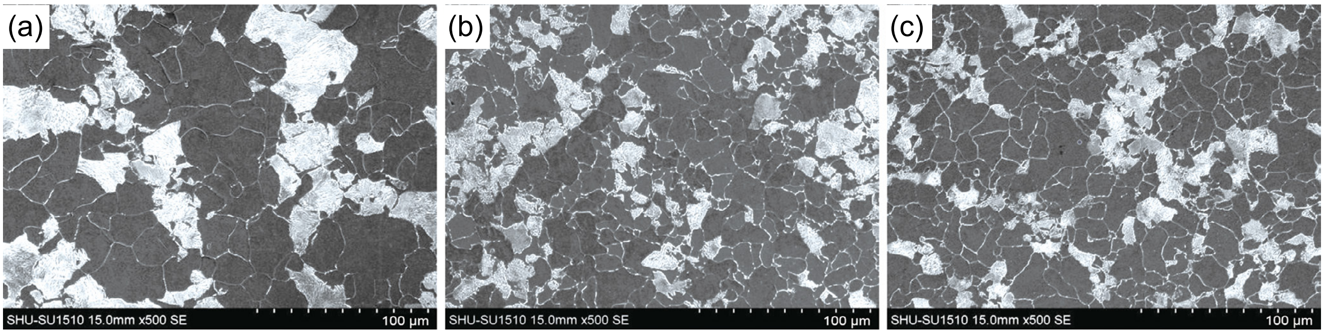

Fig. 4: SEM morphology of normalized samples with different Nb contents: (a) 0 Nb; (b) 0.2% Nb; (c) 0.4% Nb

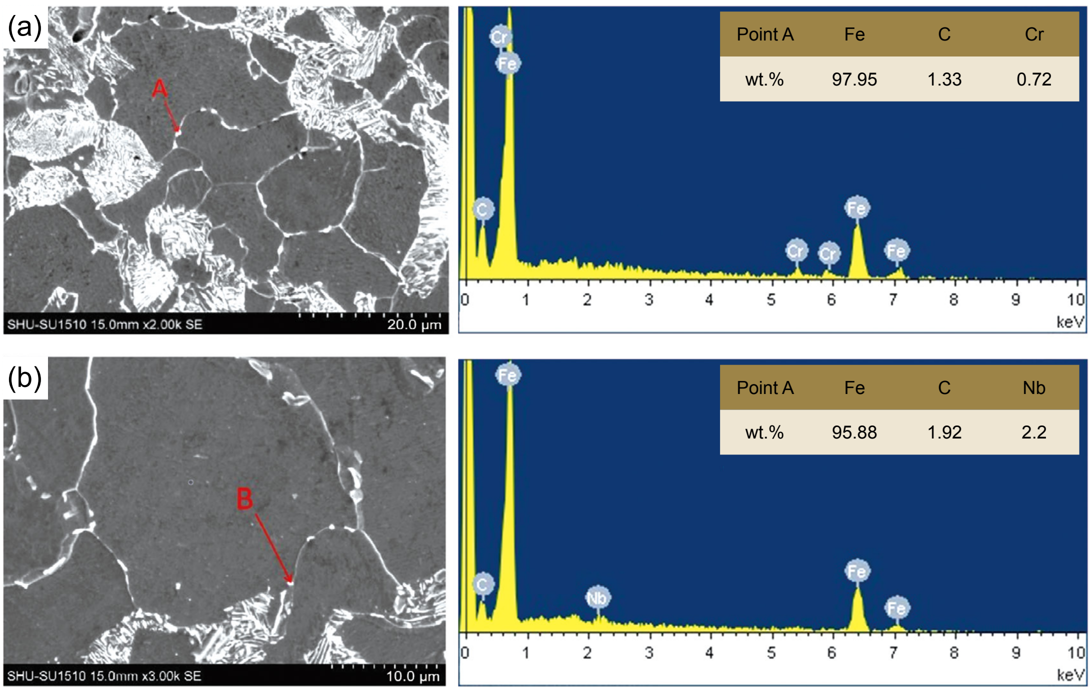

Fig. 5: SEM morphology and energy spectrum of normalized steel samples with different Nb contents: (a) 0 Nb; (b) 0.2% Nb

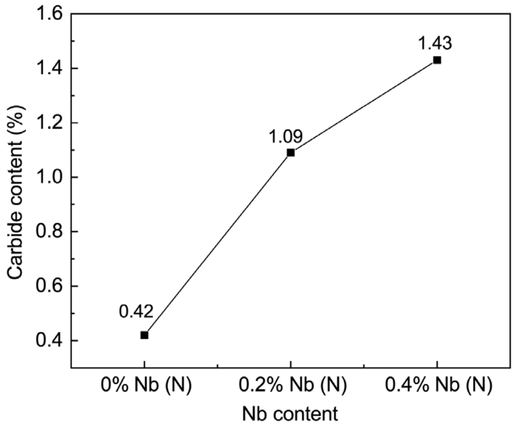

Fig. 6: Carbide content in normalized steel samples with different Nb contents

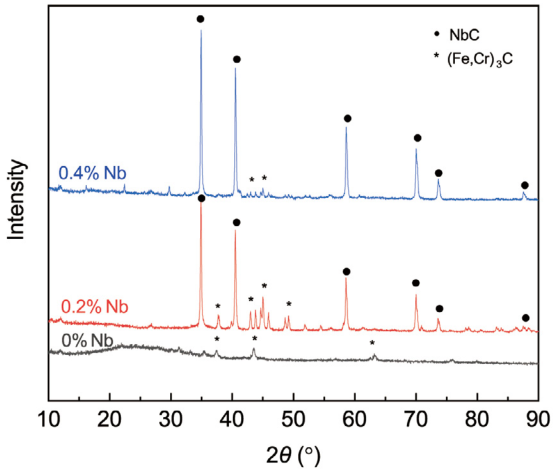

Fig. 7: XRD analysis of carbides in different Nb content normalized steels

Table 2: Change of grain diameter and hardness of test steel with different Nb contents

Nb content (wt.%)	0	0.2	0.4
Grain diameter (μm)	16.67	9.79	10.21
Hardness (HBW)	128.1	139.1	133.0
Main type of carbide	$(Fe,Cr)_3C$	NbC	NbC

In the process of friction and wear, there are complex stress distribution and plastic deformation in the worn surface layer of materials. Meanwhile, the influence of friction heat on the surface layer induced changes in the microstructure of the worn surface layer of materials. The pins after the wear test were cut along the wear direction to explore the influence of microstructure deformation in the wear surface layer on the wear performance, and the SEM morphology of the wear section is shown in Fig. 8. Under the action of normal pressure and shear stress, obvious plastic rheology appears along the wear direction. The closer to the wear surface, the more serious the rheology, and the elongated grain and surface tend to be parallel. These consequences are similar to those of Kato [14] and Trumper [15]. There are various expressions of the deformation layer in the process of the friction deformation layer. At present, the classic and commonly used one is the friction deformation layer proposed by Rice et al. [16-18], which is composed of a severe plastic deformation layer (SPDL), a small amount of plastic deformation layer (PDL), and a matrix layer. In this work, the plastic deformation of Nb-free samples is relatively uniform from the friction surface layer to the base layer, and there is no obvious serious plastic deformation layer. The depth of the plastic deformation layer is about 30 μm. For the steel containing 0.2% Nb, the grain size decreases significantly with the increase of Nb content. Grain refinement improves the toughness of the test steel and the its ability to resist the plastic deformation caused by the normal pressure and shear stress on the wear surface [18]. Furthermore, the carbide with high hardness is evenly distributed and precipitated along the crystal, facilitating the matrix structure to resist the influence of rheology. Therefore, the depth of plastic deformation layer of 0.2% Nb steel decreases to about 20 μm, 33.3% lower than that of Nb-free steel. By observing the wear section [Fig. 8(c)] and scanning energy spectrum (Fig. 9) of the specimen with 0.4% Nb content, it can be recognized that there are many microcracks along the sliding direction near the severe plastic deformation area of the specimen [as shown in the rectangular frame of Fig. 8(c)], and most of the cracks originate in the pearlite structure. This is because that there is NbC coarsening and growth and aggregation in pearlite when the Nb content is too high (Fig. 9). In repeated cyclic wear experiments, the coarse NbC and the pearlite structure with high brittleness are prone to fragmentation

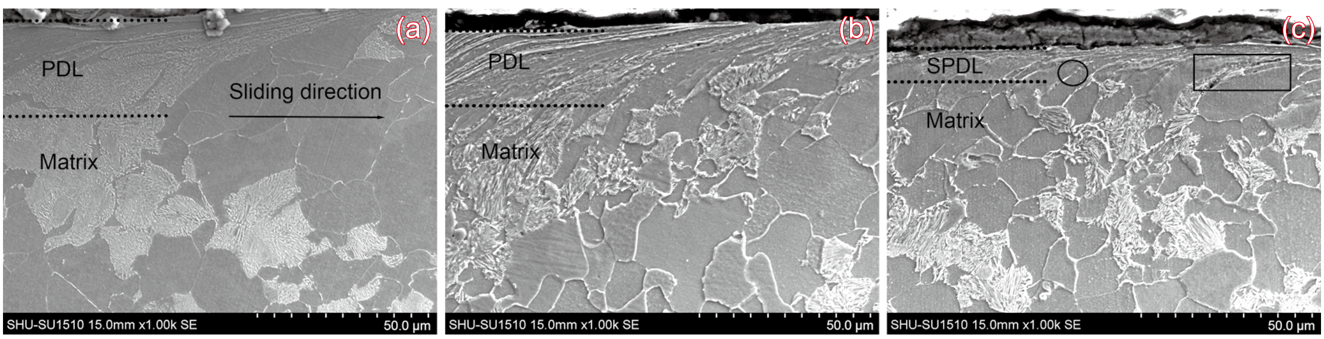

Fig. 8: SEM analysis of wear section: (a) 0 Nb; (b) 0.2% Nb; (c) 0.4% Nb

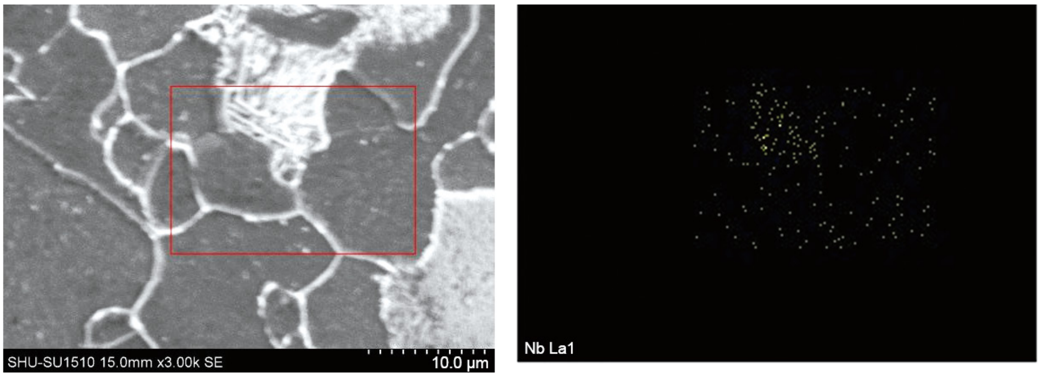

Fig. 9: Scanning energy spectrum of niobium element on the surface of 0.4% Nb normalized sample

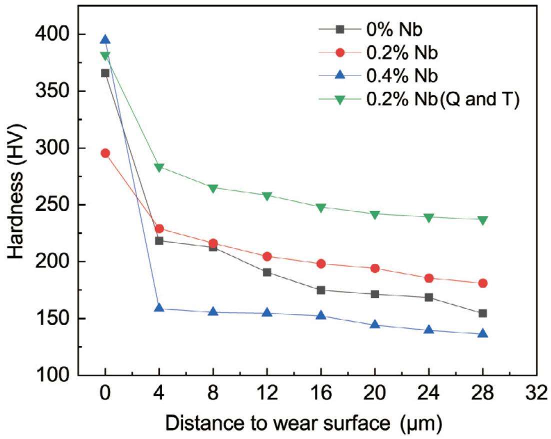

Fig. 10: Microhardness of specimen section with different Nb content

and generate crack sources at the interlayer or ferrite interface. After the crack initiation, under the continuous action of normal stress and shear stress, the crack opening increases and propagates to the surface, resulting in the peeling off of worn surface materials.

In order to explore the mechanical properties of the friction and deformation layer, microhardness tests were performed on the wear section of the samples with different Nb contents and heat treatment process along the depth direction to analyze the strain hardening behavior. The results are shown in Fig. 10. The microhardness values pictured in the figure were regarded as valid measurement results of the ferrite section to avoid the microhardness measurement error of the subsurface layer caused by the heterogeneity of the structure. It can be known that the hardness value decreases and gradually approaches the hardness of the matrix with the increase of the depth of the cross section of the same sample, indicating that the change in the structure caused by plastic deformation gradually decreases with the increase of the depth. The surface hardness of each sample is higher than that of the sub-surface layer due to the work hardening during wear. The hardness of the sample containing 0.2% Nb in the normalized state is higher than that without Nb and with 0.4% Nb. It is mainly related to the fine grain strengthening effect of Nb and the content and distribution of carbides. The hardness test results are consistent with the research conclusions of Tuckart [19] and Singla [20].

3.3 Effect of wear conditions on wear properties of test steel

Figure 11 displays the worn morphology of 0.2% Nb normalized specimen under different wear conditions. As can be observed in Fig. 11, the oxide adhered to the wear surface increases when the load changes from 30 N [Fig. 3(b)] to 50 N and the rotating speed is 100 rpm [Fig. 11(a)]. Meanwhile, it mainly shows adhesive wear and oxidation wear [Figs. 11(c) and (d). When the load is 50 N and the rotating speed changes from 100 rpm to 150 rpm, the oxide surface expands due to wear, and cracks appear on the oxide surface along the wear direction, making the oxide layer fall off from the matrix. The oxide repeatedly forms and falls off, resulting in more severe oxidation wear. On the surface of the matrix, some black island structures are formed by grinding and sintering of spalling debris. The boundary are bright and flat, reflecting the adhesive wear is the main wear form. With the distribution of NbC on the wear surface, the steel with 0.2% Nb maintains good wear resistance, though the change in wear conditions deteriorate the wear resistance of the steel.

4 Conclusions

(1) The addition of Nb to the normalized low carbon steel containing Nb steel can refine the grain size, reduce the plastic deformation of the wear surface, and generate NbC with high hardness, contributing to enhancing the wear resistance of the material. The steel achieved better wear resistance after 0.2% Nb is added. In the same composition, the average wear loss of the quenched and tempered test steel reaches 86.7% of the normalized state.

(2) Due to the fine grain strengthening and the precipitation of NbC, the steel containing 0.2% Nb improves the plastic deformation resistance during the wear process, reduces the depth of the friction-affected layer, and reinforces wear resistance. As Nb content increases to 0.4%, the carbides become coarse, and cracks occur during repeated wear, leading to severe plastic deformation layers and weakened wear resistance.

(3) At the same rotating speed, the rise of load increases the amount of oxide adhering to the wear surface of the

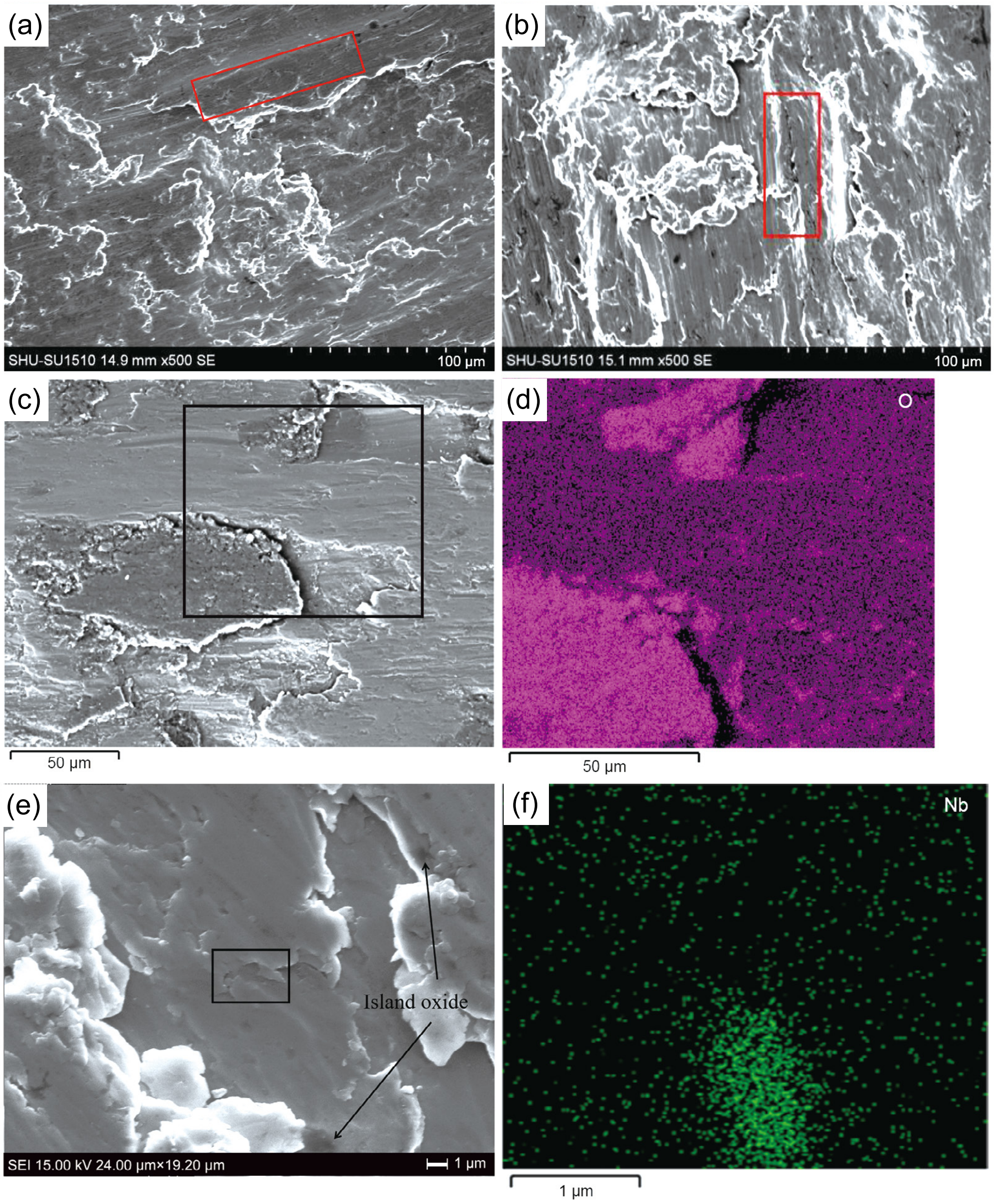

Fig. 11: Wear morphology of 0.2% Nb normalized samples: (a) 50 N-100 rpm; (b) 30 N-150 rpm; (c) 50 N-100 rpm-EDS; (d) EDS analysis of Nb in Fig. 11(c); rectangular frame (e); 30 N-150 rpm-EDS; (f) EDS analysis of Nb in Fig. 11(e) rectangular frame

0.2% Nb sample steel. The wear mechanism changes from slight oxidation wear to adhesion wear and oxidation wear. While, under the same load, the adhesive wear and oxidative wear are worsened by increasing the rotation speed that will make the adhesive wear and oxidative wear continue to be severe, and cracks along the wear direction deteriorates the wear resistance. In a comprehensive comparison, the effect of increased rotate speed is greater compared to the load on the wear behavior of the test steel.

Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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