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Research Letter

Effect of modulation period on the mechanical and tribological behavior of (CrNbTiAlV)CN/MoN coatings

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Abstract
This work is to deposit (CrNbTiAlV)CN/MoN coatings by alternating sputtering with multiple targets. The hardness and elastic modulus of the (CrNbTiAlV)CN/MoN multilayer coatings gradually increase with the decreasing of the modulation period from 420 to 110 nm; however, the values decrease on the multilayer coating with a modulation period of 50 nm. As the modulation period decreases, the characteristic of MoO_3 debris changes from large flakes to smooth transfer tribolayers and then to small particles. The coating with a modulation period of 110 nm has a minimum friction coefficient and wear rate of about 0.11 and 4.4×10^{-6} $mm^3/(N\ m)$ at 500°C, respectively. This can be attributed to the fact that it has the best resistance to plastic deformation and tends to form smooth transfer tribolayers.

Introduction
To extend the service life of moving mechanical parts such as bearings, hinges, and gears, surface coating technologies with a range of physical–mechanical advantages have become one of the available solutions. Advanced hard coatings such as nitrides of transition metals are reported to be effective in improving hardness and wear resistance because of the formation of mixed ion-metal-covalent bonds and nanocomposite structures.[1,2] Given their excellent tolerance of oxidation at elevated temperature, transition metal nitride coatings such as CrN, CrAlN, and TiAlN are good candidates for extending the life of moving parts in high-temperature applications.[3–5] Moreover, the high-temperature tribological properties of the coating are linked with the selected composition. Elements, such as Ti, V, Mo, and W, can generate the Magnéli phase with a layered crystal structure at high temperatures, which can improve the tribological properties of the coating. For example, Tao et al.[6] investigated the effect of Mo doping on the tribological behavior of CrAlSiN coatings, and found that the friction coefficient of the coatings decreased with increasing Mo content. It was found that the oxide lubricant layer formed on the surface of the abrasive tracks had a significant effect on the tribological properties of the coating.

Recently, high-entropy nitride coatings with good mechanical properties, wear resistance, and corrosion resistance have received a lot of attention.[7,8] However, few reports have investigated the high-temperature tribological properties of high-entropy nitride coatings. For example, Lo et al.[9] fabricated (AlCrNbSiTiMo)N coatings by magnetron sputtering technique and found that the average friction coefficient value and the lowest wear rate of the coatings were 0.48 and 1.2×10^{-6} $mm^3/(N\cdot m)$, respectively, in wear tests at 700°C. It was also noted that due to the appearance of the MoO_3 Magnéli phase on the surface during high-temperature wear led to a significant decrease in the friction coefficient at high temperatures. It follows that the addition of Mo elements to high-entropy nitride coatings is of interest for high-temperature tribological applications due to the formation of lamellar structured MoO_3. Besides, Lin et al.[10] investigated the mechanical and tribological properties of $(Cr_{0.35}Al_{0.25}Nb_{0.12}Si_{0.08}V_{0.20})N$ coatings. It was concluded that the mean friction coefficient value and wear rate were 0.63 and 5.4×10^{-6} $mm^3/(N\cdot m)$ at 600°C, respectively.

Nevertheless, there continues to be interest in coatings with further improved mechanical and tribological properties. Multilayer microstructural coatings offer superior mechanical and tribological properties compared to single-layer coatings. The multilayer microstructure is composed of alternating layers with different interface numbers, compositions, and thicknesses. In general, multilayer coatings typically exhibit increased hardness as the modulation period decreases. The increase in performance is attributed to the resistance of the multilayer microstructure to dislocations gliding across the interface.[11] For example, Shao et al.[12] studied the influence of modulation period on the microstructue and mechanical properties of the (AlSiTiVNbCr)N/(AlSiTiVNbCr)CN coatings. It was found that the hardness of the coating increased as the modulation period decreased, which was attributed to a reduction in grain size and an increase in the interface. Yang et al.[13] fabricated the TiN/TiAlN multilayers and found that both the hardness and plastic deformation resistance of the coating increased as the modulation period decreased. Besides, constructing coatings with multilayer microstructures allows for an effective synergy between strength and lubrication properties. The hard high-entropy nitride coatings have high load-bearing capacity to reduce abrasive wear and prolong wear life. In addition, the MoN coatings are anticipated to reduce the coefficient of friction by forming lubricant Magnéli phase.[14,15]

Therefore, in this work, (CrNbTiAlV)CN/MoN multilayer coatings with different modulation period are prepared in order to obtain coatings with excellent mechanical properties and tribological properties. And this study provides an insight into the modulation period for enhanced mechanical properties and high-temperature tribological properties of the coatings.

Experimental details

Coating deposition

(CrNbTiAlV)CN and (CrNbTiAlV)CN/MoN coatings were deposited on Si wafers and 8Cr4Mo4V steels. Prior to coating deposition, the substrate was ultrasonically cleaned using petroleum ether and alcohol in sequence, blown dry, and then placed in the deposition chamber and evacuated. To remove contaminants from the substrate, the substrate surface was first cleaned with Ar^+ bombardment sputtering when the chamber pressure was below 3E-3 Pa. The Cr adhesion layer was deposited preferentially with a deposition current of 4.5 A and a chamber pressure of 0.23 Pa. The (CrNbTiAlV)CN coating was prepared by introducing a N_2 flow rate of 38 sccm. The current of the splicing target and C target were set as 4.5 A and 4 A, respectively. By controlling the deposition time of each modulation period, the MoN coating was introduced onto (CrNbTiAlV)CN layer to construct multilayer structural coating. The deposition time for each modulation period was 40 min, 20 min, 10 min, and 5 min, respectively. And the corresponding modulation periods are 420 nm, 190 nm, 110 nm and 50 nm, respectively. For the convenience of the following discussion, the (CrNbTiAlV)CN coating was named S1, and the samples were named S2, S3, S4, and S5 in descending order of preparation time for each modulation period.

Analysis

The microstructure of the coatings was observed by scanning electron microscopy (JSM-7610F). The hardness (H) and elastic modulus (E) of the coatings were demonstrated by a nanoindenter (NHT3) in load-unload mode. The maximum indentation load was set as 3 mN with a Berkovich tip diamond indenter. The high-temperature tribological behavior was performed on a ball and disk tribometer (MS-HT1000). The Si_3N_4 ball (Φ6 mm) was available as the counterpart for high-temperature applications at 500°C, and the load was 20 N. A three-dimensional surface profiler (UP-Sigma) was used to determine the wear rate of the coatings.[16–18] Energy-dispersive spectroscopy (OXFORD) was utilized for the observation of the elemental composition in the wear tracks.

Results

Figure 1(a–f) presents the cross-sectional microstructure of the coatings. As shown in Fig. 1(a), it can be observed that the thickness of (CrNbTiAlV)CN monolayer coating is about 2.17 µm, and there is a Cr adhesion layer between the coating and the substrate with a thickness of about 200 nm. It is evident that all coatings are well adhered to the substrate and no cracks are observed at the interface. The S1 sample displays a dense microstructure. The multilayer composite coating layer interface is flat, and the periodic structure is clear and dense. The (CrNbTiAlV)CN/MoN multilayer coating mainly consists of periodic alternating light and dark, where the brighter part of the bottom layer near the substrate is the Cr adhesion layer (about 200 nm) to improve the adhesion behavior. The darker part above the Cr adhesion layer is the (CrNbTiAlV)CN layer, and the brighter layer above it is the MoN layer. The bilayer modulation periods are controlled in a range decreasing from 420 to 50 nm.

Figure 1(g) shows the results for hardness and elastic modulus. The hardness and elastic modulus of the (CrNbTiAlV)CN/MoN multilayer coatings gradually increase with the decreasing of the modulation period from 420 to 110 nm; however, the values decrease on the multilayer coating with a modulation period of 50 nm. Due to the difference in shear modulus possessed by each layer of material, the stress required for dislocations to glide across the interface increases, and therefore, dislocation blocking effects occur at the interface between layers. Thus, the high interfacial density of the multilayer structure helps block the gliding of dislocations across the interface between layers. However, as the modulation period continues to decrease to 50 nm, the hardness and elastic modulus of the coating instead decrease. This can be attributed to the lack of interface effect in the coatings.[19,20]

Figure 1(h) shows the trend of the ratio of H to E and H^3 to E^2 with the modulation period. The values of H/E and H^3/E^2 can represent the resistance to wear and plastic deformation, respectively.[21,22] Compared to the S1 sample, the H/E and H^3/E^2 values of the S2 sample decrease, which reflect the decreased resistance to plastic deformation of multilayer-structured coatings with larger periods. However, as the modulation period decreases, the H/E and H^3/E^2 values of the samples first increase and then decrease. This indicates that the elastic energy of fracture and resistance to plastic deformation of (CrNbTiAlV)CN/MoN nano-multilayers are improved by adjusting the modulation period.

Figure 1(i, j) reveals the tribological properties results. The average friction coefficient of substrate at 500°C is 0.33, while the deposition of a single high-entropy nitride coating (S1 sample) does not reduce the friction coefficient in the high-temperature environment. However, the

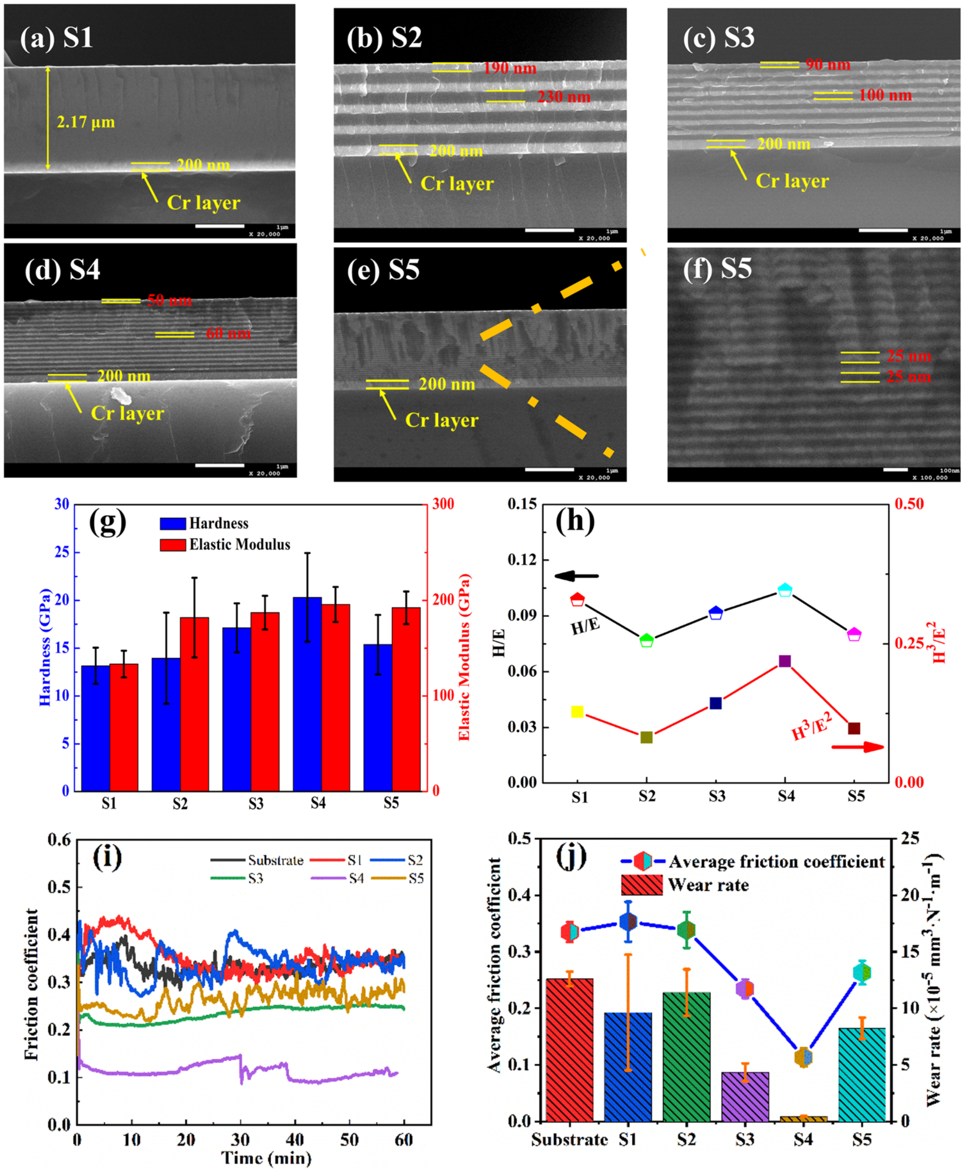

Figure 1. (a–f) SEM cross-sectional images of coatings. (g) Hardness and elastic modulus of coatings. (h) H/E and H^3/E^2 values of coatings. (i) Friction coefficient curves. (j) Average friction coefficient and wear rate of steel substrate and coatings.

friction coefficient of the multilayer composite coating tends to decrease and then increase with decreasing modulation period, where the S4 sample presents the lowest friction coefficient of about 0.11. This indicates that the introduction of MoN coating is able to reduce the friction coefficient of the coating in the high-temperature environment and the lubricating phase is generated at high temperature. The largest wear rate is 1.26×10^{-4} $mm^3/(N{\cdot}m)$ for the substrate in high-temperature environment (500°C). The protective coating is able to reduce the wear rate of the coating, where the S4 sample has the lowest wear rate of 4.4×10^{-6} $mm^3/(N{\cdot}m)$. It is noteworthy that the wear rate of the S4 sample is two order of magnitude lower than that of the steel substrate. However, the wear rate of the coating starts to increase as the modulation period is further reduced. The highest H/E (or H^3/E^2) ratio of the (CrNbTiAlV)CN/MoN multilayer coating with a modulation period of 110 nm indicates its best wear resistance.

The worn surface is further investigatd by FE-SEM to elucidate the lubrication mechanism of the coating. The wear morphology of coatings is presented in Fig. 2. What can be seen is that there are plenty of particles as well as abrasive debris on the S1 and S2 wear track with deeper furrows. This is due to the fact that during the friction process, the particles appear to be peeled off and enter the contact surfaces of the counterpart and are broken into small abrasive particles, which act as pushing or cutting in the subsequent friction and form furrows. In addition, the S1 sample presents some degree of adhesive characteristics, suggesting that an adhesive wear mechanism

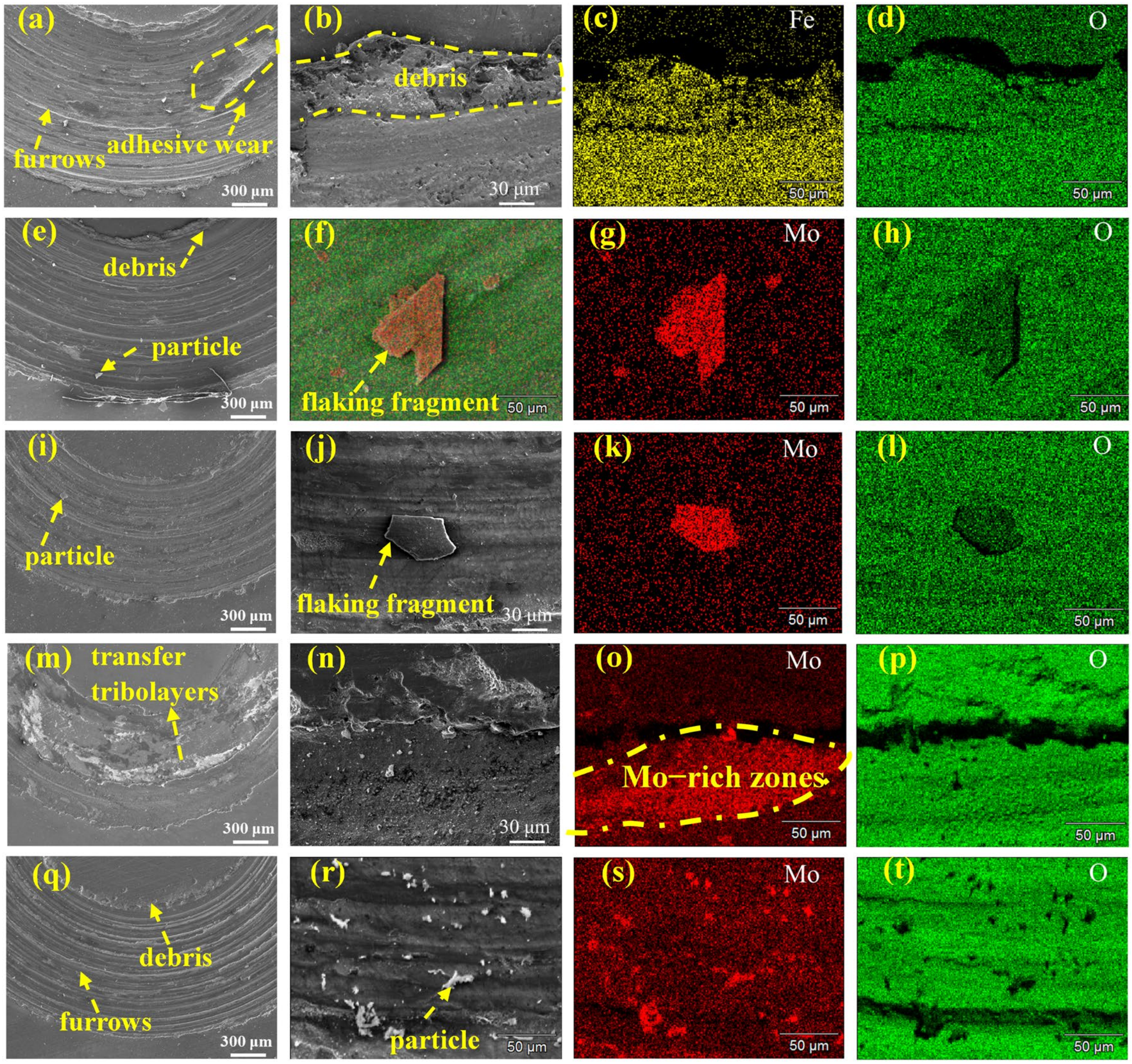

Figure 2. Wear track images and image mapping analysis of (a–d) S1 coating. (e–h) S2 coating. (i–l) S3 coating. (m–p) S4 coating. (q–t) S5 coating.

also occurs. Both S2 and S3 samples show furrows in the wear trajectory and abrasive chip accumulation at the ends of the furrows, showing their obvious abrasive mechanisms. However, judging from the images of the wear tracks in Fig. 2(m), the S4 sample shows a different adhesion characteristic, indicating an adhesive wear mechanism. Wear debris in the center of the wear track is smoothed out and then accumulates in the middle of the wear surface, which helps reduce the friction coefficient of the coating in high-temperature environments. The S5 sample has a plenty of grooves on the surface of abrasive tracks, and the edge of the abrasive tracks shows the accumulation of abrasive chips, which leads to severe abrasive wear, and therefore, it also has a high wear rate.

Image mapping is performed to study the distribution of elements on the wear track. Figure 2 shows the wear track images and image mapping of the coatings. Figure 2(a–d) shows the wear track images and image mapping of S1 coating. As shown in Fig. 2(a–d), a large amount of debris appears at the edge of the abrasive tracks, accompanied by a large number of holes and cracks. Besides, Fe and O are rich in the wear debris. This demonstrates the poor oxidation resistance of a single (CrNbTiAlV)CN coating at high temperatures. For the S2 sample, a plenty of flaking fragments are produced on the surface of the wear tracks. The EDS mapping shows that the fragments are rich in Mo elements, which lead to the inference that MoO_3 particles were produced. The similar phenomenon occurs on the wear tracks of S3 sample. This indicates that the coatings prepared at larger modulation periods are more brittle, easily flake off during friction, and form large abrasive fragments. As the modulation period decreases, no larger fragments are created in the abrasive tracks. The wear track for the S4 sample appears as a smooth transfer tribolayers with some adhered and transferred material. Therefore, the wear mechanism is predominantly adhesive wear. As shown in Fig. 2(o), the Mo-rich zones are formed on the wear track, which serves to reduce the friction. There is no significant change in the O content on the wear track, which indicates a uniform O content distribution and the generation of MoO_3 lubricant phase in the Mo-rich region at 500 ℃. This indicates that the coating is subject to oxidative wear at high temperatures. It is reported that the lamellar oxides of the MoO_3 are regarded as lubricating phases, which can reduce the friction coefficient on the contact surfaces in relative motion.[23] However, as the modulation period further decreases, a high amount of small particles rich in Mo elements appear on the abrasive tracks of S5 coating. The wear mechanism is the combination of abrasive wear and oxidative wear. The abrasive wear generated is detrimental to the tribological properties of the coating.

Figure 3(a) presents the illustration of lubricating mechanisms of the (CrNbTiAlV)CN/MoN coatings with different modulation periods. At larger modulation periods, the two interacting surfaces generate large diameter MoO_3 fragments that scrape or are crushed, resulting in a higher friction coefficient. As the modulation period decreases, an adhesive layer rich in MoO_3 lubricant phase is generated on the friction surface, reducing the friction coefficient of the coating. However, as the modulation period further decreases, a large number of MoO_3 particles do not form a lubricant adhesion layer, but are individually distributed on the coating wear trajectory. This can generate abrasive wear and deteriorate the tribological properties.

To better reflect excellent tribological performance of the multilayer coating deposited in this work, the optimum tribological performance of the coating obtained in this work is compared with that of a single high-entropy nitride coating and a single MoN-based coating prepared by other researchers.[9,10,24–27] As shown in Fig. 3(b), the coatings deposited in this work have excellent synergetic tribological properties compared to the results in the literature. The hard high-entropy nitride coating provides a high load-bearing resistance to abrasive wear, but a high friction coefficient and poor oxidation resistance at high temperatures. The MoN-based coating has good high-temperature lubrication performance and forms shear-prone MoO_3 lubricant phase at high temperature, and the coating has low friction coefficient at high temperature, but poor wear resistance performance. Therefore, the construction of nano-multilayer structure can realize the effective synergy between wear resistance and lubrication performance of the coating.

Conclusions

In this study, (CrNbTiAlV)CN/MoN coatings are fabricated by alternating sputtering with multiple targets. The hardness and elastic modulus of the (CrNbTiAlV)CN/MoN multilayer

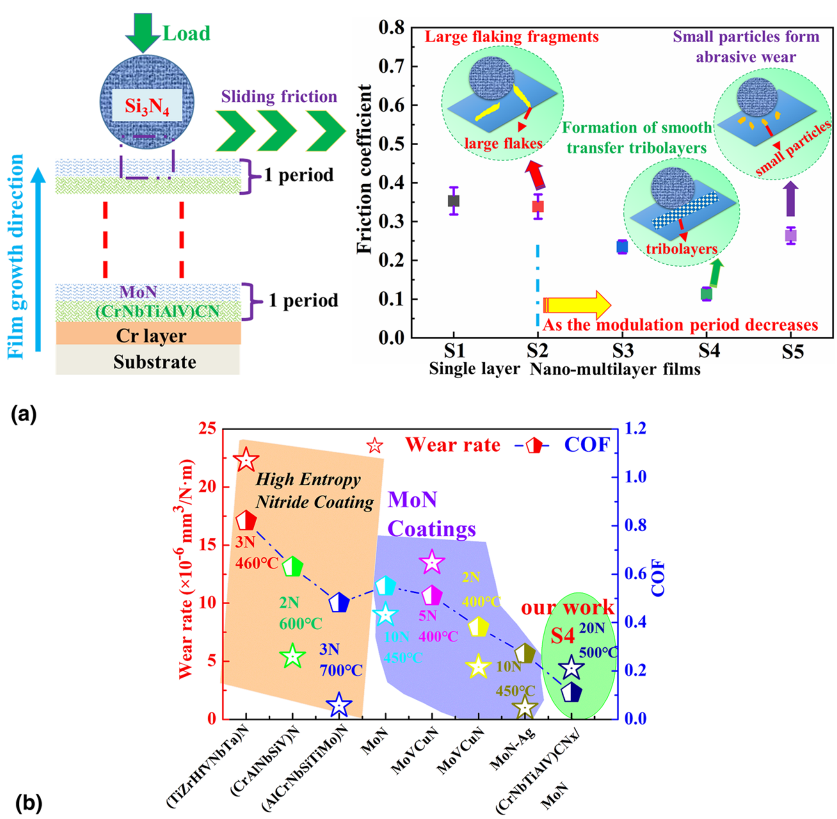

Figure 3. (a) The schematic illustration of lubricating mechanisms of the (CrNbTiAlV)CN/MoN coatings with different modulation periods. (b) Comparison of wear rates of (CrNbTiAlV)CN/MoN coatings with coatings deposited by other researchers.

coatings gradually increase with the decreasing of the modulation period from 420 to 110 nm; however, the values decrease on the multilayer coating with a modulation period of 50 nm. As the modulation period decreases, the characteristic of MoO_3 debris changes from large flakes to smooth transfer tribolayers and then to small particles. It is revealed that the friction coefficient decreases gradually with the decrease in the modulation period from 420 to 110 nm, and the value increases when the modulation period is 50 nm. The coating with a modulation period of 110 nm has the best tribological properties, with the lowest friction coefficient and wear rate of about 0.11 and 4.4×10^{-6} $mm^3/(N\ m)$, respectively, at 500°C. This work experimentally reveals the optimal modulation periods of multilayer coatings in obtaining high hardness and excellent high-temperature tribological properties.

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Author contributions

XL: Methodology, Investigation, Writing—original draft HZ, CW, XZ, and WW: Methodology, Investigation XS and JH: Writing—review and editing, Project administration.

Data availability

The data that support the findings of this research are available from the corresponding author upon reasonable request.

Declarations

Conflict of interest

The authors declare that they have not conflict of interest.

Ethical approval

This study does not contain any studies with human or animal subjects perform by any of the authors.

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