

Characterization of Al₂O₃ Coating on AISI 316 Stainless Steel Using Polyaniline (PANI) Synthesis Method

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Abstract - Stainless steel exhibits corrosion resistance under ordinary atmospheric conditions; however, corrosion attacks can occur at certain temperatures. Ceramic material coatings are required to improve the corrosion resistance at elevated temperatures. This study aimed to coat stainless steel with Polyaniline/Al₂O₃/Epoxy and Polyaniline/Al₂O₃/Al/Epoxy. The research methodology includes sanding specimens with dimensions of 40 mm x 40 mm x 1.5 mm using 60-grit sand paper. The Polyaniline/Al₂O₃/Epoxy slurry was prepared with an Al₂O₃ to Polyaniline powder ratio of 3:7. For every 1 gram of Polyaniline/Al₂O₃ mixture, 15 mL of epoxy was added as the binder. The slurry was applied to the substrate using the wet spraying method, with a spray gun nozzle distance of 100 mm from the substrate and a spraying pressure of 2 bar. The adhesion of the coatings was evaluated using a Hot-Cross-Hatch Adhesion tester. Adhesion tests were conducted on unsanded and sanded substrate specimens. The surface roughness of the unsanded specimen was 0.374 μm, while the sanded specimen showed a surface roughness of 1.521 μm. The hot cross-hatch adhesion tester adhesion test results showed no delamination for the sanded specimens, while the unsanded specimens delaminated by 2.163%.

Keywords: Al₂O₃, polyaniline, SS 316, coating, Stainless Steel, Adhesion.

I. INTRODUCTION

Stainless steel 316 is known to have good corrosion resistance under normal conditions. However, at elevated temperatures, its corrosion resistance decreases significantly, and research has shown that 316 L stainless steel undergoes severe oxidative corrosion when exposed to a lead-bismuth eutectic (LBE) alloy at 500°C for 1000 h, resulting in the formation of Fe₃O₄ and Pb₂O on its surface. Uncoated 316 L steel specimens experience a significant increase in mass (0.04 g) owing to severe oxidative corrosion after corrosion testing [1]. At temperatures above 600°C, the corrosion rate of the 316 stainless steel increased dramatically. When the temperature reached 700°C, intergranular corrosion became evident after immersion in KCl-MgCl₂ molten salt for 100 h [2].

316 stainless steel pipes are often used as ceramic coatings to improve corrosion and wear resistance in corrosive or high-temperature environments. Al₂O₃- and Y₂O₃-based ceramic coatings can slow the corrosion rate by forming a protective layer that reduces oxygen penetration at high temperatures [3]. TiC on the surface of stainless steel effectively increases the hardness and wear resistance, with a wear reduction reaching 99% compared to uncoated steel [4]. The use of ceramic particles such as TiB₂ on 316 L stainless steel drastically increases its hardness and resistance to heavy wear conditions [5]. In addition, the addition of Ti/C by laser cladding on stainless steel can improve the tribological behavior and wear resistance of the base metal surface and increase the durability and frictional stability at high temperatures [6].

Al₂O₃ as a coating material on stainless steel provides many advantages, particularly in improving the oxidation and corrosion resistance at high temperatures. One of the main advantages of Al₂O₃ is its stability at high temperatures, which makes it ideal for protecting stainless steel from oxidation in intensive industrial applications such as boilers and energy components [7]. Al₂O₃ can create an inert protective layer, inhibiting the formation of nonprotective oxides that often damage material structures in oxidative environments [8]. In addition, Al₂O₃ has a high hardness that increases its resistance to mechanical wear, making it superior to other materials such as Cr or Ni, which are more susceptible to high-temperature degradation in the long run. On the other hand, Al₂O₃ coatings have self-healing characteristics that can repair micro-damage, which provides longer durability to the stainless steel substrate it is coated on, thereby extending the life of the component in harsh environments [9].

Polyaniline (PANI) is used as an ideal matrix for Al₂O₃ coatings on stainless steel owing to its tunable conductivity and high chemical stability, which favor anti-corrosion protection and improved mechanical performance. The modification of polyaniline with TiO₂ nanoparticles, including Al₂O₃, improves the corrosion resistance of stainless steel through its physical barrier effect and in-situ anodic passivation properties [10]. In another study, PANI was successfully modified with Al₂O₃ through electrochemical

deposition, demonstrating that the addition of Al_2O_3 can improve the oxidation resistance and thermal stability of the coating under various environmental conditions [11]. The PANI structure integrated with Al_2O_3 also provided enhanced conductivity, thermal stability, and strong mechanical properties, making it superior to other conductive polymers [12]. Moreover, in photocathodic protection applications, polyaniline composites with Al_2O_3 can store electrons for protection in dark environments, thereby strengthening the protection ability of stainless steel under various light and corrosion conditions [13].

While there has been much research on PANI- and Al_2O_3 -based coatings, there are gaps in the development of effective coatings for application in 316 stainless steel pipes, especially in extreme environments. One of the main gaps is the lack of studies on the use of epoxy as a bonding agent in PANI/ Al_2O_3 composites. Epoxy can improve the adhesion of the coating to the 316 stainless steel substrate as well as the even distribution of the coating on the metal surface when applied using the wet spraying method. This study aims to produce and observe the surface of PANI/ Al_2O_3 -based

coatings with epoxy as a slurry binder applied to 316 stainless steel pipes using the wet spraying method. The focus of this study is to evaluate the ability of the coating to resist corrosion and abrasion. This research can make an important contribution to the development of industrial coatings that are more durable, efficient, and cost-effective. The advantages of more durable coatings will help reduce the maintenance frequency and improve the operational efficiency, particularly in the energy sector, which relies heavily on material resistance to corrosion and extreme mechanical stress.

II. MATERIALS AND RESEARCH METHODS

The materials employed in this research included PANI and Al_2O_3 powders, plates with dimensions of 40 mm × 40 mm × 1.5 mm, 316 stainless steel pipe measuring 1.5" × 65 mm, a spray gun, and a pneumatic compressor. The materials used to prepare the nanocomposite (PANI/ Al_2O_3) consisted of Aluminum Oxide (Al_2O_3), aluminum (Al), and aniline hydrochloric ($C_6H_5NH_2.HCL$), Ammonium Persulfate ($(NH_4)_2S_2O_8$), hydrochloric acid (HCL), n-butanol (C_4H_9O), and aquades (H_2O) with the composition and purity of the materials are shown in Table 1.

Table 1: PANI/ Al_2O_3 Polymerization Chemicals

Material	Chemical formula	Use of	Composition	Purity
Aluminum Oxide	Al_2O_3	Filler	0,2 gram	99.9%
Aluminum	Al	Filler	0,05 gram	99.9%
Aniline hydrochloric	$C_6H_5NH_2.HCL$	Monomer	1,82 mL	≥ 99%
Ammonium Persulfate	$(NH_4)_2S_2O_8$	Oxidizing agent	5,71 gram	≥ 98%
Hydrochloric acid	HCL	Polymerizing acid medium	50 mL	37%
n-Butanol	C_4H_9OH	Solvent	10 mL	≥ 99%
Acetone	C_3H_6	Cleaner	1 L	≥ 99.5%
Aquades	H_2O	Solvent	50 mL	-
Epoxy Resin	-	Binding agent	15 mL	-

Table 2 shows the composition of the 316 stainless steel material. Stainless Steel 316 has a weight percentage composition of C (0.08), Mn (2.00), Si (0.75), P (0.45), S (0.03), Cr (16.0-18.0), Mo (2.00-3.00) and Ni (10.00-14.00). PANI/ Al_2O_3 /Epoxy and PANI/ Al_2O_3 /Al/Epoxy slurry coatings were carried out on pipe-shaped substrates with dimensions of 1.5" × 65 mm, whereas coating adhesion testing specimens were carried out on plate-shaped stainless steel specimens with dimensions of 40 mm × 40 mm × 1.5 mm.

Table 2: Composition of SS 316

Material	SS 316
C	0.08
Mn	2.00
Si	0.75
P	0.45

S	0.03
Cr	16.0-18.0
Mo	2.00-3.00
Ni	10.00-14.00

2.1 Substrate Surface Roughening Process

The pipe surface was subjected to roughening treatment through sandblasting using the specified parameters. The glass bead served as the abrasive medium, with a particle size of 30/60 mesh. Particle firing was performed at a pressure of 4 bar while maintaining a horizontal angle perpendicular to the specimen surface. Particle firing was performed at a distance of 1.5 cm from the specimen surface under room temperature conditions. Coating adhesion test specimens were prepared by surface roughening using a sandpaper grit-60. Next, the substrate was immersed in acetone for 15 min to facilitate

contaminant removal, followed by drying. The substrate that had undergone the surface roughening procedure with sandblasting and sandpaper was then measured using a Mitutoyo SJ-210 roughness meter.

2.2 PANI/Al₂O₃ Coating on Stainless Steel 316

PANI/Al₂O₃/Epoxy and PANI/Al₂O₃/Al/Epoxy coatings, starting with the synthesis of PANI by preparing Solution A: Aniline with 1 M HCl, and Solution B: ammonium persulfate with deionized water. Both solutions were left to cool for 1 h. Solutions A and B were mixed and stirred, then kept at 0°C, and allowed to fully polymerize the aniline, resulting in a dark green PANI precipitate. The PANI precipitate was filtered and washed with 0.2 M HCl and acetone to remove unreacted monomers and oxidants and then dried at 60°C for 20 h. For the synthesis of PANI/Al₂O₃ composite, PANI and Al₂O₃ powder ratio of 7:3 was applied. Al₂O₃ was dissolved in n-butanol (10 ml of n-butanol and stirred for two hours. Next, PANI powder was added and stirred continuously for 2 h. The PANI/Al₂O₃ powder was then dried at 60°C for 24 h. Epoxy, solvent, and PANI/Al₂O₃ powders were mixed and stirred. The slurry was applied to the substrate surface using the tool shown in Fig. 1 and dried at room temperature.

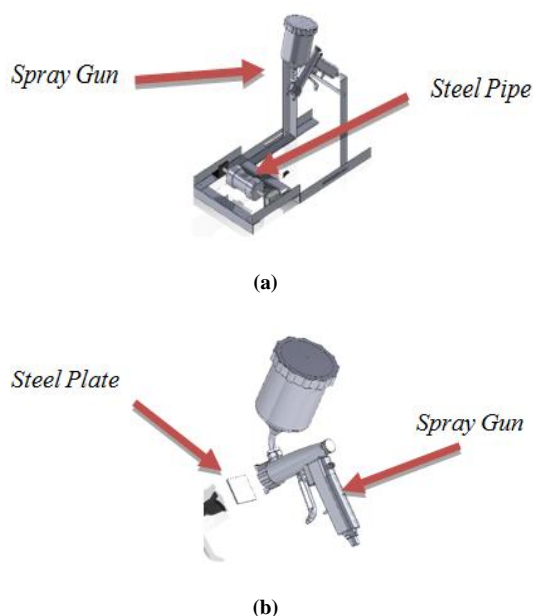


Figure 1: Illustration of Coating on SS 316 Substrate, (a) Coating on Pipe Specimen and (b) Coating on Plate Specimen

A 150 RPM DC motor drive (Figure 1a) was used to rotate the pipe specimen to be sprayed with a compressor spray gun at a distance of 100 mm as a coating tool with a spraying pressure of 2 bar. Figure 1b illustrates the coating on a plate-shaped specimen sprayed with a compressor spray gun at a distance of 100 mm with a spraying pressure of 2 bar.

III. RESULTS AND DISCUSSIONS

3.1 Substrate Surface Roughening Results

Roughness is an important parameter for understanding the effect of surface topography on the corrosion resistance of materials [14]. Figure 2a-c shows the graphs of the surface roughness test results before coating. Table 3 presents the average values of the obtained roughness parameters.

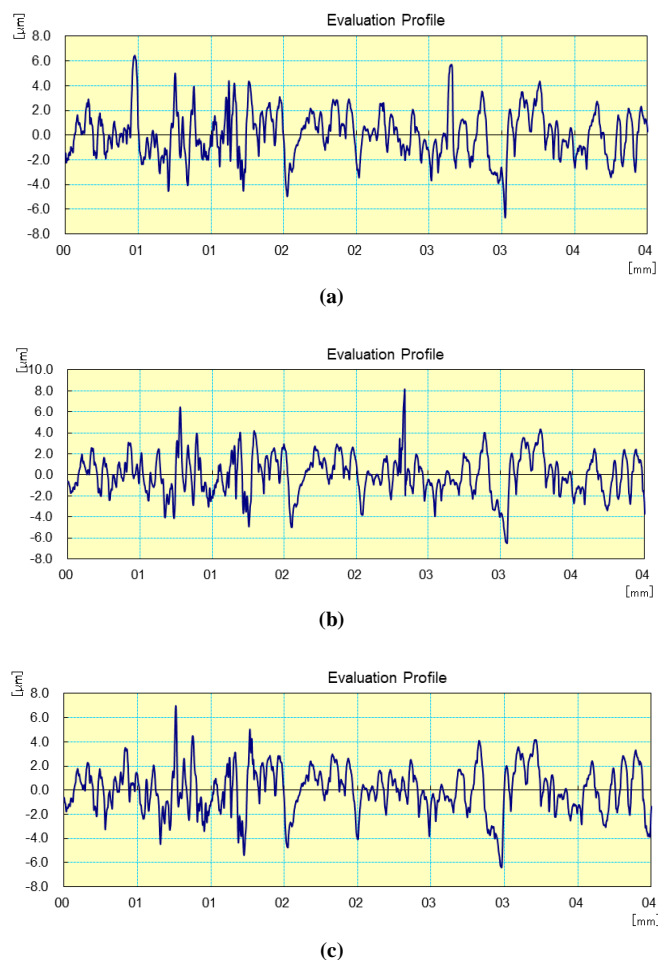


Figure 2: Graph of Surface Roughness Test Results of 60 Grit Sanding, (a) First Test (b) Second Test (c) Third Test

Table 3: Surface Roughness Test Results Sanding Grit 60

Measurement Condition	Ra (µm)		
	Measurement	Average	
λc = 0,8 mm	I	1.525	1.521
λs = 2,5 µm	II	1.517	
N = 5 times	III	1.522	

From the analysis of Figure 3a, it can be seen that after the sanding process with sandpaper grit-60, the roughness of the sample increased compared to that of the specimen without sanding (Figure 3b). This increase in roughness provides a larger surface area for coating adhesion [15][16].

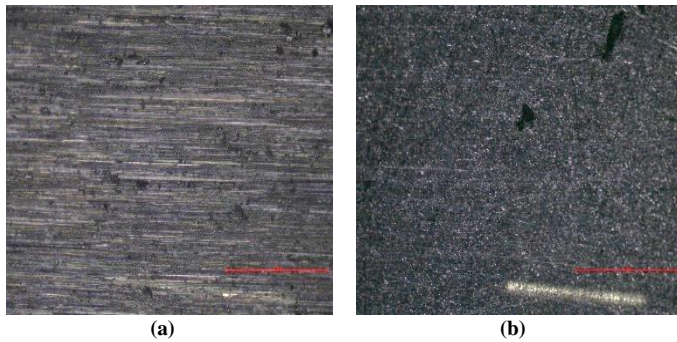


Figure 3: Surface Morphology (a) Sanding (b) Without Sanding

Sanding with 60 grit sandpaper produces significant changes in the surface morphology of stainless steel; wavy grooves and valleys are formed on the surface, and the surface becomes rough owing to the presence of micro scratches, and plastic deformation occurs in the metal surface layer [17]. The morphology resulting from rough sanding has significant implications for the subsequent coating process, as it can improve the adhesion of the coating owing to the rougher surface, facilitate the formation of discontinuous pores within the coating layer, and affect the distribution of elements in the transition layer between the substrate and the coating [18][19].

3.2 Results of PANI/Al₂O₃ Coating on Substrate

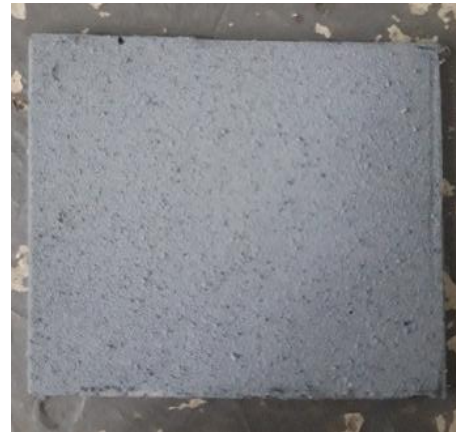
Figures 4a-c shows the results of the coating on stainless steel, showing a uniform and smooth surface texture, with a consistent granular pattern. No cracking, flaking, or significant surface defects were observed in the coating layer [20]. This indicates that the coating process proceeded well and produced a neat and stable surface. Overall, the coating results on stainless steel showed good visual quality with adequate uniformity owing to proper surface preparation using a combination of sandblasting and mechanical roughening. This reinforces the effectiveness of the coating in providing a high coating rate on the substrate.



(a)



(b)



(c)

Figure 4: Coating Results of (a) PANI/Al₂O₃/Epoxy, (b) PANI/Al₂O₃/Al/Epoxy on SS 316 Substrate and (c) PANI/Al₂O₃/Epoxy on Plate Substrate

3.3 PANI/Al₂O₃ Coating Layer Adhesion Test Results

The Hot Cross Hatch Adhesion Tester Adhesion Test results showed no delamination for the sanded specimens, while the unsanded specimens delaminated by 2.163%. Figure 5 shows the surface morphology of the substrate coated with the PANI/Al₂O₃ composite coating and treated with 60 grit sanding. This observation shows excellent adhesion between the coating and substrate. Based on the results of the cross-cut test, the coating surface showed optimal adhesion characteristics, where no peeling of the coating layer was detected in the test area, the edges of the test pieces remained smooth and showed no signs of delamination [21], and the percentage of peeled coating area was 0% [22].

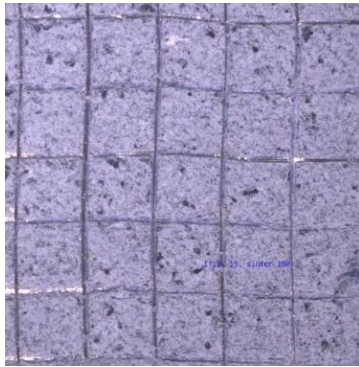


Figure 5: Morphology of PANI/Al₂O₃/Epoxy Coating Layer by Sanding

The measured peeling area of 2.163% on the surface of the substrate coated with the PANI/Al₂O₃ coating without the sanding treatment indicates a fairly good level of adhesion. The peeling that occurred was still within acceptable limits according to the testing standards. With a peeling percentage of <5%, this result is classified as category 4B according to ASTM D3359-17 [23], which indicates good adhesion between the coating and the substrate, minimal peeling along the scratches and cuts, and adequate bond strength between the coating layer and the substrate surface.

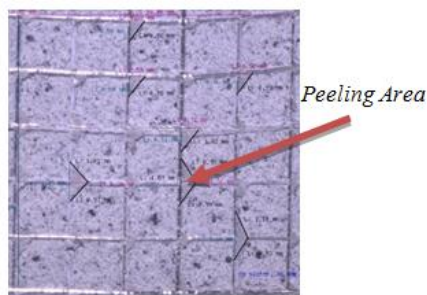


Figure 6: Morphology of PANI/Al₂O₃/Epoxy Coating Layer without Sanding

The PANI/Al₂O₃ coating showed good adhesion properties even without sanding treatment. This can be attributed to the formation of good bonds between the PANI molecules and Al₂O₃ particles and the even distribution of the particles on the substrate surface [24].

IV. CONCLUSION

In this study, PANI/Al₂O₃ nanocomposites were synthesized using a chemical oxidative polymerization method and the adhesion strength of the nanocomposites was investigated as a coating parameter for 316 stainless steel. Surface roughening of the 316 stainless steel substrate with 60-grit sandpaper significantly increased the surface roughness from 0.374 μm to 1.521 μm. This increase in roughness provides a larger area for coating adhesion, which in turn increases the adhesion strength between the coating and substrate. The PANI/Al₂O₃/Epoxy coating on stainless

steel exhibited a uniform and smooth surface texture with no cracks, flaking, or significant surface defects.

The Hot Cross-Hatch Adhesion test results showed that the sanded specimens did not delaminate, while the unsanded specimens exhibited a delamination of 2.163%. The PANI/Al₂O₃/Epoxy coating on the sanded substrate exhibited excellent adhesion, with 0% peeling off of the coating area. Meanwhile, without sanding, the PANI/Al₂O₃/Epoxy coating still showed satisfactory adhesion with only 2.163% peeling, which falls into category 4B according to the ASTM D3359-17 standard.

Overall, these results indicate that the developed PANI/Al₂O₃/Epoxy coating method effectively improves the adhesion of the coating on 316 stainless steel, especially when combined with the surface roughening of the substrate. This indicates that surface roughening is a key factor in improving the adhesion performance of protective coatings on stainless-steel substrate materials.

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