A New Method for Electroplating of Crack-Free Chromium Coatings

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ABSTRACT: *In this study, different amounts of MoS_2 particles and an anionic surfactant were added to the conventional chromium electroplating bath in order to electrodeposite crack-free chromium coatings and the structure, morphology, tribology and corrosion behavior of the deposited coatings were investigated using X-ray diffraction spectroscopy, scanning electron microscopy, pin on disk wear test method, dynamic polarization, and electrochemical impedance spectroscopy techniques, respectively. The results showed that the incorporation of MoS_2 particle into the electrodeposited Cr coatings is impossible and no chromium coating can be electroplated from baths containing more than 5 g/L MoS_2 particles. In addition, as the MoS_2 concentration in the bath increases up to 1 g/L the corrosion and wear resistance of the deposited coatings increases. Moreover, those coatings that were electroplated from the baths containing more than 1 g/L exhibited less performance regarding their corrosion and wear behavior.*

KEYWORDS: *Electroplating; Crack-free chromium coating; MoS_2 particles; Corrosion; Wear.*

INTRODUCTION

Chromium coatings are widely used due to their good corrosion and wear resistance in different service conditions [1, 2]. Electrodeposition process is recognized as a widely used technique to produce different types of coatings [3, 4]. Electroplating of the Cr crack-free deposits are also of great importance since conventional Cr coatings commonly suffer from corrosion, mainly as a result of the cracks, rather than wear [5]. In order to produce such coatings, electroplating bath temperature should be raised above 70 ^oC which in turn can give rise to a decrease in hardness, nonetheless; the corrosion performance would improve even 10 times higher than hard Cr deposits [5, 6]. In this regard, it was reported that it would be impossible to fabricate a chromium coating that is simultaneously hard and crack-free [7]. It was reported that incorporation of particles like WC [8], SiC and Al_2O_3 [9, 10] into the metallic matrix of pure Cr coatings would enhance the corrosion and wear resistance of such coatings. Molybdenum disulfide (MoS_2) can be used for a wide range of applications including nanofilms [11] and top layer [12] for field emitters and improving wear and corrosion resistance of Mg alloys, respectively. Moreover, MoS_2 is recognized for its lubrication properties that is attributed to its graphite-like structure [13]. Thus, it would be considered as a good candidate to improve wear properties of electroplated coatings as it was reported

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earlier for some metallic coatings such as zinc [14] and nickel [15].

MoS_2 is a semiconductor compound that is considered as a good catalyst for Hydrogen Evolution Reaction (HER) [14]. Since chromium electroplating process involves the high degree of hydrogen evolution, so MoS_2 particles could serve as good sites for taking place of such reaction at the cathode surface leaving the remaining cathodic area for Cr deposition. Thus, the presence of such particles in the bath would alter the properties of the electrodeposited coatings.

There are not enough published studies concerning the influence of MoS_2 particles on the electroplating of hard Cr coatings, the main objective of the present work is to investigate the feasibility of electrodeposition of crack-free Cr coatings using SDS surfactant plus MoS_2 particles in the bath and to evaluate their respective properties. In this regard, various amounts of MoS_2 particles were added to the conventional chromium plating bath and structure, morphology, tribology and corrosion behavior of the deposited coatings were investigated.

EXPERIMENTAL SECTION

Different experimental conditions that were employed to electrodeposit chromium coatings on copper substrates (with an area of 2×2 cm^2) are presented in Table 1. Besides, Sodium Dodecyl Sulfate (SDS), as an anionic surfactant, was added to the bath in order to lower the possibility of the MoS_2 particle incorporation into the Cr coating. The SEM micrograph of the MoS_2 particles is also shown in Fig. 1. Before each electrodeposition experiment, the baths, containing different amounts of MoS_2 particles (0-10 g/L), were stirred for one hour with magnetic stirrer followed by ultrasonication for another one hour. The prepared electrolytes where then employed immediately for electroplating process while they were stirred continuously during electroplating in order to keep the particles suspended.

Surface morphology and chemical composition of the coatings were investigated using a Scanning Electron Microscope (SEM) instrument equipped with an Energy Dispersive X-ray (EDX) system. Moreover, a MitutoyoSuftest 201 device was used to measure the surface roughness of the samples. The microhardness values of the coatings were measured under 100 g applied load and indentation time of 15 seconds using a Leitz L137 microhardness tester. A Bruker D8 advance diffractometer was used to obtain X-ray diffraction patterns of the samples with Cu Kα radiation at a scan rate of 5º/min. The wear behavior of the coatings was investigated using pin on disk method with 5 N applied a normal load, the rotation speed of 96 rpm and 500 m sliding distance. The coefficient of friction was simultaneously recorded as a function of sliding distance during the wear tests.

All the electrochemical tests were carried out in a conventional three electrode cell, containing 3.5 wt% NaCl solution at room temperature, with an Ag/AgCl reference electrode, a platinum counter electrode and the electroplated chromium coatings as the working electrode, using a μAutolab3 potentiostat/galvanostat device based on the the experimental conditions

Table 1. The electroplating parameters and baths chemical compositions.

Bath constituent	Concentration (g/L)
CrO_3	250
H_2SO_4	2.5
SDS	0.1
MoS_2	0, 0.5, 1, 2.5, 5, 10
Electroplating parameters	
Current density (A/cm^2)	0.3
Temperature (ºC)	55
Electroplating time (min)	300
pH	1-1.5

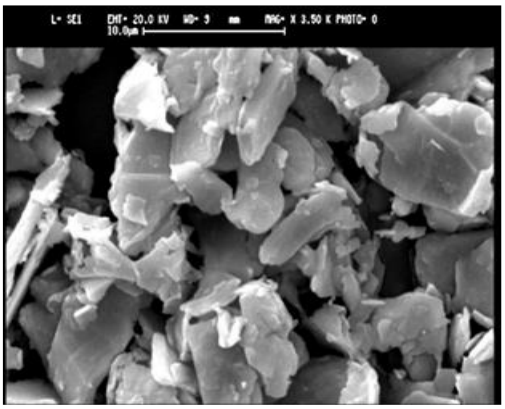

Fig. 1: SEM micrograph of the MoS_2 particles.

Table 2: The cathode current efficiency (CCE) and thickness of the Cr coatings that were electrodeposited from baths containing different amounts of MoS_2 particles.

MoS_2 concentration in bath (g/L)	Cathodic current efficiency (%)	Coating thickness (µm)
0	14.84	93
0.5	14.71	87
1	14.40	75
2.5	12.89	70
5	12.19	61

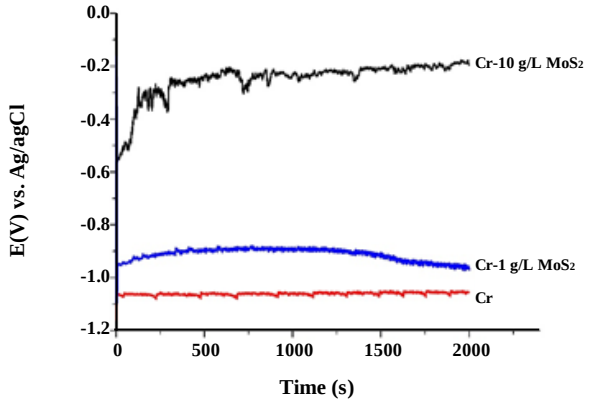

Fig. 2: Chronopotentiometry test results attributed to the galvanostatic electrodeposition of Cr coatings from the baths containing different amounts of MoS_2 particles.

mentioned by *K. Morshed-Behbahani et al.* [16]. The results of the corresponding potentiodynamic polarization and EIS tests were analyzed by GPES and FRA software (version 4.9), respectively.

RESULTS AND DISCUSSION

Electrodeposition test results

As said above, the coatings were electrodeposited from the baths containing different amounts of MoS_2 particles, but it was observed that the Cathodic Current Efficiency (CCE) of the baths decreases as the amount of MoS_2 particles in them increases (Table 2) and the galvanostatic electrodeposition of Cr from the baths containing more than 5 g/L MoS_2 is not possible. The chronopotentiometry test results attributed to the galvanostatic electrodeposition of Cr coatings from the baths containing different amounts of MoS_2 particles are illustrated in Fig. 2. As seen, by increasing the MoS_2 concentration in the bath the deposition voltage approaches more positive values in such a way that galvanostatic electrolysis of the bath containing 10 g/L MoS_2 took place at an approximate voltage of hydrogen evolution. In other words, no chromium will be electroplated from a bath containing 10 g/l MoS_2 because of severe hydrogen evolution that in turn inhibits Cr^{+6} ions reduction at the cathode surface. Such behavior would be related to the fact that MoS_2 compound is an efficient catalyst for hydrogen evolution reactions as it was reported previously [17].

The XRD patterns attributed to as-deposited coatings, which were electroplated from the baths containing different amount of MoS_2 particles, are shown in Fig. 3a. As seen, all the as-deposited coatings exhibit X-ray diffraction patterns similar to those that are typical of amorphous materials i.e. include no distinctive diffraction peaks in their XRD patterns. Nevertheless, sharp diffraction peaks related to pure crystalline chromium and Cr_2O_3 compound are present in the XRD patterns of the coatings which were heat treated in a vacuum furnace at 900 °C for 3 hours (Fig 3b). Moreover, no diffraction peaks that can be attributed to the MoS_2 compound is present in XRD patterns shown in both as-deposited and heat treated coatings (Fig. 3a, b). In this regard, it would be concluded that chromium is electroplated as an amorphous metallic phase on the substrate with little or no incorporation of MoS_2 particles in it. Such observations are in good agreement with the EDX spectra of the coatings that were electroplated from the baths containing different amounts of MoS_2 particles (Fig. 4), in which there is no evidence regarding the presence of Mo and S elements.

The SEM micrographs from the surface of the coatings that were electrodeposited at various experimental conditions are shown in Fig. 5. As seen, by increasing MoS_2 concentration in the bath from 0 to 1 g/L, coatings with finer morphology will be electroplated and

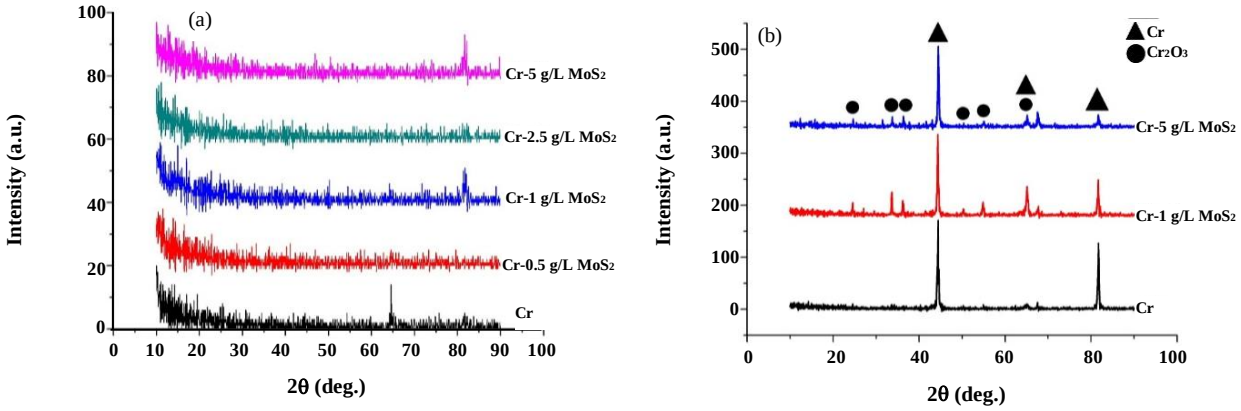

Fig. 3: XRD patterns of Cr coatings electroplated at different experimental conditions a) as-deposited condition and b) after annealing in the vacuum furnace at 900 ºC for 3h.

cracks disappear at 0.5 g/L MoS_2 concentration (Fig. 5 a-c). Such behavior would be related to the more available nucleation sites that are provided by MoS_2 particles attached temporarily to the substrate surface during the electrodeposition process. In fact, the amount of MoS_2 concentration and its surface area influence the morphology of the Cr electrodeposites by affecting the hydrogen evolution reaction. Increasing the concentration of the MoS_2 particles at the cathode surface enhances hydrogen evolution reaction resulting to have less available cathodic sites for Cr electrodeposites to nucleate and grow. This phenomenon would be the cause of having finer morphology in the samples that were electroplated from baths containing MoS_2 particles.

On the other hand, by increasing the concentration of MoS_2 particles in the bath above 1 g/L, the electrodeposited coatings exhibit more rough surface morphology (Fig. 5d, e); it can be attributed to the enhanced hydrogen evolution resulting from high amounts of MoS_2 particles at the electrode surface in such experimental conditions. These observations are in good accordance with the surface roughness values of the coatings that are shown in Fig. 6.

Microhardness and wear test results

The microhardness values of the coatings that were electroplated at various experimental conditions are presented in Table 3. As seen, all the coatings that were electroplated from the baths containing MoS_2 particles have more hardness values than that of the coating which was electroplated from a bath containing no MoS_2 particle. Such high hardness values would be related to the presence of Cr_2O_3 inclusions in their microstructure that is proved by their respective XRD patterns (Fig. 3b). Moreover, the maximum hardness value is attributed to that coating that was electroplated from the bath containing 1 g/L MoS_2 particles. It was reported that the amount of residual stress in the electroplated Cr coatings decreases as the amount of cathodic charge that were consumed for Cr^{+6} ions decreases [5]. In this regard, the lower hardness values of the coatings that were electroplated from the baths containing more than 1 g/L MoS_2 particles would be attributed to their less residual stress resulted from lower current efficiencies of their respective electroplating baths.

The wear test results of the coatings that were electrodeposited from the baths containing different amounts of MoS_2 particles are presented in Table 3. As seen, the wear resistance of the electroplated coatings increases as the amount of MoS_2 particles in their respective electroplating baths increases from 0 to 1 g/L. Moreover, those samples that were electroplated from the baths containing more than 1 g/L MoS_2 exhibited less wear resistance than the other ones.

Fig. 7 shows the SEM micrographs from the worn surfaces of the coatings that were electrodeposited at various experimental conditions. As seen, all the wear tracks include either fine grooves surrounded by debris and relatively flat areas which are typical of abrasive wear behavior. Moreover, some cracks are observed in the worn surfaces of the coatings that were deposited from baths containing less than 1 g/L MoS_2 particles (Fig. 7a-d).

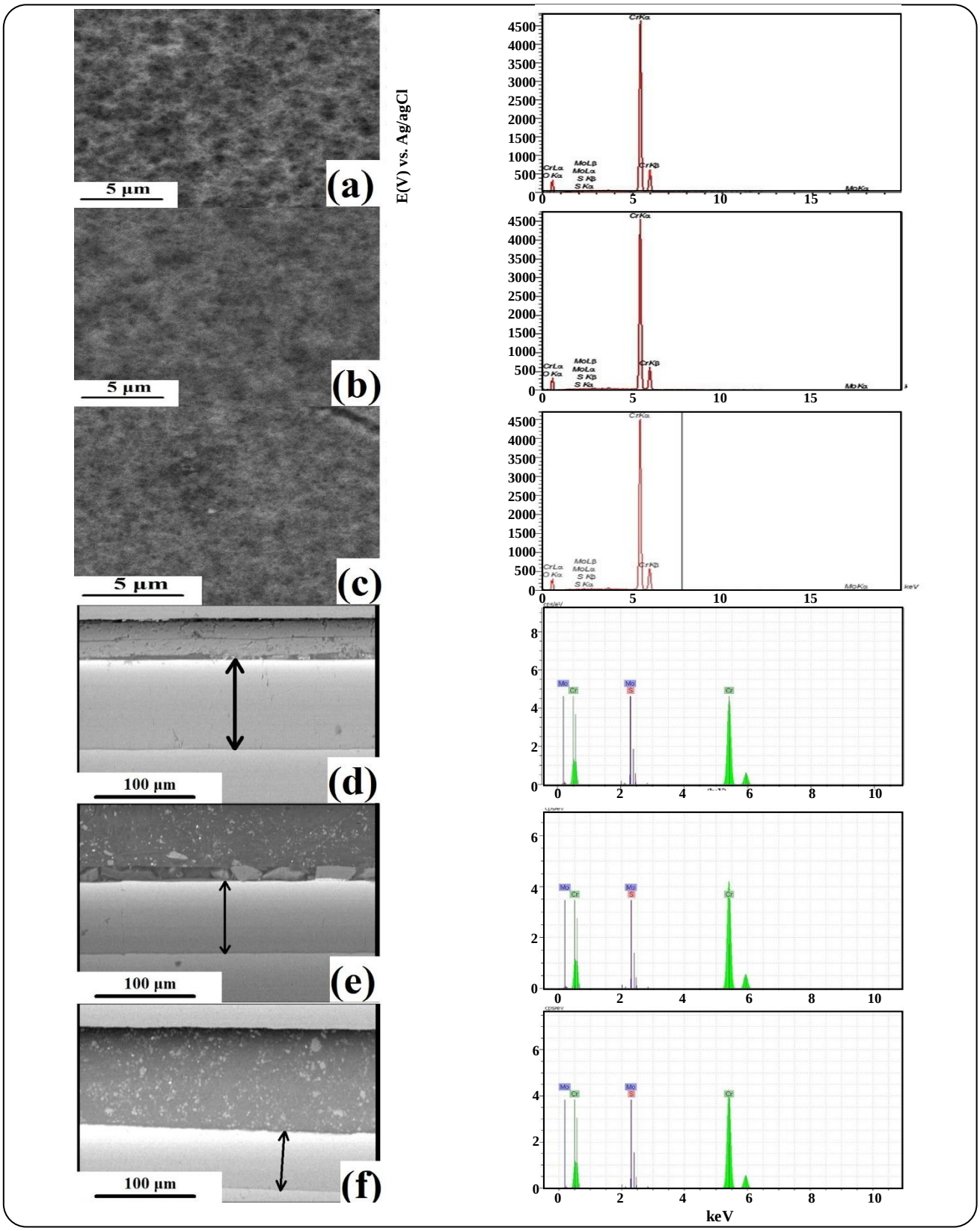

Fig. 4: EDX analysis taken from the surface and cross section of the Cr coatings electroplated from baths containing a,d) 0.5 g/L, b,e) 1 g/Land c,f) 5 g/L MoS_2 particles.

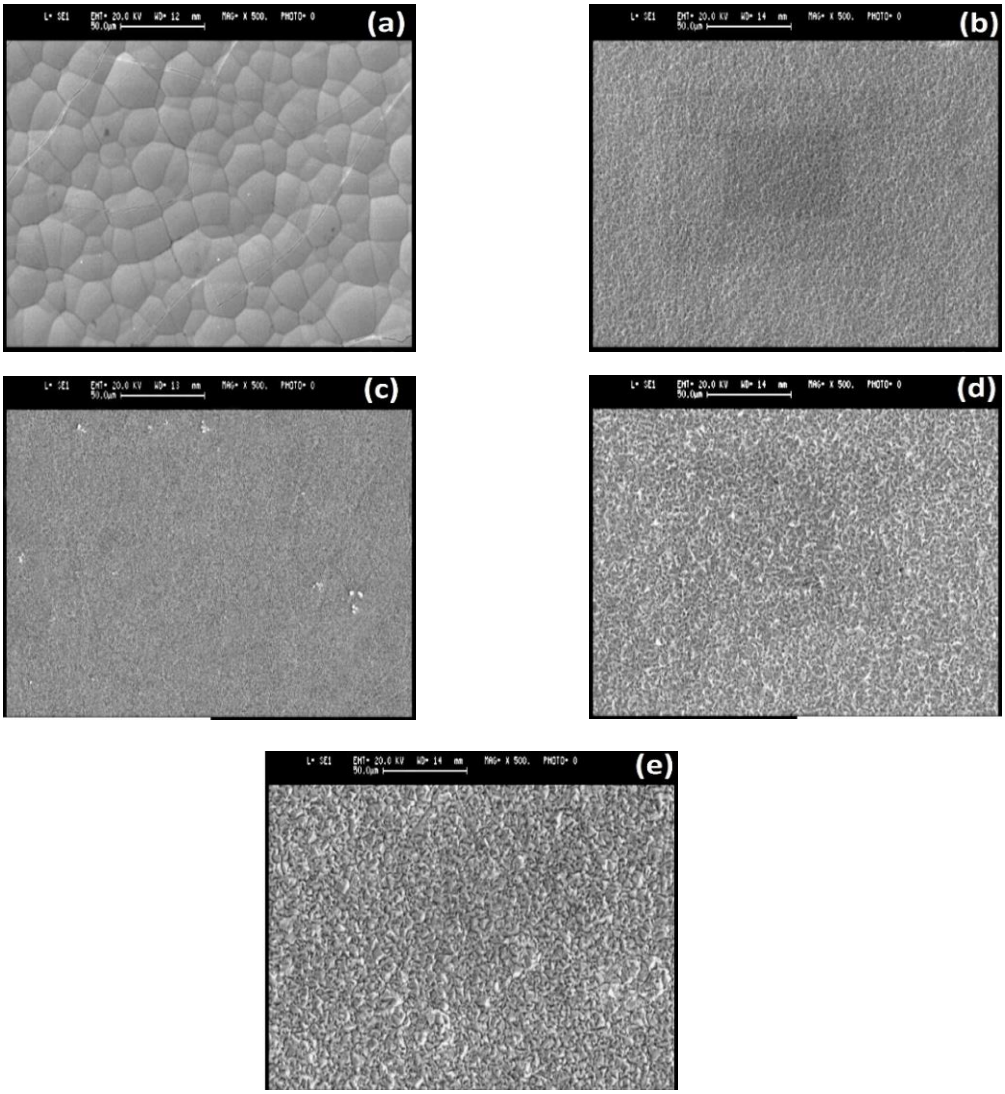

Fig. 5: SEM micrographs from the surfaces of the Cr coatings electroplated from baths containing a) 0 g/L, b) 0.5 g/L, c) 1 g/L, d) 2.5 g/L and e) 5 g/L MoS_2 particles.

Generally, the presence of the microcracks results in having poor wear resistance since they can cause coating spallation during the wear test. Such cracks may be present in the coating prior to the wear test (conventional Cr coating, Fig. 5a) or they can be produced as a result of shear stresses during the test (Fig. 7d), especially in the cases that surface roughness is still high (Cr coatings that were electroplated from the bath containing 0.5 g/L MoS_2). Moreover, there is no sign of stress-induced cracking in the worn surfaces of the coatings that were electroplated from baths containing more than 1 g/L MoS_2 particles (Fig 7e-j). In this regard, it would be concluded that the dominant wear mechanism, attributed to such electroplated coatings, has changed from fragmentation to cutting mode when the amount of MoS_2 particles in the electroplating bath is increased from 0 to 5 g/L.

The poor wear resistance of the coatings that were electroplated from the baths containing MoS_2 particles less than 1 g/L would be attributed to the occurrence of fragmentation and spalling during the wear test of such samples due to the presence of pre-existing or stress-induced microcracks in their morphology (Fig. 7b, d). In addition, in spite of the fact that no cracking and fragmentation were observed in the worn surfaces

Table 3: Microhardness and wear test results of the Cr coatings that were electrodeposited from baths containing different amounts of MoS_2 particles.

MoS_2 concentration in bath (g/L)	Coatings coefficient of friction	Wear weight loss (mg/m)	Coating microhardness (HV)
0	0.601	0.0014	916.7
0.5	0.487	0.0008	985.3
1	0.231	0.0005	1077.7
2.5	0.556	0.0018	948.3
5	0.808	0.0036	926.3

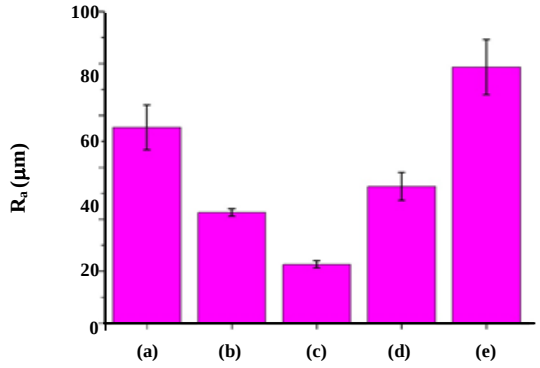

Fig. 6: Surface roughness (R_a) values of the Cr coatings electroplated from baths containing a) 0 g/L, b) 0.5 g/L, c) 1 g/L, d) 2.5 g/L and e) 5 g/L MoS_2 particles.

of the samples that were electroplated from the baths containing more than 1 g/L MoS_2 (Fig. 7e-j) their low wear resistance (high weight losses) would be attributed to their high surface roughness (Fig. 6) and friction coefficient (Table 3) leading to have harsher wear conditions for them in comparison with those of the other coatings. Last but not least, since those coatings that were electroplated from the bath containing 1g/L MoS_2 are crack free prior to the wear test and have low surface roughness, so no spallation occurred during their wear test which is in accordance with their lowest wear weight loss value (Table 3).

Electrochemical test results

The potentiodynamic polarization test results attributed to the chromium coatings that were electroplated at various experimental conditions are shown in Fig. 8 and the corresponding data are presented in Table 4. As seen, by increasing the amount of MoS_2 particles in the electroplating bath up to 1 g/L, the resulting coatings exhibit more positive (nobler) corrosion potentials ($E_{corrosion}$) and less corrosion current densities ($i_{corrosion}$). The lower corrosion resistance of the coatings that were electroplated from baths containing more than 1 g/L MoS_2 particles would be related not only to the chemical heterogeneities resulting from the presence of Cr_2O_3 inclusions that leading to the formation of more defective passive layer in such coatings, but also to the higher surface roghness that increases the effective surface of the specimens. Such an adverse effect of the chemical heterogeneities on the corrosion behavior was also reported previously [18].

The AC impedance responses of the coatings that were electroplated at various experimental conditions are shown in Fig. 9. As seen, all the responses include only a single capacitance loop and the equivalent circuit that was used to fit such experimental data is shown in Fig. 10 in which the element "R_s" is the solution resistance, "R_{ct}" is the charge-transfer resistance and "CPE" is the constant phase element. The impedance value of CPE is given by Equation (1):

$$Z_{CPE} = Q^{-1} \times (i\omega)^{-n} \quad (1)$$

In which Q is a constant and n is an empirical exponent with values between 0 to 1 [20]. The fitted values to such experimental EIS data are presented in Table 5 and the results show that the maximum charge-transfer resistance (the highest corrosion resistance) is attributed to the coating that was electroplated from a bath containing 1 g/lMoS_2 particles. It is in good accordance with the results of polarization tests that were discussed above. Moreover, it was reported that as the surface roughness decreases the value of parameter "n" approaches unity [8]. In this regard, the coating that was electroplated from a bath with 1 g/L MoS_2 would be

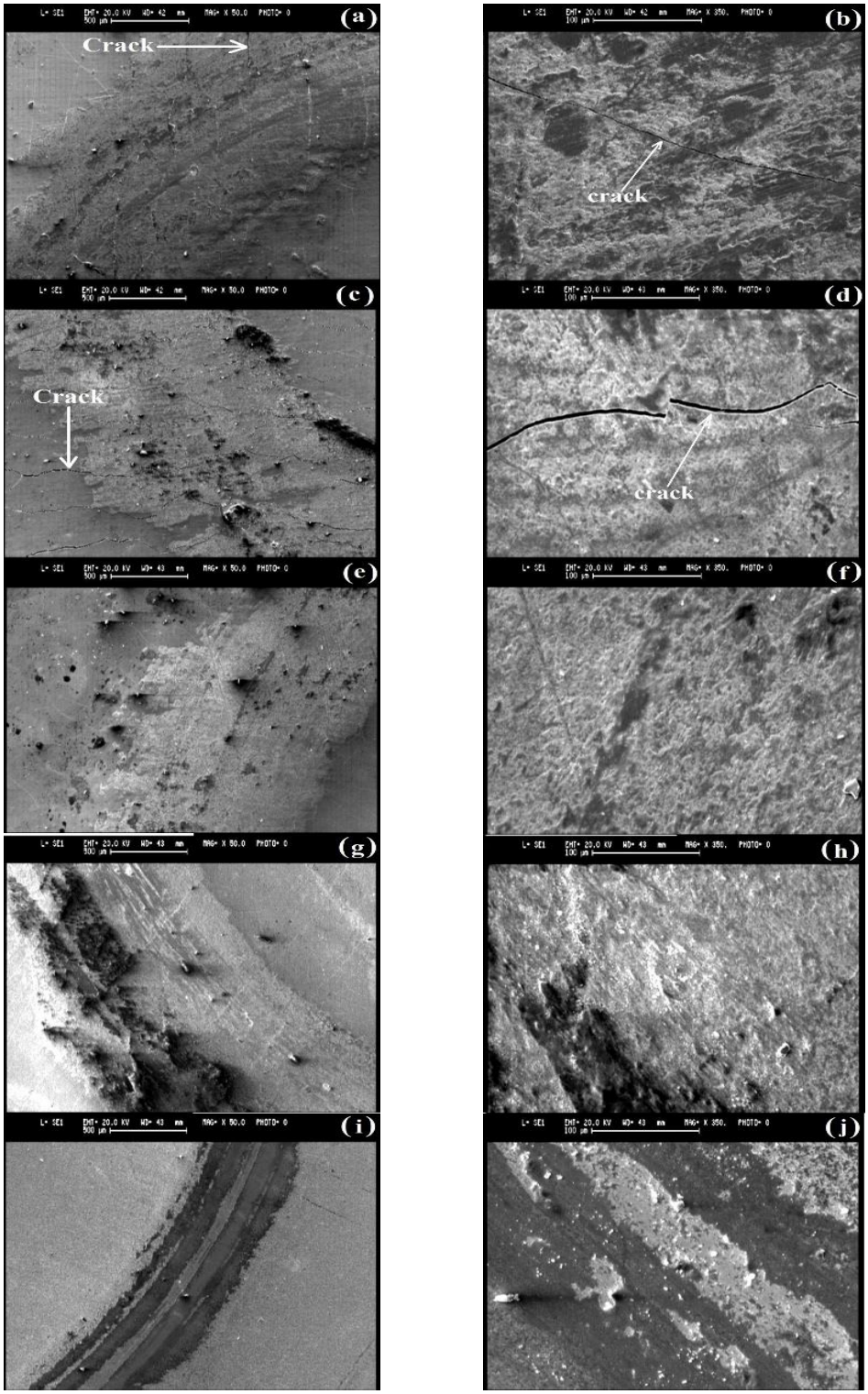

Fig. 7: SEM micrographs from the worn surfaces of the Cr coatings electroplated from baths containing: a) 0 g/L (50×), b) 0 g/L (350×), c) 0.5 g/L (50×), d) 0.5 g/L(350×), e) 1 g/L (50×), f) 1 g/L (350×), g) 2.5 g/L (50×), g) 2.5 g/L (350×), h) 5 g/L (50×) and i) 5 g/L (350×) MoS_2 particles.

Table. 4: Tafel polarization test results of the Cr coatings that were electrodeposited from baths containing different amounts of MoS_2 particles.

MoS_2 concentration in bath (g/L)	$i_{corrosion}$ (A/cm^2)	$E_{corrosion}$ (V) vs. Ag/AgCl	$-\beta_c$ (mV/dec)	β_a (mV/dec)
0	2.42 E-7	-0.437	217	23
0.5	1.24 E-7	-0.507	342	234
1	2.82 E-8	-0.579	245	249
2.5	1.07 E-7	-0.478	42	85
5	2.69 E-7	-0.457	23	80

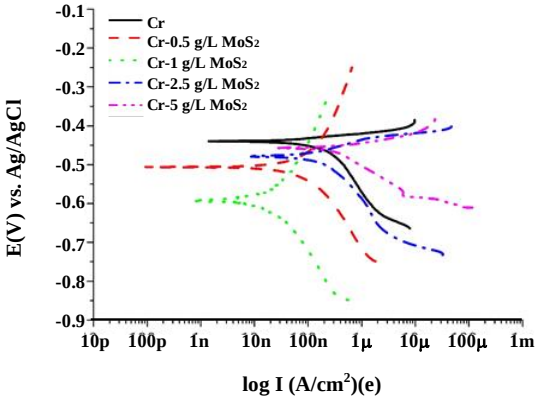

Fig. 8: Polarization curves of the Cr coatings electroplated from baths containing different concentrations of MoS_2 particles.

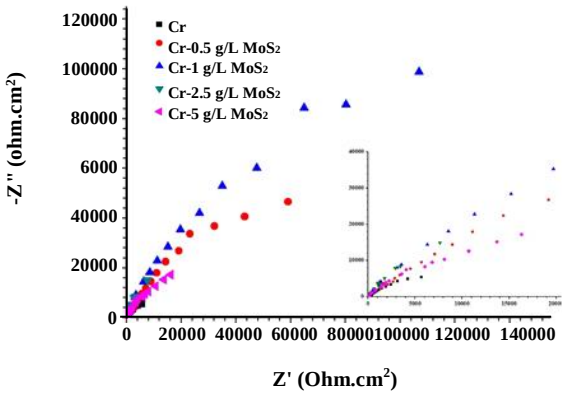

Fig. 9: AC responses of the Cr coatings electroplated from baths containing different concentrations of MoS_2 particles.

regarded as the one with the lowest amount of surface roughness since it has the highest fitted value of parameter n among the others. Such observation is in accordance with the surface roughness test results that are shown in Fig. 6.

CONCLUSIONS

Different amounts of MoS_2 particles in addition to the anionic surfactant (SDS) were added to the conventional chromium electroplating bath to electroplate crack-free chromium coating. The results showed that the Cathodic Current Efficiency (CCE) of the baths decreases as the concentration of the MoS2 particles in them increases and no chromium will be deposited from the baths containing more than 5 g/l MoS_2. According to X-ray diffraction spectroscopy and EDX results of the electrodeposited coatings, no of MoS_2 particle was incorporated into the Cr deposits. On the other hand, crack-free Cr coatings were electroplated as the MoS_2 concentration of the bath increased up to 0.5 g/L. The crack-free coatings showed an increase in hardness values; however, there would be a decline in such values for the coatings electroplated from baths containing MoS_2 concentrations above 1g/L as a result of less residual stress in the deposits caused by more consumption of the cathodic charges. The corrosion test results indicated that the corrosion resistance of the crack-free coatings would be increased; nontheless, higher concentrations of the MoS_2 particles in the baths above 1 g/L led to lower corrosion resistance (still higher than the hard Cr coating) mainly due to more surface active sites imposed by higher surface roughness. Last but not least, the bath temperature of the present study could be maintaind at 55 ^oC which is well below the temperatures suggested for the electroplating of the crack-free Cr coatings. Thus, the crack-free Cr coating electrodeposited from 1 g/L MoS_2 bath could be a potentially good candidate for applications that simultaneously require wear and corrosion resistances.

Table. 5: The fitted results to the experimental EIS data of the Cr coatings that were electrodeposited from baths containing different amounts of MoS_2 particles.

MoS_2 concentration in bath (g/L)	R_s ($\Omega.cm^2$)	R_{ct} ($k\Omega.cm^2$)	n	Q (F/cm^2)
0	4.89	31.19	0.679	3.92 E-4
0.5	2.80	153.76	0.773	6.12 E-5
1	5.60	248.23	0.851	1.94 E-5
2.5	7.83	73.35	0.739	8.07 E-5
5	6.76	44.6	0.702	1.73 E-4

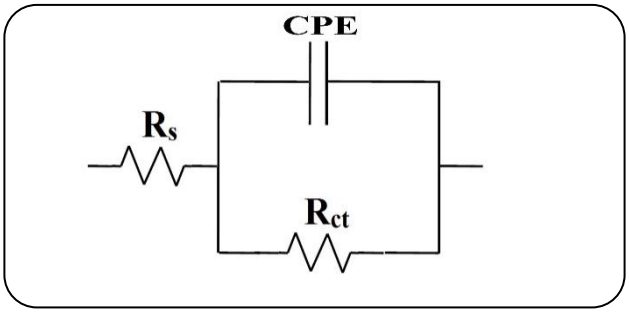

Fig. 10: The equivalent circuit that was used for fitting the experimental EIS data are shown in Fig 9 [19].

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