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Characterization of Oxide Film Formed on Ck45 Steel by Plasma Electrolytic Oxidation Method

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Abstract: Plasma electrolytic oxidation (PEO) is the new, environmentally and cost-effective method for coating the metals. This method vastly has been studied for coating Aluminum, Titanium and Magnesium and their alloys and recently for steel. In present study, Ck45 steel samples were coated by PEO method in order to improve corrosion resistance. During this process specimens were immerged in carbonate solution contains sodium dehydrogen phosphate (NaH_2PO_4) and sodium carbonate (Na_2CO_3) with PH equaled 8.3. Composition and microstructure of the coating were investigated by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The electrochemical corrosion behavior of coated and uncoated species was verified in 3.5% NaCl solution by potentiodynamic polarization technique. The result of XRD and SEM test showed that nanocrystale particles of Fe_3O_4 were successfully formed on the surface of samples while the average size of particles and phase were less than 800nm. Grain size was estimated about less than 80 nm. Corrosion test exhibited the remarkable improvement (about three times) in corrosion resistance of coated samples (16023 Ω) compared with those without coating (5200.4 Ω).

Keywords: Nanocrystal-Particle; Plasma Electrolytic Oxidation; Corrosion; Ck45 Steel

1. Introduction

Steel are easy to work, especially for their high tensile strength and excellent plastic and tough characteristics. However, the wear and corrosion resistance of steel are much lower than those of ceramic materials. So, preparation ceramic coatings on steel for protection purposes has been being a common thought of scientists and engineers for many years. Plasma electrolytic oxidation (PEO) is relatively new surface treatment technique (from 1970), which is both cost-effective and environmentally friendly [1,2]. PEO is characterized by high temperature of 103–104 K and high local pressure of 102–103 MPa in the discharge channels [3]. The coatings consist typically of porous top layer, dense intermediate layer and thin inner layer. The coating synthesized through the PEO process has exhibited superior mechanical properties, specially high corrosion resistance and excellent adhesive strength superior to plasma sprayed ceramic coating [3,4].

PEO coating on Al, Mg and Ti in order to achieving proper corrosion resistance have been widely studied during recent years and some encouraging results have been obtained [5-7]. This method also has been used for steel [8-12] and some improvements in both physical and mechanical properties of sample were reported. The results obtained during these researches shown that mechanical properties such as thermal shock resistance, bonding strength, friction coefficient and corrosion resistance of specimens were acceptably improved. In present study, Ck45 steel was used as substrate and electrolyte was contained of sodium carbonate and sodium dehydrogen phosphate with prepare composition and equivalent PH value 8.3. DC voltage (up to 300 V) was applied on samples. After treatment, microstructure and thickness of coating was investigated by SEM and composition of layer was evaluated by XRD pattern. Grain size also, has been calculated and reported such that this amount was less than 80 nm. In order to estimating the corrosion behavior of coating,

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potentiodynamic polarization test in 3.5 Wt.% NaCl solution was used.

2. Experimental details

2.1. Preparations of specimens

Substrates (Ck45 steel) were prepared in the form of cylinders, 20 mm in diameter and a certain height. Before deposition, specimens were polished and washed thoroughly with distilled water. The samples were connected to the holder such that only their cross section had contact with the electrolyte and the effect of edges was completely omitted. The solution was made of proper concentration of Na_2Co_3 and NaH_2Po_4 that PH value was 8.3. The DC voltage was applied to the samples such that current density varied in the range 25-310 A/dm^2. During the PEO process, the bath cooled by cooling system and temperature of electrolyte was kept at room temperature.

2.2. Specimen examination

Microstructure, morphology and thickness of coating were examined by SEM method (model: XL30-Philips), average thickness of coatings was estimated by eddy current method (model: MAC-175). Composition and grain size of coating was investigated by x-ray diffraction (XRD, Equinox3000, Inel France, Cu target). Corrosion resistance of samples was evaluated by potentiodynamic polarization technique. The conventional three electrodes system was used as the electrochemical cell, which contains a saturated calomel electrode (SCE, the reference electrode), a Pt foil electrode (the auxiliary electrode) and a working electrode (samples with the exposed area of 0.25 cm^2). A saline (3.5 Wt. % NaCl) solution was used as the corrosive electrolyte. Polarization curves were potentiodynamically measured alone by the Autolab-PGS.30 potentiostat.

3. Results and discussion

3.1. Microstructure and composition of coating

3.1.1. Phase composition

XRD pattern of uncoated (a) and coated (b) samples has been exhibited in Fig. 1. The figure illustrates that, there are only α-Fe peaks on the X-ray pattern of untreated sample, while samples that were coated in carbonate solution through PEO method, contains crystal phase of Fe_3O_4 and there is no peak of Fe available in this condition. The grain size also was evaluated by Scherer's equation about 80nm. The results induce that coating process has been done successfully and crystal phase of magnetite composition was formed on the surface of sample.

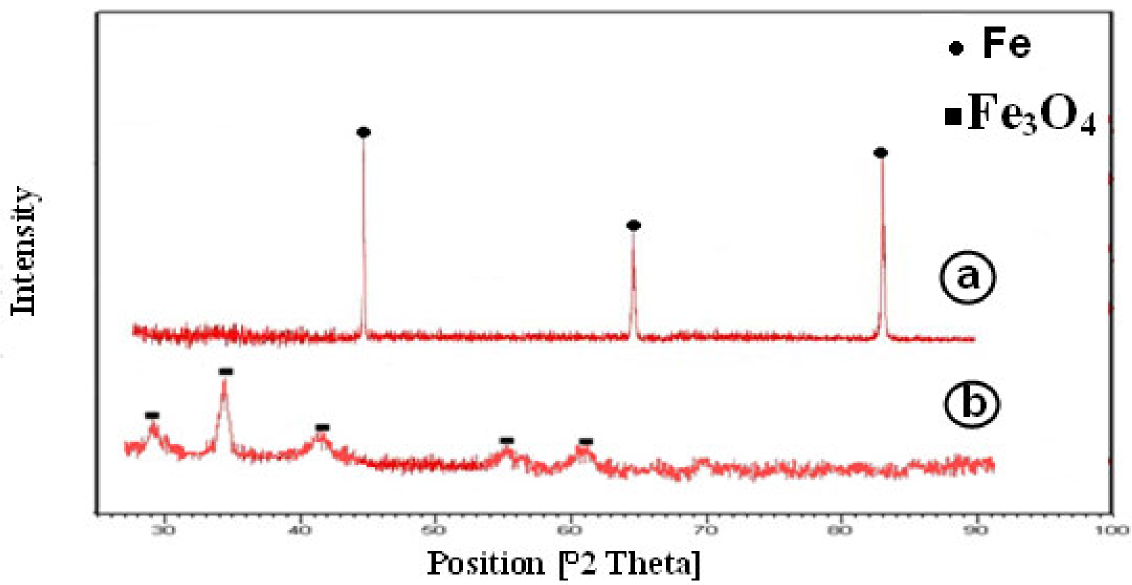


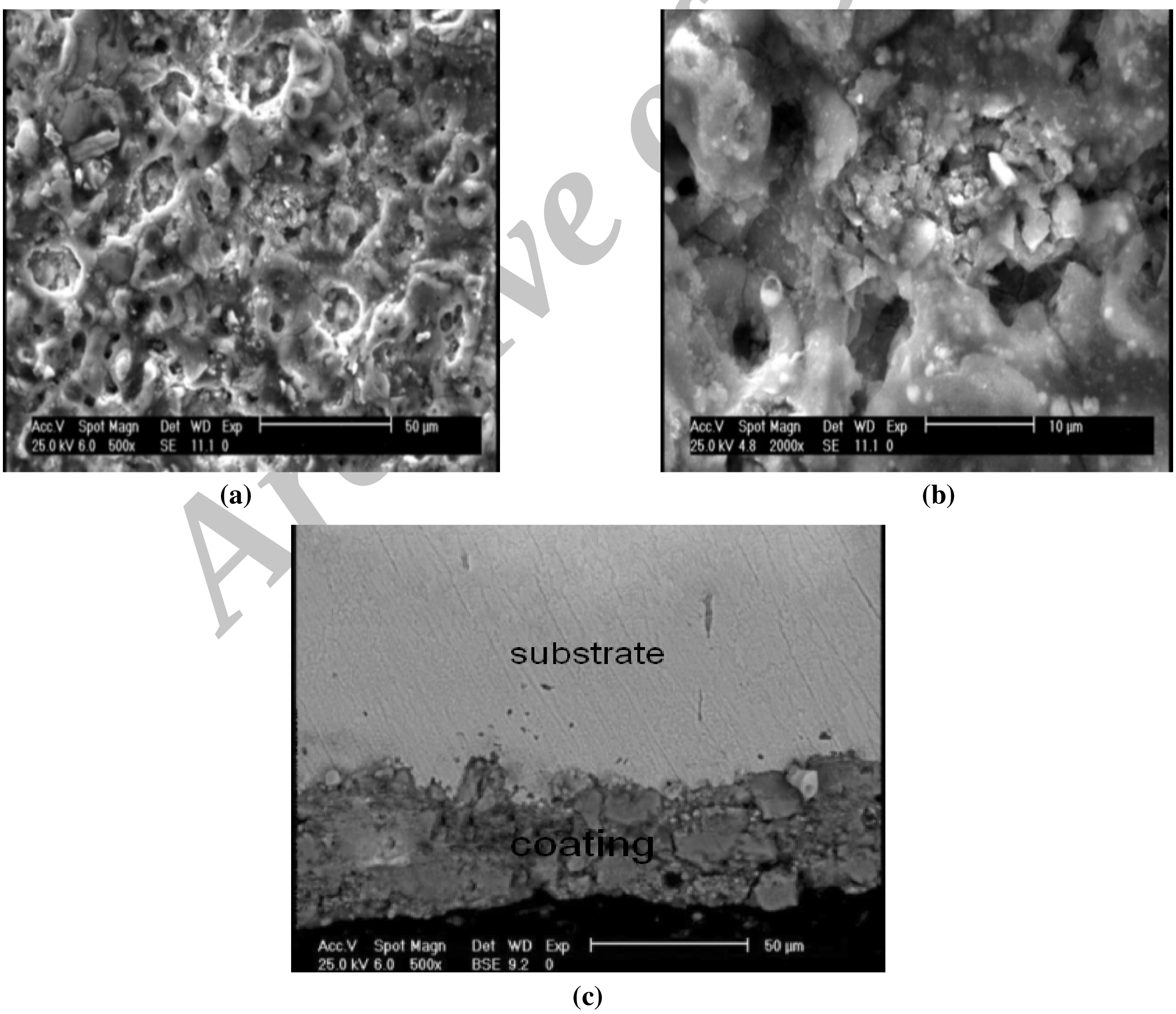
Fig. 1. X-ray pattern of (a) uncoated and (b) coated sample through PEO process in carbonate solution.

3.1.2. Surface and cross section morphology

The surface and cross section morphology of PEO coating have been illustrated in Fig. 2 (a & b). Parts (a) and (b) exhibit the coating surface is coarse and porous. There are some large circular dark holes distributed on the coating surface whose mouths were surrounded by pancake shaped sintered particles. These large holes were intervaled by many irregular pits. These pits are just like the volcanic crater with caliber below 10µm. In fact, these holes and pits are residual discharge channels during the plasma spark reaction and pancakes around the circular discharge channels were formed due to the rapid cooling effect of electrolyte [10]. Some considerable cracks also can be seen in the structure of the coating. Cross section of coating was shown in fig. 2 (c), which demonstrated that the coating was porous but continuous. The average thickness of coatings was estimated around 35 µm by eddy current method (model: MAC-175).

3.2. Corrosion test

The results of potentiodynamic polarization test in 3.5% NaCl solution for both coated (green) and uncoated (black) samples were exhibited in Fig. 3. As this figure illustrates, Tafel diagram for treated sample has been shifted in left side, which shows decreasing in corrosion current of coated specimen. The values of corrosion current density for coated and uncoated sample were 5.82×10^{-6} A/cm^2 and 3.81×10^{6} A/cm^2. Furthermore, absolute value of Ecorr has been declined from 0.4734 V for uncoated sample to 0.3987 V for treated sample that both results demonstrate improvement in corrosion resistance.



(a) **(b)**

(c)

Fig. 2. (a) and (b) SEM pattern of surface morphology in 50 µm and10 µm and (c) cross section of sample was coated through PEO process in carbonate solution.

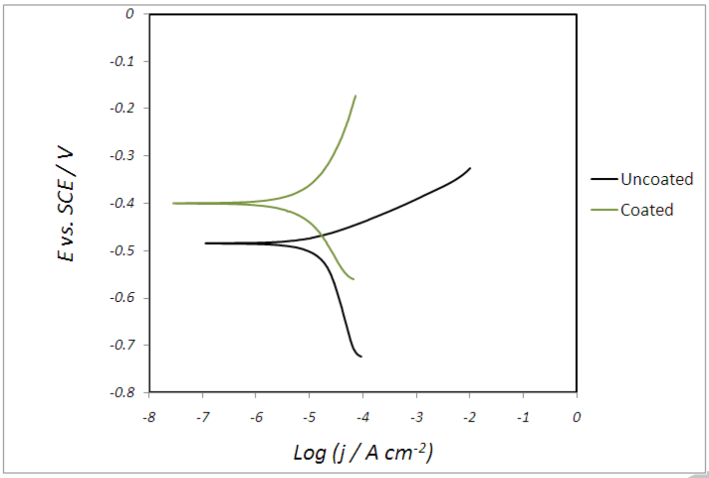


Fig. 3. The results of potentiodynamic polarization test in 3.5% NaCl solution for coated and uncoated samples.

Polarization resistance and rate of corrosion also were calculated in Table. 1. It can be seen that polarization resistance has been increased from 5200.4 Ω for untreated samples to 16023 Ω for coated specimen, which shows more than three times improvement. In addition, Rate of corrosion has declined from 0.04423 mm/year to 0.01724 mm/year, which illustrates acceptable enhancement in corrosion behavior of coated samples.

Table 1. Results of polarization test.

Sample	Ecorr (V)	Icorr (A/cm^2)	ba (v/dec)	R (Ω)	Rate (mm/year)
uncoated	-0.4734	3.81×10^{-6}	0.2923	5200.4	0.04423
coated	-0.3987	5.82×10^{-6}	0.4654	16023	0.01724

4. Conclusions

According to XRD pattern result, nanocrystal particles of magnetite (Fe_3O_4) layer, with grain size of less than 80 nm, were formed successfully on Ck45 steel through plasma electrolytic oxidation (PEO) method. Morphology of surface by SEM method illustrates that coating was coarse with many circular dark holes distributed on the coating surface whose mouths were surrounded by pancake shaped sintered particles. The average thickness of coating was estimated about 35 µm. Corrosion resistance of coating improved by PEO process such that, polarization resistance of coated sample has been enhanced more than three times.

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