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WEAR RESISTANCE OF ELECTRODEPOSITED Fe-W ALLOY COATINGS UNDER DRY CONDITIONS AND IN THE PRESENCE OF RAPESEED OIL

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Abstract: Amorphous Fe-W alloys with 25 at.% of W were electrodeposited under direct and pulse modes from glycolate-citrate bath with and without addition of polyethylene glycol. The tribological behavior of the coatings was studied at 1, 2 and 5 N loads under dry friction and in the presence of rapeseed oil films of 0.2-5.0 μm thickness. The tribological behavior of obtained coatings at dry friction reveals their severe tribo-oxidation resulting in a high wear depth and coefficient of friction. Observed groove like surface with well-adhered particles inside the wear track point out on abrasive-adhesive wear mechanism of Fe-W alloys. In the presence of rapeseed oil films the wear mechanism changes, and values of coefficient of friction decrease up to 10 times compared to dry friction conditions. The optimum thickness of rapeseed oil film was 1 μm. This film has the satisfactory adhesion and uniform distribution on the surface, and could withstand up to 2 000 cycles.

Keywords: iron alloys, tungsten alloys, tribooxidation, abrasive wear, rapeseed oil

1. INTRODUCTION

Recently, electrodeposited tungsten alloys with iron group metals (Fe, Co, Ni) become the subject of extensive studies due to their attractive properties: high hardness [1], corrosion and wear resistance [2, 3]; thermal stability [4]; catalytic activity for hydrogen evolution reaction [5], methanol oxidation [6] and reduction of NO_x [7]. This makes these alloys appealing alternative materials for different industrial branches, including the fabrication of protective coatings for Cr replacement, barrier layers for Cu- and Sn-containing interconnects [8] and different microelectromechanical systems (MEMS) [9]. In addition, recent works show that the magnetic properties of these alloys can be tuned by fine control of the deposition parameters. Thus, indicating that owing a certain combination of the properties, these alloys could be potential candidates for the biomedicine application [10]. Furthermore, the usage of Ni for devices that might be in contact with human skin was currently restricted (European Union directive EN 1811), since it was recognized to be highly allergenic. Also, in 2007 the Cobalt REACH Consortium Ltd. was created with the purpose of preparing the registration dossiers for cobalt and cobalt compounds which may cause long lasting harmful effects on humans. Taken this into account, electrodeposition of Fe-based coatings is of great interest for "green" manufacturing of advanced materials for targeted applications.

Fe-W is more eco-friendly material and it has higher hardness and thermal stability than Co- and Ni-based alloys. Thus, the hardness of electrodeposited Fe-W coatings can reach up to ~13 GPa (at low loads) which is comparable to that of electrodeposited chromium [3, 10]. Usually, Fe-W alloys with higher W content (up to 30 at.%) have better mechanical properties due to grain boundary strengthening, solid solution strengthening and nanostructurization processes. Thus, increasing the percentage of W in alloys leads to the significant grain size refinement and hence the structure transforms form nanocrystalline to amorphous. Remarkable, the electrodeposited Fe–W alloys containing high amount of tungsten retain their structure even after annealing up to 800 °C [4].

Many researchers report over the years the importance of high hardness for the good wear resistance and show the strong linear relationships between these two properties for different polycrystalline materials like Ni-W [11], Ni-Fe-W [11], Ni-P [12] and other [13]. However, this is not always the rule.

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The hardness is an intrinsic parameter, i.e. it depends on the composition and structure of the material only, while the wear resistance is an extrinsic property and depends on the certain tribo-system used. Therefore, for the alloys which undergo abrasive wear the resistance remains relatively unaffected with increasing the hardness and, in some cases, even decreased. For instance, the wear resistance of hard electrodeposited Fe-W alloys at dry friction is rather low, due to severe oxidation of the coating during fretting (tribooxidation) [1]. Formed oxides act as the third body, thus leading to the increase of coefficient of friction (COF) and larger wear volume, as compared to Co-W alloys evaluated at similar conditions.

Furthermore, it was shown that lubricants can reduce the oxygen penetration into the sliding pairs, thus lowering the tribooxidation and improving the tribological performance of the coating (N. I. Tsyntsaru et al., 2010). Different types of liquid lubricants were investigated, e.g. vegetable oils, mineral oils, ionic liquids, etc. As the metallic surfaces generally are hydrophilic, it is of particular importance to ensure the ability of the lubricant to penetrate into the area of the wear and remain there. The use of sugar films as a solid lubricant for the Co-W alloy was discussed in [15]. As a different approach, the co-deposition of solid lubricants (like graphene, WS_2, MoS_2) into the metallic matrix was also reported [16]. In this case, the second phase soft particles can reduce the asperity contact between the coating and the counter body, therefore reduce the wear. However, the electrodeposition conditions should be optimized carefully in order to achieve uniform particles distribution, low porosity and appropriate microstructure of the coating.

In this work, the rapeseed oil was used as a lubricant, as it is known to be potential candidate to replace some of mineral oils. The investigation of the lubricating properties of the rapeseed oil is recently actively studied due to its inherent biodegradability, non-toxicity (as compared to motor oils) and good lubricity of metallic surfaces [17]. It also has some advantages in terms of sustainability. Among all other oil plants, the rapeseed gives more oil per unit of land area compared to other oil sources, such as soybeans and canola. Across the Europe, in Germany, France and Poland around 15 millions of tons of rapeseed are grown every year mostly for the production of biodiesel and cooking oils (according to Food and Agriculture Organization (FAO).

For this study, the Fe-W alloys have been electrodeposited from the recently developed [10, 18] glycolate-citrate bath based on Fe(III)-salt. The bath is considered as environmentally-friendly one, while Fe-W deposits are considered as green and sustainable materials. This study aimed to investigate the tribological behavior of obtained coatings under dry and lubricating conditions keeping the main concepts of green tribology.

2. MATERIALS AND METHODS

Fe-W samples having 25 at.% of W were electrodeposited onto Si wafer with sputtered Cu conductive layer from glycolate-citrate electrolytes of the following composition: 1 M glycolic acid, 0.3 M citric acid, 0.1 M $Fe_2(SO_4)_3$ and 0.3 M Na_2WO_4 (bath 1), and with addition to the bath 1 of 0.25 g polyethylene glycol (PEG) (bath 2). Fe-W alloys were electrodeposited under direct current (DC) and pulse current (PC) modes and the coating thickness was cca 10 µm. The corresponding baths and deposition conditions are given in Table 1.

In the case of pulse current, the average current density was calculated from:

$$i_{avg} = \frac{i_p d.c.}{100\%} \quad (1)$$

$$d.c. = \frac{\tau_{pulse}}{\tau_{pulse} + \tau_{pause}} 100\% \quad (2)$$

where i_p is the pulse current density, *d.c.* is the duty cycle, t_{pulse} and t_{pause} is the duration of pulse and pause periods, respectively.

Table 1. Electrodeposition conditions for Fe-W alloys.

Sample	Bath	pH	T°C	$-i_{avg}$, mA/cm^2	d.c., %	t_{pulse}, s	t_{pause}, s
1_DC	1	6.5	65	15	100	-	-
2_PEG	2				100	-	-
3_PC	1				95.2	1	0.05

For the investigation of tribological behavior of Fe-W electrodeposited coatings the pin-on-disc Tribometer (Anton Paar TriTec SA, Switzerland) was employed by using a ball-on-flat configuration. The corundum ball of 6 mm diameter was the counter-body that oscillated against rigidly fixed coated samples for 2 000 cycles at 2 Hz frequency of reciprocating motion, resulting in a track length of 1 mm and the total distance of 2 mm for one reciprocal friction cycle. All the tests were performed in ambient air at 23±2 °C and 48 % relative humidity. After the sliding tests, the samples were cleaned in acetone and ethanol in order to remove debris before measuring the wear track profiles. Scanning electron microscope (SEM) imaging was performed using Hitachi TM3000 instrument and elemental analysis of the wear tracks was determined with the energy dispersive X-ray spectroscopy (EDS) analysis tool attached to the SEM.

Rapeseed oil was used as a lubricant, and films of fixed thickness (0.2, 0.5, 1.0, 2.0 and 5.0 μm) were applied on Fe-W coatings before tribological tests. In order to obtain the oil film of particular thickness, the corresponding amount (10÷250 μL/cm^2) of oil stock solution (100 μL oil in 50 mL of diethyl ether) was applied on electrodeposited samples and kept until full evaporation of ether.

3. RESULTS AND DISCUSSIONS

All investigated Fe-W coatings were electrodeposited under conditions elaborated earlier (Nicolenco et al., 2018) in order to assure 25 at.% of W in deposits; this tungsten content leads to amorphous structure and high hardness revealed for these alloys. The typical SEM images of the Fe-W surface are shown in Fig. 1. The surfaces are free of cracks and smooth. It was expected that the addition of PEG to the plating bath should assists hydrogen release from the surface, thus lowering the imbedded stress into electrodeposited coatings. Indeed, as it is seen from Fig. 1b, the surface of the alloy deposited from the bath containing PEG appears with some small globules, which probably indicates the different nucleation and growth of the coating. Also, it was expected, that deposition under PC mode should result in essential improvement of tribological behavior of tungsten alloys due to the microstructural changes, as it was shown for Co-W alloys [19]. However, in the case of Fe-W alloys deposition, the corrosion processes occur during the pause, thus resulting in a higher oxidation and porosity of Fe-W coating (Fig. 1 c). Nevertheless, three Fe-W coatings are bright and mirror-like in appearance, and the average roughness of the coatings is less than 100 nm (Fig. 2).

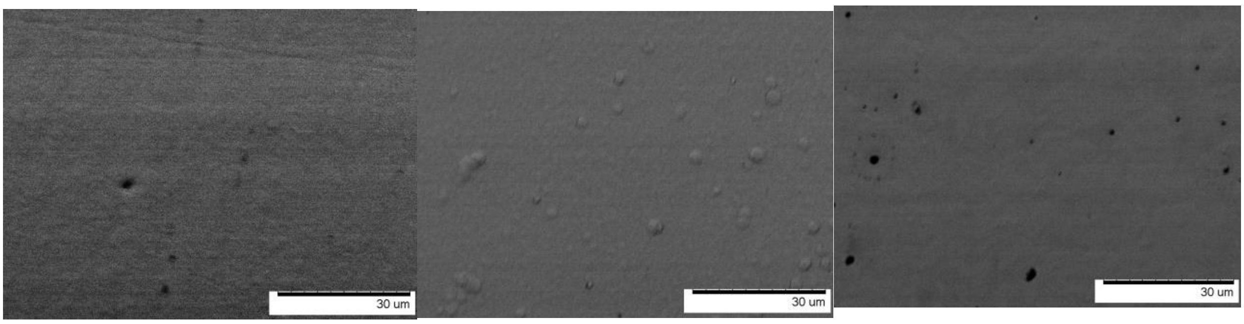


Figure 1. SEM images of Fe–W alloys deposited under conditions stated in Table 1: 1_DC (a); 2_PEG (b) and 3_PC (c).

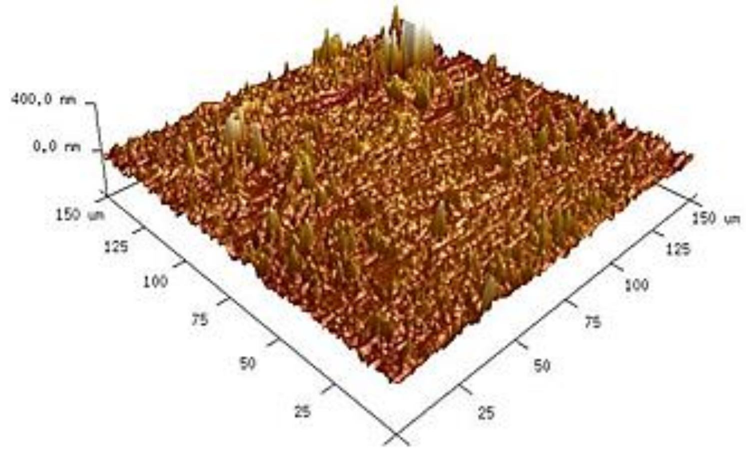


Figure 2. Typical AFM image of Fe–W alloy, sample 1_DC.

The evaluation of tribological behavior of Fe-W coatings deposited at different conditions was carried out under dry friction conditions applying 1, 2 and 5 N normal loads. As it can be seen from the Fig. 3, the COF increases sharply at the first 50–100 cycles, then reaches the maximum and remains constant. Furthermore, the maximum of the COF decreases with an increase in applied load (Fig. 3). The COF reaches the similar values between 0.8 and 1.2 after run-in period for all of three investigated coatings independently from deposition conditions. This could be associated with the tribooxidation of Fe-W alloys at the early stages of fretting test, as it was noticed earlier [1]. Abrasive particles –iron oxides, may act as the third body particles that slide against the hard corundum counter body and result in high values of COF. These particles form independently on the deposition conditions applied, apparently, due to the direct contact of Fe-W coatings with the ambient air, which contains the molecules of water and oxygen.

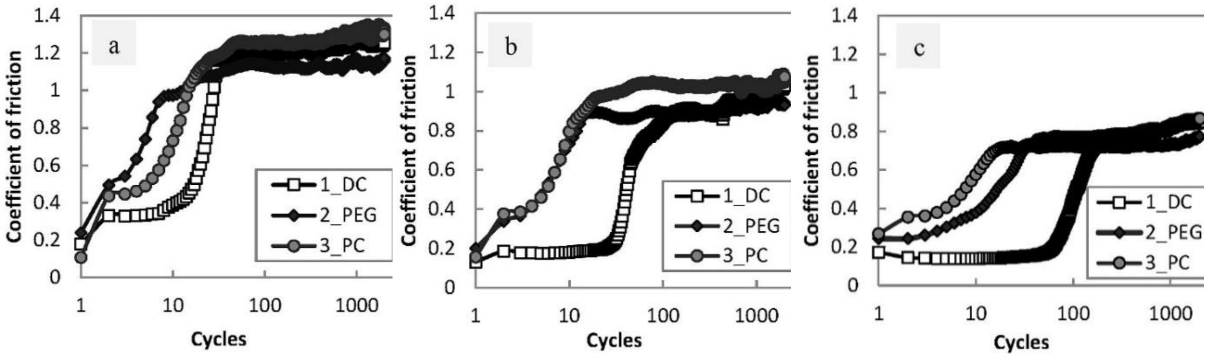


Figure 3. Evolution of COF of Fe-W samples at dry friction under applied loads: 1 N (a), 2 N (b), 5 N (c).

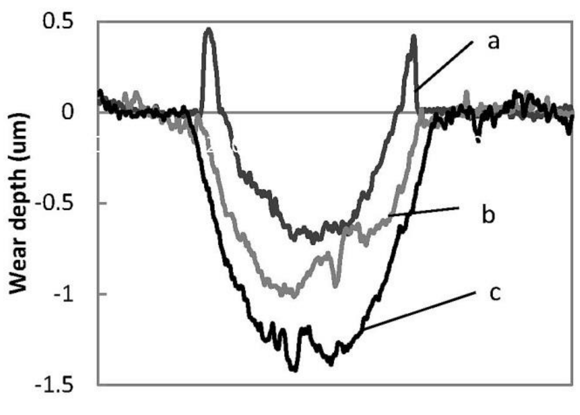


Figure 4. Depth profiles of wear tracks of Fe-W alloys (sample 1_DC) after dry friction at 1 N (a), 2 N (b) and 5 N (c).

In order to understand the nature of wear mechanism involved, the wear tracks were examined by 2D profilometer, SEM and EDS analysis after 2 000 fretting cycles performed at different loads. Depth profiles presented in Fig. 4 show that despite the COF was decreasing with increase of the applied load, the severe wear propagation was noticed at higher loads. The lowest COF and maximum wear depth of ~1.4 μm were obtained at 5 N load. The profiles of Fe-W worn surfaces are rather sharp, denoting the abrasive type of wear.

SEM images (Fig. 5) are attesting the results discussed above on COF and the important role of third body particles on tribological behavior of Fe-W coatings. When sliding against the hard corundum counter body, these abrasive particles leave deep grooves, which eventually increase the wear volume. Indeed, freshly formed debris are agglomerated in piles at low loads and distributed on the surface of the wear track. Thus, resulting asperities lead to a high COF (up to 1.2) at these conditions (Fig. 5 a, b). While at higher loads, abrasive particles could be incorporated inside the wear track therefore reducing the COF, but causing cracks propagation (Fig. 5 c). The diminishing of cracks propagation was noticed while using PEG for Fe-W deposition (Fig. 5 d). However, for the investigated system, pulse deposition mode did not improve the tribological behavior (Fig. 5 e), in the contrary to Co-W alloys [19], probably due to the corrosion processes occurring during the pause.

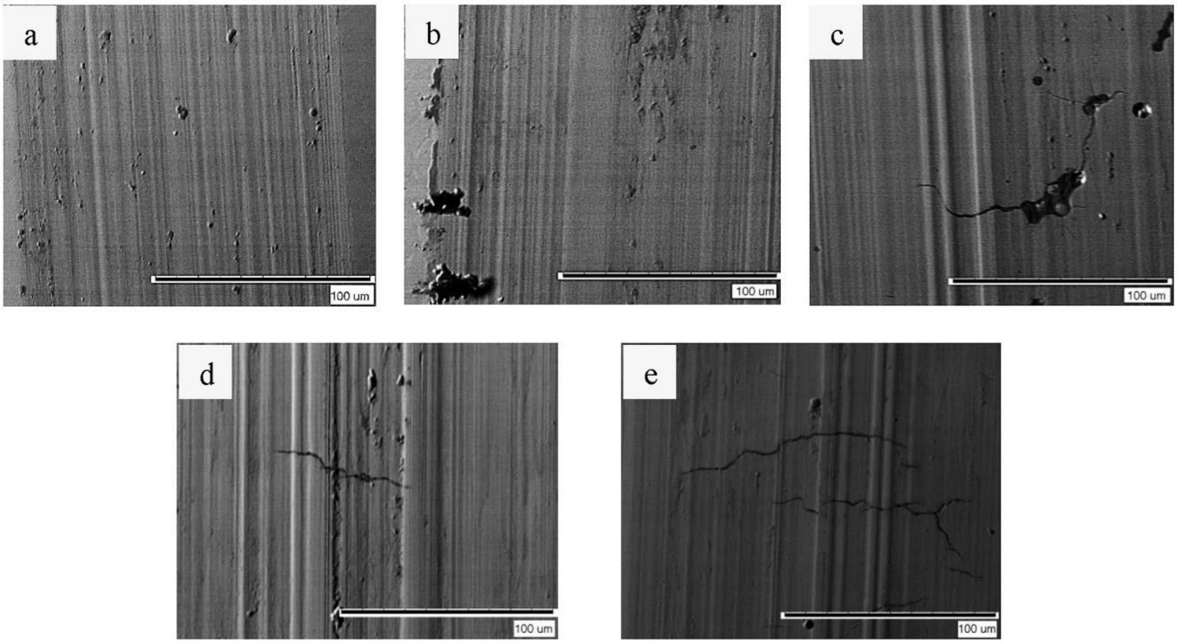
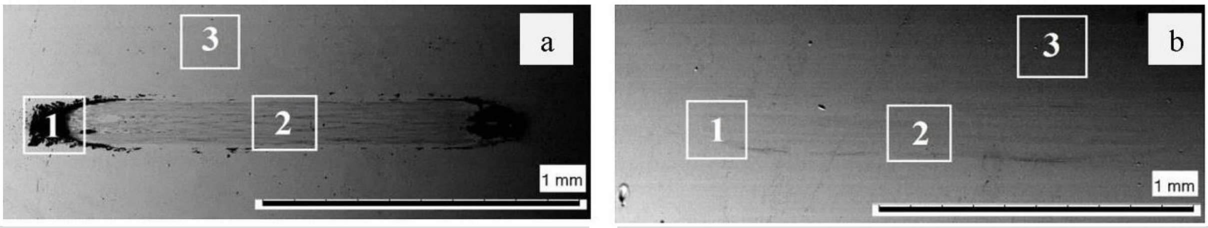


Figure 5. SEM images of wear tracks on Fe-W alloys at dry friction applied on sample 1_DC: 1 N (a), 2 N (b) and 5 N (c); and sample 2_PEG (d) and sample 3_PC (e) tested both at 5 N load.

It is noticeably seen from SEM images that some of wear particles remain adhered to the surface even after cleaning. Therefore, we could suggest that the wear mechanism of Fe-W alloys at dry friction can be ascribed to a combination of abrasive and adhesive wear.

In order to clarify the chemical content of the debris and confirm whether the high wear is caused by the tribooxidation or not, the EDS analysis on three different areas was performed (Fig. 6). Chemical analysis shows that the oxygen content in debris is increased up to 50 at.% after dry friction, which corresponds to the atomic fraction of oxygen in the mixed iron oxide Fe_3O_4 (Fig. 6 a). The oxygen content is also increased inside the wear track, due to the presence of adhered oxide particles. Moreover, the clear brown particles were observed on the surface of Fe-W samples after the fretting, thus attesting that tribooxidation under dry friction is indeed the driving factor which influence on the wear propagation.



Dry friction	1	2	3
O at.%	50	28	19
Fe at.%	38	54	60
W at.%	12	18	21

1 µm RO	1	2	3
O at.%	29	26	21
Fe at.%	52	56	59
W at.%	19	18	20

Figure 6. SEM images and EDS analysis of wear tracks on sample 1_DC after fretting test at 2 N: dry friction (a); in the presence of 1 µm rapeseed oil (b); 1, 2 and 3 denote corresponding EDS analyzed zones.

In order to diminish the Fe-W tribooxidation the surface was covered with thin film of the lubricant. Previously, the reduction of Fe-W tribooxidation was achieved by using the M-10 G2K engine oil [20]. In this study, the rapeseed oil was used as a lubricant in order to increase the environment sustainability of the process.

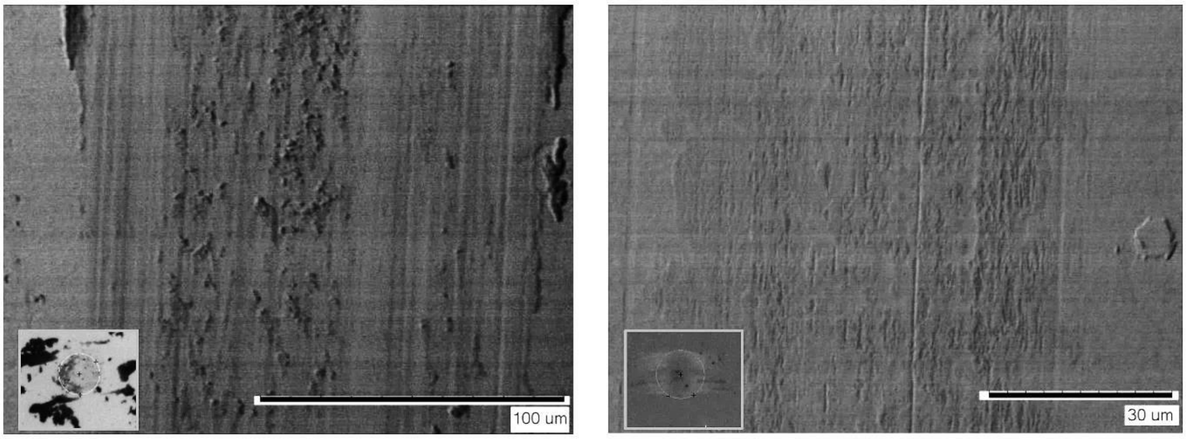


Figure 7. SEM images of the wear track after 2 N applied on Fe-25W sample after dry friction (a) and in the presence of 1 µm thick layer of rapeseed oil (b). In insert is the microscopic image of the corundum ball after the test.

Indeed, EDS analysis of the wear track on the sample 1_DC under lubricated conditions showed only slight increase in the oxygen content, while after dry friction it was increased more than 2 times on the edges (Fig. 6 b). The use of lubricant significantly reduces the contact of the Fe-W surface with aggressive medium, i.e. ambient air, which in fact is of special importance for Fe-based alloys. Due to substantial lowering of tribooxidation, the wear depth decreases significantly in comparison to dry friction and the surface becomes only slightly "polished" (Fig. 7). It was reported [15] that the oxidation can occur even when the oil film is applied, but in significantly smaller extent. Thus, no oxide particles neither adhered at the worn surface nor on the counter body were observed, while the average roughness after the test with the rapeseed oil was comparable to the roughness of as-deposited alloy. Therefore, it could be proposed that the wear mechanism changes from adhesive-abrasive at dry friction to abrasive under lubricating conditions, where produced fretting debris acts as an "in-situ" polishing agent.

In order to estimate the minimum thickness of rapeseed oil that should be applied on Fe-W coatings, the interval from 0.2 to 5.0 µm was evaluated. Obviously, the values of COF will decrease in the presence of lubricating films. Under lubricating conditions, the COF has been reduced by approximately five times for Fe-W coatings covered with rapeseed oil films on top compared to the dry friction conditions (Fig. 8). The minimal thickness of oil film when COF begins to decrease is 0.2 µm, but it could not withstand more than 50 cycles and therefore, 0.5 µm should be applied in order to retain the experiment for 2 000 cycles (Fig. 8 a). Moreover, the optimal thickness of rapeseed oil film

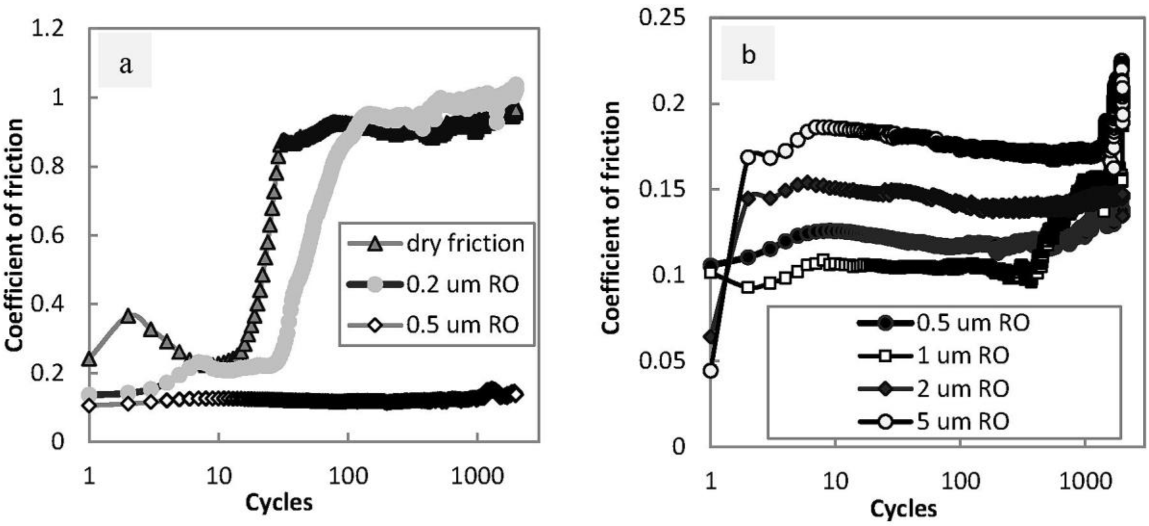


Figure 8. Evolution of the coefficient of friction at 2 N load applied on Fe-W sample 1_DC in the presence of rapeseed oil (RO) film of 0÷0.5 μm (a) and 0.5÷5.0 μm (b) thickness. Lubricating conditions are indicated on the graph.

that provides the lowest COF is 1 μm. That could be related to the better adherence of such film to the Fe-W coating and its uniform distribution on the surface, what ensure the ability of the lubricant to penetrate into the area of the wear and remain there during the test.

4. CONCLUSIONS

The wear resistance of electrodeposited Fe-W alloys having 25 at.% of W obtained under direct and pulse current modes was studied under dry and lubricating conditions using rapeseed oil. It has been shown that high COF obtained under dry friction can be linked to the combination of abrasive and adhesive wear. Abrasive particles are generated during early stages of sliding as a result of Fe-W tribooxidation.

The tribooxidation has been revealed to be the driving factor which influences on the wear propagation, which was inhibited by covering the surface with thin rapeseed oil film. Thus, the coefficient of friction was reduced by ten times from ~1.0 at dry friction to ~ 0.1, when the surface was covered with rapeseed oil film. It has been shown, that the optimum film thickness of rapeseed oil that has satisfactory adhesion to the surface and could withstand up to 2 000 cycles was 1 μm.

The roughness of the wear track after friction under lubricated conditions was found to be at the range of the initial surface roughness and no adhered particles on the worn surface were observed. Hence, the wear mechanism changes from adhesive-abrasive at dry friction to abrasive under lubricating conditions.

Taking into account the green and sustainable origin of produced Fe-W coatings they could be considered for engineering applications, where lubricating conditions can be applied. Particularly, the rapeseed oil can be used as a lubricant increasing the sustainability of the process.

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