Tribological properties of MoS_2 thin films deposited by RF magnetron sputtering technique

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Abstract: This study aims to reduce the friction and wear losses of AISI440C steel, which is preferred in some machine elements like bearing balls, valves, oil pumps, cutters, springs, cams and scissors. The AISI440C substrates were coated with thin film MoS_2 which provides low friction and wear. During the coating, steel disc groups were obtained by changing the parameters of deposition including the radio frequency sputtering power and the deposition temperature. The thickness value of the lubricating MoS_2 films were determined with a scanning electron microscope. The hardness and elasticity modules of the MoS_2 coatings were determined. The data showing friction coefficient-distance relation of wear tests was obtained and the average friction coefficients, wear amounts and wear trace widths were reached. In this way, how changes in substrate deposition temperature and RF sputtering power affect tribological properties were interpreted. As a result, tribological optimisation of the most suitable coating pair parameter was provided.

Keywords: thin film coating; physical vapour deposition; wear; friction.

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1 Introduction

Solid lubricant coatings are commonly used (Singh et al., 2015) including molybdenum disulfide, graphene derivatives, polymers, soft metals and so on. Sufficient adhesion strength is obtained by using surface modification techniques namely physical vapour deposition (Navinsek et al., 2002; Xu et al., 2004). Effect of applied load and sliding speed on wear mechanism has been investigated (Sentyurikhina et al., 1987). In the literature, there are studies regarding the change in tribological properties of lubricant coatings at different temperature points (Dellacorte et al., 1999).

Unbonded films are mostly used for space applications. Since the pioneering work of Spalvins, sputtered films are primarily used. The conditions of the deposition process is related to the chemical and physical properties of MoS_2 layers (Spalvins, 1969, 1984). Therefore, the coefficient of friction of MoS_2 should be less than 0.05 (Vadiraj and Kamaraj, 2010; Vadiraj et al., 2011). On the other hand, the tribological measurements of the film coatings were evaluated using a tribometer. In dry atmospheric conditions, the coefficient of friction of the MoS_2 film coating is about to 0.05 at Ar RH = 75%, the wear rate of the MoS_2 coating was 2.20×10^{-5} mm^3/Nm (Ding et al., 2010). The AISI440C stainless steel, ball bearing with sputtered MoS_2 films were evaluated in a vacuum bearing chamber. Bearing test results in vacuum showed that endurance lives of more than 1,000 hours were obtained with bearings directly sputtered with MoS_2. When air was admitted into the bearing chamber, the bearing failed approximately in an hour (NASA, 1975).

RF sputtered MoS_2 coatings exhibit lower coefficients of friction and wear rates (Waghray et al., 1995). These RF-sputter deposition techniques can alter certain coating properties which control friction and wear behaviour (Singer et al., 1996).

A study was carried out regarding sources of contamination. The quality of the coating can be affected by impurities created by undesirable chemical reactions which can occur during the deposition process due to impurities existing in the residual gas atmosphere (Buck, 1986). Even traces of water during sputtering lead to the substitution of sulphur by oxygen on lattice sites. The structure of the resulting $MoS_{2-X}O_x$ films is the usual MoS_2 2H structure; the dimensions of the unit cell remain unchanged. Geometrical considerations via the smaller atomic diameter of oxygen in comparison to sulphur can explain a decrease in the lattice constant a_0 with increasing oxygen content. The increase in the lattice constant c_0 with rising oxygen content is caused by a change in the density of states of the electrons. A variation in the growth velocities in the 001 and 100 directions with a varying contamination parameter causes the change in texture at an extremely low level of water contamination (Buck, 1991).

In this paper, it is aimed to reduce the wear and friction losses. Therefore, the AISI440C substrates are deposited with thin film MoS_2 which provides low friction and wear. The experimental methods, materials and results are described below.

2 Experimental methods and materials

All AISI440C substrates, 32 mm in diameters, were polished and ultrasonically rinsed in alcohol for 10 minutes and then dried in air.

By using the physical vapour deposition system with an RF magnetron sputtering technique, MoS_2 film coatings were deposited on to AISI440C steel substrates. During coatings; RF powers of 100, 140 and 200 W were determined to apply on the surface of AISI440C. The deposition parameters are tabulated in Table 1.

The morphology of cross sections of MoS_2 coatings and film coating thickness were investigated by a scanning electron microscope (SEM). Moreover, the hardness and elasticity modulus measurements were carried out.

Table 1 The parameters of RF magnetron sputter deposition

Coating parameter	*RF power (W)*	*Substrate temperature (°C)*	*Flow rate of Ar (sccm)*	*Deposition time (min)*
1.1	100	25	30	50
1.2	100	70	30	50
1.3	100	120	30	50
1.4	140	25	30	50
1.5	140	70	30	50
1.6	140	120	30	50
1.7	200	25	30	50
1.8	200	70	30	50
1.9	200	120	30	50

Tribological tests were performed through Anton Paar Nano Tribometer. AISI420 steel was used as abrasive balls. AISI440C steel sputtered with MoS_2 film in different coating parameters under 1 mN and 5 mN loading stages, maximum linear speed of 0.3 and 0.6 mm/s, 0.10 mm full amplitude, total 0.5 metre wear distance, 25°C ambient temperature and 30% humidity and total abrasion tests were performed at 2,500 cycles. For each accumulated RF power, the hardest MoS_2 coatings obtained as a result of hardness measurement were subjected to tribological tests. The samples with 1.3 coating parameters deposited at 100 W, 1.6 coating parameters deposited at 140 W and 1.9 coating parameters deposited at 200 W are the hardest samples of the powers they are accumulated. The average friction coefficients were reached for the maximum linear speeds of 0.3 and 0.6 mm/s of the loading stages of these film coatings of 1 mN and 5 mN, and the wear rates and wear trace widths were determined. Graphs of variation of friction coefficients according to the same loads and maximum linear speeds of film coatings deposited in different parameters are presented in the following section.

3 Results and discussion

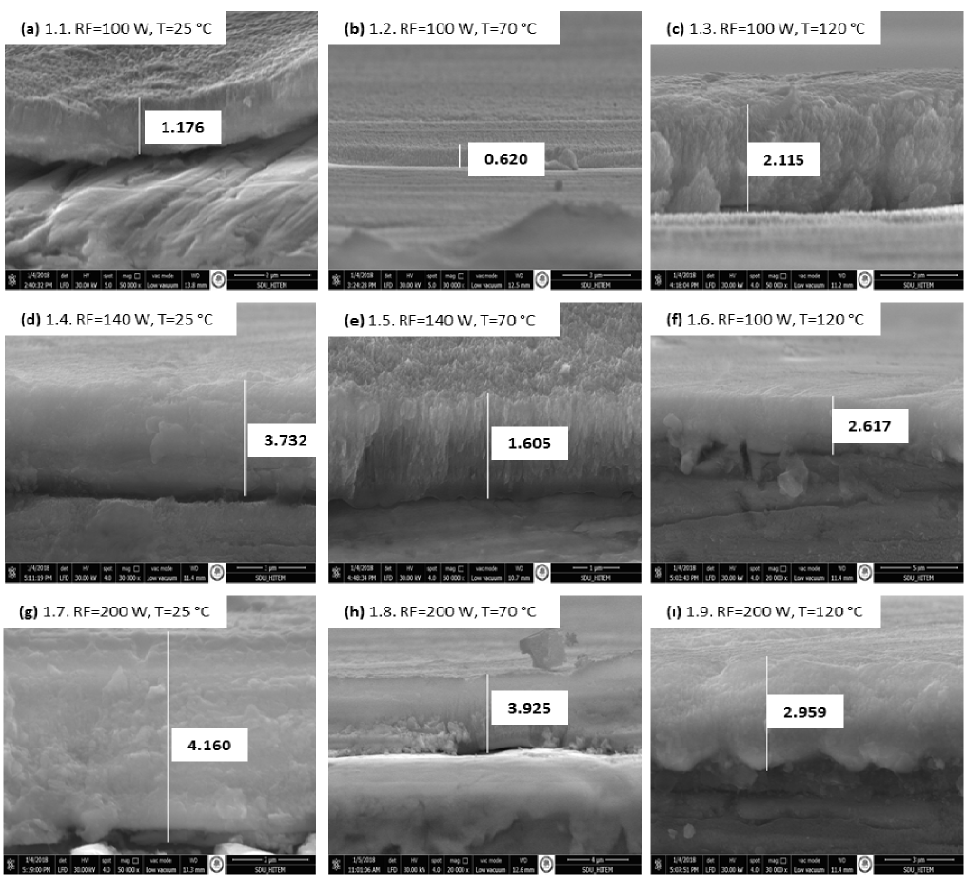
The thickness measurements presented in Figure 1 were taken from a single point. As a result of these SEM section images, Table 2, which shows the thicknesses, was constructed. Table 2 also shows the average values of the hardness and elasticity modulus of films (Poyraz, 2019).

As seen in Figure 1, the thickness values of MoS_2 film coatings were obtained between 0.62 micrometers and 4.16 micrometers. At the same deposition temperature; increasing the thickness of the coating by increasing the radio frequency sputtering power was consistent with the literature (Arslan et al., 2005; Jiao et al., 2018).

Table 2 Mechanical properties of deposited films

Coating parameters	*Hardness (H) (GPa)*	*Elasticity (E) (GPa)*	*Thickness (µm)*
1.1	0.7901	89.7471	1.1763
1.2	1.1032	67.6150	0.6202
1.3	1.3861	118.0711	2.1152
1.4	1.0353	68.1591	3.7321
1.5	1.7562	94.3022	1.6053
1.6	3.6624	132.4683	2.6172
1.7	1.5722	89.0722	4.1604
1.8	1.7951	87.7021	3.9251
1.9	3.7972	108.7424	2.9593

Figure 1 SEM cross-section views of MoS_2 films, (a) parameter number 1.1 (b) parameter number 1.2 (c) parameter number 1.3 (d) parameter number 1.4 (e) parameter number 1.5 (f) parameter number 1.6 (g) parameter number 1.7 (h) parameter number 1.8 (i) parameter number 1.9

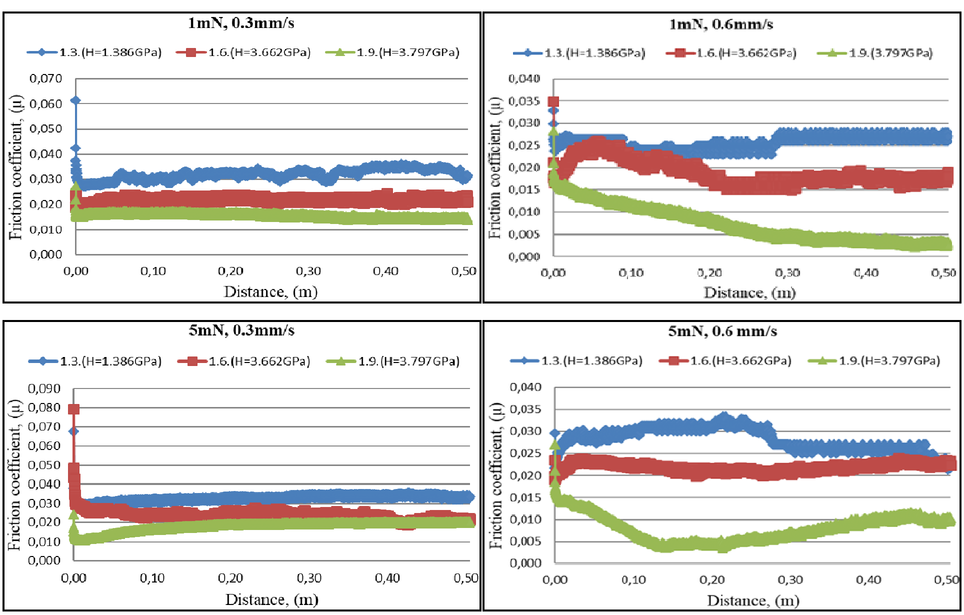


The lowest and highest values of hardness were obtained as 0.79 GPa and 3.79 GPa, respectively. The hardest values were obtained as 1.386 GPa at 100 W, 3.662 GPa at 140 W and 3.797 GPa at 200 W. Coatings of this hardness deposited at coating parameters 1.3, 1.6 and 1.9 respectively were obtained at 120°C, the highest deposition temperatures at the radio frequency power at which they were deposited. At room temperature, 70°C and 120°C deposited coatings; the hardest coating values of the temperatures at which they were deposited were obtained at 200 W, the highest radio frequency sputtering power. Due to the increase of deposition temperatures and radio frequency sputtering power, increasing the hardness of the coating, the hardest MoS_2 coating 1.9. Coating parameters were obtained.

The nano hardness of the MoS_2 film coating is 1.50 GPa (Zhu et al., 2012). The value of the hardness for the MoS_2 layer was 5.20 GPa (Chien et al., 2010). In this study, the nano hardness values obtained by the radio frequency magnetron sputtering technique were lower when compared to the direct current UMS, and this study is again compatible with the literature.

Figure 2 shows the mean coefficient of friction-distance graphs of the hardest sputtered with MoS_2. Wear rates of the coatings under applied loads and speeds are given in Figure 3. The comparative wear rates are collectively shown in Figure 4. The abrasion trace widths are given in Figure 5 (Poyraz, 2019). The friction coefficients, wear rates and abrasion trace widths are given in Table 3. Graphs showing the effect of speed changes under the same load and load changes under the same speed on the coating wear rates are given in Figure 6.

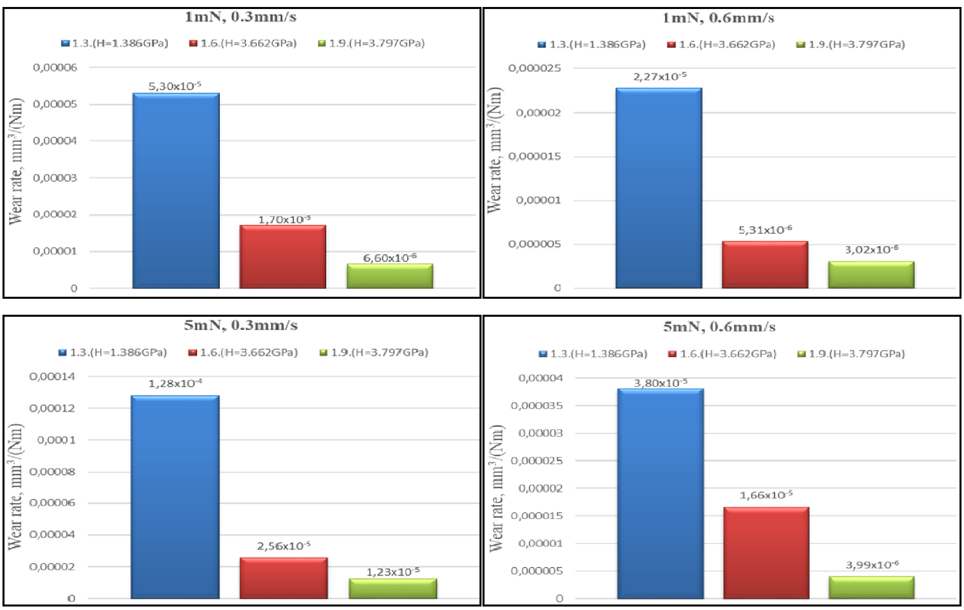
Figure 2 Friction coefficient-distance graphs of MoS_2 coatings (see online version for colours)



It is seen from Figure 2 that the average coefficients of friction of coatings with a coating parameters of 1.3, 1.6 and 1.9 under a load of 1 mN and a speed of 0.3 mm/s are 0.0320, 0.0219 and 0.0157; under 1 mN load and 0.6 mm/s are 0.0262, 0.0188 and 0.0073; under 5 mN load and 0.3 mm/s are 0.0329, 0.0240 and 0.0181; under 5 mN load and 0.6 mm/s are 0.0272, 0.0218 and 0.0080 respectively.

As seen in Figure 3, the wear rates of coatings with 1.3, 1.6 and 1.9 coating parameters under 1 mN load and 0.3 mm/s speed are 0.0000530, 0.0000170 and 0.00000660 mm^3/Nm; under 1 mN load and 0.6 mm/s are 0.0000227, 0.00000531 and 0.00000302 mm^3/Nm; under 5 mN load and 0.3 mm/s are 0.000128, 0.0000256 and 0.0000123 mm^3/Nm; under 5 mN load and 0.6 mm/s are 0.0000380, 0.0000166 and 0.00000399 mm^3/Nm respectively.

Figure 3 Wear rates of MoS_2 coatings (see online version for colours)



The hardest film with 1.9 coating parameters can be seen from Figure 4, where it gives the lowest wear rates compared to other MoS_2 coatings. It shows that the lowest wear rate is 3.02×10^{-6} mm^3/Nm in MoS_2 coating deposited in 1.9 coating parameter at 1 mN load and 0.6 mm/s speed. The highest wear rate was determined as 1.28×10^{-4} mm^3/Nm in 1.3 coating parameters at 5 mN load and 0.3 mm/s speed. It has been observed that coating wear rates decrease with increasing hardness.

As it shown on Figure 5, it is seen that the wear trace widths of coatings with coating parameters 1.3, 1.6 and 1.9 under a load of 1 mN and a speed of 0.3 mm/s are 33.80, 22.80 and 19.80 µm; under a load of 1 mN and a speed of 0.6 mm/s are observed to be 30.30, 16.10 and 14.20 µm; under 5 mN load and 0.3 mm/s speed are 37.30, 33.80 and 27.10 µm; under 5 mN load and 0.6 mm/s speed are 32.80, 20.40 and 18.80 µm, respectively. Tribological tests showed that harder MoS_2 coatings give smaller abrasion trace widths.

Figure 4 The comparative wear rates of MoS_2 coatings (see online version for colours)

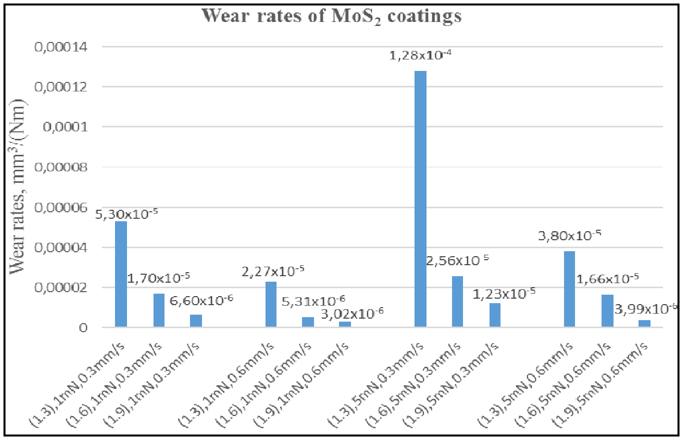
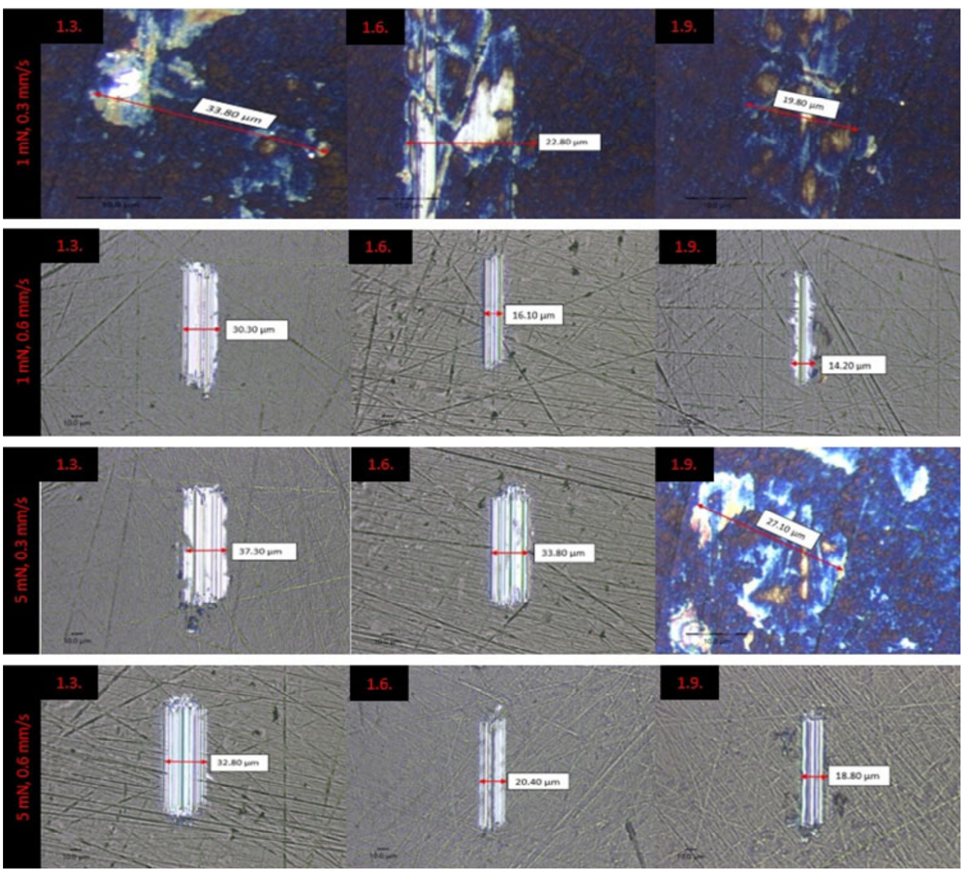


Figure 5 The abrasion trace widths of MoS_2 coatings (see online version for colours)

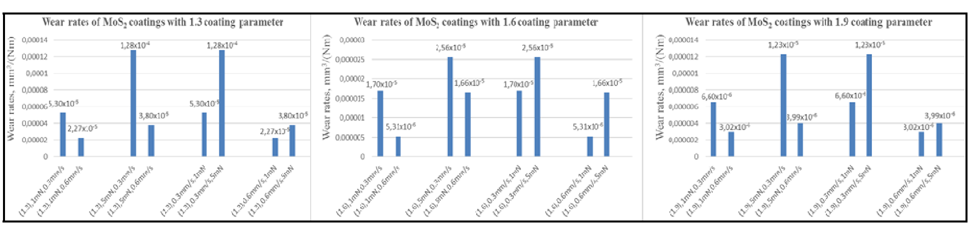


According to Table 3, MoS_2 coating deposited in 1.9 coating parameter, which is the hardest coating for applied loads and speeds, gives the lowest friction coefficients, wear rates and trace widths at each loading and speed. The highest friction coefficients, wear rates and trace widths were shown with the smallest hardness of 1.3 coating parameters. Thus, the smallest friction coefficient was 0.0073, the smallest wear rate was 3.02×10^{-6} mm^3/Nm and the smallest wear trace width was 14.20 µm; MoS_2 coating with 1.9 coating parameters was obtained at a loading of 1 mN and a speed of 0.6 mm/s. The highest coefficient of friction was 0.0329, the highest wear rate was 1.28×10^{-4} mm^3/Nm and the highest wear trace width was 37.30 µm; MoS_2 coating with 1.3 coating parameters was obtained at a loading of 5 mN and a speed of 0.3 mm/s.

Table 3 Tribological properties of MoS_2 coatings

Coating parameter	*Hardness (GPa)*	*Load (mN)*	*Speed (mm/s)*	*Friction coefficient*	*Wear rate mm^3/(Nm)*	*Trace widths (µm)*
1.1	1.3861		0.3	0.0320	5.30×10^{-5}	33.80
1.6	3.6624	1	0.3	0.0219	1.70×10^{-5}	22.80
1.9	3.7972		0.3	0.0157	6.60×10^{-6}	19.80
1.3	1.3861		0.6	0.0262	2.27×10^{-5}	30.30
1.6	3.6624	1	0.6	0.0188	5.31×10^{-6}	16.10
1.9	3.7972		0.6	0.0073	3.02×10^{-6}	14.20
1.3	1.3861		0.3	0.0329	1.28×10^{-4}	37.30
1.6	3.6624	5	0.3	0.0240	2.56×10^{-5}	33.80
1.9	3.7972		0.3	0.0181	1.23×10^{-5}	27.10
1.3	1.3861		0.6	0.0272	3.80×10^{-5}	32.80
1.6	3.6624	5	0.6	0.0218	1.66×10^{-5}	20.40
1.9	3.7972		0.6	0.0080	3.99×10^{-6}	18.80

Figure 6 Variation of wear rates of MoS_2 films depending on load and speed (see online version for colours)



The wear rate of MoS_2 coating with a coating parameter of 1.3 is 5.30×10^{-5} mm^3/Nm at 1 mN load, 0.3 mm/s; the speed of the same load to 0.6 mm/s, 2.27×10^{-5} mm^3/Nm leads to decrease. The wear rate with a coating parameter of 1.3 is 1.28×10^{-4} mm^3/Nm at 5 mN load, 0.3 mm/s; the speed of the same load to 0.6 mm/s, 3.80×10^{-5} mm^3/Nm is seen to decrease. The wear rate with a coating parameter of 1.3 is 5.30×10^{-5} mm^3/Nm at 0.3 mm/s speed, 1 mN loading; the load at the same speed to 5 mN, 1.28×10^{-4} mm^3/Nm is seen to increase. The wear rate with a coating parameter of 1.3 is 2.27×10^{-5} mm^3/Nm at 0.6 mm/s speed, 1 mN loading; the load at the same speed to 5 mN, 3.80×10^{-5} mm^3/Nm is seen to increase.

As it was observed on Figure 6; it was observed that coating wear rates decreased with increasing speed at constant loads. Coating wear rates increased with increasing loading at constant speeds.

4 Conclusions

The results and conclusions of this experimental study can be summarised as follows.

- Thicker MoS_2 film coatings were obtained at the same deposition temperatures as the radio frequency sputtering power increased. The thickest of the films whose coating thicknesses were measured was obtained from MoS_2 with 1.7 coating parameters. So, the highest sputtering power of 200 W RF and 25°C substrate deposition temperature were applied and the coating thickness was measured as 4.160 µm.
- As a result of thickness measurements, the smallest thickness was obtained from MoS_2 coating with 1.2 coating parameter. In this parameter, the lowest sputtering power of 100 W RF and 70°C substrate deposition temperature were applied and the thickness was measured as 0.620 µm.
- The hardness values were obtained between 0.790 GPa and 3.797 GPa. The nano hardness value of the MoS_2 coating deposited in the 1.7 coating parameters was found to be 1.572 GPa and was consistent with the literature.
- In the MoS_2 coatings deposited at the same radio frequency sputtering power, it was shown that the hardness values increased as the deposition temperatures increased. The hardest values were obtained as 1.386 GPa at 100 W RF, 3.662 GPa at 140 W RF and 3.797 GPa at 200 W RF. Coatings of this hardness deposited at coating parameters 1.3, 1.6 and 1.9 respectively were obtained at 120°C.
- In the MoS_2 coatings deposited at the same deposition temperature, it was shown that the hardness values increased as the radio frequency sputtering power increased. The hardest values of 1.572 GPa at 25°C, 1.795 GPa at 70°C and 3.797 GPa at 120°C were obtained. Coatings of this hardness deposited at coating parameters 1.7, 1.8 and 1.9 respectively were obtained at 200 W.
- Harder coatings were obtained due to the increase in substrate deposition temperature and RF sputtering power. Among the coatings whose hardness values were determined, the highest hardness was obtained from the one with 1.9 coating parameters.
- Friction coefficients ranging from 0.0073 to 0.0329 were obtained from MoS_2 film coatings. The highest coefficients of friction, wear rates and wear trace widths were shown with the smallest hardness of 1.3 coating parameters.
- It was found that friction coefficient, wear rates and wear track widths decrease with increasing speed at constant loads, while they increase with increasing load at constant speeds.

- MoS_2 coating deposited in 1.9 coating parameter, which is the hardest coating for applied loads and speeds, gives the lowest coefficients of friction, wear rates and wear trace widths at each loading and speed. The smallest coefficient of friction obtained from tribological tests was 0.0073, the smallest wear rate was 3.02×10^{-6} mm^3/Nm and the smallest wear trace width was 14.20 µm; MoS_2 coating with 1.9 coating parameters was obtained at a loading of 1 mN and a speed of 0.6 mm/s. Since MoS_2 film with 1.9 coating parameters shows the lowest coefficients of friction, wear rates and wear widths compared to other MoS_2 coatings, 1.9 coating parameter was optimised as the most suitable coating pair in terms of tribological properties in this study.

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