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A Study of the Tribological Properties of Sputter-deposited MoS_X/Cr Coatings

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ABSTRACT

In this investigation, MoS_x/Cr coatings were deposited by direct-current magnetron sputter onto Ck45 (AISI 1045) plain carbon steel substrates. The MoS_x/Cr ratio in the coatings was controlled by sputtering the composite targets. The chemical characterization was performed using EDX (energy dispersive X-ray analysis); the structural characterization was accomplished by X-ray diffraction (XRD) studies. The mechanical properties of coatings were analyzed by nanoindentation experiments. The tribological behavior of the coatings was investigated using the pin on disc test at room temperature. MoS_x/Cr coatings with Cr atomic percentage of 13% was found to possess the optimum wear resistance and durability. The MoS_x/Cr coatings showed a maximum hardness of 12.5 GPa at a dopant content of 13 at.% Cr. Moreover, the films exhibited a steady state friction coefficient from 0.15 to 0.19 and the main wear mechanisms of the MoS_x/Cr coating in air were abrasive, adhesive, and oxidation.

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1. INTRODUCTION

Molybdenum disulfide (MoS_2) is the predominant materials used as solid lubricant has been widely used in many tribological applications. The layered structure is constructed by unit S-M-S atomic tri-layers. The bonding between Mo and S is covalent, and the tri-layers themselves are held together by weak van der Waals forces. This weak bonding between layers of MoS_2 structure is responsible for its good lubricating property [1-3].

MoS_2 coating is strongly influenced by the test atmosphere. It has been mostly used as a solid lubricant in space and vacuum application. MoS_2 is too highly sensitive to humid air. The temperature limitation at 400ºC is restricted by oxidation. The tribological performance of MoS_2 coating depend on structure, thickness, impurity, density and bonding strength and can be controlled by the deposition parameters [4, 5].

In order to improve the properties of MoS_2 coating using the co-sputtering method, several authors have studied adding metals and materials into MoS_2 matrix structure. The metals studied include Au [6, 7], Zr [8, 9], Cu [10], Ag [11], Nb [12, 13], W [14], Ti [15, 16], Ta [12], Cr [15, 17], Al [18].

The composite coatings showed better performance compared to pure MoS_2 coating in terms of hardness, wear resistance and adhesion to the substrate. The addition of Cr into the MoS_2 coating in recent years has drawn attention because of the significant improvement of the oxidation resistance and tribological performance dependent on the humidity in ambient air.

Although the tribological properties of MoS_2 composite coatings have received extensive attention, the levels of metal doped into MoS_2 matrix structure have been seldom addressed. The investigation of wear mechanism of MoS_x/Cr composite coatings, with different chromium content, have not been presented in any paper. In this investigation, MoS_x/Cr composite coatings were investigated to give some insight in the effects Cr doped in MoS_x/Cr composite coatings on the microstructure and mechanical properties and tribological performance in the ambient air.

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2. EXPERIMENTAL

Samples of Ck45 (AISI 1045) plain carbon steel, measuring 10 mm× 5 mm× 2 mm, were used as substrates. The chemical composition of steel substrate is listed in Table 1.

Coupons were polished with 320-grit up to 600-grit paper, ultrasonically cleaned in ethanol and then dried. After mechanical polishing the roughness of surfaces was less than 0.2 µm.

The MoS_2/Cr coating was fabricated in DC magnetron sputtering ion plating equipment (model DST3 – S.

The vacuum system was pumped down to an ultimate base pressure of 5×10^{-4} Torr using a combination of diffusion and rotary pumps. Ar (99.95%) was used as the sputtering gas across the target surface. Prior to deposition, the chamber was pumped down to less than 5×10^{-5} Torr, and the substrates were cleaned in the argon plasma for 10 minutes. The MoSx/Cr ratio in the coatings was controlled by sputtering the composite targets.

MoS_2 (purity 99.8%) and Cr (purity 99.99%) with 0, 5, 10, and 15 wt% composite targets of a 50 mm diameter fixed on a magnetron-effect cathode were used. The composite targets were fabricated by ball milling the mixture of pure MoS_2 and Cr powders, followed by pressing the mixture under a pressure of 60 MPa in an Ar atmosphere at 850K.

The microstructure and chemical composition of the surface and cross-section of the coatings were analyzed using a scanning electron microscopy (SEM) (Camscan MV2300) with energy dispersive spectroscopy (EDS). X-ray diffraction (XRD) was utilized to identify the phases that formed in the surface layer of the coated sample, using Cu Kα radiation (λ=1.5405 Å).

The hardness and Young's modulus were measured using the nano indentation test (CSM) developed by Oliver and Pharr[19]. The maximum indentation depth, load, and max loading and unloading rate were 130 nm, 5000 µN, and 60 mN/min, respectively. Six indentations were applied on each coating and the average value was presented.

The adhesion of the coatings on the substrate was performed using a diamond 90° conical probe with a 100 nm radius of curvature. Each scratch consisted of a 6 µm scratch length and a 30-second duration.

Sliding wear tests were conducted using a pin-on-disc machine. This equipment is controlled by its PC software, which allows the evolution of the friction coefficient to be observed.

TBLE 1. Chemical composition of plain carbon steel

S	Si	Mn	C	Fe	Element
0.02	0.22	0.92	0.15	Bal	Concentration (wt.%)

During the tests, the treated samples were rotating against a stationary AISI 52100 steel pin (with 4.576 mm hemispherical tip radius and hardness of 800 HV_{30}) at a linear speed of 0.1 m/s under a load of 3, 5 and 7 N.

The wear test was carried out at a constant sliding velocity of 0.01 m/s (70 rpm) and under three normal load of 5 N. The friction coefficient values were measured by a load cell equipped with the pin-on-disc apparatus. The SEM/EDS techniques were used to determine the wear mechanisms.

3. RESULTS AND DISCUSSION

An example spectrum from EDS elemental analysis of MoS_2/Cr coatings is given in Figure 1. As it can be seen Sulfur and Molybdenum peaks are overlapped due to the energy levels of the Mo-L*α* and S-K*α*.

The quantitative results of elemental analyses of coatings determined by EDS are presented in Table 2. The Oxygen in the coatings probably was incorporated during the deposition as a contamination. The coatings are formed as nonstoichiometric MoS_x, in which the value of x is in the range of 1.5 to 1.9. The sulfur deficiency is caused by either the Resputtering of sulfur atoms of the growing film though the impact of particles with high energy, or by the chemical reactions of sulfur with the residual atmosphere [20].

The stoichiometric ratio and Oxygen content of MoS_2/Cr composite coatings are shown in Figure 2. As shown, with an increase of the Cr content, the number of sulfuratoms to themolybdenum atoms increases from 1.2 (pure MoS_2) to 1.8 (MoS_2-Cr, 20 at.% Cr). Due to the preferential re-sputtering effect of sulfur, oxygen easily substitutes the sulfur deficient sites and forms the Mo-O-S structure. Chromium atoms move into interstitial or substitutional of MoS_x during coating deposition and act as the barrier for oxygen diffusion and lead to decrease of the oxygen content [21]. Figure 3 shows XRD pattern of pure MoS_X coating.

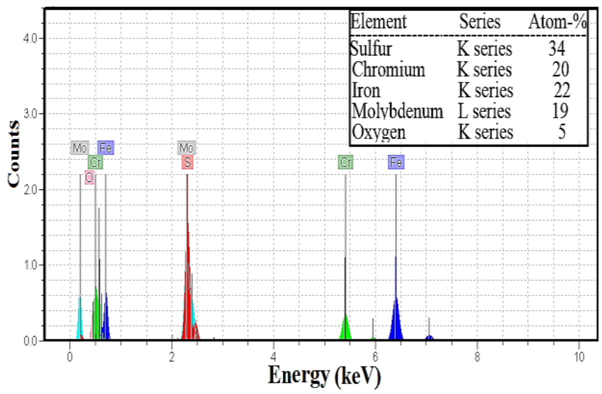

Figure 1. An example EDS analysis of film composition

TABLE 2. Film thickness, elemental composition for various MoS_x/Cr coatings

Coating	Film thickness (µm)	Elemental composition (at.%)				
		Cr	Fe	Mo	S	O
MoS_x	4	0	37	23	28	12
MoS_x/Cr 7 at.%	5	7	22	28	33	10
MoS_x/Cr 13 at.%	5	13	28	23	27	9
MoS_x/Cr 20 at.%	5	20	22	19	34	5

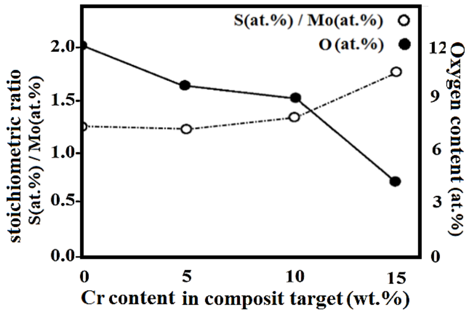

Figure 2. The stoichiometric ratio and Oxygen content of MoS_x/Cr composite coatings with different Cr content.

In addition to the diffraction peak that arose from the substrate, peaks were evident at approximately 2θ from 14°, 32°, 33°, 35° and 39° for the pure MoS_2 coating, which is assigned respectively to the MoS_2 (002), (100), (101), (102), and (103) planes (according to JCPDS-ICDD card No 87-2416).

Pursuant to chemical stability and crystallinity, the MoS_x coatings are classified into three types: type I, type II and amorphous coatings. Type I films have randomly oriented basal planes, with a large proportion of the basal planes perpendicular to the substrate (edge plane orientation). Type II films have a parallel basal plane orientation. The MoS_x coatings with the basal planes parallel to the sliding direction not only supply good lubrication properties but are also more resistant to oxidation given that the edge sites are protected [22, 23]. MoS_x coating is further identified as random-oriented coating.

Since the peaks after 60° in the MoS_X coating have very weak intensities, XRD patterns are reported only between 10° and 50° in Figure 4 MoS_x/Cr coatings. The shape of the broad reflection in the 10–50° range is very similar to that found by Rigato et al. [14] and is ascribed to random stacking of S-Mo-S sandwich layers in the structure. When Cr is added, more than 13 at.% to the MoS_2, the picks reflection of MoS_2 disappear and only the substrate material and pure chromium are detected in the XRD patterns of the MoS2/Cr coatings.

From the coatings of XRD patterns, it can be concluded that the Cr-Doped MoS2 Composite Coatings are characterized by a very disordered microstructure consisting of randomly distributed. The degree of crystallization of the Cr-Doped MoS_2 composite coatings decreased with an increase of Cr content, and the structure of the MoSx/Cr composite coatings turned into the amorphous structure. There is no clear evidence of Cr sulfides or mixed Cr–Mo sulfides in the XRD patterns which may be due to the content of these phases that are too low to be detected. Moreover, no considerable scattered intensity of Mo and Cr oxides were detected.

Figure 5 shows the hardness and Young's modulus of the MoS_2/Cr composite coatings as a function of the Cr content.

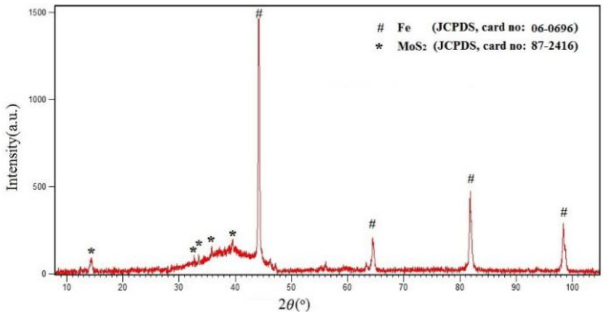

Figure 3. XRD patterns of MoS_2 coating

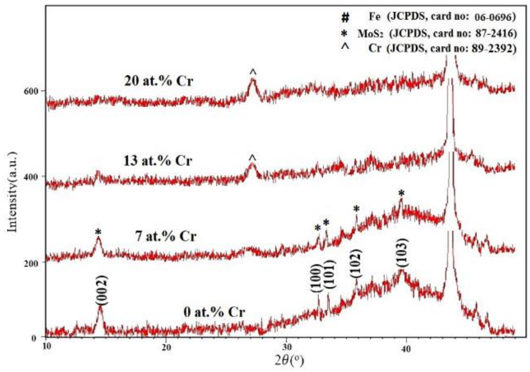

Figure 4. XRD patterns of MoS_2/Cr composite coatings with different Cr content

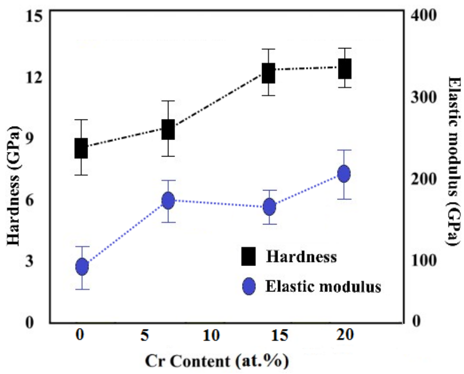

Figure 5. The hardness and elastic modulus results of the MoS_x/Cr composite coatings as a function of Cr content

As can be seen, increasing the Cr content from 0 to 13 at.% led to the significant increase of hardness of the coatings. For the pure MoS_2 coating, the hardness was only about 8 GPa, while with the Cr content of 13 at.%, it increased to13 GPa. Beyond this threshold value of 13 at.% Cr, the over rich doped soft Cr atoms in turn caused the structure deterioration and led to the decrease of hardness and young's modulus.

Addition of chromium content more than 13 at.% caused the MoS_2 lattice to be distorted. Lince et al. [24] reported that the doping of Cr atoms in the MoS_2 coatings caused a denser coating structure. The hardness increase in the MoS_2/Cr composite coating could be understood by the solid solution hardening effect and can be attributed to its dense structure. When the Cr doping content increased more than 13 to 20 at.%, the coating hardness remain constant. This could be due to either the structure deterioration or the possible formation of discrete metallic particles.

Figure 6 shows the critical load of the MoS_x/Cr composite coatings as a function of the Cr content. 3-D in-situ SPM image of MoS_2/Cr coating after a 4000 µN ramping force nanoscratch test is shown in Figure 7. Since the scratch groove depth is less than 1 µm the strength of adhesion is higher than coating cohesion. Thus, it can be deduced that Cr concentration seemed to play a significant role in coating cohesion.

As can be seen in Figure 6, the addition of Cr content from 0 to 13 % led to significant increase of the adhesion of the coatings. Further increase in Cr content from 13 to 20 % resulted in the densification deterioration of the coating, which in turn leads to lowering the coating hardness and adhesion.

In order to investigate the effect of doped Cr content on the tribological behavior of the MoS_2/Cr composite coatings, pin-on-disc friction tests were performed in the ambient air. During the wear tests friction coefficients were continuously recorded. Figure 7 shows the variation of the friction coefficient for MoS_x (Figure 8(a)) and MoS_x/Cr (Figure 8(b)) as a function of wear distance.

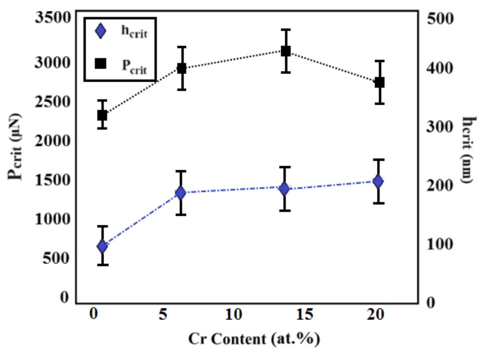

Figure 6. Adhesion test results of MoS_x/Cr composite coatings with different Cr contents

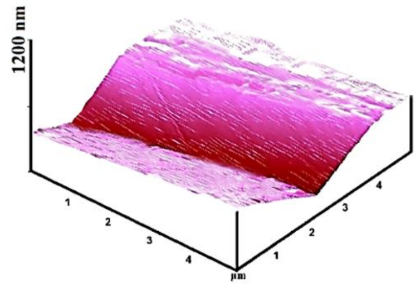

Figure 7. 3-D in-situ SPM image of MoS_x/Cr, 13 at.% Cr after a 4000 µN ramping force nanoscratch test

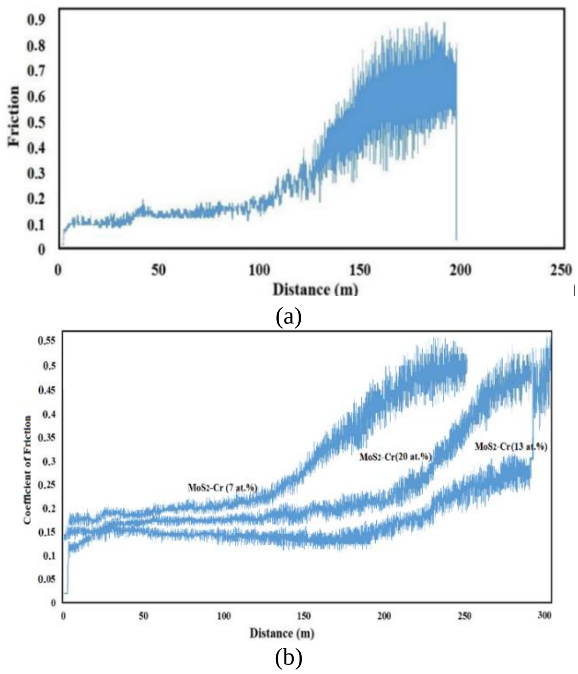

Figure 8. Variation of the friction coefficient as a function of wear distances for (a) Pour MoS_x and (c) MoS_x/Cr composite coatings during the pin-on-disc wear test.

The lifetime of the pure MoS_x coatings is short and is deteriorated after a few friction cycles and the friction coefficient rapidly increased from 0.1 to 0.8 in less than 50m corresponding to the sharp increasing in the friction coefficient. This phenomenon may be ascribed to the bad adhesion between the coating and substrate according to the result from scratch tests (Figure 6). As can be seen in Figure 7 the friction coefficient curves of MoS_x composite coatings can be divided into two parts. In the first part, the friction coefficient was low and stable, with the value ranging from 0.15 to 0.2. The second part is a fluctuating curve with a high friction coefficient. As the sliding distance increases, further micro cracks developed, and wear debris was generated so friction coefficient increased. At the end of this stage (170m), the accumulation of large wear particles initiated, and led to increase in the frictional noise and vibration values. The frictional noise of the MoS_x/Cr coatings is much lower than the pure MoS_x coating.

Figure 9 shows the average friction coefficient of MoS_2/Cr composite coatings with different Cr contents. As can be seen, with the increase of Cr content, the average friction coefficient of the coating reduced from 1.85 (pure MoS_2) to 1.55 (MoS_2-Cr, 13 at.% Cr). Doping Cr (13 %) into the MoS_x coatings presented a relatively steady and low friction coefficient that was lower for the pure MoS_x. Furthermore, Figure 9 shows the endurance of coatings under the load of 5 N as a function of Cr content. Addition of Cr to MoSx/Cr (<13 at.%) increased in the endurance of MoS_2 coatings. The MoS_2 coatings failed after approximately 900 cycles during the pin-on-disc wear tests. The loss of endurance for MoS_2 is believed to be related to the reaction with oxygen and counter-face materials, which change the wear mode of the coating and provides no lubrication effect [25].

Figure 10 shows the wear coefficients of MoS_x/Cr composite coatings. Within the Cr content region of 0–13 at.%, increasing the Cr content led to a significant decrease of wear coefficient of the coating. This indicates that the doped Cr improved the tribological properties of pure MoS_x in the air environment. A reasonable explanation is that with an increase in chromium content to 13 % both hardness and adhesion of the MoS_x/Cr coatings is increased.

In order to determine dominant wear mechanisms, the wear tracks of pure MoS_2 and MoS_2/Cr with 13% coatings which were tested at the normal load of 5 N and after 800 wear cycles were examined by SEM and EDX. As can be seen in Figure 11(a), many wide and deep grooves were found in the direction of slip. These grooves parallels are also typical features associated with abrasive wear, in which hard oxide particles between the pin and disc, plough or cut into the coating surface causing wear by the removal of small particle. In addition of grooving, some locally plastic deformation can be seen in Figure 11(b). Worn surface of the MoS_x/Cr coating is smoother and shows fine grooves and significant plastic flow. However, severe plastic deformations were existed in MoS_x/13 at.% Cr coating. The main wear mechanisms in the MoS_x and MoS_x/Cr coatings were therefore abrasive and adhesive, respectively.

Furthermore, the EDS analysis of the wear tracks showed that they had a high level of oxygen, which indicated tribochemical wear in the sample. The formation of oxidation products led to an increase of coefficient friction and a decrease in wear life in the MoS_x composite coatings. The presence of chromium atoms within the MoS_x structure prevented the erosion of the water vapor and oxygen. With increasing Cr content, more MoS_x was protected. The major wear mechanism in MoS_x coatings is generally a mixture of abrasive and oxidation reaction. In general, the loose structure of MoS_x is prone to water adsorption and easily oxidized in the ambient environment, causing a decrease of coating friction life time and an increase in friction coefficient.

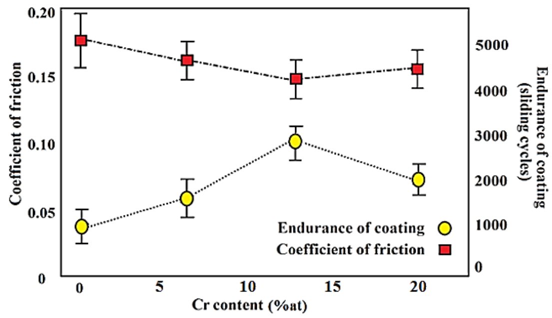

Figure 9. Average friction coefficient and endurance of coatings test results of MoS_2/Cr composite coatings with different Cr contents

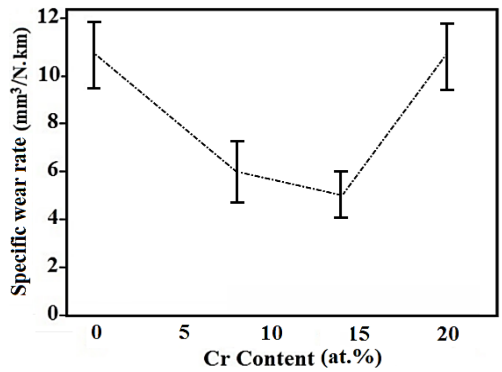

Figure 10. Wear coefficient of MoS_x/Cr composite coatings as a function of Cr content

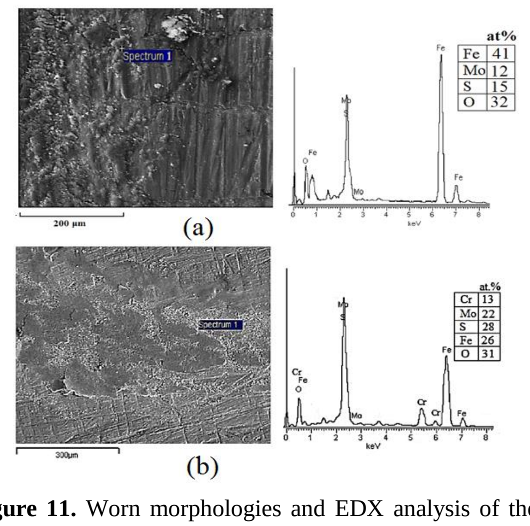

Figure 11. Worn morphologies and EDX analysis of the (a) pour MoS_x (b) MoS_2/Cr, 13 at.% Cr after 800 wear cycles

The appropriate chromium addition led to high coating adhesion and hardness combined with the densification. In addition, some investigation suggested that the formation of chromium oxides in surface of the coating could effectively prevent the oxidation of MoS_x; thus, improving the wear lifetime of the coating [17, 26, 27].

4. CONCLUSION

In this investigation, MoS_x/Cr composite coatings with Cr contents varying from 0 to 20 % were deposited onto steel substrate using a DC magnetron sputter process. The following conclusions can be drawn from the results:

1. The properties of MoS_2 coatings can be significantly improved through the codeposition of an appropriate amount of chromium.
2. Within the Cr content region of 0-13 at.%, increasing the Cr content led to a significant increase in cohesion strange and hardness of coatings.
3. A Cr metal atomic percentage at 13 % in the MoS_2/Crx% coating was found to possess the optimum wear resistance and durability.
4. The degree of crystallization of the MoS_2/Cr coatings decreases with an increase in chromium content.
5. Appropriate doped Cr (13 at.%) led to high hardness and adhesion combined with the densification and compaction of the coating.
6. The MoS_x/Cr coatings exhibited a steady state friction coefficient from 0.15 to 0.19.
7. Adding Cr to MoS_x coatings increased cohesion strange and hardness of coatings. It also increased the wear performance of MoS_x coatings under atmospheric conditions.
8. The main wear mechanisms in the MoS_x and MoS_x/Cr coatings were abrasive and adhesive, respectively.

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Solid Lubricant

چکیده

در این تحقیق پوشش‌های MoSx/Cr به روش کندوپاش مغناطیسی جریان مستقیم روی زیرلایه فولاد کربنی ساده Ck45 (AISI 1045)رسوب گردید. نسبت MoSx/Cr در پوشش‌ها با تغییر ترکیب ماده‌های هدف کنترل گردید. .ترکیب شیمایی پوشش‌ها با استفاده از طیف‌سنجی پراش انرژی پرتوایکس (EDX) و ساختار فازی با استفاده از پراش پرتوایکس(XRD)بررسی شد. با استفاده از آزمون نانو فرورونده خواص مکانیکی پوشش‌ها مورد بررسی قرار گرفت. رفتار تریبولوژی پوشش با استفاده از آزمون پین روی دیسک در دمای اتاق ارزیابی شد. نتایج نشان داد که بالاترین پایداری و مقاومت سایشی با افزودنکروم به میزان 13 درصد حاصل می‌گردد. بالاترین میزان سختی پوشش با افزودن کروم به میزان 13 درصد اتمی ایجاد گردید. مقدار ضریب اصطکاک پوشش‌ها از 0/15 تا 0/19 متغیر بود و سایش خراشان، چسبان و اکسیداسیون به‌عنوان مهم‌ترین مکانیزم‌های حاکم بر سایش پوشش MoSx/Cr تعیین شد.

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