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Effect of H_2O Addition on the Corrosion Resistance of Trivalent Chromium-Carbon Coatings Electrodeposited from Choline Chloride Deep Eutectic Solvent

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The chromium coatings are successfully electrodeposited from the deep eutectic solvent with content of H_2O from 0 wt.% to30 wt.%. In lower added content of H_2O (0-5 wt.%) in deep eutectic solvent, the electrodeposited coatings belong to hexavalent chromium. When the higher added content of H_2O (10-30 wt.%) in deep eutectic solvent, the electrodeposited coatings belong to trivalent chromium. The surface roughness of coatings decrease from 2.12 um to 0.69 um with an increase of H_2O content in the deep eutectic solvent due to the conductivity of the deep eutectic solvent becomes better during the electrodeposition process. The coatings electrodeposited from the deep eutectic solvent with added 10 wt.% H_2O has the lowest corrosion current density approximately at $4.02 \text{ X } 10^{-6}$ A/dm^2.

Keywords: deep eutectic solvent, hexavalent chromium, trivalent chromium

1. INTRODUCTION

Hexavalent chromium coating has been widely used in industry for a long time for the purposes of decoration, anti-wear and anti-corrosion. Owing to the manufacture process of hexavalent chromium coating is highly noxious and may bring about serious environmental concerns [1], therefore, European Union had passed the ‘‘RoHs’’ and ‘‘WEEE’’ directives to restrict hexavalent chromium in electrical and electronic equipment [2,3]. In order to improve the harmful problems caused by hexavalent chromium, a newly and low toxicity manufacture process of trivalent chromium coating [4-8] or trivalent chromium-carbon coating [9-13] were developed by researchers in recently years. However, in aqua-trivalent chromium bath system, the complexes agent must to be added into the bath to promote the reduction reaction of trivalent chromium ions [14], and those complexes agent such as formats, aminoacids, glycine have low thermodynamic stability and high kinetic inertness will affect plating performance [15,16]. Survilienė et al. [17] also indicated that the trivalent chromium plating solution still exist some problems that are not easy to solve such as a very complicated solution chemistry of Cr(III) complexes in water solutions. In order to improve the above shortcomings of the traditional trivalent chromium electroplating aqueous solution, a new generation of deep eutectic solvent (DES) has been developed in recent years [18-21].

Deep eutectic solvent is a eutectic mixture composed of hydrogen bond donors and acceptors in a certain stoichiometric ratio through hydrogen bonds [22]. Deep eutectic solvent has higher thermal fixation and good stability to water and atmosphere than that of aqueous solutions and ionic liquids [23], it also has some advantages such as non-toxic, biodegradable and biocompatible can be classified as a green solvent [24]. The composition of deep eutectic solvent is divided into main salt, hydrogen bond donor (HBD) and hydrogen bond acceptor (HBA). Previous study indicated that used choline chloride as HBA and ethylene glycol as HBD to compose a deep eutectic solvent is more stable, higher conductivity and lower viscosity than that of reline or glyceline [25,26]. Even so, the viscosity of the deep eutectic solvent will be very high without adding water, which will cause great restrictions on industrial applications. If you want to improve the applicability of deep eutectic solvents in electrodeposition, the plasticizer effect of water must be used, i.e. to add small amounts of water can significantly modifies the viscosity without impairing its properties. In the past, there are very few studies on the use of deep eutectic solvents to prepare electrodeposited trivalent chromium coatings. Even if there are few research literatures, they only discuss the theories of electrochemical reactions, and there is almost no actual comparison and research on the performance of the coatings. Therefore, the use of deep eutectic solvents to prepare electrodeposited trivalent chromium coatings, the mechanical properties and corrosion resistance of the obtained coatings are worthy of in-depth discussion.

In this study, the effect of content of H_2O in deep eutectic solvent in the corrosion resistance and microstructure of electrodeposited trivalent chromium coatings are study. The effect of content of H_2O on the physic property of deep eutectic solvent were also analyzed by measurements of viscosity.

2. EXPERIMENTAL

In this study, the trivalent chromium coatings were electrodeposited on copper substrates with a dimension of 30 × 30 × 2 mm from the deep eutectic solvent with different added content of H_2O. The chemical composition of deep eutectic solvent and electroplating parameters are shown in Table 1. The HBD and HBA of deep eutectic solvent are choline chloride (1.0 mol) and ethylene glycol (2.0 mol) which mole ratio is 1:2, the addition quantity of main salt ($CrCl_3 \cdot 6H_2O$) is 0.4 mol. The addition content of H_2O is from 0 to 30 wt.%, electrodeposited at 25°C with a stirring speed at 400 rpm and current density at 15 A/dm^2 for 15 min.

Table 1. Plating bath composition and operating conditions

Composition and operating conditions	
$CrCl_3·6H_2O$	0.4 mol
Choline chloride	1.0 mol
Ethylene glycol	2.0 mol
Added content of H_2O	0-30 wt.%
Temperature	25℃
Stirring speed	400 rpm
Current density	15 A/dm^2
Electroplating time	15 min

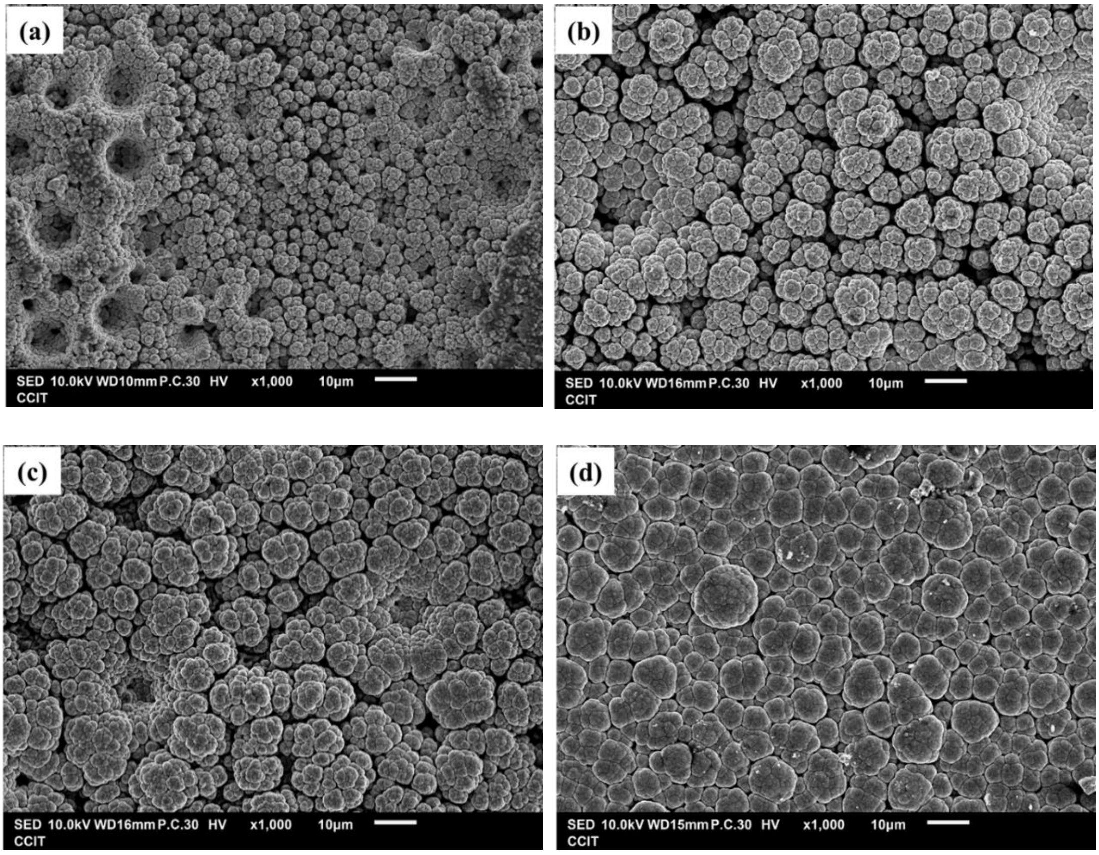
The viscosity of deep eutectic solvents with different added content of H_2O was measured with a viscometer (Walter Her200, Germany). The measurement method of Saybolt viscosity is to drop the liquid to be tested from the drip hole into the scribe line of the standard collection cup (60 ml), and time it. The distance between the collection cup and the drip hole is 12 cm. When the timing is stopped, the time obtained is called the "Saybolt second". The "Saybolt viscosity" can be calculated by following formula: $\upsilon = A \bullet t - B/t$, where υ is "Saybolt viscosity", A and B are constant and t is "Saybolt second". The morphology and cross-sectional images of trivalent chromium coatings prepared from deep eutectic solvent bath were analyzed using a scanning electron microscopy (SEM, JEOL JSM-IT100, operating at 10 kV), the chemical composition of trivalent chromium coatings prepared from deep eutectic solvent bath were confirmed by an energy dispersive spectrometer (EDS). The phase identification of trivalent chromium coatings were examined by a X-ray diffractometer (XRD, BRUKER D2 PHASE) using Cu Kα radiation (λ=0.15405 nm) with a scanning range from 20° to 95°. The potentiodynamic polarization of trivalent chromium coatings were analyzed using a standard three-electrode cell system carried out with an Autolab-PGSTAT30 potentiostat/galvanostat controlled by a GPES (General Purpose Electrochemical system) software and stabilized at open circuit potential (OCP) before analysis. The analyzed parameters of potentiodynamic polarization test including using a 3.5% NaCl solution at room temperature, the potential range between −1.5 V and 0.5 V with a scanning rate of 0.5 mV s^{-1}. Before the measurement, the samples were degreased and rinsed with DI water. The roughness of trivalent chromium coatings prepared from deep eutectic solvent bath were examined by a 3D surface profilometer (Chroma 7503, Taiwan), each sample was measured five times.

3. RESULTS AND DISCUSSION

The calculated "Saybolt viscosity" of deep eutectic solvent with content of H_2O are shown in Table 2. The Saybolt viscosity of the deep eutectic solvent without adding H_2O is approximately at 277.0 cs, the viscosity value significantly drops down to 116.3 cs after adding 5 wt.% H_2O. When the added content of H_2O is 10, 15, 20, 25, 30 wt.%, the viscosity of deep eutectic solvent is approximately at 56.6, 33.9, 22.7, 16.4 and 11.4, respectively. The viscosity change trend of deep eutectic solvent with increasing content of H_2O is agreement with the study of Gabriele et al. [27].

Table 2. The analysis of Saybolt viscosity of deep eutectic solvent with content of H_2O

Sample code	Saybolt viscosity (cs)
without adding H_2O	277.0
adding 5 wt.% H_2O	116.3
adding 10 wt.% H_2O	56.6
adding 15 wt.% H_2O	33.9
adding 20 wt.% H_2O	22.7
adding 25 wt.% H_2O	16.4
adding 30 wt.% H_2O	11.4



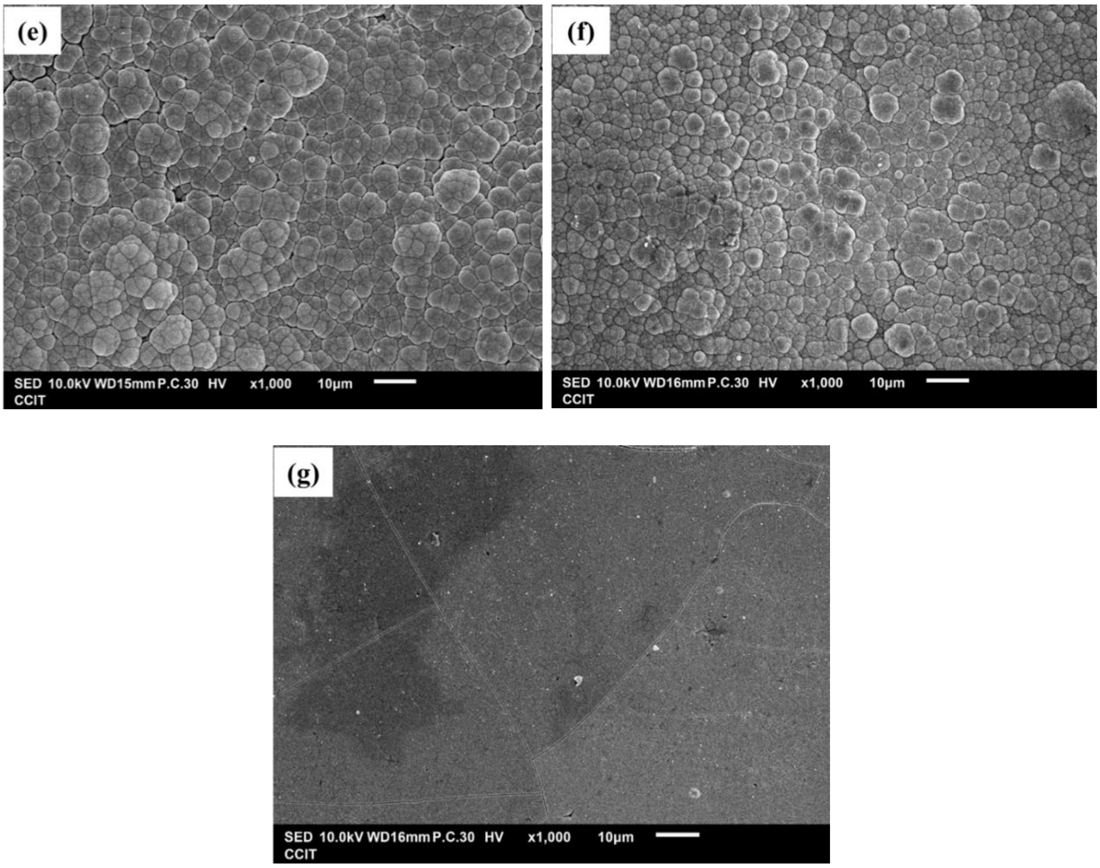


Figure 1. The SEM morphology images of trivalent chromium coatings prepared from deep eutectic solvent bath with content of H_2O: (a) without adding H_2O, (b) adding 5 wt.% H_2O, (c) adding 10 wt.% H_2O, (d) adding 15 wt.% H_2O, (e) adding 20 wt.% H_2O, (f) adding 25 wt.% H_2O, (g) adding 30 wt.% H_2O.

Fig. 1 presents the SEM morphology images of trivalent chromium coatings deposited from deep eutectic solvent bath with various content of H_2O. The coatings deposited from the deep eutectic solvent without H_2O has a large number of obvious pores formed by hydrogen evolution reaction (Fig. 1(a)). As the content of H_2O is 5 and 10 wt.%, the pores caused by hydrogen evolution reaction significantly decreased, the microstructure of trivalent chromium coatings appear a cauliflower structure (Fig. 1(b) and (c)). When the content of H_2O in the deep eutectic solvent is from 15 to 25 wt.%, the gaps in the cauliflower structure and the pores caused by hydrogen evolution reaction disappeared, and the trivalent chromium coatings showed a complete morphology dominated by a nodules structure (Fig. 1(d) and (f)). When the content of H_2O in the deep eutectic solvent increase to 30 wt.%, the surface morphology of trivalent chromium coatings becomes very smooth and appears cracks (Fig. 1(g)).

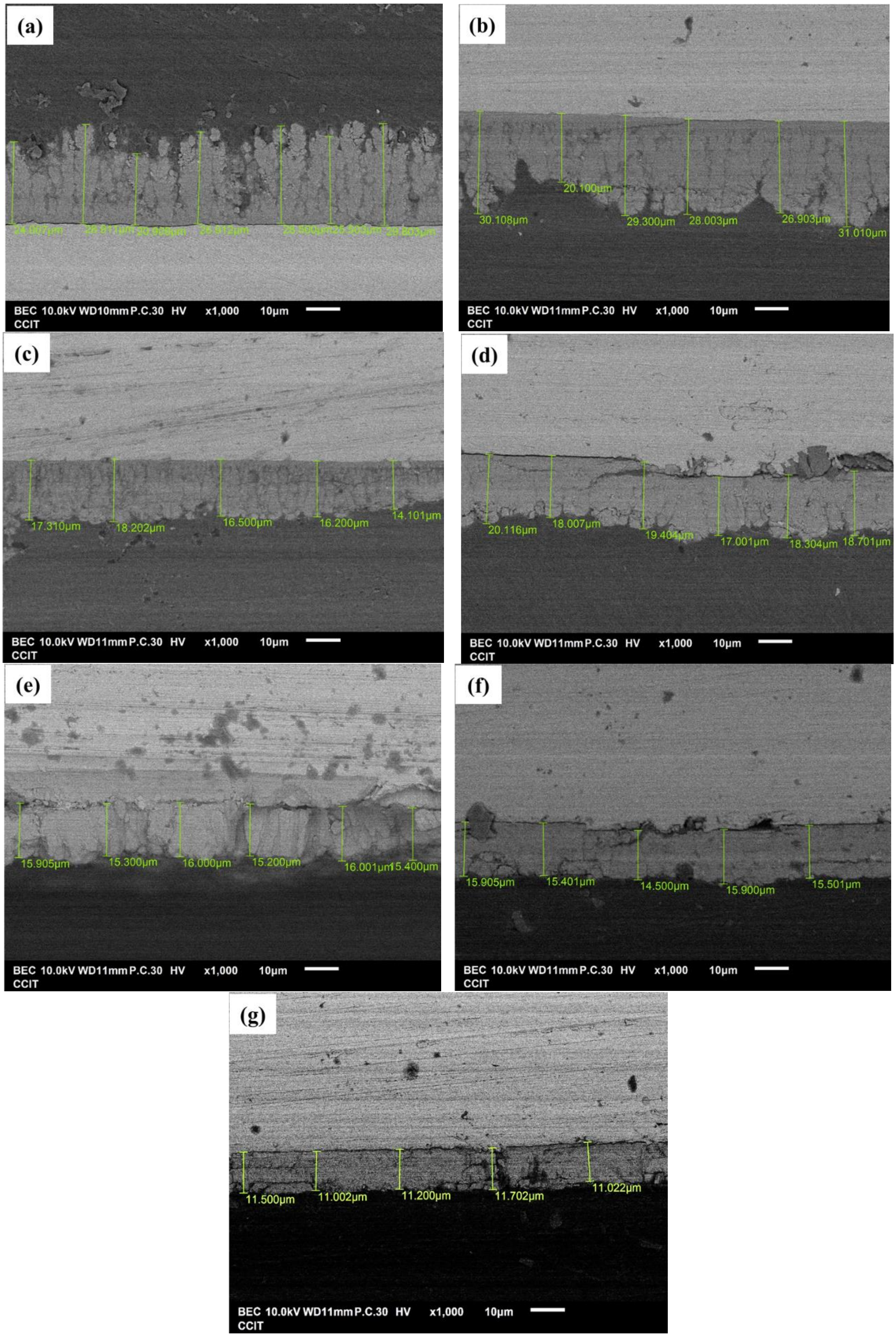


Figure 2. The SEM cross-sectional images of trivalent chromium coatings prepared from deep eutectic solvent bath with content of H_2O: (a) without adding H_2O, (b) adding 5 wt.% H_2O, (c) adding 10 wt.% H_2O, (d) adding 15 wt.% H_2O, (e) adding 20 wt.% H_2O, (f) adding 25 wt.% H_2O, (g) adding 30 wt.% H_2O.

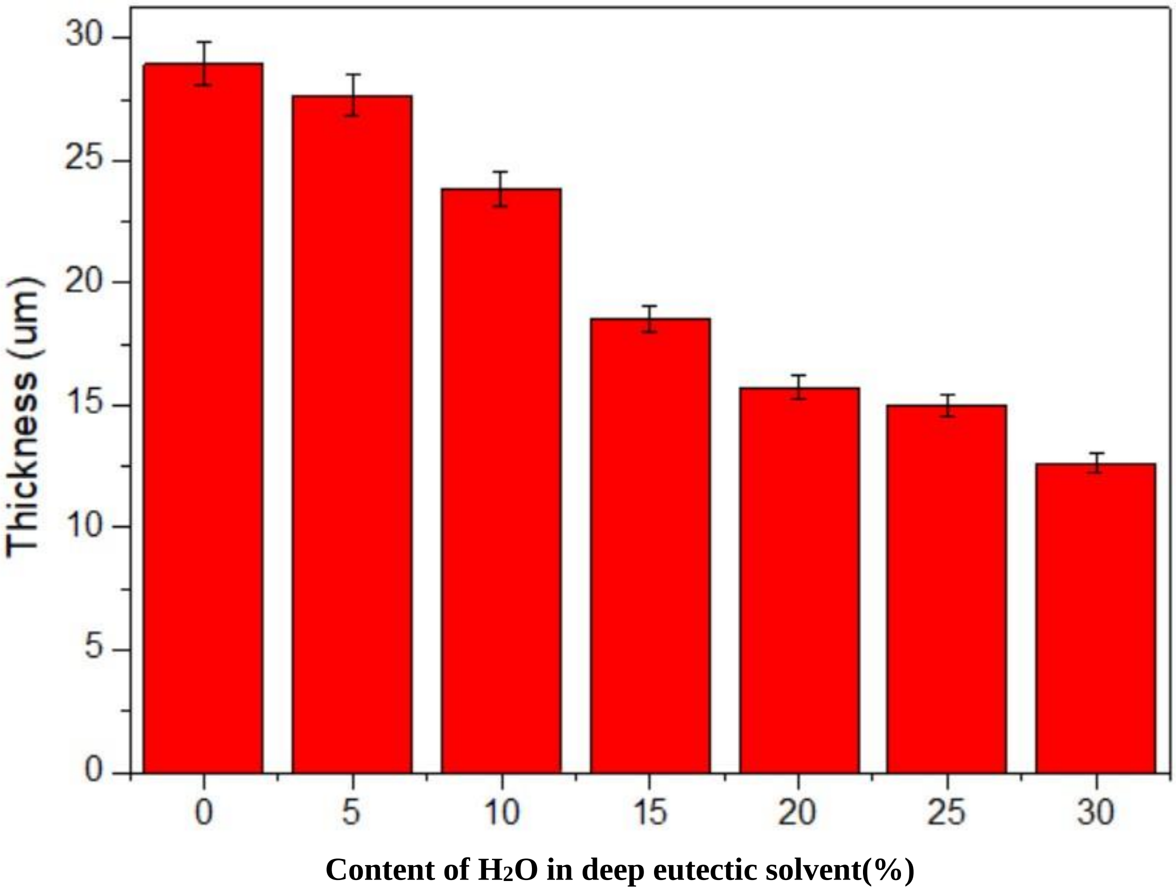


Figure 3. The thickness of trivalent chromium coatings prepared from deep eutectic solvent bath with content of H_2O.

Fig. 2 presents the SEM cross-sectional images of trivalent chromium coatings prepared from deep eutectic solvent bath with content of H_2O. It can be observed that the coating has a dendrites structure, when the water addition in the deep eutectic solvent ranges from 0 to 10 wt.% (Fig. 2(a)-(c)), and their microstructure is loose. Fig. 2(d) shows the cross-sectional SEM image of trivalent chromium coatings prepared from deep eutectic solvent bath with the content of H_2O is 15 wt.%, the loose dendritic structure is reduced a lot and replaced by a denser structure. When the content of H_2O in the deep eutectic solvent is 20-30 wt.%, the dendritic structure disappears and the coating exist a denser structure. This phenomenon can be attributed to the conductivity of deep eutectic solvent become better with the increased adding content of H_2O will facilitate the reduction of metal ions during the electrodeposition process [27]. Protsenko et al. [28] also indicated that the conductivity of deep eutectic solvent are affected both by temperature and water content, indicating the suitable H_2O content can improve the reduction reaction of chromium ions.

Fig. 3 shows the thickness of trivalent chromium coatings prepared from deep eutectic solvent bath with content of H_2O. The thickness of trivalent chromium coatings decrease with an increase of content of H_2O in the deep eutectic solvent. Although the thickness of the coating can be thicker in a deep eutectic solvent with a H_2O content of 0 and 5 wt.%, the structure of the coating is relatively loose.

This is directly related to the conductivity of the deep eutectic solvent. The previous studies indicated that the low electrical conductivity indicates reduced covering power of the plating bath, which is extremely undesirable for chromium electroplating [29, 30].

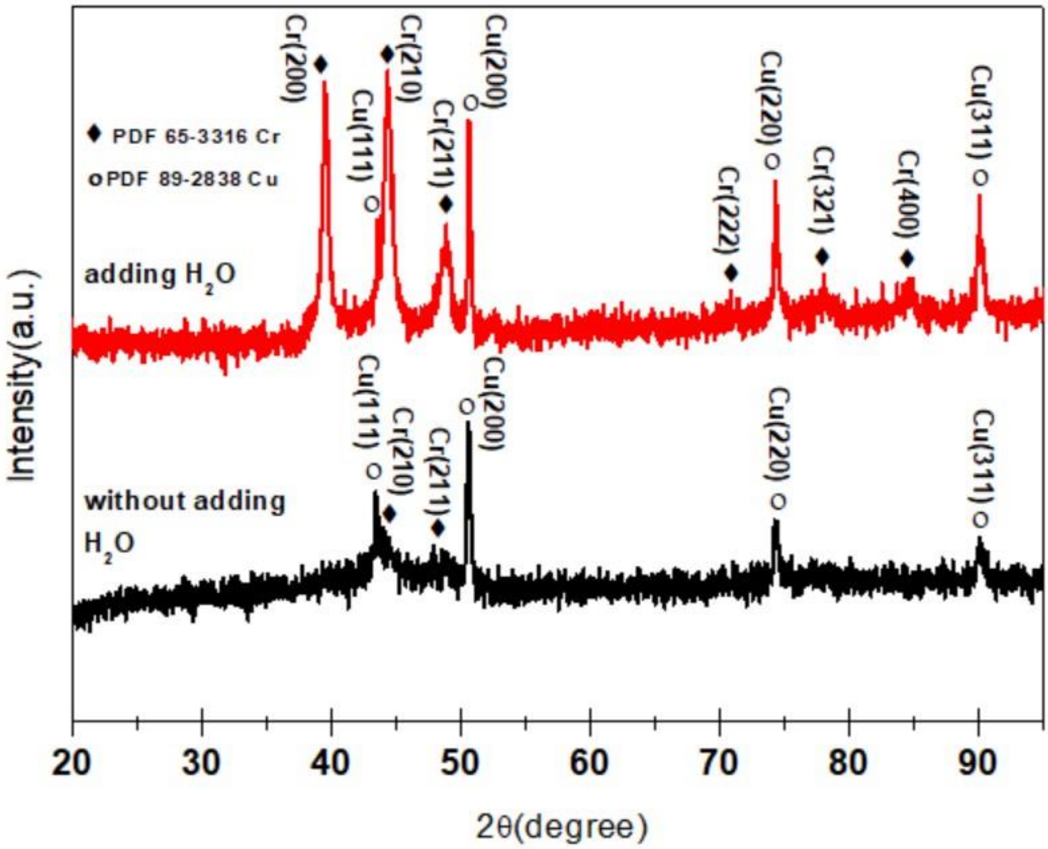
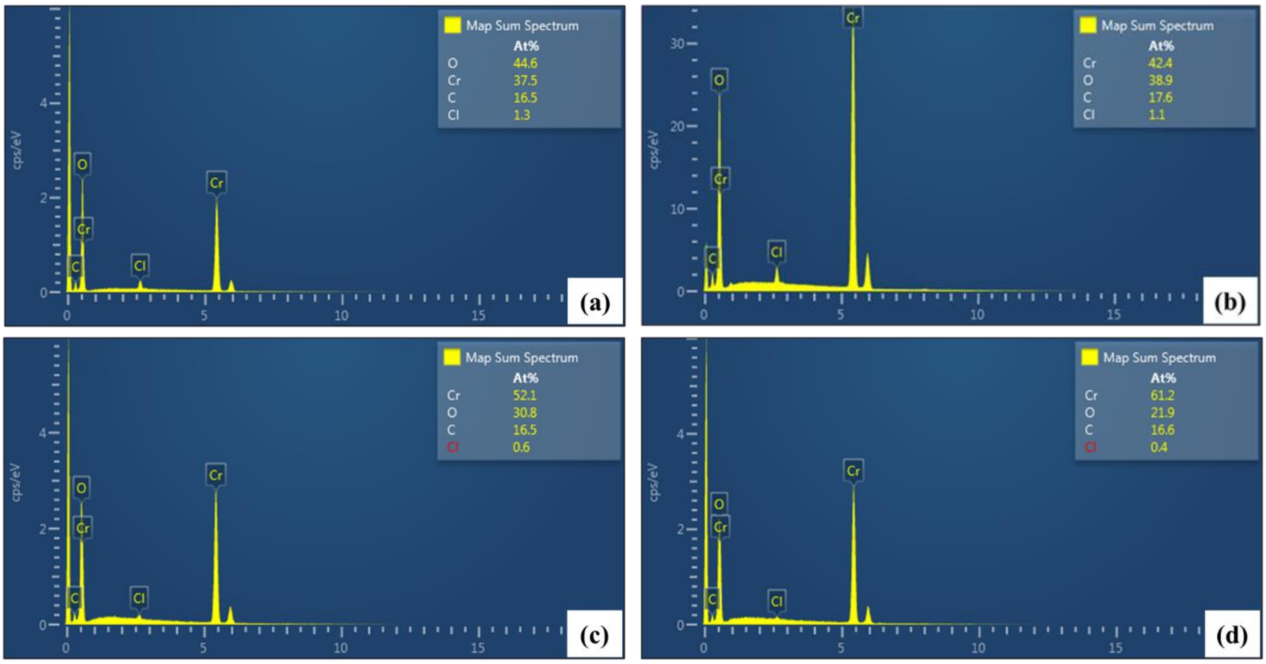


Figure 4. The X-ray patterns of trivalent chromium coatings prepared from deep eutectic solvent with added H_2O and without added H_2O.



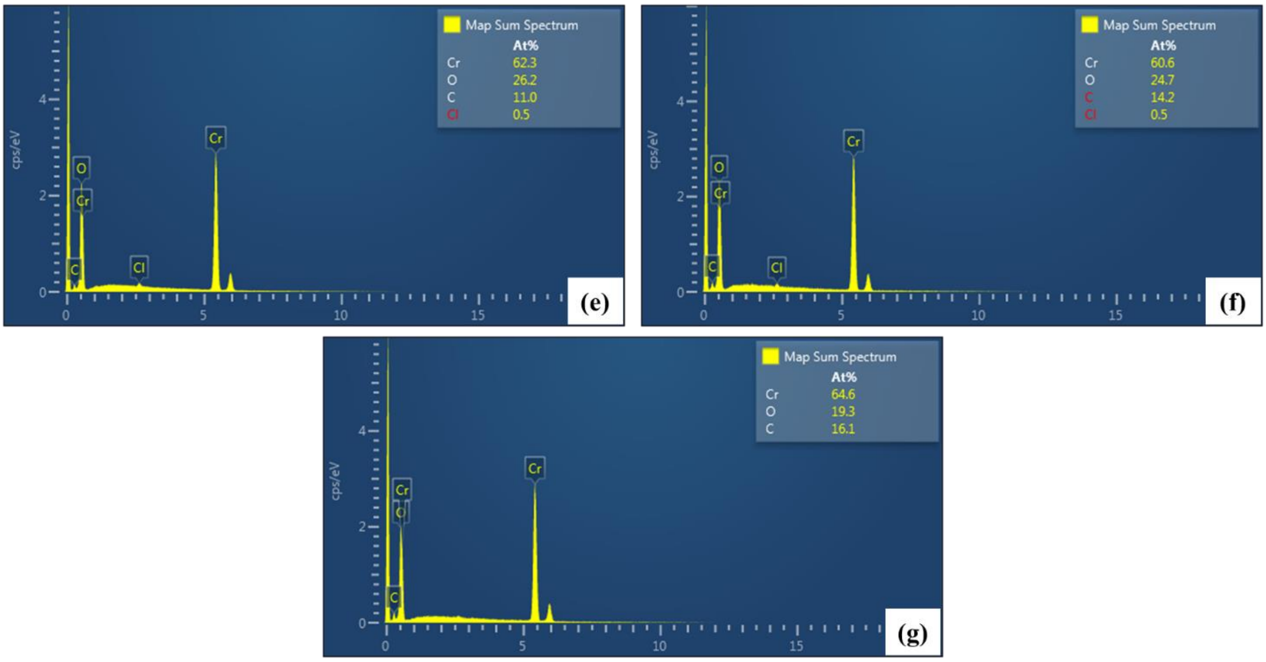
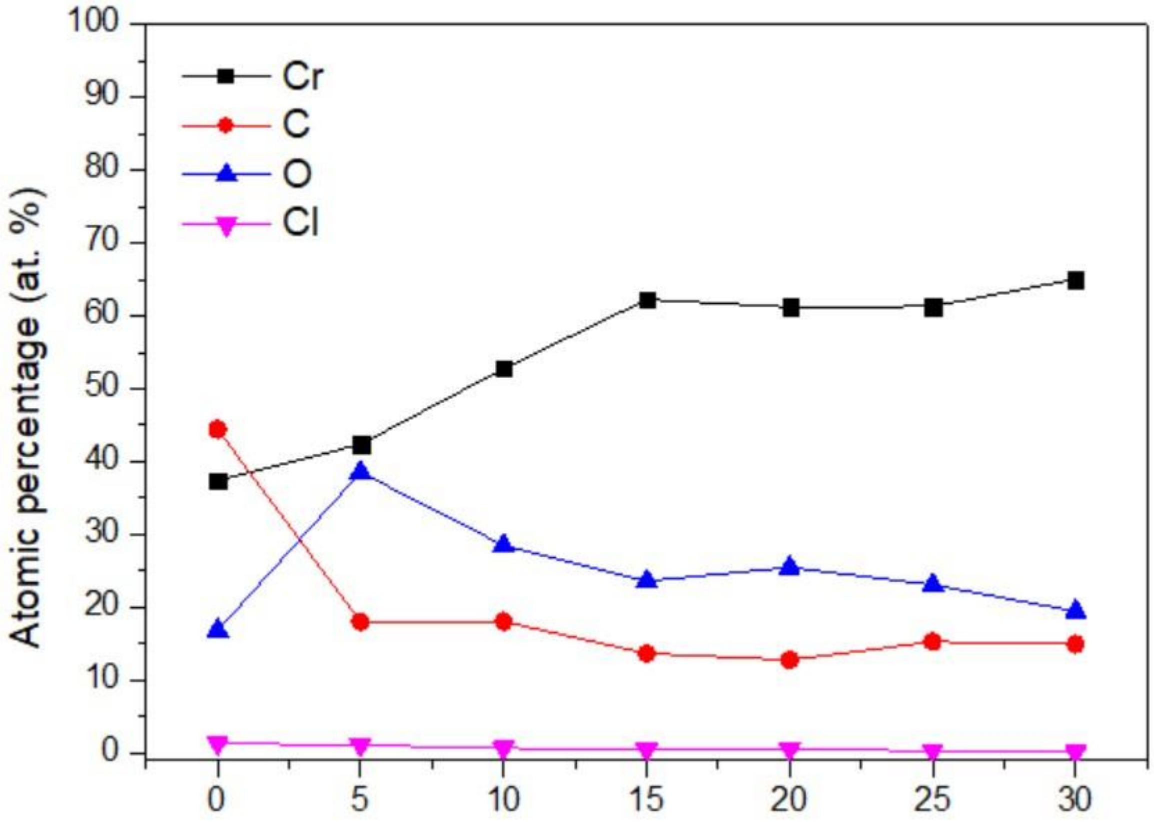


Figure 5. The EDS analysis of trivalent chromium coatings prepared from deep eutectic solvent bath with content of H_2O.

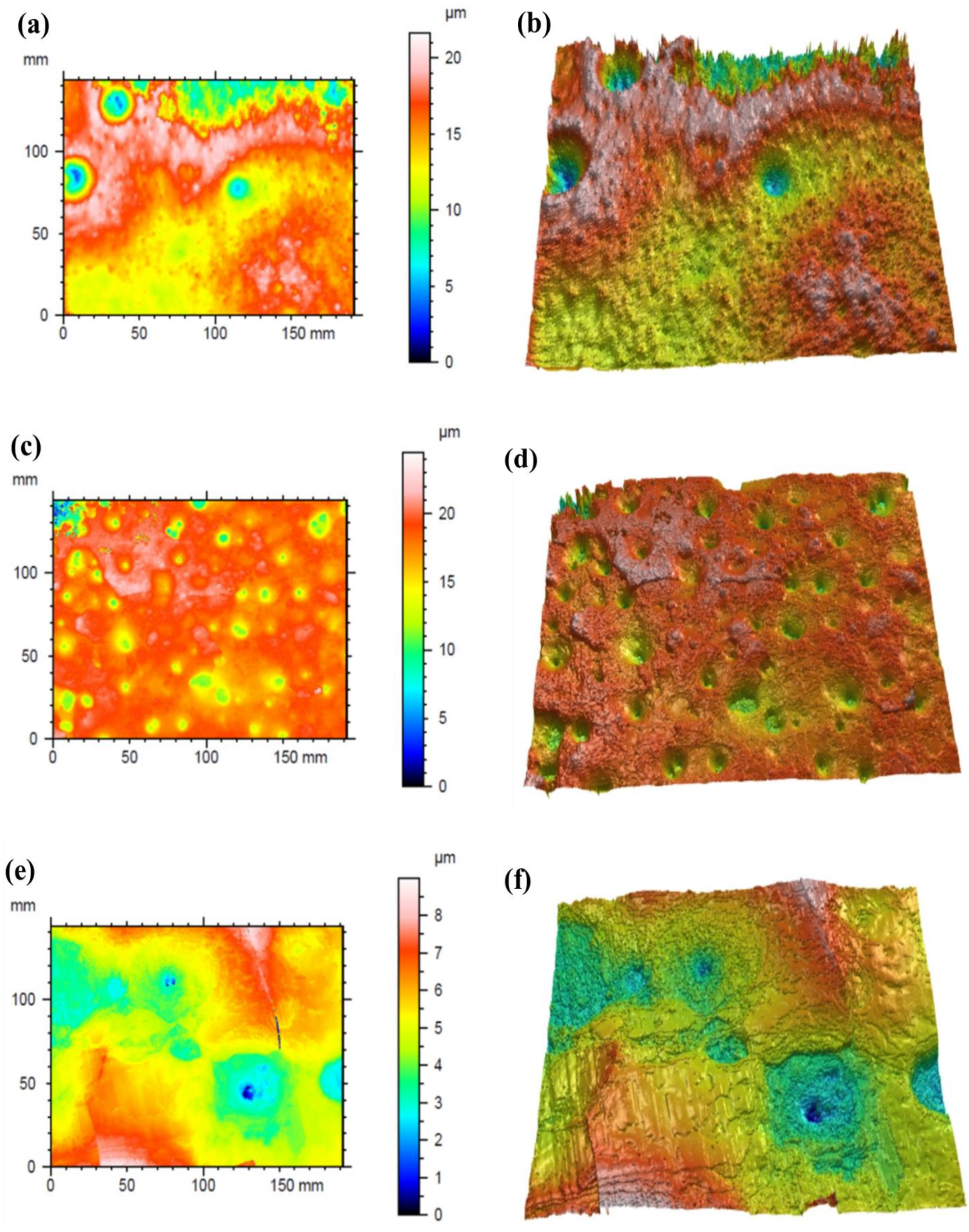
Fig. 4 presents the X-ray patterns of trivalent chromium coatings prepared from deep eutectic solvent with added H_2O and without adding H_2O. The diffraction angle (2θ) occurs at 43.35°, 50.41° and 74.08° indicating the copper (JCPDS # 89-2838), 39.26°, 44.09°, 48.68°, 71.11°, 77.8° and 84.49° indicating the chromium (JCPDS # 65-3316). The diffraction intensity of chromium of the coatings prepared from deep eutectic solvent bath without adding H_2O is very weak, indicating the structure of the coating is close to the amorphous state. The formation of amorphous deposits in trivalent chromium plating electrolytes had been observed in the previous studies [31,32]. The diffraction intensity of trivalent chromium coatings become stronger such as the "Miller index" plane of (200), (210), (211), (222), (321) and (400) for chromium. On the other hand, the crystallinity of the trivalent chromium coatings becomes higher in the condition of adding H_2O into deep eutectic solvent. The composition analysis of the EDS composition of the trivalent chromium coating prepared under different conditions is shown in Fig. 5.

The changes of elements such as chromium, carbon, oxygen, and chlorine in the trivalent chromium coatings deposited under different conditions are also sorted into a curve as shown in Fig. 6. The highest concentration of carbon (44.40 at.%) and chlorine (1.32 at.%), and the lowest concentration of chromium (37.44 at.%) occurs at the trivalent chromium coatings prepared from deep eutectic solvent without H_2O, chlorine comes from choline chloride which is incompletely reacted, the high concentration of carbon comes from the instantaneous high temperature formed by the electrochemical reaction of the cathode when the ethylene glycol is not added with water, which leads to the carbonization of ethylene glycol. When the H_2O addition amount of the deep eutectic solvent is increased from 5 wt.% to 30 wt.%, the carbon content of trivalent chromium coatings significantly drops to 12.76-18.06 wt.%. The concentration of chromium increase up to 42.38 and 52.76 at.% when the added content of H_2O is 5 and 10 wt.%. With the addition of water from 15 wt.% to 30 wt.%, the chromium concentration in the coating is maintained in the range of 61.22 at.% to 65.02 at.%. The concentration of oxygen of all samples is maintained in the range of 16.84 at.% to 38.54 at.%. The concentration of chlorine decrease with the increase of the added content of H_2O, this is because the increase of H_2O content in the deep eutectic solvent will increase the conductivity of the solvent, so that choline chloride can fully react, and the concentration of chlorine will gradually decrease.



Content of H_2O in deep eutectic solvent(%)

Figure 6. Chemical composition of trivalent chromium coatings prepared from deep eutectic solvent bath with content of H_2O.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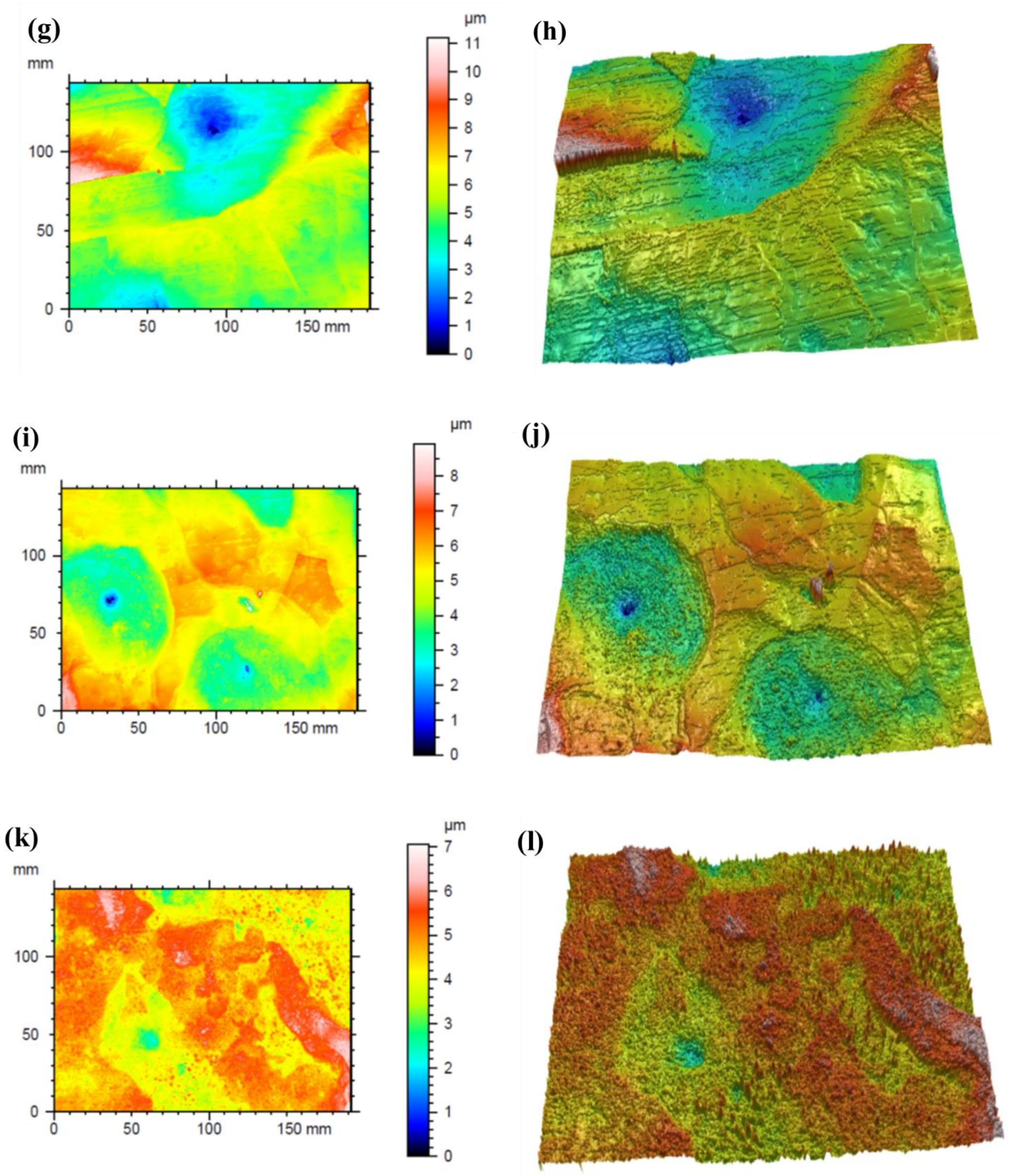
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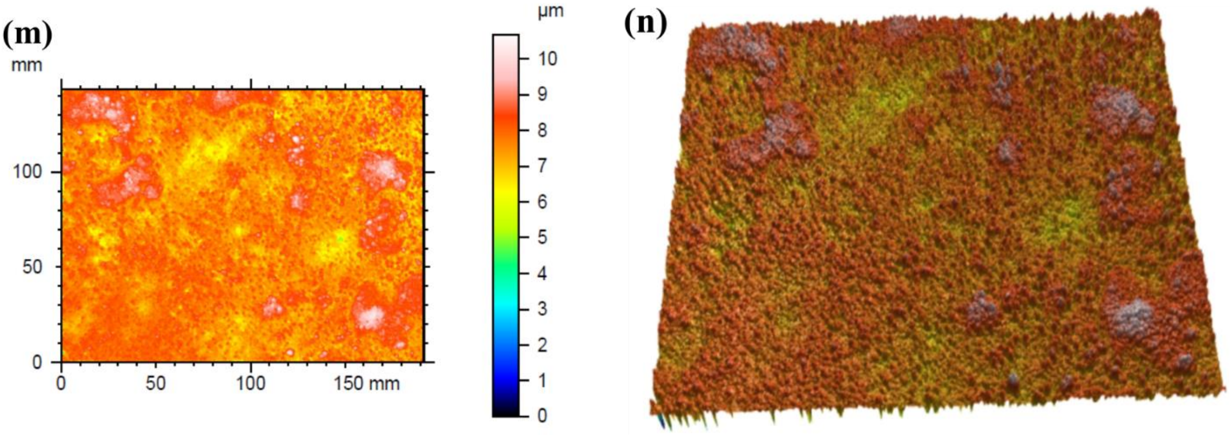

Figure 7. The surface profiles of the 2D top view and 3D profiles of trivalent chromium coatings prepared from deep eutectic solvent bath with content of H_2O.

Fig. 7 shows the surface profiles of the 2D top view and 3D profiles of trivalent chromium coatings prepared from deep eutectic solvent bath with content of H_2O. The surface roughness (S_a) of coatings are also shown in Fig. 8. The coating deposited by the deep eutectic solvent bath without H_2O has a maximum surface roughness approximately at 2.12 um, which can be attributed to the large number of obvious pores formed by the hydrogen evolution reaction. When 5 wt.% and 10 wt.% of H_2O are added to the deep eutectic solvent, the surface roughness of the deposited trivalent chromium coating is 1.89 and 1.20 um, respectively, indicating the addition of H_2O will reduce the formation of pores and decrease the roughness of coatings. When the water content in the deep eutectic solvent increases to 30 wt.%, the coatings can obtain the smallest roughness approximately at 0.69 um.

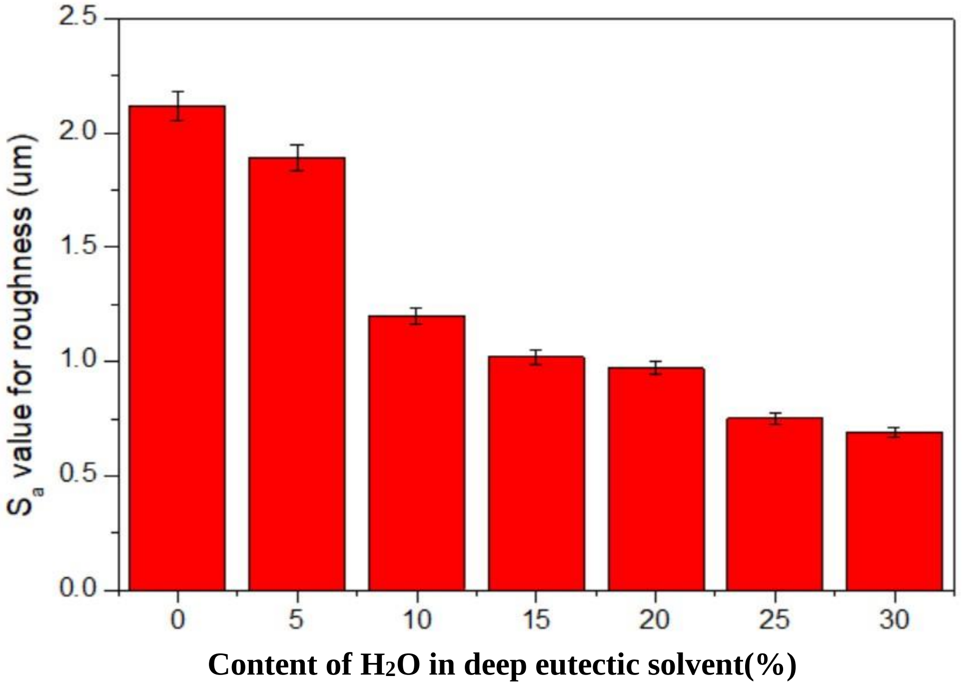

Figure 8. The roughness of trivalent chromium coatings prepared from deep eutectic solvent bath with content of H_2O.

Fig. 9 presents the potentiodynamic curves of trivalent chromium coatings deposited from deep eutectic solvent bath with content of H_2O, the values of E_{corr} and i_{corr} of various coatings are also shown in Table 3. The value of E_{corr} measured from the trivalent chromium coatings deposited from deep eutectic solvent without H_2O and with added 5wt.% H_2O is -0.95 and -0.99 V, respectively. The corrosion potential in this range belongs to the corrosion reaction of hexavalent chromium (Cr^{6+}), and this result is agreement with the previous research literature [33].

Table 3. Corrosion resistance of trivalent chromium coatings prepared from deep eutectic solvent bath with content of H_2O analyzed from 3.5 wt.% NaCl solution.

Sample code	I_{corr} (A/dm^2)	E_{corr} (V vs. SEC)
Cu substrates	2.53×10^{-5}	-0.242
without adding H_2O	3.0×10^{-5}	-0.954
adding 5 wt.% H_2O	8.32×10^{-5}	-0.992
adding 10 wt.% H_2O	4.02×10^{-6}	-0.571
adding 15 wt.% H_2O	8.45×10^{-6}	-0.374
adding 20 wt.% H_2O	2.06×10^{-5}	-0.453
adding 25 wt.% H_2O	1.01×10^{-5}	-0.456
adding 30 wt.% H_2O	1.06×10^{-5}	-0.432

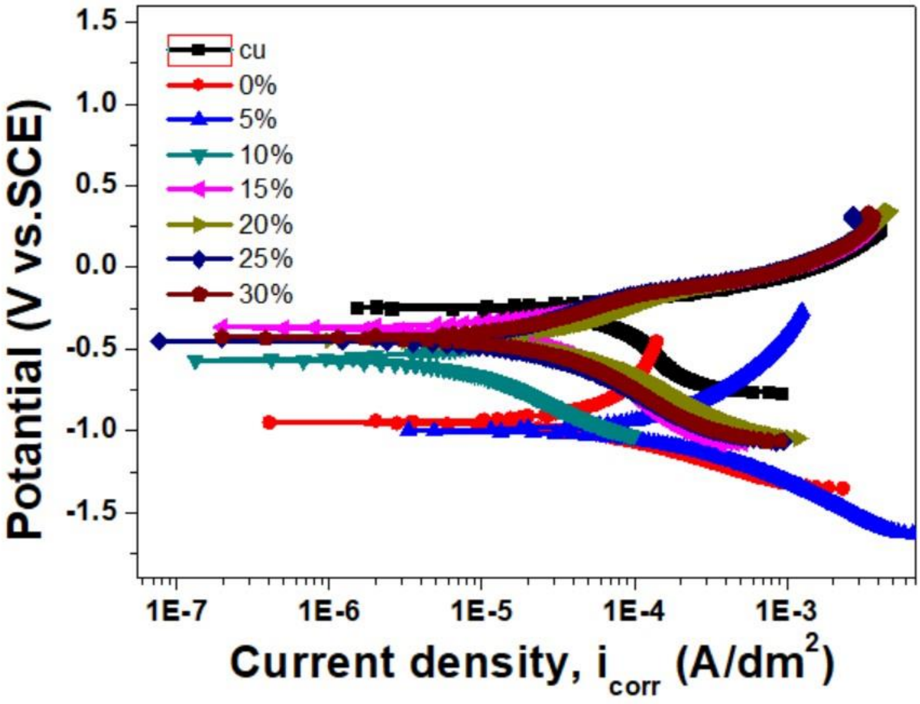

Figure 9. Potentiodynamic curves of trivalent chromium coatings prepared from deep eutectic solvent bath with content of H_2O in 3.5% NaCl solution

Sheu et al. [34] studied the corrosion behavior of trivalent chromium carbon coatings electroplated from both different temperature and concentration of saccharose in the bath, indicating the value of E_{corr} is range from -0.27 V to -0.47 V. In this study, the value of E_{corr} of the coatings prepared from deep eutectic solvent with added content H_2O from 10 wt.% to 30 wt.% is range from -0.37 V to -0.57 V, indicating the coatings belong to trivalent chromium. Therefore, the X-ray pattern of coatings electrodeposited from the deep eutectic solvent without adding H_2O belong to hexavalent chromium, and the coatings electrodeposited from the deep eutectic solvent with higher addition content of H_2O belong to trivalent chromium (Fig. 5). The corrosion current density (i_{corr}) of coatings prepared from deep eutectic solvent without adding H_2O and with added 5 wt.% H_2O is 3.0 X 10^{-5} and 8.32 X 10^{-5} A/dm^2, respectively. The corrosion current density of coatings prepared from deep eutectic solvent with added 10 wt.% and 15 wt.% H_2O is 4.02 X 10^{-6} and 8.45 X 10^{-6} A/dm^2, respectively. When the added content of H_2O in the deep eutectic solvent is 20, 25 and 30 wt.%, the corrosion current density of coatings drop down to 2.06 X 10^{-5}, 1.01 X 10^{-5} and 1.06 X 10^{-5} A/dm^2, respectively. The lower i_{corr} value of coatings electrodeposited at the condition of without adding H_2O and with added 5 wt.% H_2O is caused by the large number of pores and loose microstructure of coatings. When the added content of H_2O in the deep eutectic solvent is 10 and 15 wt.%, the microstructure of electrodeposited coatings become dense and the pores of coatings significantly decrease, leading to a better corrosion resistance. However, when the water content in the deep eutectic solvent is higher than 20 wt.%, the phenomenon of excessive internal stress in the coating caused by the traditional electroplating process reappears, causing cracks in the coating and worsening the corrosion resistance of the coating.

4. CONCLUSIONS

The effect of H_2O content on the microstructure, chemical composition and corrosion resistance of coatings electrodeposited from deep eutectic solvent are studied. The chemical composition of coatings electroplated from the content of H_2O with 0 and 5wt.% in the deep eutectic solvent belong to hexavalent chromium, the content of H_2O increase from 10 to 30 wt.%, the chemical composition of coating belong to trivalent chromium. The surface roughness of coatings decrease from 2.12 um to 0.69 um with an increase of H_2O content in the deep eutectic solvent. The coatings electrodeposited from the deep eutectic solvent with the content of H_2O is 10 wt.% has the lowest corrosion current density approximately at 4.02 X 10^{-6} A/dm^2.

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