

Analysis Variation of Concentration and Pickling Time in SA-312 304 on Oxide Layer Thickness

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Abstract—Austenitic stainless steel SA-312 Grade 304 offers good corrosion resistance, but welding can form oxide layers that degrade its performance. Pickling with nitric acid (HNO₃) and hydrofluoric acid (HF) is commonly used to remove oxides and restore corrosion resistance. This study examines the effects of varying HNO₃ concentration and pickling time on oxide layer thickness. Optical and scanning electron microscopy (SEM) were performed. The optimal result was achieved with 15% HNO₃ + 4% HF for 35 minutes, producing a 5.6658 μm oxide layer, while 25% HNO₃ + 4% HF for 35 minutes yielded only 3.0082 μm. Results show that proper pickling parameters effectively reduce oxide scale.

Keywords—microscope optic, nitric acid, oxide layer, pickling, SEM, stainless steel.

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I. INTRODUCTION

Stainless steel is specifically designed for demanding applications requiring resistance to heat, chemicals, and various forms of corrosion. The term “stainless steel” refers to a group of metal alloys generally composed of iron, carbon, and chromium. Chromium plays a critical role in forming a strong, homogeneous passive layer that protects the alloy from its surrounding environment. This passive layer consists of a hard chromium oxide film overlying a metal layer with reduced chromium content, typically only a few nanometers thick [1]. Chromium depletion in this layer can reduce its corrosion resistance. To improve specific characteristics such as hardness, strength, and corrosion resistance, additional elements like nickel, manganese, and others are often added to the alloy [2]. Global demand for stainless steel products continues to grow, particularly in the food and beverage, chemical, nuclear, and oil and gas industries. Moreover, its use is expanding in building protection, transportation systems, and energy infrastructure to address corrosion-related issues [3].

Although austenitic stainless steel has high corrosion resistance, its welded joints are not always free from the risk of pitting corrosion [4]. This may occur due to improper welding processes that damage

the protective oxide layer composed of chromium oxide. However, the protective oxide layer can reform if the welding oxide or burn scale is properly removed [5]. One way to eliminate oxides and contaminants is through chemical surface treatment, a process aimed at repairing damage to the oxide layer caused by welding using acid solutions. The method most commonly used in this process is pickling [6].

The pickling process is commonly applied in industry to ensure the quality of stainless steel weld joints. Technically, pickling is superior for stainless steel surface treatment because it can optimally restore corrosion resistance [7]. However, its drawback lies in the use of strong acid solutions that are corrosive and toxic, requiring special care and handling during application [8].

This research aims to investigate the thickness of oxide layer of the welded joints TIG stainless steel 304 due to the effect of variation concentration and pickling time and to investigate the suitability of the test results with microscope optic and SEM (scanning electron microscope). Specifically, this research focuses on how these pickling parameters influence the thickness of the oxide layer, as well as comparing specimens with and without pickling treatment. The objective of this study is to determine the effect of varying pickling solution concentration and treatment duration on the thickness of the oxide layer, as well as to compare specimens with and without pickling treatment.

II. METHOD

A. Object of Research

Material used in this research is stainless steel 304 pipe.

B. Research Parameters Material

1. Material : Stainless steel 304 pipe
2. Welding Type : TIG
3. Groove : Single-V
4. TIG Weld Angle : 60°

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These materials were fit-up into two welded joints according to the specified dimensions, with detailed configuration shown in the following Figure 1.

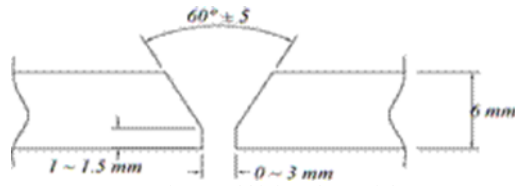


Figure 1. Fit up weld joint of materials

C. Pickling Process

The pickling process was carried out after welding using the immersion pickling method, in which the welded specimens were immersed in a solution of HNO_3 (68%) and HF (55%). This process applied variations in solution concentration and pickling duration to the specimens. The detailed application of these variations to the test specimens is presented in Table 1, and image specimen for pickling are shown in Figure 2.

TABLE 1.
CODE SPECIMEN FOR APPLICATION OF PICKLING

Code Specimen	Concentration Solution HNO_3 + HF	Time Pickling
A1	HNO_3 15% + HF 4%	35 Minutes
A2		25 Minutes
A3		15 Minutes
A4		5 Minutes
B1	HNO_3 20% + HF 4%	35 Minutes
B2		25 Minutes
B3		15 Minutes
B4		5 Minutes
C1	HNO_3 25% + HF 4%	35 Minutes
C2		25 Minutes
C3		15 Minutes
C4		5 Minutes

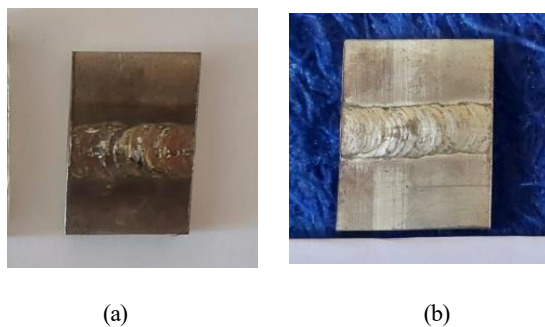


Figure 2. Specimen Pickling a) Before Pickling, b) After Pickling

D. Testing of Specimen

The testing process in this research involves optical microscope and scanning electron microscope (SEM) to determine the average thickness of the oxide layer formed after the pickling process.

E. Microscope Optic

Optical microscope was conducted to analyze the surface structure and oxide layer thickness of the specimens formed after the pickling process. The optical microscope specimens are shown in Figure 3.

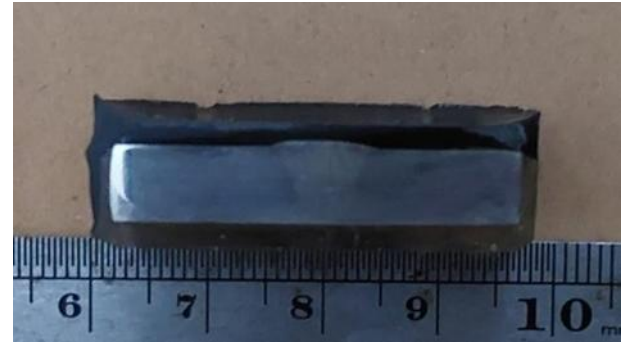


Figure 3. Specimen for Microscope Optic Testing

F. Scanning Electron Microscope (SEM)

Scanning Electron Microscopy (SEM) was performed after the pickling process to analyze the thickness of the oxide layer formed. SEM enables detailed observation of the material's surface microstructure, allowing evaluation of the quality and uniformity of the oxide layer produced by pickling. The SEM test specimens are shown in Figure 4.



Figure 4. Specimen for SEM

III. RESULTS AND DISCUSSION

A. Oxide layer thickness results of specimens pickled with 15% HNO_3 + 4% HF solution for 5 minutes

The oxide layer thickness data for specimens pickled with 15% HNO_3 + 4% HF at pickling times for 5 minutes shown in Figure 5. Specimens pickled in 15% HNO_3 + 4% HF for 5 minutes (A4) exhibited an oxide layer thickness of 3,9527 μm .

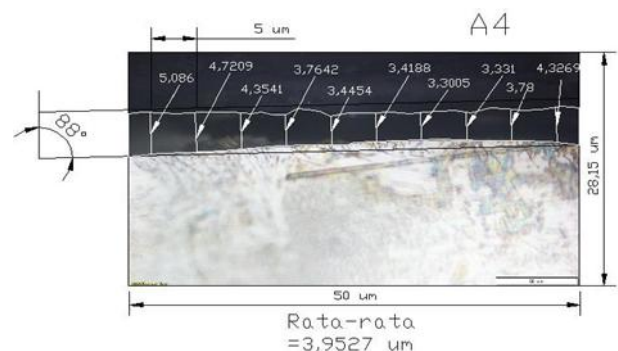


Figure 5. Oxide Layer Thickness of Specimens Pickled in 15% HNO_3 + 4% HF Solution for 5 Minutes

B. Oxide layer thickness results of specimens pickled with 15% HNO_3 + 4% HF solution for 15 minutes

The oxide layer thickness data for specimens pickled with 15% HNO_3 + 4% HF at pickling times for 15 minutes shown in **Figure 6**. Specimens pickled in 15% HNO_3 + 4% HF for 15 minutes (A3) exhibited an oxide layer thickness of 4,4191 μm .

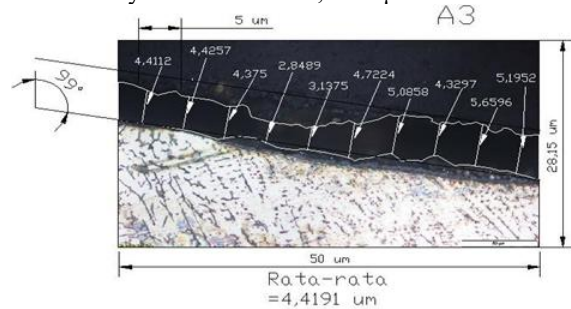


Figure 6. Oxide Tayer Thickness of Specimens Pickled in 15% HNO_3 + 4% HF Solution for 15 Minutes

C. Oxide layer thickness results of specimens pickled with 15% HNO_3 + 4% HF solution for 25 minutes

The oxide layer thickness data for specimens pickled with 15% HNO_3 + 4% HF at pickling times for 25 minutes shown in **Figure 7**. Specimens pickled in 15% HNO_3 + 4% HF for 25 minutes (A2) exhibited an oxide layer thickness of 5,1888 μm

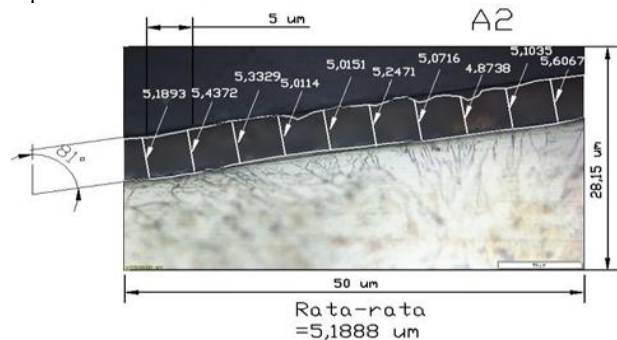


Figure 7. Oxide Layer Thickness of Specimens Pickled in 15% HNO_3 + 4% HF Solution for 25 Minutes

D. Oxide layer thickness results of specimens pickled with 15% HNO_3 + 4% HF solution for 35 minutes

The oxide layer thickness data for specimens pickled with 15% HNO_3 + 4% HF at pickling times for 35 minutes shown in **Figure 8**. Specimens pickled in 15% HNO_3 + 4% HF for 35 minutes (A1) exhibited an oxide layer thickness of 5,6658 μm .

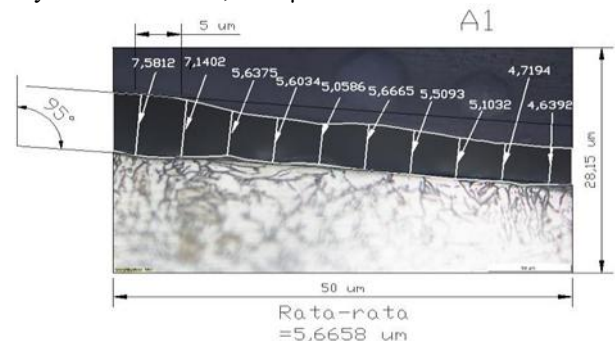


Figure 8. Oxide Layer Thickness of Specimens Pickled in 15% HNO_3 + 4% HF Solution for 35 Minutes

Analysis of the oxide layer thickness for specimens pickled with 15% HNO_3 + 4% HF indicates that increasing the pickling duration leads to the formation of a thicker oxide layer. This behavior is attributed to the relatively low HNO_3 concentration, which slows and stabilizes the oxide dissolution process, thereby facilitating the development of a homogeneous and stable passive layer primarily composed of Cr_2O_3 [9]. In contrast, shorter pickling durations result in insufficient oxide removal and hinder the formation of an optimal passive layer [1]. The oxide layer thickness data for specimens pickled with 15% HNO_3 + 4% HF can be seen in **Table 2**.

TABLE 2
OXIDE LAYER THICKNESS DATA OF SPECIMENS PICKLED WITH 15% HNO_3 + 4% HF

Code Specimen	Pickling Time	Oxide Layer Thickness per Segment (μm)										Average (μm)
		Seg 1	Seg 2	Seg 3	Seg 4	Seg 5	Seg 6	Seg 7	Seg 8	Seg 9	Seg 10	
A4	5 Minute	5,08	4,72	4,35	3,76	3,44	3,41	3,30	3,33	3,78	4,32	3,9527
A3	15 Minute	4,41	4,42	4,37	2,84	3,13	4,72	5,08	4,32	5,65	5,19	4,4191
A2	25 Minute	5,18	5,43	5,33	5,01	5,01	5,24	5,07	4,87	5,10	5,60	5,1888
A1	35 Minute	7,58	7,14	5,63	5,60	5,05	5,66	5,50	5,10	4,71	4,63	5,6658

E. Oxide layer thickness results of specimens pickled with 20% HNO_3 + 4% HF solution for 5 minutes

The oxide layer thickness data for specimens pickled with 20% HNO_3 + 4% HF at pickling times for 5 minutes shown in **Figure 9**. Specimens pickled in 20% HNO_3 + 4% HF for 5 minutes (B4) exhibited an oxide layer thickness of 4,9788 μm

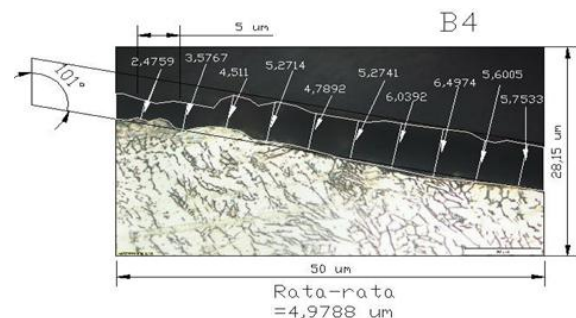


Figure 9. Oxide Layer Thickness of Specimens Pickled in 20% HNO_3 + 4% HF Solution for 5 Minutes

F. Oxide layer thickness results of specimens pickled with 20% HNO_3 + 4% HF solution for 15 minutes

The oxide layer thickness data for specimens pickled with 20% HNO_3 + 4% HF at pickling times for 15 minutes shown in **Figure 10**. Specimens pickled in 20% HNO_3 + 4% HF for 15 minutes (B3) exhibited an oxide layer thickness of 4,2012 μm

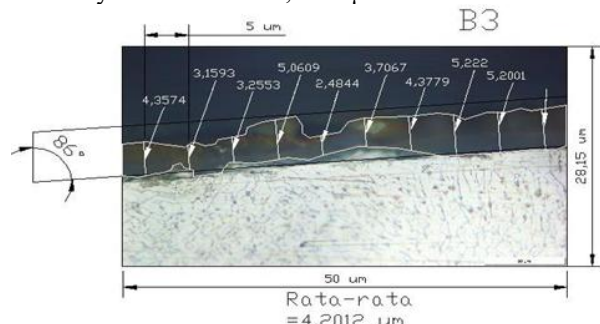


Figure 10. Oxide Layer Thickness of Specimens Pickled in 20% HNO_3 + 4% HF Solution for 15 Minutes

G. Oxide layer thickness results of specimens pickled with 20% HNO_3 + 4% HF solution for 25 minutes

The oxide layer thickness data for specimens pickled with 20% HNO_3 + 4% HF at pickling times for 25 minutes shown in **Figure 11**. Specimens pickled in 20% HNO_3 + 4% HF for 25 minutes (B2) exhibited an oxide layer thickness of 3,3550 μm .

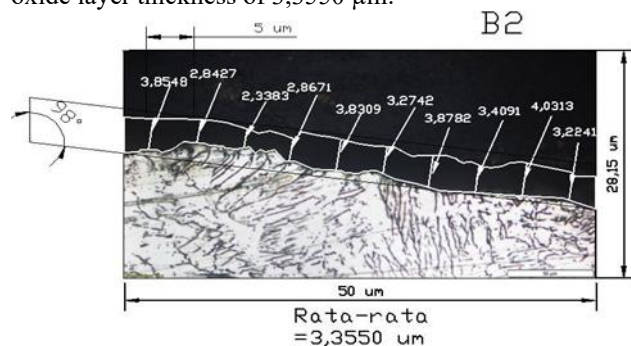


Figure 11. Oxide Layer Thickness of Specimens Pickled in 20% HNO_3 + 4% HF Solution for 25 Minutes

H. Oxide layer thickness results of specimens pickled with 20% HNO_3 + 4% HF solution for 35 minutes

The oxide layer thickness data for specimens pickled with 20% HNO_3 + 4% HF at pickling times for 35 minutes shown in **Figure 12**. Specimens pickled in 20% HNO_3 + 4% HF for 35 minutes (B1) exhibited an oxide layer thickness of 3,2614 μm .

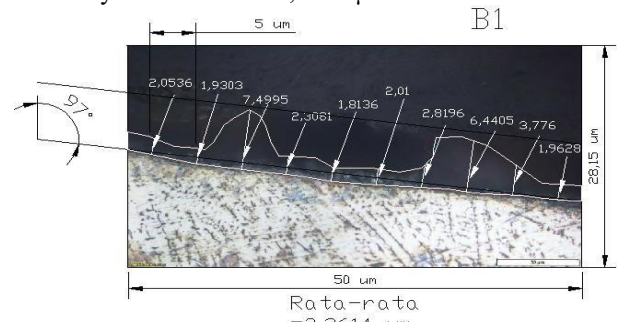


Figure 12. Oxide Layer Thickness of Specimens Pickled in 20% HNO_3 + 4% HF Solution for 35 Minutes

The oxide layer thickness results for specimens pickled with 20% HNO_3 + 4% HF indicate that longer pickling durations produce thinner oxide layers. This is attributed to the initial rapid and aggressive reaction between the metal surface and the HNO_3 solution, which facilitates quick formation of a new, homogeneous, and stable passive layer predominantly composed of Cr_2O_3 in a short time [1]. However, at higher HNO_3 concentrations, prolonged pickling causes the oxide dissolution reaction to become excessively aggressive, leading to over-etching, where the acid solution begins to excessively dissolve the base metal itself [10]. The oxide layer thickness data for specimens pickled with 20% HNO_3 + 4% HF can be seen in **Table 3**.

TABLE 3
OXIDE LAYER THICKNESS DATA OF SPECIMENS PICKLED WITH 20% HNO_3 + 4% HF

Code Specimen	Pickling Time	Oxide Layer thickness per Segment (μm)										Average (μm)
		Seg 1	Seg 2	Seg 3	Seg 4	Seg 5	Seg 6	Seg 7	Seg 8	Seg 9	Seg 10	
B4	5 Minute	2,47	3,57	4,51	5,27	4,78	5,27	6,03	6,49	5,60	5,75	4,9788
B3	15 Minute	4,35	3,15	3,25	5,06	2,48	3,70	4,37	5,22	5,20	5,19	4,2012
B2	25 Minute	3,85	2,84	2,33	2,86	3,83	3,27	3,87	3,40	4,03	3,22	3,3550
B1	35 Minute	2,05	1,93	7,49	2,30	1,81	2,01	2,81	6,44	3,77	1,96	3,2614

I. Oxide layer thickness results of specimens pickled with 25% HNO_3 + 4% HF solution for 5 minutes

The oxide layer thickness data for specimens pickled with 25% HNO_3 + 4% HF at pickling times for 5 minutes shown in **Figure 13**. Specimens pickled in 25% HNO_3 + 4% HF for 5 minutes (C4) exhibited an oxide layer thickness of 4,7685 μm .

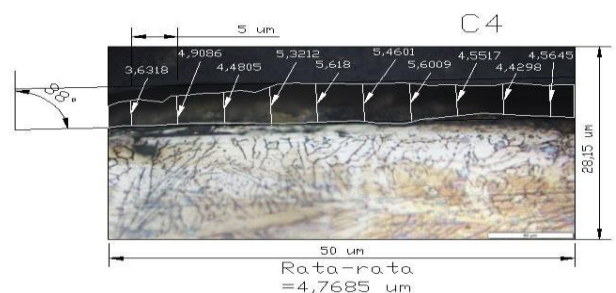


Figure 13. Oxide Layer Thickness of Specimens Pickled in 25% HNO_3 + 4% HF Solution for 5 Minutes

J. Oxide layer thickness results of specimens pickled with 25% HNO_3 + 4% HF solution for 15 minutes

The oxide layer thickness data for specimens pickled with 25% HNO_3 + 4% HF at pickling times for 15 minutes shown in **Figure 14**. Specimens pickled in 25% HNO_3 + 4% HF for 15 minutes (C3) exhibited an oxide layer thickness of 3,7394 μm .

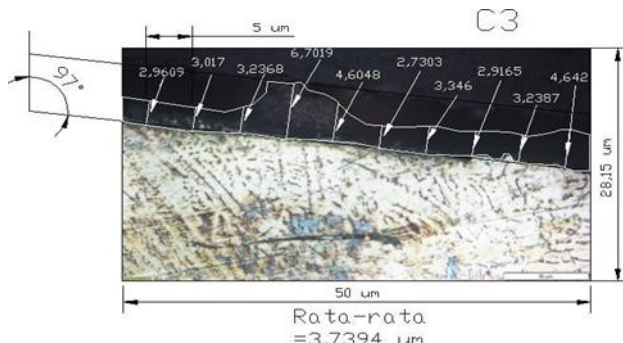


Figure 14. Oxide Layer Thickness of Specimens Pickled in 25% HNO_3 + 4% HF Solution for 15 Minutes

L. Oxide layer thickness results of specimens pickled with 25% HNO_3 + 4% HF solution for 35 minutes

The oxide layer thickness data for specimens pickled with 25% HNO_3 + 4% HF at pickling times for 35 minutes shown in **Figure 16**. Specimens pickled in 25% HNO_3 + 4% HF for 35 minutes (C1) exhibited an oxide layer thickness of 3,0082 μm .

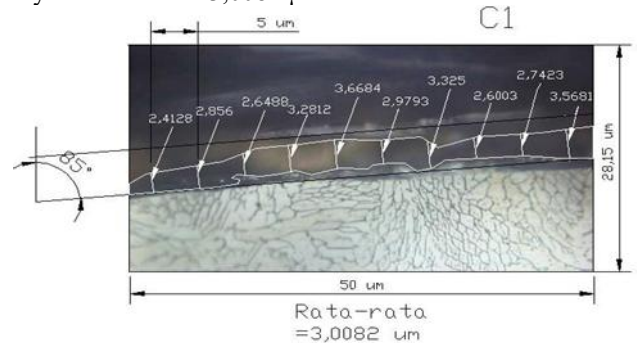


Figure 16. Oxide Layer Thickness of Specimens Pickled in 25% HNO_3 + 4% HF Solution for 35 Minutes

K. Oxide layer thickness results of specimens pickled with 25% HNO_3 + 4% HF solution for 25 minutes

The oxide layer thickness data for specimens pickled with 25% HNO_3 + 4% HF at pickling times for 25 minutes shown in **Figure 15**. Specimens pickled in 25% HNO_3 + 4% HF for 25 minutes (C2) exhibited an oxide layer thickness of 3,1314 μm .

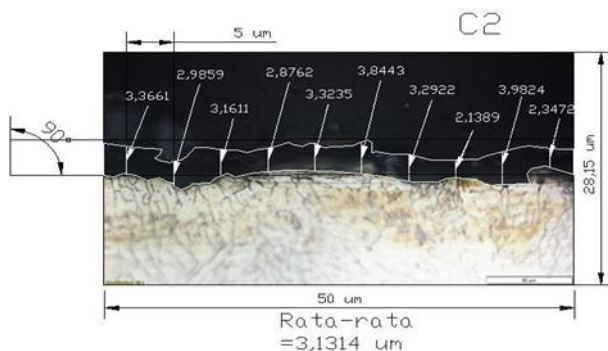


Figure 15. Oxide Layer Thickness of Specimens Pickled in 25% HNO_3 + 4% HF Solution for 25 Minutes

The oxide layer thickness results for specimens pickled with 25% HNO_3 + 4% HF show that longer pickling durations lead to progressively thinner oxide layers. At this high HNO_3 concentration, a new passive oxide layer predominantly composed of Cr_2O_3 can form rapidly, creating a more homogeneous and stable surface in the early stages [1]. However, the high aggressiveness of the 25% HNO_3 pickling solution promotes excessive dissolution (over-etching). This can increase induce micro-pit formation, and even cause chromium depletion at the surface, ultimately reducing the material's ability to form a high quality passive layer [10]. The oxide layer thickness data for specimens pickled with 25% HNO_3 + 4% HF can be seen in **Table 4**,

Thus, the graphics data of oxide layer thickness results for each specimen pickled with 15%, 20%, and 25% HNO_3 + 4% HF for 5, 15, 25, and 35 minutes are presented in Table 5 below. While the graphics data for oxide layer thickness shown in **Figure 17**.

TABLE 4
OXIDE LAYER THICKNESS DATA OF SPECIMENS PICKLED WITH 25% HNO_3 + 4% HF

Code Specimen	Pickling Time	Oxide Layer thickness per Segment (μm)										Average (μm)
		Seg 1	Seg 2	Seg 3	Seg 4	Seg 5	Seg 6	Seg 7	Seg 8	Seg 9	Seg 10	
C4	5 Minute	3,63	4,09	4,40	5,32	5,61	5,46	5,60	4,55	4,42	4,56	4,7685
C3	15 Minute	2,96	3,01	3,23	6,70	4,60	2,73	3,34	2,91	3,23	4,64	3,7394
C2	25 Minute	3,36	2,98	3,16	2,87	3,32	3,84	3,29	2,13	3,98	2,34	3,1314
C1	35 Minute	2,41	2,85	2,64	3,28	3,66	2,97	3,32	2,60	2,74	3,56	3,0082



Figure. 17. Graphics of Oxide Layer Thickness for All Picking Variations

Figure 17 presents the oxide layer thickness for all pickling variations. In specimens treated with 15% HNO_3 + 4% HF, increasing the pickling duration leads to thicker oxide layers. This behavior reflects a slower and more controlled oxide dissolution process at low acid concentration, which facilitates the gradual formation of a homogeneous and stable Cr_2O_3 passive layer [1]. In contrast, specimens pickled with 20% and 25% HNO_3 + 4% HF exhibit thinner oxide layers with longer exposure times. The high acid concentration initiates a rapid reaction with the metal surface, forming a Cr_2O_3 passive film almost immediately [10]. However, as pickling continues, the dissolution process becomes excessively aggressive. This over-etching not only removes the oxide but also begins to attack the underlying metal, degrading surface integrity and potentially reducing corrosion resistance [5].

M. SEM Micrograph of Specimen A2

The SEM images obtained using the Secondary Electron (SE) mode to observe surface morphology are shown in **Figure 18**, while the Backscattered Electron (BSE) mode to analyze material contrast for specimens pickled with 15% HNO_3 + 4% HF for 25 minutes (A2) is presented in **Figure 19**.

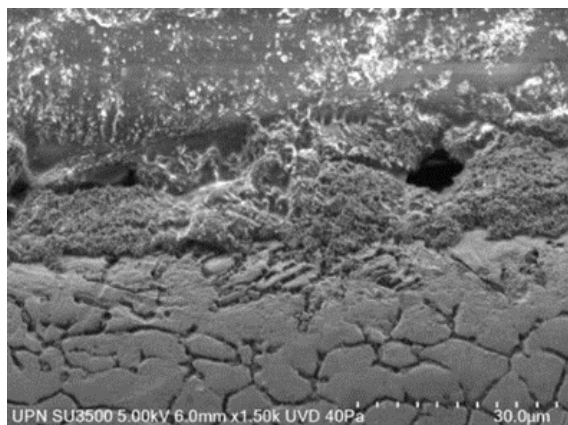


Figure 18. SE Mode for Surface Morphology of A2

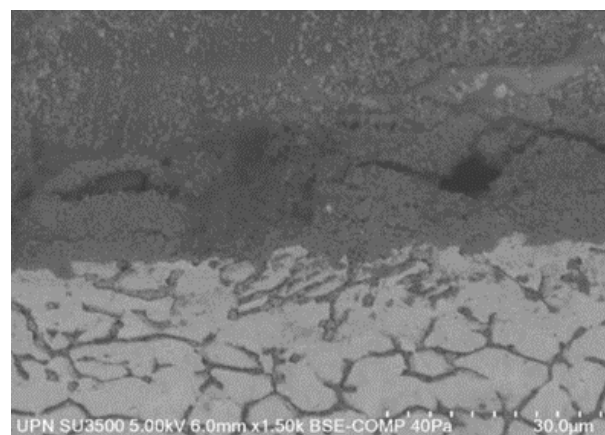


Figure 19. BSE Mode for Material Contrast of A2

Based on the observations in **Figure 18** and **Figure 19**, the surface morphology of the specimen pickled with 15% HNO_3 + 4% HF for 25 minutes appears homogeneous and smooth. The oxide layer formed is thick, uniform, and exhibits low porosity. This is attributed to the relatively low HNO_3 concentration (15%), which slows and stabilizes the oxide dissolution process, enabling the formation of a new passive oxide layer that is more homogeneous and stable, predominantly composed of Cr_2O_3 [1]. As a result, the surface becomes cleaner with finely distributed oxide particles, making this condition more effective in producing a high-quality oxide layer morphology.

N. SEM Micrograph of Specimen B3

The SEM images obtained using the Secondary Electron (SE) mode to observe surface morphology are shown in **Figure 20**, while the Backscattered Electron (BSE) mode to analyze material contrast for specimens pickled with 20% HNO_3 + 4% HF for 15 minutes (B3) is presented in **Figure 21**.

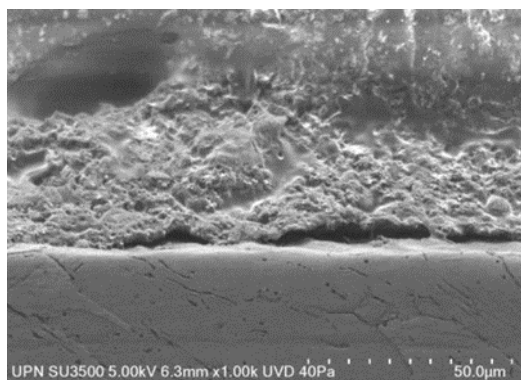


Figure 20. SE Mode for Surface Morphology of B3

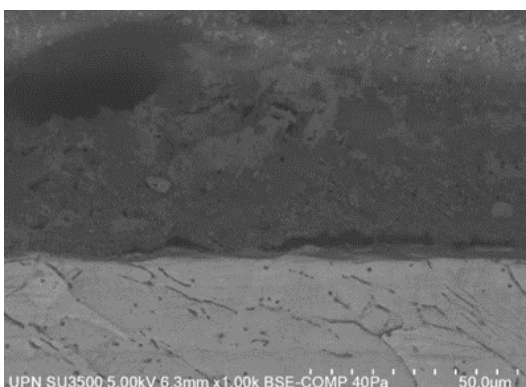


Figure 21. BSE Mode for Material Contrast of B3

Based on the observations in **Figure 20** and **Figure 21**, the surface morphology of the specimen pickled with 20% HNO_3 + 4% HF for 15 minutes shows a less uniform and porous structure. The oxide layer formed appears thick and evenly distributed, with low porosity within a short time, which facilitates the formation of a new, more homogeneous and stable passive oxide layer predominantly composed of Cr_2O_3 [1]. However, higher acid concentration combined with longer pickling duration can lead to excessive dissolution (over-etching), where the acid solution begins to attack not only the oxide layer but also the base metal itself [10].

O. SEM Micrograph of Specimen Without Pickling (X)

The SEM images obtained using the Secondary Electron (SE) mode to observe surface morphology are shown in **Figure 22**, while the Backscattered Electron (BSE) mode to analyze material contrast for specimens without pickling process is presented in **Figure 23**.

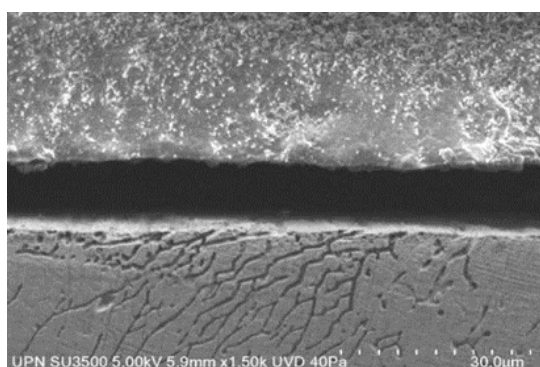


Figure 22. SE Mode for Surface Morphology of X

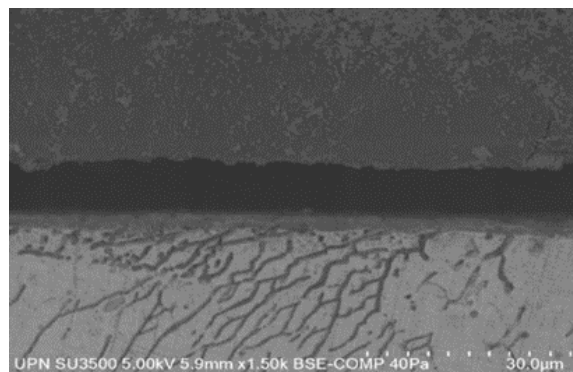


Figure 23. BSE Mode for Material Contrast of X

Based on the observations in **Figure 22** and **Figure 23**, the surface morphology of the specimen without pickling treatment appears smooth due to the presence of a natural oxide layer formed during production or storage [2]. Since this layer has not undergone chemical dissolution, the surface appears flat, without prominent roughness, pores, or protrusions [7]. However, such naturally formed oxide layers are typically non-uniform and contain micro- defects, inclusions, and contaminants that serve as preferential sites for localized attack [10]. Moreover, the passive film is thin and heterogeneous, predominantly composed of Fe_2O_3 and Fe_3O_4 formed by high- temperature oxidation of iron. Unlike Cr_2O_3 -based passive layers, these iron oxides are brittle, less protective, and prone to cracking, which compromises their barrier properties and facilitates chloride penetration, making them susceptible to pitting corrosion initiation [1].

IV. CONCLUSION

Based on the data presentation and analysis in this study, the following conclusions can be drawn. The oxide layer thickness measurements indicate that lower HNO_3 concentrations combined with longer pickling times result in thicker oxide layers. Conversely, higher HNO_3 concentrations with longer pickling times produce thinner oxide layers. The most optimal oxide layer thickness was observed in the specimen pickled with 15% HNO_3 + 4% HF for 35 minutes (A1), which formed an oxide layer of 5.6658 μm . In contrast, the least optimal thickness was obtained in the specimen pickled with 25% HNO_3 + 4% HF for 35 minutes (C1), where the oxide layer measured only 3.0082 μm .

The oxide layers formed on specimens subjected to pickling appear thicker than those formed naturally. This is because the pickling process accelerates the formation of oxide layers in a short time, which facilitates the formation of a new, more homogeneous and stable passive oxide layer predominantly composed of Cr_2O_3 . Whereas the naturally formed oxide layer on specimens without pickling is typically thin and non-homogeneous. The dominant oxides in the natural layer are Fe_2O_3 and Fe_3O_4 , which result from high-temperature iron oxidation. These oxides are brittle, poorly protective, and prone to cracking, making them potential initiation sites for pitting corrosion.

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