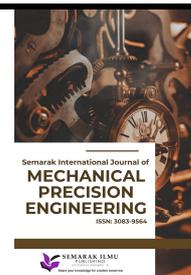




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Properties Trivalent Chromium Coating Influenced by Surface Activation Pretreatment

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ABSTRACT

This study presents a novel approach of optimizing trivalent chromium coating development to achieve enhanced properties, focusing on the interplay between surface activation and coating adhesion. Chromium coating aggrandized their application to aircraft, automobile, and machinery industries. However, their application is impeded by insufficient coating adhesion strength. To enhance coating adhesion strength between metal coating and substrate, a surface activation before electroplating is proposed. Advanced characterization techniques, including Scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX) and surface topography testing were employed to investigate the surface morphology, and roughness of the coating. The key innovation lies in correlating these parameters to achieve coating with compact and uniform morphology, resulting in exceptional performance. The activation was done by acid dipping using different types of acid Sulfuric acid (H₂SO₄) and Hydrochloric acid (HCl) at different concentrations and dipping duration. The trivalent chromium coating has been deposited at 25A/dm² for 10 minutes. The study identifies optimal surface activation by utilizing HCl with a concentration of 30 vol% dipped for 120 seconds to achieve superior coating adhesion. These findings significantly advance the understanding of trivalent chromium coating processes and their potential applications in industries demanding high durability. In addition, it was confirmed that a coating layer of trivalent chromium was successfully deposited by electroplating with a thickness more than 5µm with the addition of the activation method. It is concluded that a meticulously developed activation pretreatment procedure is essential before the electroplating process to create a high-quality coating with notable coating protection.

Keywords:

Trivalent chromium; activation; electrodeposition; coating

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1. Introduction

Chromium coating offers excellent corrosion resistance, wear resistance, and aesthetic appeal when used as a coating [1]. Excellent characteristics have made chromium coating a preferred choice in various industries such as the automotive, aerospace, electronics, and decorative sectors. Traditional electrodeposition methods have heavily relied on hexavalent chromium (Cr^{6+}). However, the use of hexavalent chromium raises significant environmental and health concerns due to its toxicity. The toxic compounds involved in hexavalent chromium plating resulted in severe health issues, including respiratory problems and cancer [2].

Additionally, the fumes of hexavalent chromium into the environment during plating lead to air pollution. Meanwhile, the disposal processes contribute to water and soil pollution. In response to these environmental and health hazards, trivalent chromium (Cr^{3+}) has gained popularity and acts as a safer and environmentally friendly alternative in modern times [3]. Trivalent chromium electrodeposition is highly regarded as an environmentally friendly option because of its reduction in toxicity concern. Trivalent chromium compounds are significantly less harmful compared to hexavalent chromium, making them safer for both workers and the environment. This shift towards trivalent chromium baths represents a significant step toward greener and more sustainable coating processes.

Traditionally, coatings electrodeposited from hexavalent chromium baths have been well-known for their excellent properties. This distinction arises because hexavalent chromium baths can operate at higher current densities and lower temperatures resulting in thicker chromium coatings. In contrast, depositing thick and high-quality chromium layers from trivalent chromium baths has posed significant challenges with the existence of the highly stable $[\text{Cr}(\text{H}_2\text{O})_6]^{3+}$ ions [4]. The existence of the ions caused the complex process of developing the trivalent chromium coating [5]. To overcome this limitation, researchers have employed various complexing agents, such as formic acid, glycine, urea, dimethylformamide and others to destabilize the $[\text{Cr}(\text{H}_2\text{O})_6]^{3+}$ complex [6]. Besides, the needs of additional processes like multilayer coating are required to control the quality of the trivalent chromium.

By introducing these methods, modern trivalent chromium electrodeposition processes have made significant advancements in improving the quality and performance of coatings [7]. Recent innovations have successfully bridged the gap, enabling the production of trivalent chromium coatings with comparable quality to those obtained from hexavalent chromium baths. The different compositions of trivalent chromium baths require varying working conditions to achieve the best coating. Both methods required additional costs and were time-consuming. However, there remains a significant gap in the literature regarding the specific influence of surface activation on the deposition process of trivalent chromium electroplating. Current studies have not sufficiently explored how different surface activation techniques impact the quality and characteristics of the deposited coating.

The motivation for this study arises from the need to bridge the gap between the performance of coatings from hexavalent and trivalent chromium baths. By optimizing the development method for trivalent chromium coating with the utilization of the activation stage, this research aims to enhance the adhesion of the coatings produced from trivalent chromium baths. This optimization is crucial for applications where high-performance coatings are essential, including automotive parts, aerospace components, and machinery parts. Improved adhesion will provide better performance for components used in demanding environments, ensuring enhanced durability and protection.

To develop a durable coating, the condition of a coating is the essential first place to be considered. Defects such as cracks or pores should be avoided in the protective coating. One of the

important factors to ensure a proper coating formation is the pretreatment step on the substrate. An optimized pretreatment should be concerned to clean and activate the surface of the steel. Then, a defect-free coating can be deposited on the steel to gain a noteworthy improvement in corrosion resistance and other mechanical performance [8]. The most economical activation pretreatment that could be done for surface preparation is acid activation.

Surface pre-treatment activation is important to ensure a substrate is clean and provides a proper condition for the adhesion in the electroplating process [9]. For the best result of the final coating, the oxide layer on the substrate surface needs to be removed completely to ensure the surface is clean as the preparation for the coating to adhere to the substrate. Besides, the surface roughness plays an important role in ensuring the coating is adhered to the substrate surface. Previous work [10] has shown that activation using acid activation leads to better coating adhesion. Acid activation is important to electroplating because it removes the oxide layer, neutralizes alkaline film, eliminates impurities, and activates the modal surface that is prone to the oxide layer on the substrate [11].

The most common activator used in carbon steel surface treatment is corrosive fluid such as hydrochloric acid [12]. Another acid used in activation could be sulfuric acid [13]. The surface profile after surface pre-treatment of carbon steel also showed a significant effect on coating and corrosion protection for long-term service life. A thicker coating could be adhered to by a rough metal substrate to give better corrosion protection, however there is a limit [14]. A very rough surface might contain contaminant debris and a deep valley that is difficult to be coated. It could have an impact on the pitting corrosion [15].

Thus, the surface profile investigation is essential to ensure the quality of the coating. The above-mentioned previous study showed that the key to having a quality final coating from the electroplating process depends on the type and concentration of the activator solution for substrate surface pre-treatment. This indicate that optimizing parameter of acid activation process is important to ensure better coating quality. Besides, the textured surface quality improvement using either mechanical or chemical processes greatly enhances the surface properties [16]. Thus, the objective of this study was to investigate the optimum activation pretreatment for a trivalent chromium coating electroplated on a carbon steel substrate.

2. Methodology

Carbon steel is the most widespread form of steel in the world. AISI 1045 carbon steel disc with a diameter of 25 mm and thickness of 4 mm was used as the substrate in the present study. The AISI 1045 was purchased from Misumi Malaysia Sdn. Bhd. and has a tensile strength of 585 MPa with Vickers hardness of 210 HV.

The main parameters in this study were the types of the activator. The pretreatment was done by submerging the carbon steel plate in sulfuric acid (H_2SO_4) and hydrochloric acid (HCl) activator. The original had a concentration of 38%, and the original H_2SO_4 batch had a concentration of 95%. The batch was diluted with 300 ml of distilled water to yield a concentration of 5 vol% for H_2SO_4 and 30 vol% for HCl. The submergence was done at room temperature under a fume hood for two different time durations: 60 seconds for H_2SO_4 and 120 seconds for HCl.

The carbon steel was usually surface treated by hydrochloric or sulfuric acid. Sulfuric acid was more commonly used because lower cost than hydrochloride acid. It is known that hydrochloric acid and sulfuric acid will have different activation effects. Thus, this study explored the carbon steel's acid cleaning and activation using both acid types at the 60 s to 120 s processing time.

After the activation pretreatment, the steel plate was sent to the electroplating process. The electroplating process used an electrolyte solution from a combination of Chromium Chloride

Hexahydrate ($\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$), Ammonium Formate (NH_4HCO_2), Potassium Bromide (KBr) and Urea ($\text{CH}_4\text{N}_2\text{O}$). In the earlier investigation, the solution concentration details were provided by Huang *et al.*, [17]. The temperature was maintained at room temperature, and the electrolytic solution was continuously stirred at 180 rpm during electroplating to ensure all the particles were well dispersed. Electroplating was conducted at current densities of 25 A/dm^2 with a fluctuating voltage of 10 V. The electroplating process used a graphite plate as the anode. The electroplating process was allocated in 10 minutes to obtain a thin single layer of trivalent chromium coating. Figure 1 shows a schematic diagram of the electroplating process setup.

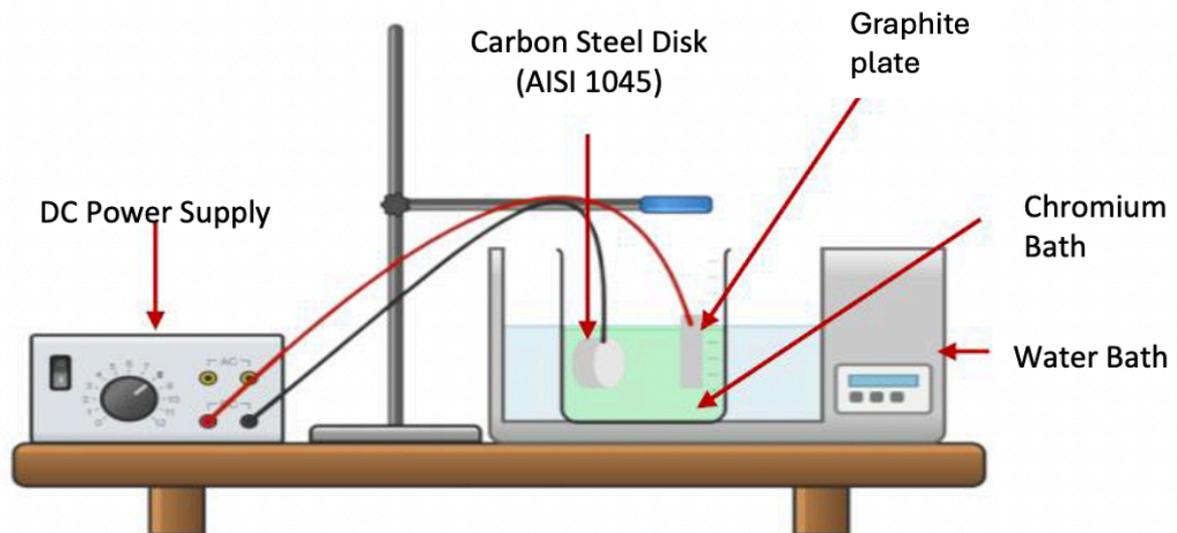


Fig. 1. Schematic diagram for electroplating set-up apparatus

Once the electroplating was done, the coated specimen was tested for its surface coating quality. The scanning electron microscope (SEM) test was used to visually inspect the coating surface. SEM allowed any small cracks and pits in the coating to be detected. The energy dispersive X-ray analysis (EDX) on the other hand, was used to examine the elements of the coated specimen. Both SEM and EDX tests were done by a single machine, JSM-6010PLUS/LA (JEOL Ltd., Japan), equipped with energy dispersion from Oxford Instruments Nano Analysis Plc in the United Kingdom.

The surface roughness of the coated specimen was measured with the assistance of a 3D optical profiler, ZeGageTM (ZYGO Corporation and Ultra Precision Technology Division of AMETEK Inc., United States), equipped with MxTM software. The coating thickness was measured with the cross-section analysis by (SEM).

3. Results

3.1 Surface Morphology

A visual inspection of the coating was carried out using SEM examination. Figure 2 displays the images from the SEM analysis for the carbon steel specimen before the coating process. Figure 2(a) shows that the surface of the specimen without pretreatment was rough and filled with granules. The granules were contaminated from the steel machining process as well as the oxide layer formed on the surface of the specimen. The oxide layer inhibited the coating from forming and adhering to the surface of the specimen.

After the grinding step, some rougher features on the steel surface were removed, and the surface appeared to be smoother as shown in Figure 2(b). After the sample undergoes the activation,

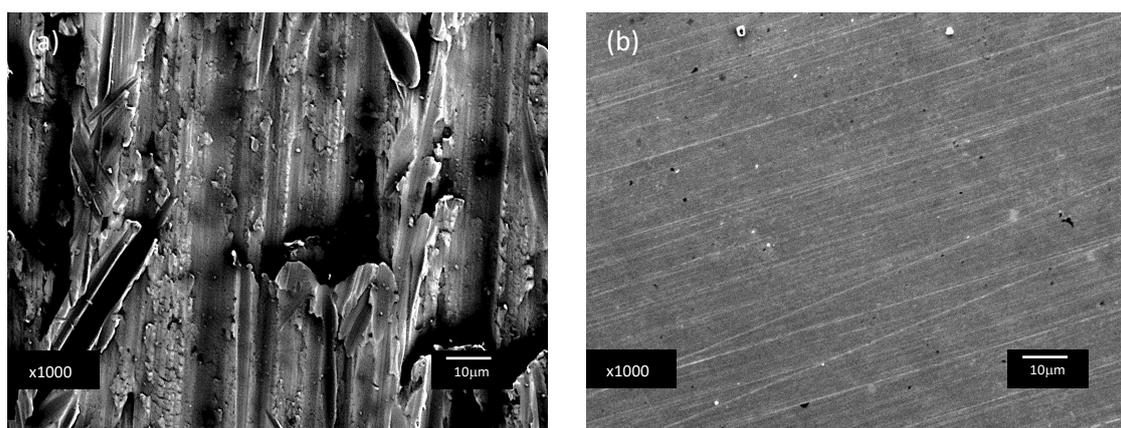
the microstructure can be seen. Pearlite and ferrite are a combination of structures that are normally found in AISI 1045 [18]. The microstructure AISI 1045 was composed of 88% pearlite and 12% ferrite, as reported by [19], with the carbon content of 6.8% and 0.02% respectively, for pearlite and ferrite.

After the sample is activated using H_2SO_4 and HCl, the feature structure can be seen with pearlite and ferrite as confirmed by SEM image analysis, different acid activations show different percentages of ferrite and pearlite structure forms on the carbon steel surface. Figure 2 (c) shows that the surface is filled with ferrite and pearlite structures when activated using H_2SO_4 . Interestingly, the predominant pearlite structure is formed on the surface activated using HCl, as shown in Figure 2 (d).

As confirmed by SEM image analysis, different acid activations show different percentages of ferrite and pearlite structure forms on the carbon steel surface. Pearlite surface covered about 70.75% and 92.66% of the sample activated with H_2SO_4 and HCl, respectively, as indicated in Figures 2 (c) and (d). An interesting finding was found on the ferrite contents, which attribute about 29.25% and 7.34% for the sample activated with H_2SO_4 and HCl, respectively, when analysed with public ImageJ software V1.53, Bethesda, MD, USA. The finding shows that the surface is filled with ferrite and pearlite structures when activated using H_2SO_4 . Interestingly, the predominant pearlite structure is formed on the surface activated using HCl.

Generally, the existence of pearlite and ferrite structures affects the surface. The different amounts of pearlite and ferrite measured after the activation may lead to different coating performances. The variant structure will give different roughness to the coating surface based on its properties as ferrite structure is softer and ductile than pearlite. Meanwhile, pearlite has a harder and more brittle structure [20].

The difference in surface roughness is further discussed based on sample topography. Besides, the difference in structure form after activation also leads to variance morphology of trivalent chromium coating. Figures 2 (e) and (f) indicate that both coated samples show the same morphology, consisting of a nodular shape that is generally found on Trivalent chromium coating. However, the nodular size of the sample activated with HCl as shown in Figure 2 (f), is more extensive than that of the sample activated with H_2SO_4 in Figure 2(e). The extensive nodular size of the sample activated with HCL facilitates difference higher growth rate compared to the sample activated with H_2SO_4 .



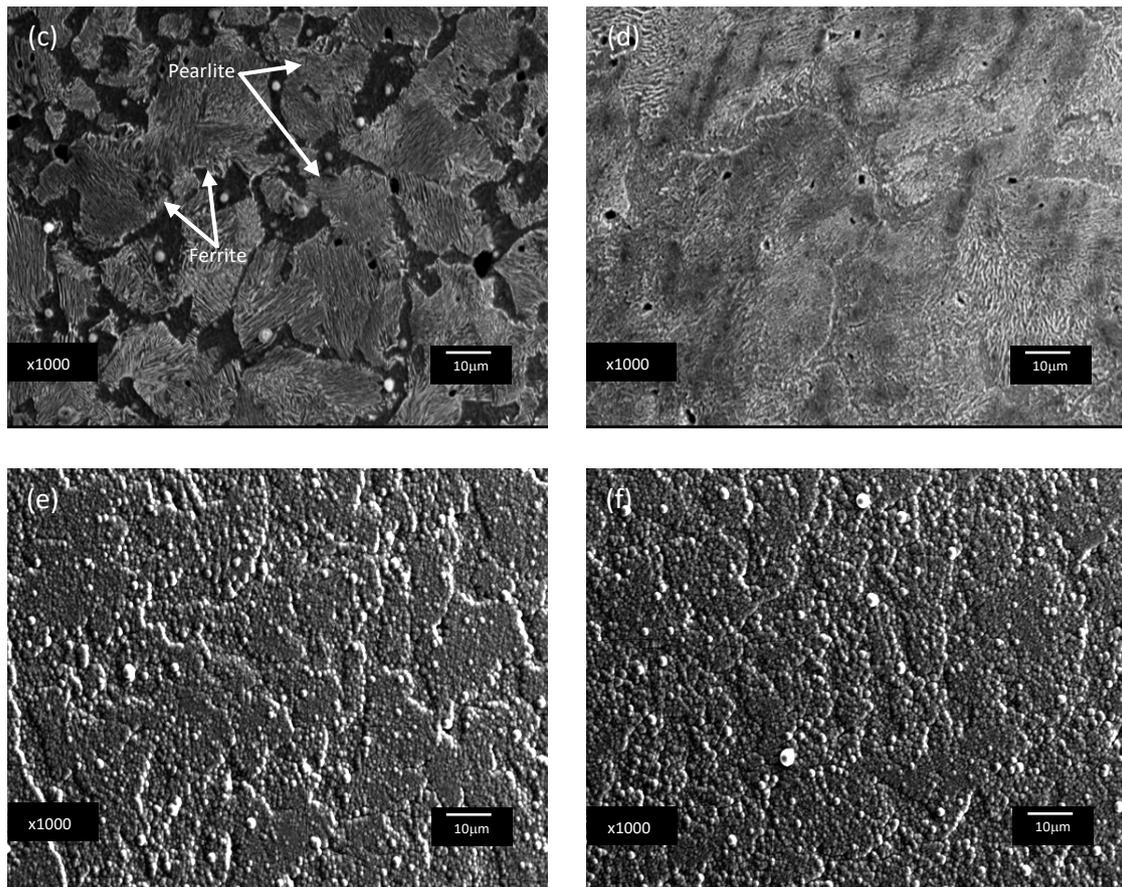


Fig. 2. Surface morphology of AISI1045 (a) as received , (b) grinded, (c) activated using sulfuric acid, (d) activated using hydrochloric acid, and trivalent coated on (e) activated using sulfuric acid, (f) activated using hydrochloric acid

3.2 3D Surface Profile

Previously, the surface activation indicates the changes in the morphology with the existence of different amounts of ferrite and pearlite. Generally, changes in morphology will show the difference in its topography. Next, the surface has been examined with the quantification of surface topography by considering its Ra value. The Ra value of the sample has been measured and tabulated as shown in Table 1. The existence of ferrite and coarsening pearlite structure on a sample activated with H_2SO_4 and HCl will show different topography and influence its surface roughness. The differences in its topography were analysed with a 3D profilometer to support this finding.

Figure 3 shows the filtered images obtained from the analysis with the 3D profilometer that indicates the sample topography. The surface topography enables quick visual evaluation of surface roughness and features such as crests and troughs. Several colour regions ordered from red, orange, yellow, green, and blue are used to denote the crests and troughs on a topographic image [21]. The blue region represents the lowest area (trough), while the red region is the highest elevation in the area (crest) [22]. In evaluating surface topographical characteristics, the required parameter is Ra, donating the average surface roughness (Ra) value indicates the average of surface heights (asperities) and depths (valleys) across the surface [21]. The number of peaks and valleys increases according to the acid activation.

Figure 3 (a) indicates the as received sample without grinding with Ra around $0.329 \pm 0.005 \mu m$. Meanwhile, after undergoing the grinding step, the inactivated sample achieved a smooth surface as

indicated in Figure 3 (b) and the Ra value reported is $0.013 \pm 0.002 \mu\text{m}$. After activation with H_2SO_4 and HCl, the Ra value increased to $0.166 \pm 0.002 \mu\text{m}$ and $0.197 \pm 0.006 \mu\text{m}$, respectively. The activated sample seems rougher, with a higher Ra value that highlights significant changes according to the surface activation compared to the inactivated sample.

The Ra value of the sample activated with HCl is higher in contrast to the Ra value of the sample activated with H_2SO_4 due to the coarsening of the pearlite structure, leading to a reduction in the volume of ferrite. This is due to the usage of HCl, which caused vigorous reactions with the oxide layer, causing rapid dissolution and aggressive activation action, building a coarse surface profile. The fully layered appearance of pearlite is accentuated after HCl activation, creating a jagged and uneven surface and highlighting the increasing average roughness (Ra value). This has been proved with changes in its topography as shown in Figure 3 (c) and (d).

After the sample undergoes the electroplating process, the topography has been changed with the existence of nodular from the trivalent chromium coating as illustrated in Figure 3 (e) and (f)

This indicates that the surface roughness gradually decreases to $0.107 \pm 0.007 \mu\text{m}$ for the sample activated with H_2SO_4 and $0.128 \pm 0.004 \mu\text{m}$ for the sample activated with HCl when undergoes the electroplating process. The reduction of surface roughness indicates that the coating adheres to the substrate. This has been proved with the existence of a coating layer that successfully forms on the carbon steel substrate as shown in Figure 4.

The overview of the 3D profiler used to conclude that the usage of different types of acid reveals different reactions related to the generalized dissolution of metals, which caused an emphasis on deep valleys due to the changes in structure on the sample surface. Different structures contributing to an increment of Ra value are affected by an increment of surface area. Besides, this finding indicates that both types of acid used in pretreatment activation are expected to be sufficient for promoting adhesion by increasing the surface area with increases in surface roughness of the activated sample.

One might consider that the acid activation using H_2SO_4 gives the benefit of increasing surface area by reducing the oxide layer on the substrate. Interestingly, activation with HCl provides significant benefits with a cleaner surface by increasing the surface area as the oxides are fully removed. Due to this, it can be concluded that the use of both types of acid might be a promising method for better coating adhesion, leading to successful electroplating with excellent coating quality.

Table 1

The average surface roughness of inactivated and activated AISI 1045 samples before and after deposition

Sample	Surface Roughness (μm)
AISI 1045 As Received Sample	0.329 ± 0.005
AISI 1045 Grind Sample	0.013 ± 0.002
Activated by Sulfuric Acid (H_2SO_4)	0.166 ± 0.002
Activated by Sulfuric Acid (HCl)	0.197 ± 0.006
Trivalent Chromium Coated Specimen Activated (H_2SO_4)	0.107 ± 0.007
Trivalent Chromium Coated Specimen Activated By (HCl)	0.128 ± 0.004

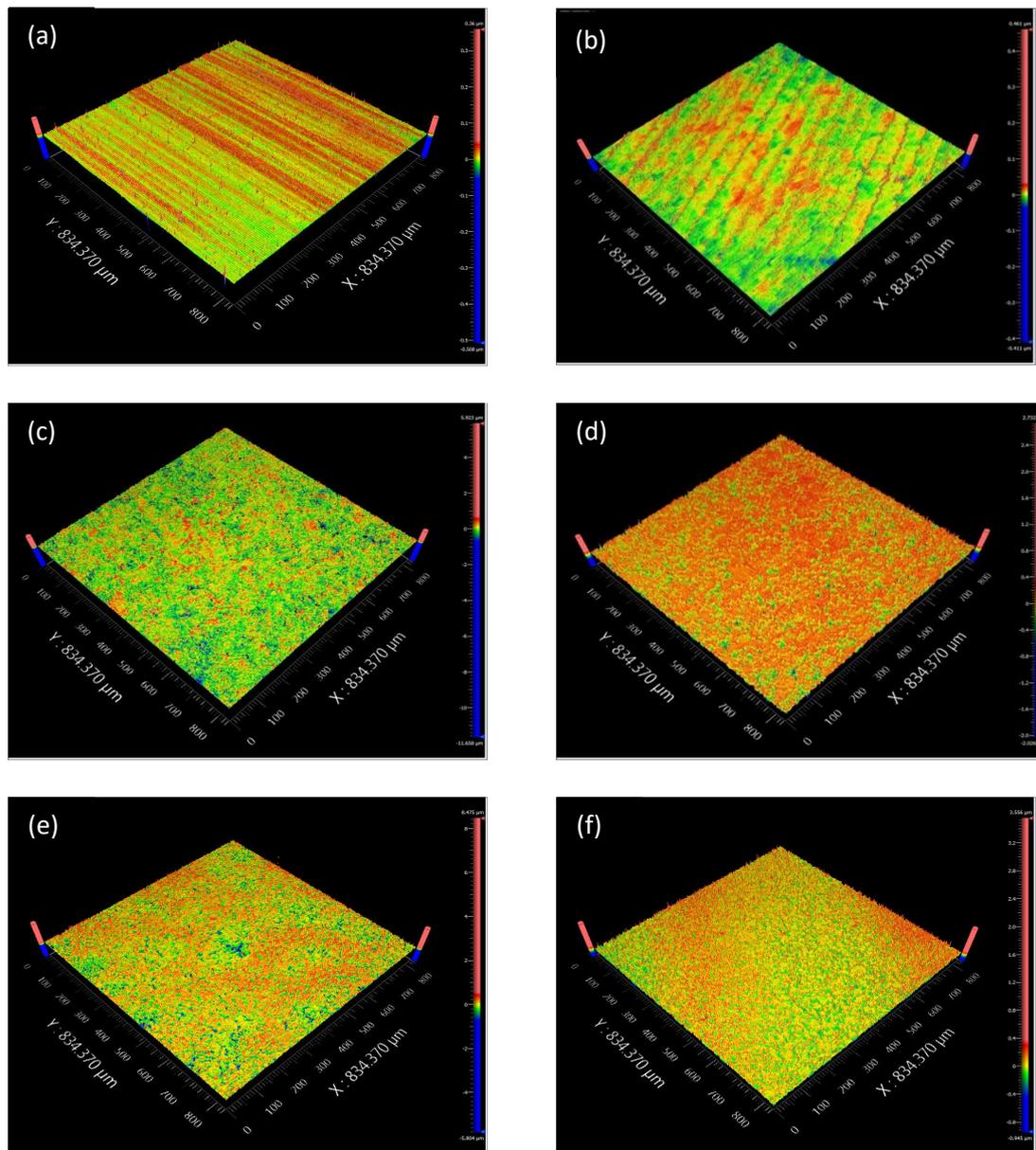


Fig. 3. Surface topography of AISI1045 (a) as received , (b) grinded, (c) activated using sulfuric acid, (d) activated using hydrochloric acid, and trivalent coated on (e) activated using sulfuric acid, (f) activated using hydrochloric acid

3.1 Cross-section of Trivalent Chromium Coating

Previously, the activation has altered the surface topography where the surface has been roughen. The roughening action leads to an increase in surface area for the deposition. The increased surface area has affected the growth coating. Figures 2 (e) and (f) indicate the nodular size of Trivalent Cr coating for the activated sample of H_2SO_4 was found smaller compared to the sample activated with HCl. The difference in nodular size was found due to the rougher surface build for the surface activated with HCl compared to the surface activated with H_2SO_4 , as reported in Table 1.

Higher surface roughness increases the number of nucleation sites and increases the energy state of the surface, facilitating the growth and enlargement of the nodules. The enlargement of the nodule facilitates the growth of the trivalent chromium coating causing differences in the coating layer form on the substrate. As shown in Figure 4 (a) and (b) the thickness of the coating is measured

at $5.19\mu\text{m}$ and $5.43\mu\text{m}$ for the sample activated with H_2SO_4 and HCl respectively. This finding indicates the effectiveness of the activation pretreatment that was demonstrated in the observation of SEM pictures, where the coating was effectively developed by eliminating the impurities and dirt on the substrate surface. Besides, thicker coatings and larger grains have been achieved for the sample activated with HCl which indicates higher growth compared to the sample activated with H_2SO_4 with the deposition of 10 minutes at $25\text{A}/\text{dm}^2$.

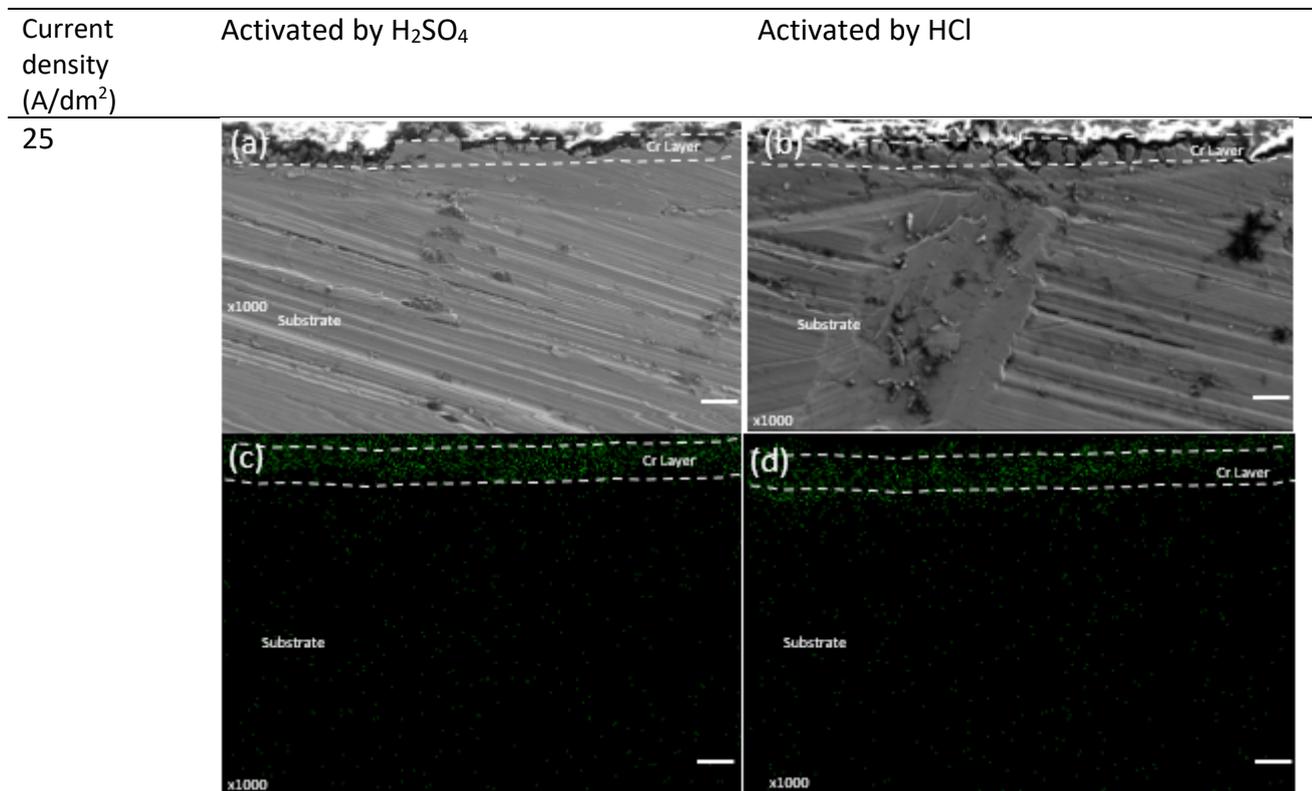


Fig. 4. SEM and EDX images for the cross-section of trivalent chromium coating deposited for 10 minutes at $25\text{A}/\text{dm}^2$ of activated sample by (a) and (c) H_2SO_4 , (b) and (d) HCl

4. Conclusions

This research reveals an effective approach that highlights the significant influence of surface activation for the electroplating of trivalent chromium coatings on carbon steel AISI 1045. The transition from hexavalent chromium (Cr(VI)) to trivalent chromium (Cr(III)) has garnered attention due to the serious health and environmental risks associated with Cr(VI) . While previous research has focused on the toxicity of hexavalent chromium and the need for safer alternatives, there is an urgent need to explore the environmental benefits of trivalent chromium, which is less toxic and generates lower hazardous waste that is easier to manage. This study specifically examines how surface activation impacts the deposition of trivalent chromium coatings, aiming to enhance electroplating processes and promote sustainable practices. Activating carbon steel with hydrochloric acid (HCl), alter the surface roughness by 18% compared to sulfuric acid (H_2SO_4) which include the increase of surface area that is crucial for effective electroplating. Additionally, increasing surface roughness enhances coating thickness due to the formation of a denser nodular structure. The increase in thickness provides various benefits for products with the utilization of this technology. Thicker coating will increase the hardness which is highly desirable as it can extend the lifespan of coated products and reduce the replacement frequency. Recognizing surface activation as a quick and

economical pretreatment method preferred by industrial users, where pre-treating substrate surfaces has been recommended to optimize coating performance. Ultimately, this research supports efficient resource use and aligns with green chemistry principles by minimizing hazardous substances and fostering safe more sustainable industrial practices. As industries shift towards sustainable practices, dynamic technologies will be essential in producing environmentally friendly, high-performance solutions for various applications.

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