

## Effect of CTAB surfactant on microstructure and oxidation behavior of Ni-TiO<sub>2</sub> Composite coating

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### ABSTRACT

In this research, ZnO nanoparticles (NPs) were synthesized using a binary Zn(II) Schiff-base complex. The complex was heat-treated to prepare ZnO nanoparticles via thermal decomposition route at 500 °C. The formation of single-phase ZnO nanoparticles and their microstructures were studied by XRD pattern, SEM and TEM observations. The photo-degradation characteristic of the as-prepared ZnO NPs was evaluated and acid blue 193 was investigated as the organic colorant in a photocatalytic reactor by advanced oxidation process (AOP). The effects of operating parameters such as initial concentration of the dye, the amount of catalyst and the pH value of the solutions were studied to determine the optimum condition of the process to improve the photo-degradation performance. According to the results, impressive photo-degradation of acid blue is conceivable by the as-synthesized ZnO NPs. The optimum operating conditions to achieve photo-degradation were found to be a solution with the pH value of 6, the catalyst concentration of 0.04 g/L, and the dye concentration of 10 g/L. The highest efficiency would be achieved at the natural pH of the solution (pH≈6). The optimum concentration of ZnO NPs is about 0.04 g/L. More or less amount of the photocatalyst could not enhance the photodegradation efficiency. At the constant photocatalyst concentration, any increase in the dye amount leads to the decrease in degradation efficiency.

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### 1-Introduction

Surface engineering involves altering the properties of the surface phase, in order to reduce the degradation over time in some process like fatigue, wear, creep and corrosion. There are various methods to deposit a coat on a substrate like physical vapor deposition (PVD), chemical vapor deposition (CVD), sputtering vacuum deposition, sol gel and

electrodeposition [1]. Among these methods, in the recent years, electrodeposition has been considered significantly between researchers and scientists because of low cost production and good reproducibility [2]. Electrodeposition of metals, polymers and ceramics within a metal matrix that produce composite coating is an effective method for improving hardness, corrosion behavior and oxidation behavior of a

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substrate [3-5]. Electrodeposition of Ni is commercially important and widely used for improving surface properties like hardness and corrosion properties [6]. Ni-plated surface has not enough resistant to oxidation and corrosion, in order to overcome this problem; codeposition of second particles have been employed [7]. For this purpose metals oxide, metals carbide, nitrides and boride like (TiO<sub>2</sub>, SiC, WC, BN, ZrB) have been used for codeposition in order to enhancing mechanical behavior [8-13] and some others one like Fe<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub> and CeO<sub>2</sub> are not only for enhancing mechanical behavior but also for thermal barriers purposes. It is also worth to mention that these coatings acts as a barrier to the ingress of oxygen and outward diffusion of cations which in turn lowers their activities thereby enhance the oxidation resistance of the substrates [14-18].

It should be mentioned that there are various parameters including electrodeposition time, electrolyte concentration, additive and current density which they affect the physical properties, mechanical properties, morphology and microstructure of electrodeposited film and subsequently indirectly control the oxidation process.

One of the most important parameters which affect the microstructure, adhesion of coating, corrosion and oxidation resistance is additive. The addition of Cetyltrimethylammoniumbromide (CTAB), hexadecylpyridinium bromide (HPB), Sodium dodecyl sulfate (SDS) and polyetherdiamine (PEDA) as an additive could be utilized for enhancing distribution and deposition of particles as well as increasing the mechanical properties like wear and hardness [19, 20]. Cationic additives such as CTAB generate a negative charge on the surface of the particles and hence by absorbing nickel ions cause positive polarity in the surface, consequently other particles will not absorb [19]. Also the

addition of CTAB surfactant to the plating bath causes to the smoother surface, more codeposition and decrease of agglomeration.

It is suggested that cationic surfactant inhibited adsorption of cations and enhanced adsorption of anions on the positive charged carbide surface. It was attributed to the CTAB adsorption realized predominantly by hydrophobic interactions between aliphatic chain of the molecule and second phase surface with the positive head group of CTA<sup>+</sup> pointed toward the bulk solution. It was concluded that enough strong attraction of the particles to the cathode surface provided by the cationic surfactant plays the most important role in the particles codeposition, since it allows keeping particles at the cathode surface for the time sufficient to their incorporation into the growing metal matrix. However, the presence of CTAB on the particles has negative effect on the current efficiency due to decrease in the amount of metal ions adsorbed on the particles and thus available for reduction. CTAB inhibits the cathodic process, but the polarization was slightly affected by the additive concentration. It resulted in fine grained nickel matrix of the composite deposits characterized [21].

The goal of this research is to investigate the effect of CTAB surfactant on the microstructure and oxidation behavior of Ni- TiO<sub>2</sub> composite coating on AISI 430 ferritic stainless steel.

## 2- Experimental procedure

A pure nickel with dimensions of 20mm×20mm×2mm was used as the anode, and an AISI 430 stainless steel specimen with dimensions of 10 mm ×10 mm ×2 mm was employed as a cathode. They were ground to a final 3000# SiC paper. After ultrasonically cleaning in acetone, they were electrodeposited from a nickel sulfate-chloride bath. The composition of bath is shown in Table1.

**Table1.** Bath composition of electrolyte

| Composition                          | Mass concentration(g/L) |
|--------------------------------------|-------------------------|
| NiSO <sub>4</sub> .6H <sub>2</sub> O | 240                     |
| NiCl <sub>2</sub> .6H <sub>2</sub> O | 45                      |
| H <sub>3</sub> BO <sub>3</sub>       | 30                      |
| Temperature                          | 55°C                    |
| Time                                 | 10 Min                  |
| pH                                   | 5                       |

The current density used was 5 A/dm<sup>2</sup>, the pH was 4. The temperature was 50 °C and TiO<sub>2</sub> concentration of bath 30 g/L during electrodeposition procedure. Also in order to investigate the effect of CTAB on microstructure and oxidation behavior, 0.1 g/Lit of CTAB surfactant was added to bath separately. Before the electrodeposition, the samples were degreased ultrasonically in Aceton, dipped in acid (10% HCl) and finally washed with distilled water and then immersed immediately in the plating bath to allow for the electrodeposition of coatings. In order to prevent the agglomeration of TiO<sub>2</sub> powders, TiO<sub>2</sub> powders were dispersed ultrasonically in solution for 20 min before electrodeposition. During the electrodeposition, magnetic stirring was employed to maintain the uniform particles concentration and prevent the sedimentation. Ultrasonic cleaning of the deposits in distilled water for 5 min was followed by the electrolytic codeposition to remove loosely adsorbed TiO<sub>2</sub> particles. The surface morphology of Ni-TiO<sub>2</sub> layers was examined by scanning electron microscopy (SEM) equipped with EDS analysis which was used for the determination of chemical composition of the deposits. The structure of coatings were studied by X-ray diffraction analysis by means of Cu K $\alpha$  X-ray source

Isothermal oxidation test was carried out in air at 800 °C up to 250 h in static air in order to obtain mass gain. The samples were weighed after: 1, 5, 10, 24, 50, 80, 110, 150, 200 and 250h. Cyclic oxidation at 800 °C up to 60 cycles were exerted to coated steel and each cycle consisted of 50 min of heating and 10 min of cooling in air. Mass measurement of uncoated

and coated steel before and after oxidation was conducted using a balance with 0.0001 g sensitivity.

Microstructure and chemical composition of coated specimens before and after oxidation were analyzed. The techniques used to characterize the structure and composition of tested specimens included scanning electron microscopy (SEM) (Tscan MV2300) with energy dispersive spectroscopy (EDS), Energy dispersive X-ray (EDX) analyses and X-Ray diffraction (XRD) with a Philips X'Pert High Score diffractometer using Cu K $\alpha$  ( $\lambda = 1.5405 \text{ \AA}$ ). The step angle and time step was kept 0.02° and 1 sec/degree, respectively in all the measurements.

### 3- Results

#### 3-1- Morphology of composite coatings

Figure 1 shows surface morphology of Ni-TiO<sub>2</sub> (Figure 1a) and CTAB-Ni-TiO<sub>2</sub> coating (Figure 1b). As it can be seen, the Ni-TiO<sub>2</sub> (Figure 1a) coating is well condensed and it is also well bounded to the substrate moreover it did not show any cracks but some agglomeration sites can be seen which are common phenomenon in electrodeposition process. It is worth to mention that not only TiO<sub>2</sub> particles can act like a nucleation place for Ni but also like an obstruction place for Ni grain growth so smaller grain size is expected when particles are present in the deposit. CTAB-Ni-TiO<sub>2</sub> coating (Figure 1b) demonstrated a condensed surface and no crack or spallation is observed. Furthermore, no agglomeration at presence of CTAB occurred and the grains are bigger in comparison with the grains at absence of CTAB surfactant.

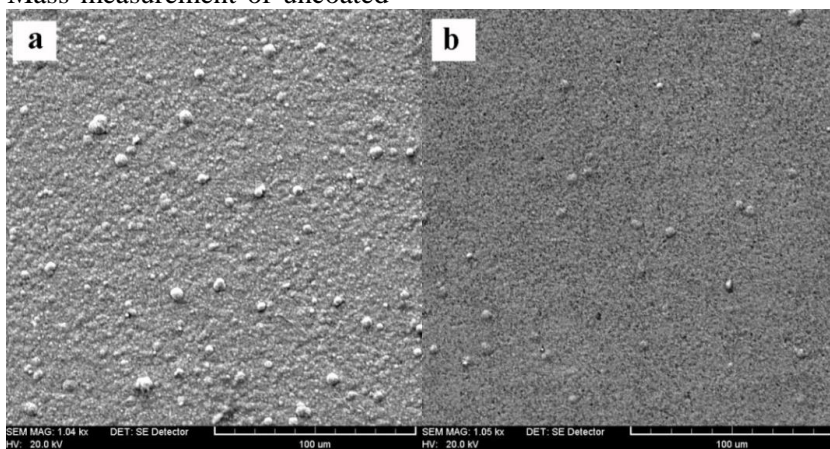


Fig. 1. SEM surface morphology of as deposited Ni-TiO<sub>2</sub> (a) and CTAB- Ni-TiO<sub>2</sub> (b) coated steel.

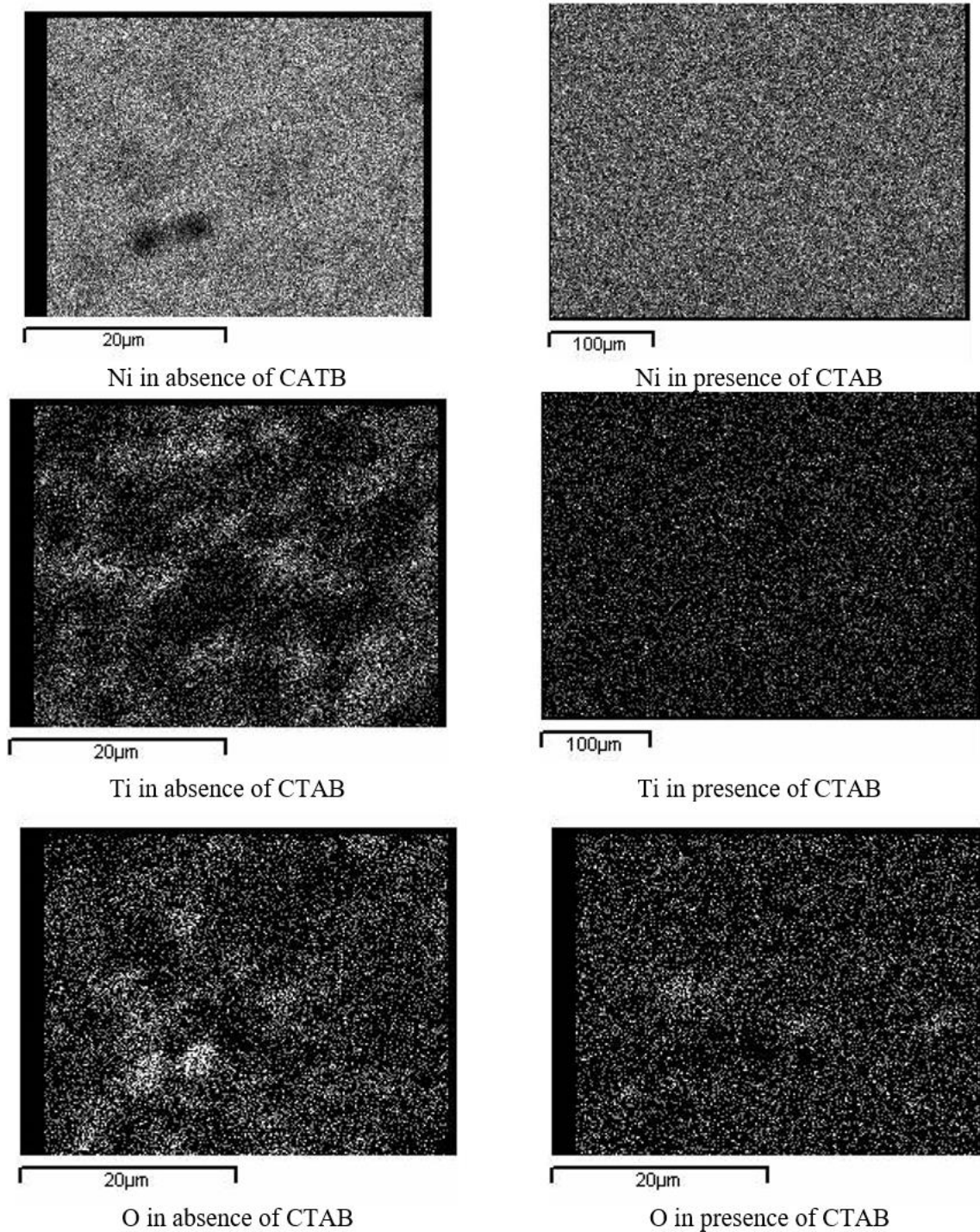


Fig. 2. Elemental distribution of Ni, Ti and O for Ni-TiO<sub>2</sub> composite coating in the absence of CTAB and in the presence of CTAB

Figure 2 shows distribution of Ni, Ti and O in lack of CTAB and in presence of CTAB. The distribution of elements which have been demonstrated in Figure 2 verifies how elements

are well distributed better in the CTAB-coated layer.

When CTAB surfactant is added to bath, surfactant would adsorb on the surface of  $\text{TiO}_2$  particles and the zeta potential of  $\text{TiO}_2$  particles will be positive [22-24]. The positive zeta potential offers an extra adhesion force between inert particles and cathode, which would result in increasing the amount of embedded  $\text{TiO}_2$  particles in the matrix and the grain size will decrease. Also better distribution will occur. The weak and strong adsorption effect between cathode and particles would increase when the amount of surfactant adsorbed increase on the surface of  $\text{TiO}_2$  particles. As a result, the amount of co-deposited  $\text{TiO}_2$  particles in composite coatings would enhance until reaching the optimum level [23].

Figure 3 shows XRD pattern of Ni- $\text{TiO}_2$  coated and CTAB-Ni- $\text{TiO}_2$  coated steel. In XRD

pattern of Ni- $\text{TiO}_2$  coating peaks of Ni,  $\text{TiO}_2$  and Fe are observed. Due to the penetration of the X-Ray beam into the depth of the sample it should be mentioned that Fe phase which observed in XRD pattern is related to the AISI 430 stainless steel substrate. In XRD pattern of CTAB-Ni- $\text{TiO}_2$  coated steel also peaks of Ni,  $\text{TiO}_2$  and Fe are observed. Sharper peaks of  $\text{TiO}_2$  in CTAB-Ni- $\text{TiO}_2$  coated steel in comparison with Ni- $\text{TiO}_2$  coated emphasizes that the deposition of  $\text{TiO}_2$  is higher in CTAB-Ni- $\text{TiO}_2$  coating. XRD patterns were evaluated by Maud software in order to obtain the  $\text{TiO}_2$  embedded in the coating and data shows  $\text{TiO}_2$  was more deposited in the presence of CTAB (~ 6 wt. % in the absence of CTAB and (~ 8 wt. % in the presence of CTAB).

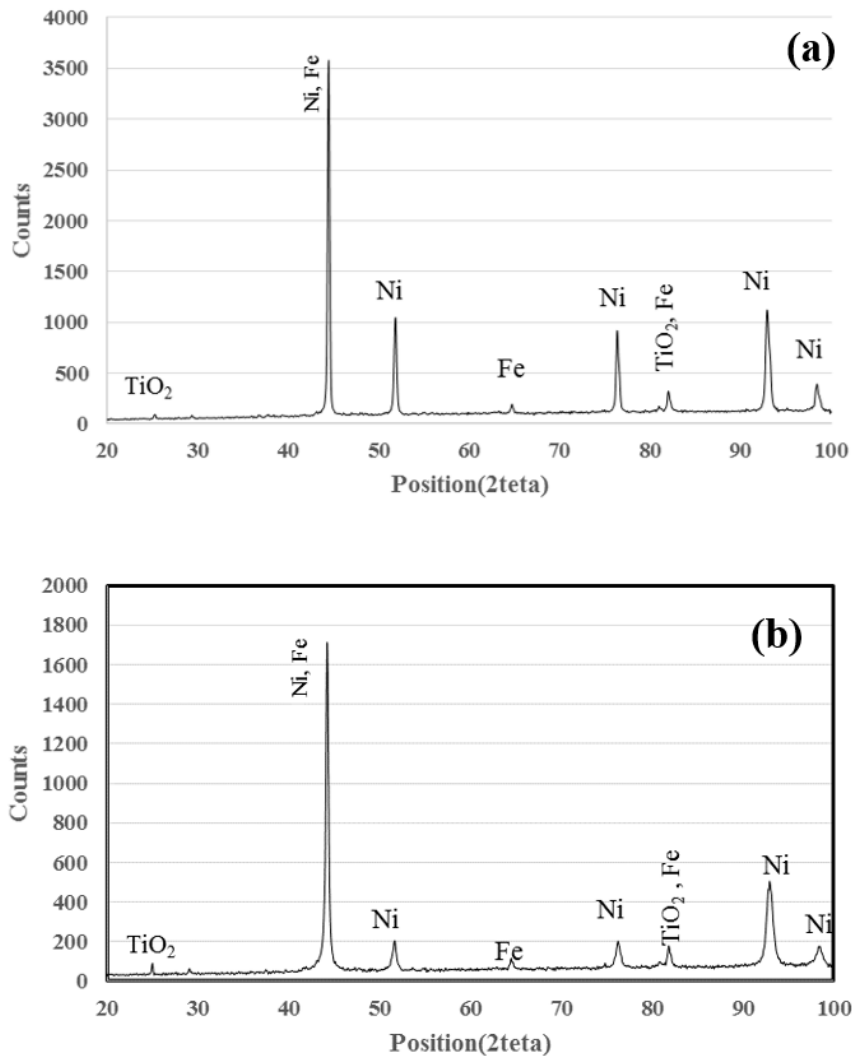


Fig. 3. XRD pattern of Ni- $\text{TiO}_2$  (a) and CTAB-Ni- $\text{TiO}_2$  coated steel (b).

### 3-2-Investigation of oxidation resistance

#### 3-2-1- Isothermal oxidation

According to the experimental results, Figure 4 shows the mass gain mass gain of bare and two types of coated steel after 250 h of oxidation at 800 °C. It is evident that the oxidation rate for bare steel in the first stage of oxidation was higher than the second stage of oxidation kinetic. This is because the lack of protective scale on the surface. The developed  $Cr_2O_3$  on

bare steel was not able to fully protect the surface [25]. In the first stage of oxidation kinetics  $Cr_2O_3$  forms when bare steel expose to oxidant atmosphere and subsequently prohibit more weight gain by acting like diffusion inhibitor but when the process progress, this scale destroy according to below interaction and bare steel gain weight again but in a lower rate compare to first stage of its oxidation.

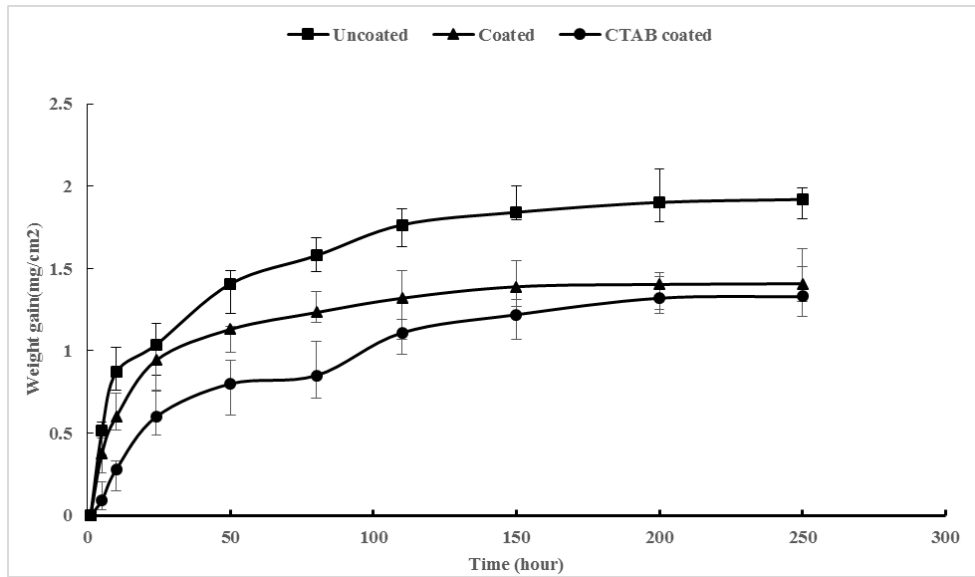
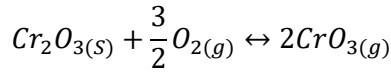


Fig.4. Mass gain of bare and coated steel after 250 h in the 800 °C

After 250 h of oxidation in 800 °C the bare steel approximately gains 1.92 g/cm<sup>2</sup>. The weight gain for Ni-TiO<sub>2</sub> and CTAB-Ni-TiO<sub>2</sub> coated steel after 250 h of oxidation was 1.408 g/cm<sup>2</sup> and 1.33 g/cm<sup>2</sup>, respectively. CTAB-Ni-TiO<sub>2</sub> coated sample had a suddenly weight increase after 80 h of oxidation. As it was mentioned earlier however better distribution is achieved in presence of CTAB and it lead to better oxidation resistance but it leads to weak mechanical behavior which causes cracks in higher temperature due to intensified mismatched.

In comparison to the present study, it is worth to mention that Fe-based coating like Fe-Co- Ni, Fe-Ni and Ni-Fe<sub>2</sub>O<sub>3</sub> not only couldn't suppress the mass gain of AISI 430 stainless steel substrates but also increase the mass gain of them. The high mass gain of coated steels in

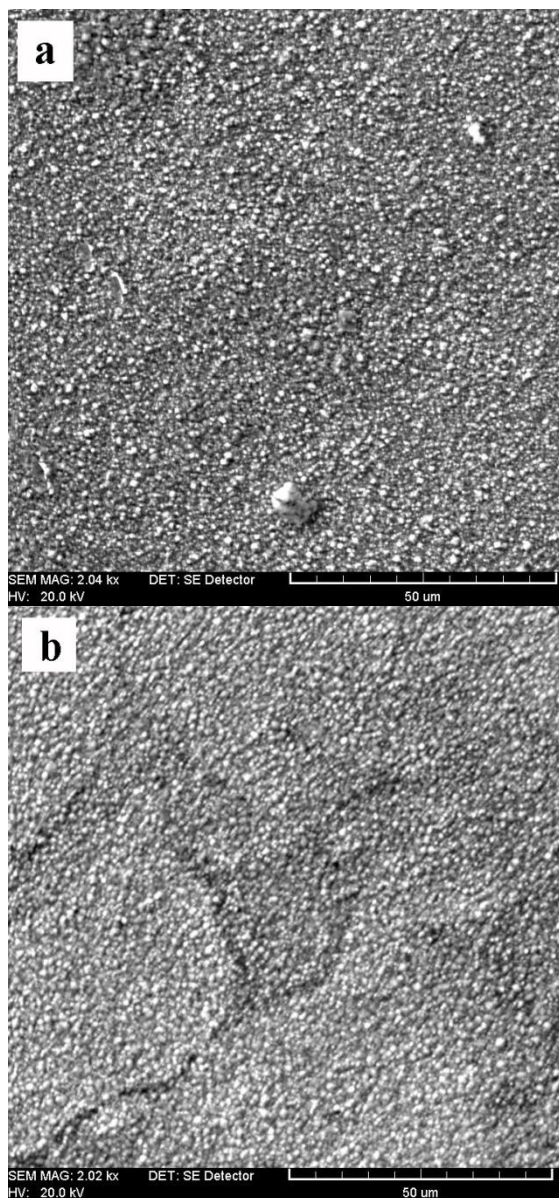
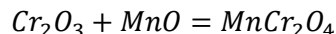
these researches were explained due to oxidation of coated layer itself however the coated steels oxidation rate after one weeks were almost similar to bare steels [17, 26-27]. In another same result, Shaigan and et. al. provided Co/La<sub>2</sub>O<sub>3</sub> nanocompsite on AISI 430 steel, although the coated sample showed more weight gain than substrate but after initial hours of oxidation process, the oxidation rate of coated sample was significantly less than uncoated one. This is due to rapid oxidation of Co in the coatings but after some time the scale, which forms on the surface of oxidized Co/LaCrO<sub>3</sub>-coated AISI 430 at 800 °C in air, consists of two layers of cobalt containing spinel, a Co-Fe spinel as the mid-layer and Co<sub>3</sub>O<sub>4</sub> as the top layer, and a chromia-rich inner layer. The mid and inner layers of the scale contain LaCrO<sub>3</sub>

particles. The coating limits the diffusion of Cr into the scale surface so improves the oxidation rate [28].

In another research, Xue and et al had developed Ni-CeO<sub>2</sub> and Ni coatings on stainless steel under different electrodeposition current (DC and PC) which better oxidation behavior were seen in all experiments. In the mentioned paper, Ni-CeO<sub>2</sub> completely shows better oxidation resistance than Ni. It is concluded that the dispersion of CeO<sub>2</sub> nanoparticles in the Ni coating blocks the outward diffusion of nickel cations and changes the oxidation growth mechanism, which causes a reduction in scaling rate. Mean while, the increased grain boundaries as a result of the grain refinement for the nanocomposite coating due to the dispersed CeO<sub>2</sub> nanoparticle acts as a barrier sites for outward diffusion. Although, compare to present study, in the same condition, more mass gain achieved but where Ni-CeO<sub>2</sub> were coated under PC condition, it shows less mass gain compare to our Ni-TiO<sub>2</sub> coating. This phenomena is related to the electrodeposition current because pulse current provide a better coating properties than direct current and also cause more codeposition of second particles[29].

Figure 5 shows SEM surface morphologies of Ni-TiO<sub>2</sub> (Figure 5a) and CTAB-Ni-TiO<sub>2</sub> coated (Figure 5b) specimens after 80 h of oxidation. As it can be seen some cracks created in the surface of CTAB-Ni-TiO<sub>2</sub> coated steel while the surface of Ni-TiO<sub>2</sub> coated steel is free crack. After cracking, diffusion simplifies and oxide scales trough the simple migration of ions grows faster. Figure 6 shows XRD patterns for oxidized Ni-TiO<sub>2</sub> (Figure6a) and CTAB-Ni-TiO<sub>2</sub> coated (Figure6b) steel in air at 800°C after 250 h of isothermal oxidation. The oxide scale formed on the both coated steel after 250 h of oxidation consisted of NiFe<sub>2</sub>O<sub>4</sub>, NiCr<sub>2</sub>O<sub>4</sub>, NiO, (Mn,Cr)<sub>3</sub>O<sub>4</sub>, TiO<sub>2</sub>, Cr<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>. Figure 7 shows SEM cross section of Ni-TiO<sub>2</sub> (Figure7a) and CTAB-Ni-TiO<sub>2</sub> coated (Figure7b) steel after 250 h of isothermal oxidation. Both coatings exhibited two-layer of oxide scale. By considering both XRD and cross section analyses, it could be concluded that the first

scale is Cr<sub>2</sub>O<sub>3</sub> while the second could be NiO. The second oxidized layer even may correspond to the growth of MnCr<sub>2</sub>O<sub>4</sub>, NiFe<sub>2</sub>O<sub>4</sub>, NiCr<sub>2</sub>O<sub>4</sub>, Fe<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub> which have been observed by other researchers too [30-32]. MnCrO<sub>4</sub> forms according to below interaction which is resulted from the re-establishment of Mn flux.



**Fig. 5.** (a) SEM surface morphology of Ni-TiO<sub>2</sub> coated steel after 80 h oxidation, B) CTAB after 80 h of oxidation.

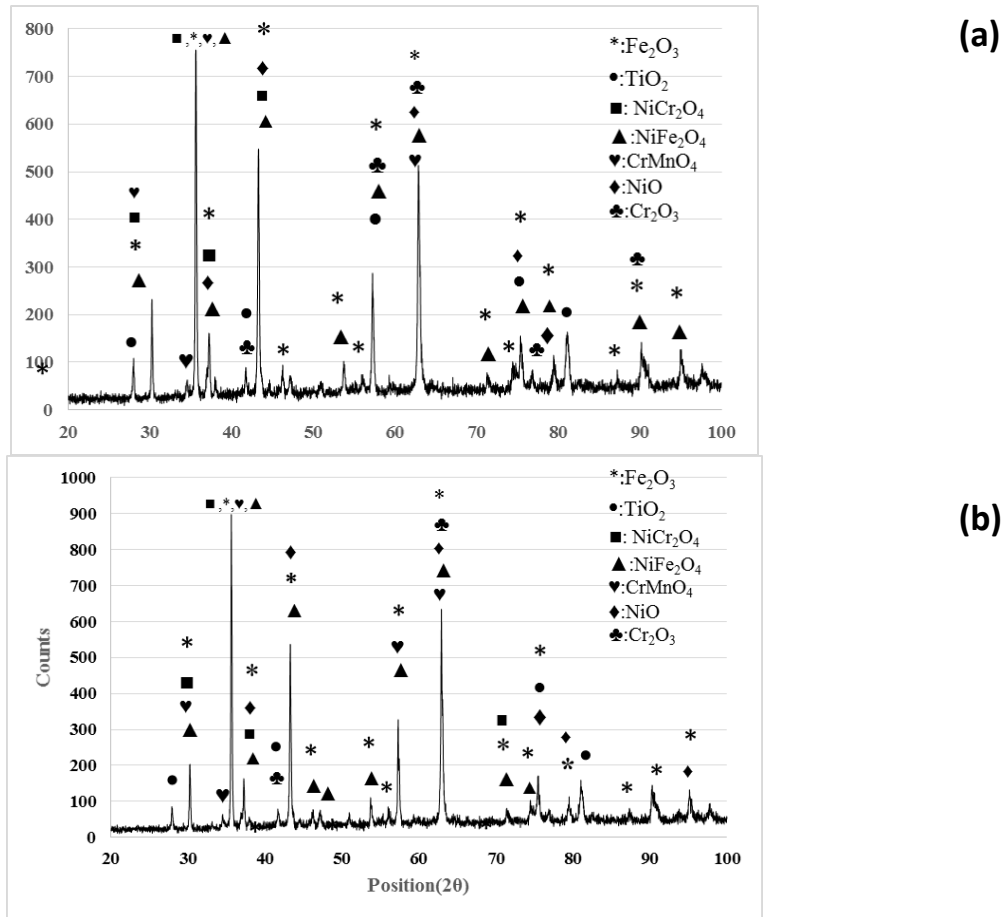


Fig. 6. XRD pattern of Ni-TiO<sub>2</sub> (a) and Ni-TiO<sub>2</sub> CTAB (b) coated steel after 250 h of isothermal oxidation.

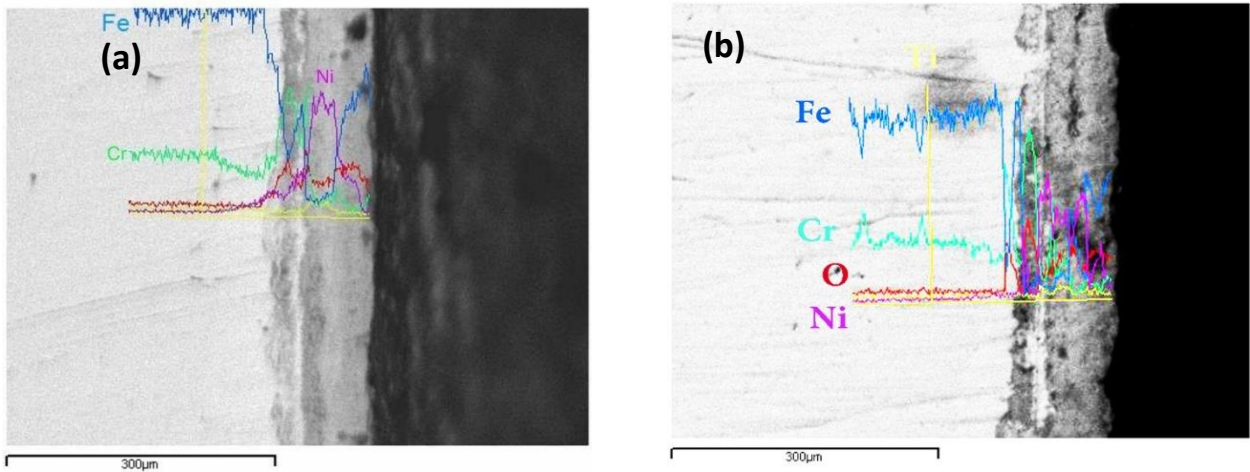


Fig. 7. Cross section of Ni-TiO<sub>2</sub> coated (a) steel after 250 h oxidation, b) CTAB-coated after 250 h

The first stages of oxidation has a higher rate compare to the second state of oxidation. The oxidation rate of the alloy is governed by the diffusion of cations passing a dense oxide scale according to Wagner theory. In the present

study, the first stage of oxidation at 800°C can be considered to correspond to the formation and growth of Cr<sub>2</sub>O<sub>3</sub> subsequent to the initial formation of other resistant phases which is responsible for higher oxidation rate. The



second stage oxidation correspond to the growth of  $\text{NiFe}_2\text{O}_4$ ,  $\text{NiCr}_2\text{O}_4$  which lead to lower outward diffusion of cations like Ni and Cr and then lower oxidation rate [6, 28]. Moreover, after  $\text{NiFe}_2\text{O}_4$ ,  $\text{NiCr}_2\text{O}_4$  formation, oxidation mechanism has been changed from outward cations diffusion to inward anions diffusion. Because anion diffusion like O is substantially

lower than outward diffusion of cations like Cr, better oxidation behavior has been observed after about 20 hours in coated steels. In spite of above discussion, it is suggest that  $\text{TiO}_2$  particles are Rutile in  $800^\circ\text{C}$  when they have the highest resistant to oxidation. So, it leads to lower local outward diffusion of cations and then lower oxidation rate has been observed in second stage of oxidation [33].

The weight gain of CTAB-Ni- $\text{TiO}_2$  coating was lower than Ni- $\text{TiO}_2$  coating. Also the thickness of chromia layer in CTAB-Ni- $\text{TiO}_2$  coated steel

after 250 h of oxidation is lower (Figure7a) compared to Ni- $\text{TiO}_2$  coated steel (Figure7b). Zeta potential increases in presence of surfactant in electrolyte bath and as a result the possibility of absorption between cathode and particles climbs. In other words, the affinity of positive particles surface charge increases the codeposited into metal matrix [10, 24]. As mentioned in presence of surfactant, the distribution of phases was uniform and composite layer was free crack and it was well bounded to substrate before oxidation process. Therefore CTAB plays a vital role in oxidation resistance in the first stage of oxidation which is due to better distribution of  $\text{TiO}_2$  and other phases.

### 3-2-2- Cyclic oxidation resistance

Figure 8 demonstrates the mass gain of substrate and coated specimens (CTAB and Ordinary) versus the number of cycles in air at  $800^\circ\text{C}$ .

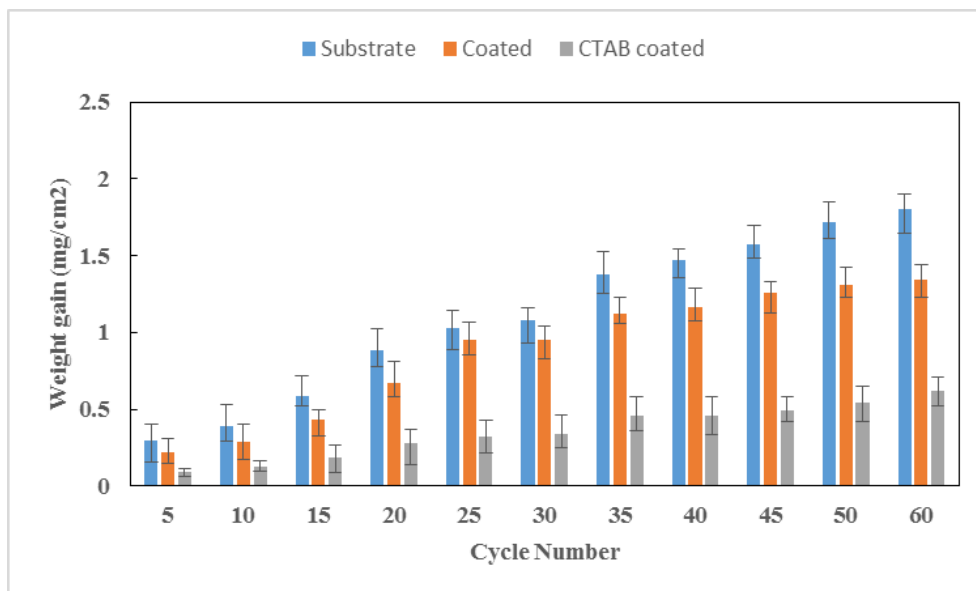


Fig. 8. Mass gain of bare and coated steel after 60 cycles of oxidation at  $800^\circ\text{C}$

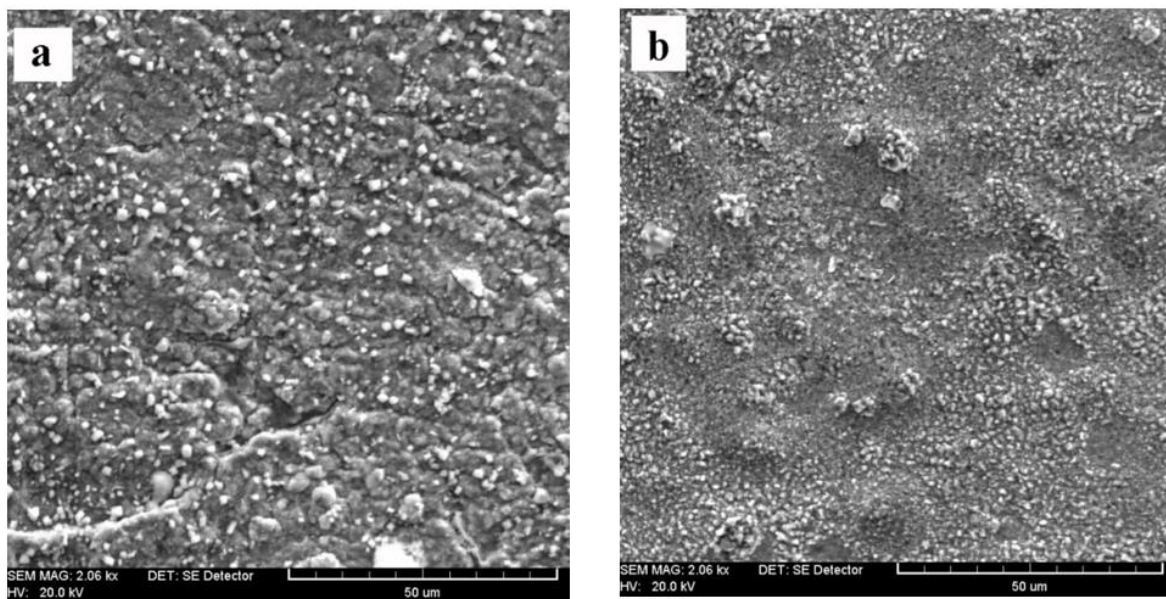
The CTAB coated specimens gain only  $0.62 \text{ mg/cm}^2$  after 60 cycles which is considerably less than mass gain for ordinary and substrate steels in all cycles, 1.341 and 1.805, respectively. **Error! Reference source not found.** shows surface morphology of Ni- $\text{TiO}_2$  (Figure 9a) and CTAB-Ni- $\text{TiO}_2$  (Figure 9b) coated specimens after 60 cycles of oxidation. Although CTAB coated steel cracked after about 80 h of isothermal oxidation, but in cyclic

oxidation probably due to less oxidation times compare to isothermal oxidation, CATB coated did not show any cracks which is obvious from figure10. Some micro cracks are observed in the surface of Ni- $\text{TiO}_2$  coating layer. These cracks may be nucleated from the sites which are reached of  $\text{TiO}_2$ . Agglomeration of  $\text{TiO}_2$  particles caused to the enrichment of some parts of surface from  $\text{TiO}_2$  and it resulted to the cracking. Thermal expansion coefficient of  $\text{TiO}_2$  is different from coating and substrate and

therefore it leads to cracking [7]. Another reason for better resistance of CTAB coating layer during cyclic oxidation is better distribution of  $\text{TiO}_2$  particles in the coating layer. Uniform distribution leads to the uniform expansion all over the surface and it resulted to better resistance to cracking and spallation [10]. So mass gain of CTAB coated steel is less compared to the ordinary coated steel.

When the morphology shows some voids as well as spallation (Figure9) in the film, oxygen can easily diffuse inward. These voids and cracks

places are favorable place for short-circuits diffusion path and thereby accelerate the oxidation of the coated steel and then bigger mass gains unavoidable [34]. It can be concluded, even though the CTAB coated steel exhibited an initially lower mass gain compare to ordinary coated steel, formation and the growth of different layers lead to local spallation, consequently, fast local outward diffusion of cations and then bigger oxidation rate has been observed because of higher mismatched in elevated temperature.



**Fig. 9.** SEM surface morphology of Ni- $\text{TiO}_2$  coated (a) steel after 60 cycles of oxidation, B) CTAB after 60 cycles at 800 °C.

#### 4- Conclusion

Ni- $\text{TiO}_2$  composite coating with CTAB and without CTAB was fabricated on AISI 430 stainless steel. The effect of CTAB on microstructure and oxidation behavior was investigated. Results showed better distribution of  $\text{TiO}_2$  particles in CTAB coated specimen compared to ordinary coating. Isothermal and cyclic oxidation at 800°C was exerted on the coated and uncoated 430 sample. CTAB coated specimen exhibited lower mass gain in comparison with other samples. However CTAB coated composite spalled after just 80 hours of oxidation and it caused to the higher oxidation rate in this composite coating compare to ordinary coated composite. In contrast to isothermal oxidation, in cyclic oxidation process, CTAB did not show any cracks up to 60

cycle and because of its effect on distribution of elements, this coated steel demonstrate better cyclic oxidation behavior and better the oxidation resistance.

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