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Investigation of the friction and wear properties of Ti/TiB$_2$/MoS$_2$ graded-composite coatings deposited by CFUBMS under air and vacuum conditions

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ABSTRACT

MoS$_2$ coatings are effectively used in vacuum and in water vapor-free environments because of increased friction coefficient and decreased service life under atmospheric conditions. A lot of different alloy elements (e.g., Ti, Nb, Cr) and compounds (e.g., TiN, TiB$_2$) are used to enhance the friction and wear properties of MoS$_2$ coatings. In this study Ti/TiB$_2$/MoS$_2$ graded-composite coatings (GCC) were deposited by closed-field unbalanced magnetron sputtering (CFUBMS). The structural properties of the coatings were analyzed by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD). The hardness of the coatings was measured with the use a microhardness tester. The tribological properties of the coatings were determined under air and vacuum conditions. Ti/TiB$_2$/MoS$_2$ (GCC) have MoS$_2$ (002) and TiB$_2$ (100) reflections. The coatings exhibited a dense and non-columnar structure. Tribological properties of the coatings under air and vacuum conditions significantly affected the hardness, thickness and stoichiometric ratio of elements in the structure.

1. Introduction

Conventionally, liquid lubricants and greases have been used to reduce friction and increase wear life in sliding applications. However, the use of liquids is not recommended because of environmental concern, cost and effectiveness [1]. Recently, considerable amount of research showed that applying a solid lubricant as a coating on the surfaces of sliding components has become an effective way to reduce friction and enhance wear life [2]. Therefore, solid lubricants have been widely used in industrial applications [3]. Among them MoS$_2$ is one of the most well-known solid lubricants [4] and widely used for space application [5,6], gear [7] and precision bearing applications [8], dies [9], cutting tools [9,10], forming tools [10,11] and milling [11] due to its favorable lubrication properties.

However, the lubricating properties of MoS$_2$ are deteriorated in humid air due to the oxidation of MoS$_2$ to MoO$_3$ [2,9,12]. So, the friction coefficient increases and coating lifetime decreases [4]. A lot of metals such as Au [11,13], Ni [13,14], Pb [15], Ti [16–18], Ta [13], Cr [18,19], Nb [20,21], mixed metal [22] or ceramic such as TiN or TiB$_2$ [23,24] were used to overcome the deterioration of MoS$_2$ coatings in humid air. These studies showed that MoS$_2$ coatings deposited with metal addition gettered oxygen partly during the wear experiments and enhanced the friction coefficient and wear resistance of the coatings relatively [16,25]. It was also determined that MoS$_2$/metal composite coatings were hard and less sensitive to water vapor than pure MoS$_2$ coatings [3,26]. Among these metal elements, incorporation of Ti to MoS$_2$ has been introduced to the market as MoST™ by Teer Coatings Ltd [16]. In this system, while Ti improves the load carrying and the adhesion of the film to the substrate, MoS$_2$ provides the solid lubrication. Considerable amount of studies was performed to investigate the friction and wear properties of MoS$_2$–Ti coatings. These studies showed that MoS$_2$–Ti composite coatings are denser, have higher load carrying capacity, have more adhesive strength and more oxidation-resistant than pure MoS$_2$ [16,25]. Furthermore titanium reacts with oxygen from the vacuum chamber and forms TiO$_2$ within the coating material during a deposition process and this reaction product behaves as a solid lubricant [11]. Also, Ti can be used as an interlayer which led to an improvement in coating adhesion [27]. This situation led to incorporation of titanium into the coating itself, resulting in improved friction and wear properties [3,26].

Recently, as a transition metal based refractory material, TiB$_2$ coatings have taken considerable attention due to its outstanding properties such as high hardness, excellent wear and corrosion resistance [27–29], high melting point [28,30], high chemical stability [27,31], and good thermal and electrical conductivity [31]. TiB$_2$ has these excellent properties making it a suitable coating for cutting and forming tools due to high performance [29,32].

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Some studies showed that co-sputtering of C and TiB_2 has given the soft MoS_2 an improved friction coefficient and wear resistance at ambient conditions [2,29]. Gilmore et al. [23] investigated the effect of MoS_2 and C amount on the friction properties of TiB_2 and revealed that incorporation of MoS_2—even at low amount—significantly reduced the coefficient of friction of the $TiB_2 + MoS_2$ coating system.

Recently due to the enhanced load carrying capacity by offering smoother transitions in mechanical properties from those of the hard and stiff coating to those of the softer and more flexible substrate, MoS_2 based application of graded composite coatings is also becoming an important research area [33,34].

In the current research, $Ti/TiB_2/MoS_2$ graded-composite coatings were deposited at different deposition parameters using the closed field unbalanced magnetron sputtering (CFUBMS) technique. Tribological properties of the coatings were determined under air and vacuum conditions. While the structural properties of the coatings were determined by XRD and scanning electron microscopy SEM–EPMA, their tribological behavior was investigated by a ball-on-disk tribometer.

2. Material and methods

$Ti/TiB_2/MoS_2$ graded-composite coatings were deposited on Cu and silicon substrates by standard Teer CFUBMS equipment. Before the deposition process all Cu substrates were polished to roughness values of Ra ≈ 0.130 μm by using SiC emery paper with a 400-mesh grit and the polished substrate surfaces were ultrasonically cleaned with ethanol bath and dried with warm air. After this process, the substrate surfaces were etched using 10% nital for 25 s. The magnetrons within the coating chamber were arranged so that two TiB_2 targets, one MoS_2 target and one Ti target were used and the Cu substrates rotated between the targets. At the beginning of the deposition process, the surface of the Cu substrates was sputter-ion cleaned using a bias voltage of 900 V with argon gas for 20 min to remove possible contaminants. To increase adhesion and to decrease residual stress in between substrates and coatings a Ti interlayer was deposited for 5 min on Cu substrates. The deposition process was continued for 90 min. The details belonging to deposition parameters are given in our previous study [35].

The microstructure and elemental analyses of the $Ti/TiB_2/MoS_2$ graded-composite coatings were conducted with a Zeiss-EVO LS10 system. The coating thickness was measured using SEM cross-section images taken after the silicon substrates were cleaved.

XRD measurements were made using Rigaku 2000 Dmax diffractometer equipment with a Cu-Kα radiation source. Measurement values were obtained in the 5 to 60° scan range at a 2°/min scan speed. Interpretation of the X-ray results was conducted using JCPDS files.

The microhardness of the $Ti/TiB_2/MoS_2$ graded-composite coatings was obtained by Knoop microhardness measurements using a Buehler microhardness tester. Deformation tracks were made using a Knoop diamond pyramidal tip (172°30′ surface angle) under a load of 10 gf for 15 s.

The ball-on-disk tribometer was used to investigate friction and wear properties of the $Ti/TiB_2/MoS_2$ coatings. The friction and wear experiments were performed at two different conditions: (i) air (1013 mbar pressure and 55% relative humidity) and (ii) 5×10^{-3} mbar vacuum condition. The normal load was applied using dead weight. During the experiments, the ambient temperature was 20 ± 2 °C. The experiments were conducted under 2 N load and 150 rev/min in 10 min, during which the corresponding sliding distance was 600 m. All experiments were performed against the Al_2O_3 ball. The wear tracks were observed using SEM. Compositional analysis of the wear tracks was also performed using EDS.

3. Results and discussion

Fig. 1 shows a typical scanning electron micrograph of the surface morphology for the $Ti/TiB_2/MoS_2$ graded-composite coatings. All coating surfaces typically showed a granular structure. A typical SEM image of the fractured surface of the coatings is shown in Fig. 2. It can be seen in Fig. 2 that the $Ti/TiB_2/MoS_2$ graded-composite coatings have dense and non-columnar structure. The details belonging to

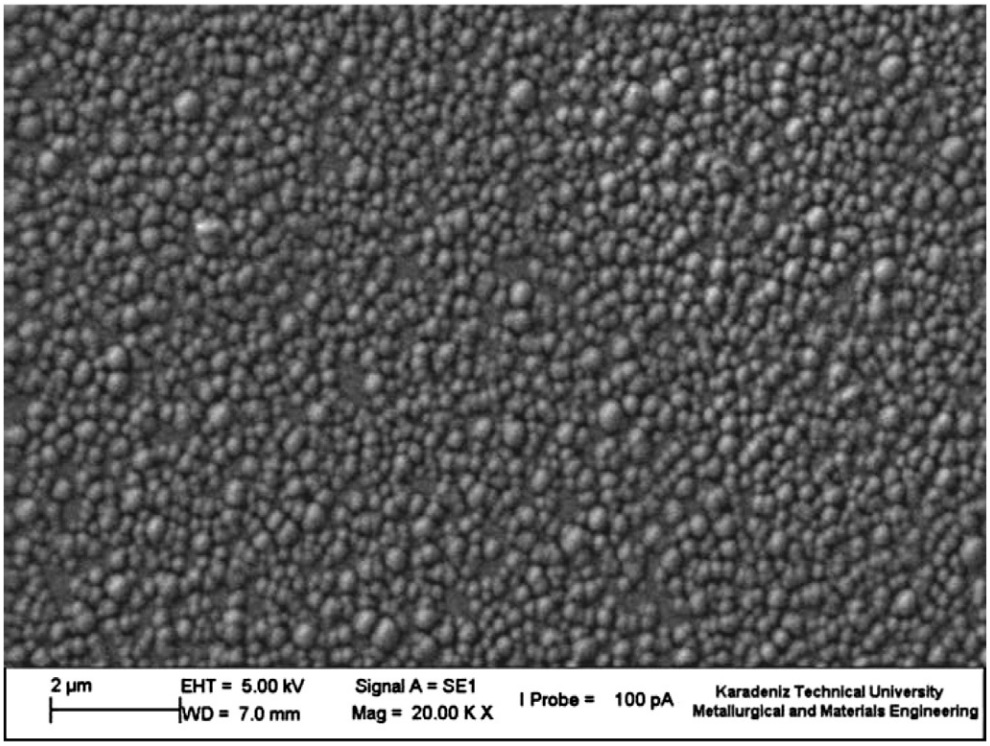

Fig. 1. The surface morphology for $Ti/TiB_2/MoS_2$ graded-composite coatings.

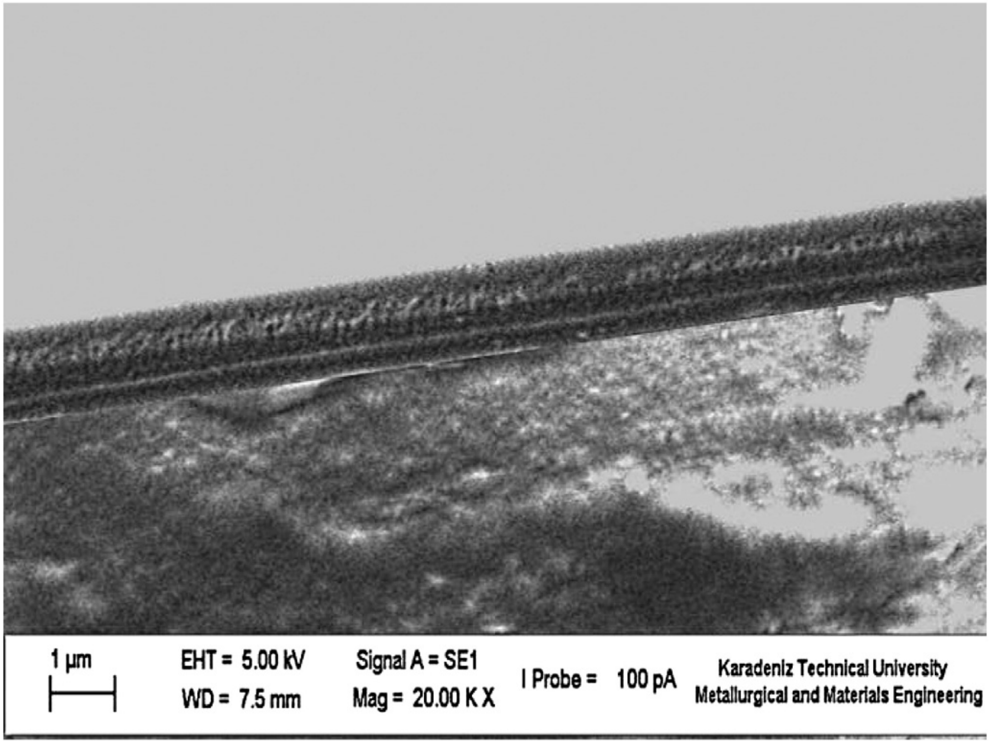

Fig. 2. A SEM image of the fractured surface for Ti/TiB_2/MoS_2 graded-composite coatings.

thickness, hardness and chemical analysis results of the Ti/TiB_2/MoS_2 graded-composite coatings were given in our previous study [35]. The highest thickness value (1.3 µm) was obtained with R3 deposition parameters. R3 has the highest MoS_2 and TiB_2 target currents. Also, the S/Mo and Ti/Mo proportions in the coatings decrease with increasing applied target currents of MoS_2 and TiB_2 (Table 3 in Ref [35]). Furthermore, hardness of the Ti/TiB_2/MoS_2 graded-composite coatings deposited by R3 coating parameters increased. This increase is also attributed to its denser structure and oriented basal plane (0 0 2) [17].

According to XRD results, the Ti/TiB_2/MoS_2 graded-composite coatings have MoS_2 and TiB_2 peaks as shown in Fig. 3. Intensively MoS_2 (0 0 2) basal plane orientation parallel to the substrate surface at about $2\theta = 12.5$ Bragg angle is observed for all of the coatings. This phase is preferable in tribological applications because it provides low friction and solid lubrication between sliding surfaces [36]. Also these basal-oriented MoS_x coatings have a dense and non-columnar structure, while random-oriented MoS_x coatings possess a columnar structure [24, 37]. Also, all coatings have MoS_2 (100) and MoS_2 (103) phases. But, MoS_2 (002) peak is the strongest peak when compared with the (100) and (103) peaks. Less-pronounced peaks were found in R1 and R2. The TiB_2 (100) peak appeared at approximately $2\theta = 35°$. At R3 that has maximum TiB_2 and MoS_2 target currents, the most prominent MoS_2 (002) and TiB_2 (100) peaks were obtained.

The highest hardness value (254 $HK_{0.01}$) for the Ti/TiB_2/MoS_2 graded-composite coatings was obtained with the highest MoS_2 and TiB_2 target currents. The amount of the atoms sputtered from the targets and ionization within the plasma increased with increasing target currents. It is known that TiB_2 is in a hard phase. The hardness of the coatings increased with increasing TiB_2 diffraction intensity [2].

Table 1
The coefficient of friction and wear rate values under different test conditions of substrate and Ti/TiB_2/MoS_2 graded-composite coatings.

	Friction coefficient, µ		Wear rates, mm^3/Nm	
	Air	Vacuum	Air	Vacuum
Cu substrate	0.95	0.443	0.000107	0.000078
R1	0.632	0.415	0.000101	0.000056
R2	0.542	0.377	0.000087	0.000039
R3	0.484	0.132	0.000074	0.000023

Coating R3 deposited with maximum target current of TiB_2 has the highest hardness and thickness values. In coatings, while the B/Ti ratio increases with the increase of TiB_2 and MoS_2 target currents, S/Mo and Ti/Mo ratio is decreased (Table 3 in ref [35]).

The friction and wear behaviors of Cu substrates and Ti/TiB_2/MoS_2 graded-composite coatings were determined by wear tests performed under air and vacuum conditions. The average friction coefficient and wear rate values obtained from different test conditions for substrates and coatings were given in Table 1. Cu substrates have the highest friction coefficient and wear rate under both conditions.

Moreover the friction coefficient and wear rate of the Ti/TiB_2/MoS_2 graded-composite coatings decreased with increasing thickness,

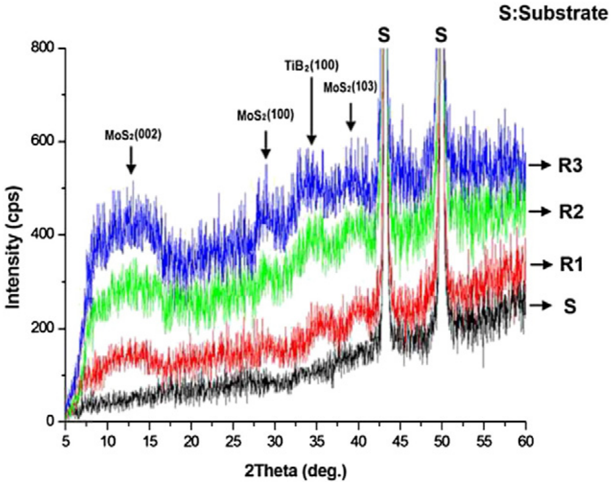

Fig. 3. XRD pattern for Ti/TiB_2/MoS_2 graded-composite coatings.

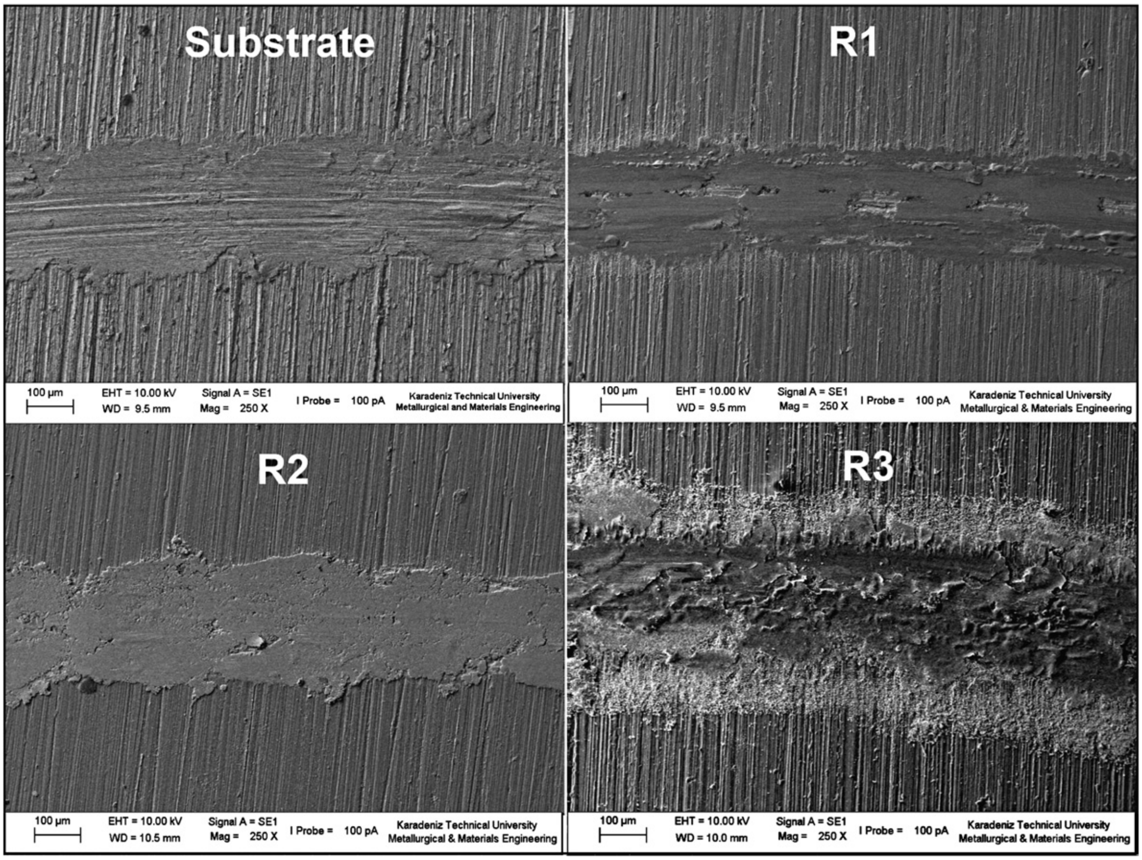

Fig. 4. SEM morphologies of wear tracks of Cu substrate and Ti/TiB_2/MoS_2 graded-composite coatings under air condition.

hardness and B/Ti ratio because of increased TiB_2 and MoS_2 target currents. The friction coefficients of the Ti/TiB_2/MoS_2 graded-composite coatings tested under air conditions had a large variation range than those tested under vacuum condition. Also, the friction coefficients are higher than vacuum at the same deposition conditions. Additionally, the lifetime of the Ti/TiB_2/MoS_2 graded composite coatings tested under air conditions is lower than vacuum conditions. This is due to the presence of oxygen in ambient air condition. He et al. [9] reported similar results for MoS_2-based Ti coatings. Also, Efeoglu and Bulbul [17] reported that the coefficient of friction and the wear life of the Mo_xS_y–Ti films decreased as a function of the environmental conditions. The increase of the coefficient of friction of the MoS_x coatings with relative humidity has been linked to the tribo-chemical reaction of MoS_x with O_2 and H_2O [37]. The tribo-chemical reaction takes place at the edge planes of MoS_2 crystals, since the edge planes of MoS_2 crystals are more reactive than basal planes. Donnet et al. [38] have noted that the effect of the atmosphere during friction played an important role, therefore, the formation of MoO_x in humid air caused an increase in the friction coefficient. So, MoO_3 particles are generated on the wear tracks and in the debris [37].

With addition of metal and non-metal to MoS_2 coatings, if the metal or non-metal entering space between the sulfur planes can reduce the amount of water vapor and/or prevent the water vapor. Also, the hardness of the coatings increases due the distortion of the MoS_2 lattice. The shear strength of these coatings due to the high hardness is higher than pure MoS_2 coatings. So, friction coefficient significantly decreases and improves tribological properties of pure MoS_2 [3].

Gangopadhyay et al. [39] reported that the low coefficient of friction value of the composite coating can be reasonably attributed to the presence of lubricious MoS_x phase throughout the coating. Similar results were also obtained in this study. The hardness, adhesion [35] and wear resistance of the Ti/TiB_2/MoS_2 graded-composite coatings increased in coating R3 with increase of the MoS_2 and TiB_2 target currents and the friction coefficients of the coatings are decreased. Research indicates [3,17] that very high titanium content adversely affects the wear properties. Also, Rigato et al. [10] reported that the coatings with Ti/Mo ratios larger than approximately 1.3 have a weaker wear resistance. Ti/Mo ratios were significantly decreased with the R3 deposition parameters and this ratio is greater than 1.3 for all of the coatings. Additionally, a sulfur deficiency in the MoS_2-based coatings improves the mechanical properties. The high friction coefficient and wear rate values for the Ti/TiB_2/MoS_2 graded-composite coatings deposited by R1 coating parameters are due to high Ti and S content. The wear rate values of the Ti/TiB_2/MoS_2 graded-composite coatings under both conditions were given in Table 1. In contrast to MoS_x coatings, the coefficient of friction of TiB_2 coatings decreases at increasing relative humidity. The low coefficient of friction for TiB_2 coatings at an atmospheric pressure has been attributed mainly to the oxygen bonding on (hydrated) TiO_2 [24]. Due to the reaction of the alloying element with oxygen, the transfer layer contained a higher amount of oxygen under atmospheric conditions. This oxide containing transfer layer has a hard and brittle structure, which leads to an increase in the wear amount of contacting surfaces under atmospheric pressure. MoS_2 exhibits lubrication effect under vacuum condition. So, the Ti/TiB_2/MoS_2 graded-composite coatings exhibited less wear under vacuum than air conditions.

Figs. 4 and 5 show the SEM worn track morphology of Cu substrates and Ti/TiB_2/MoS_2 graded-composite coatings after wear tests under air and vacuum conditions, respectively. It can be seen in Fig. 4 that the wear particles containing high-oxide re-adhered throughout wear test caused an increase in the wear via abrasion, blistering and break-up of the coating with exposure of the Cu substrate under air condition. The EDS results of wear tracks obtained from wear tests under air and vacuum conditions were given in Table 2. Mo, S, Ti, B, Cu and O were found in

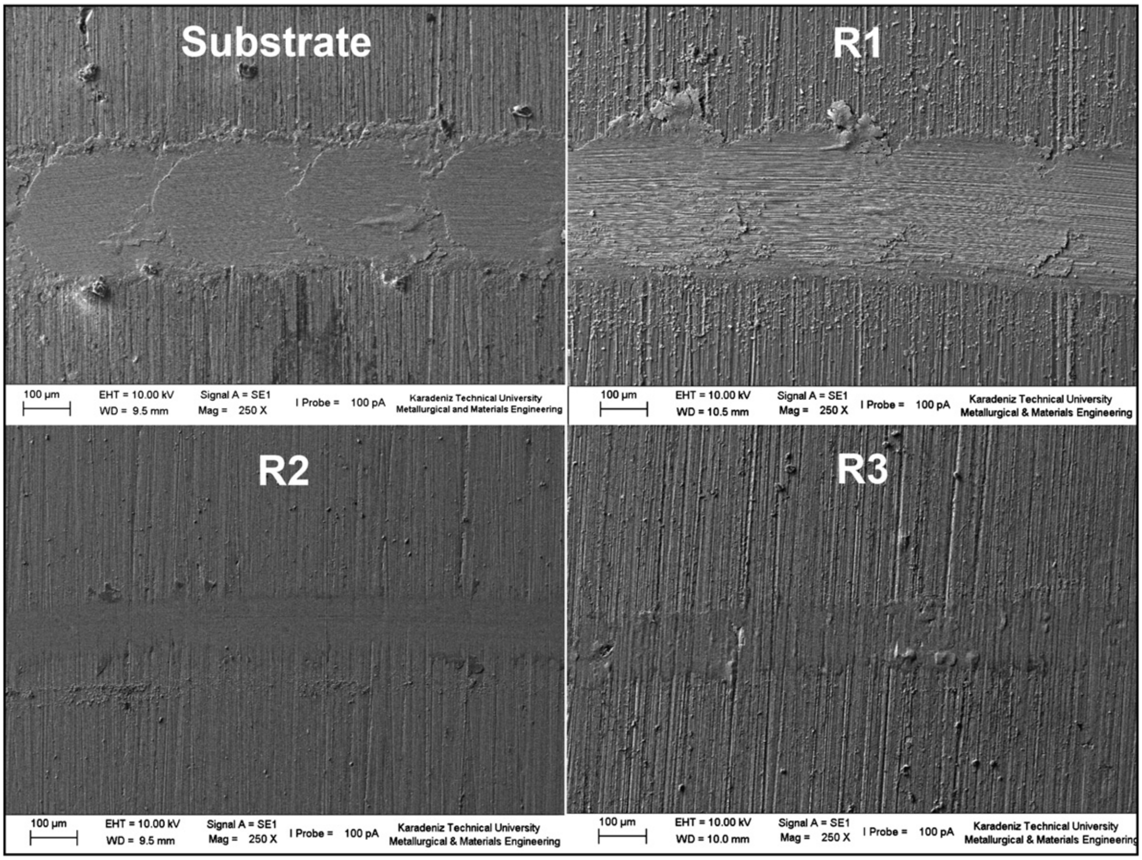

Fig. 5. SEM morphologies of wear tracks of Cu substrate and Ti/TiB_2/MoS_2 graded-composite coatings under vacuum condition.

all coatings. Also substrate and coating R1 have Zr that is within the substrate. The highest Cu and O amount was obtained from coating R1 both in wear test conditions. Cu and O amount decreased with increasing target currents of MoS_2 and TiB_2 (R3). Arslan et al. [40] reported that the oxidation products resulting from exposure to humid air cause an increase in the friction coefficient and a decrease of the wear life, thus creating an abrasive effect on the counter face.

All coatings under vacuum condition showed a smooth wear track with fine wear debris. Adhesive wear has been observed on the surface of the coatings tested under vacuum (Fig. 5). The blistering and break-up on the coatings cannot be observed in Fig. 5. Adhesive wear rate decreased with increasing hardness and decreasing Ti/Mo ratio (R3). Compared with air condition, Cu and O amount in all the coatings was lower under vacuum condition. Further, the lowest Cu and O amount was obtained from coating R3 (Table 2). The friction coefficients of these coatings deposited under the highest TiB_2 and MoS_2 target currents decreased and wear life increased due to increasing Mo, S, Ti and B amount in the coating.

4. Conclusion

In this work, the tribological properties of the Ti/TiB_2/MoS_2 graded-composite coatings deposited by the CFUBMS technique were evaluated under air and vacuum conditions. The following conclusions can be drawn:

- The highest hardness and thickness values for the Ti/TiB_2/MoS_2 graded-composite coatings were obtained with highest target currents.
- Ti/TiB_2/MoS_2 graded-composite coatings deposited with the highest TiB_2 and MoS_2 target currents exhibit most prominent MoS_2 (002) and TiB_2 (100) peaks.
- The friction coefficient and wear rate of the Ti/TiB_2/MoS_2 graded-composite coatings decreased with increasing thickness, hardness, B/Ti ratio and decreasing Ti/Mo and S/Mo ratios.
- Compared with air conditions, the Ti/TiB_2/MoS_2 graded-composite coatings exhibited less friction coefficient and wear under vacuum conditions.

Table 2
EDS analyzes results of wear tracks obtained from wear tests under air and vacuum conditions.

Wear test conditions	Air (1013 mbar)				Vacuum (5×10^{-3} mbar)			
Coatings	Elements							
	Cu	Zn	O		Cu	Zn	O	
Substrate	84.0	8.54	7.46	*Remaining*	85.37	9.15	5.48	*Remaining*
R1	46.79	3.82	30.37	Mo, S, Ti and B	33.78	4.27	16.62	Mo, S, Ti and B
R2	20.12	–	12.74		15.61	–	9.27	
R3	8.15	–	5.25		4.55	–	3.12	

- The lifetime of the $Ti/TiB_2/MoS_2$ graded-composite coatings tested under vacuum conditions significantly increased than that under air conditions.

Conflict of interest

No conflict of interest.

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