

## CORROSION RESISTANCE OF SURFACE-CONDITIONED 301 AND 304 STAINLESS STEELS BY SALT SPRAY TEST

*Temitope Olumide Olugbade, Babatunde Olamide Omiyale*

Department of Industrial and Production Engineering, Federal University of Technology, P.M.B. 704, Akure, Ondo State, Nigeria.  
E-mail: boomiyale@futa.edu.ng

### ABSTRACT

The corrosion rate of surface-conditioned 301 and 304 stainless steels (SS) was determined by salt spray test in a controlled accelerated corrosive medium (9.5 L of pure distilled water + 500 g NaCl). Surface conditioning via mechanical attrition treatment was firstly carried out before the salt spray test. The corrosion rate was determined by weight loss method before and after the salt spray test. Compared to the untreated 301 SS sample with a weight loss of 0.15 g, the surface-conditioned 301 SS samples treated for 300 s and 1200 s experienced a lower weight loss of 0.04 and 0.02 g, respectively. A similar reduction in weight loss was achieved for 304 SS sample when treated for 300, 600, and 1200 s.

Keywords: Salt spray test; 301 SS; 304 SS; corrosion rate; weight loss.

### 1. INTRODUCTION

AISI 304 and 301 stainless steels (SS) are widely used engineering materials in most manufacturing and production industries due to their good corrosion behaviour and excellent high hardness-strength combination. Despite their good corrosion properties, there is still a high tendency of material failure due to usage and exposure to corrosive environment over time [1-4]. Hence, there is a need to determine their actual corrosion rate in a monitored accelerated corrosive medium to predict their behaviour in a real-life application. Over the years, various corrosion testing methods have been adopted to determine the resistance of materials to corrosion which includes polarization tests and salt spray test [5-7].

As a form of corrosion testing method, salt spray test determines the rate of corrosion of engineering materials over a long period of time in a controlled corrosive environment. It is a promising method for identifying the time when the first sign of corrosion is evident after subjecting the material to a long corrosion test. In the past, salt spray test method has been successfully used to evaluate the corrosion behaviour of different material systems including stainless steel [5, 8-9],  $\text{Al}_2\text{O}_3\text{-ZrO}_2$  [10],  $\text{Al}_2\text{O}_3$  [11-12],  $\text{ZrO}_2$  [12-13], alumina coating [14-15], Mg-Al alloy [16], and galvanized steel [17-18].

Stainless steel types 301 and 304 are presently attracting considerable attention due to their good mechanical and corrosion properties and they find applications in most manufacturing and production industries. However, further study is still needed on their corrosion resistance especially when they are subjected to surface treatment. Hence, the corrosion resistance of treated SS in a monitored accelerated corrosive medium will be a good subject of investigation.

In the present study, 301 and 304 SS samples were first subjected to surface conditioning through mechanical attrition treatment. Thereafter, the corrosion behaviour of the untreated and treated samples was investigated through salt spray test using 5% concentration salt solution. The weight of the samples before and after salt spray test were determined and the corrosion rate was therein determined through weight loss method.

### 2. EXPERIMENTAL DETAILS

#### 2.1 Material preparation

A typical commercial 304 and 301 stainless steels (SS) samples of dimension 70 x 60 x 1 mm<sup>3</sup> were used in this study, with a chemical composition in Table 1. All samples were cut using electrical discharge machining (Model: ALN400G, Thailand) and properly cleaned using acetone before the corrosion test. The

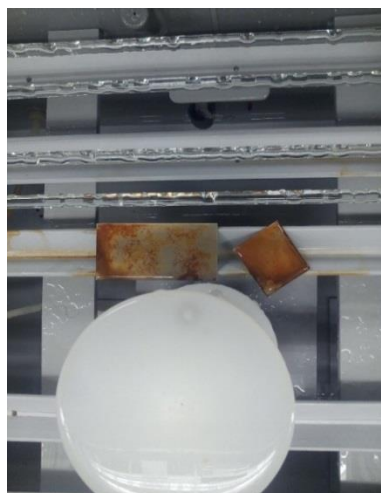
samples were then surface conditioned via mechanical attrition treatment for 300, 600, and 1200 s. The mechanical attrition procedure has been previously described in the literature [19-20].

**Table 1. Elemental composition of untreated 304 and 301 stainless steels.**

Elements	Conc. (wt. %)	
	304 SS	301
C	0.04	0.15
Si	0.52	1.00
Mn	1.18	2.00
Cr	17.59	18.00
Ni	8.03	10.00
S	0.03	0.03
P	0.04	0.05
Fe	Bal	Bal

## 2.2 Salt spray test

The corrosion rate of the untreated and treated samples was determined by subjecting the samples to salt spray test using a salt spray tester (Model: SH-90, China) with a chamber volume of 270 liters, salt solution tank size of 25 liters and spray rate of 1-2 ml/80 cm<sup>2</sup>/hour. The apparatus and the setup are shown in Figure 1.



**Figure 1. Salt spray tester and the specimens after the salt spray test.**

The chamber and standard air tank temperatures are 35°C and 47°C, respectively. The tester operated at the air compressor of 0.32 MPa, pressure adjuster of 1 kg/cm<sup>2</sup> and the pressure reducer was in the range 2 – 3 kg/cm<sup>2</sup>. The testing time was 240 h and the sample tilt angle is 10°. According to ASTM B117 standard (ASTM B117-16 2016) [21], the salt solution used for the test comprised the mixture of 9.5 liters of pure distilled water and 500 g NaCl salt (5 % concentration). The test determines the relative corrosion resistance of the untreated and treated samples for 300, 600, and 1200 s. To ensure uniform exposure to the salt spray mist, all the steel samples were frequently rotated in the test chamber. During the test, the rate of corrosion was determined by noting the time until the first sign of rust is evident on the samples. Weight loss was measured for each sample at every 24 h intervals and the mean weight loss was calculated.

The condensate collection was carried out twice a day and it was analyzed for both pH and concentration throughout the salt spray test. The pH and concentration of the collected condensate were within the range of 6.5 – 7.2 and 4 - 6 %, respectively. To replenish the salt solution inside the test chamber, fresh solution (5 % concentration) was prepared every 24 h of the test.

Before the test, the samples were arranged in the salt test chamber in such a way that they were not in contact with any metallic material or with each other and placed parallel to the direction of the fog flow. After the salt sprat testing, the treated samples were examined according to ASTM D1645-02 method, which provides a means of comparing and evaluating the common corrosion performance of the samples. To remove salt deposits from the treated sample surface after the salt spray test, the samples were carefully removed from the holder and gently washed in clean warm running water at about 38°C, and then immediately dried naturally in air. Thereafter, the weight of the treated samples after test were taken and the weight loss was subsequently determined.

### 3. RESULTS AND DISCUSSION

#### 3.1 Corrosion rate for 304 steel samples

Figure 2 shows the cumulative weight losses of the untreated and treated 304 SS samples after the salt spray test for 240 h while the comparison of weights of untreated and treated 304 SS samples before and after salt spray test, at the end of 240 h is shown in Figure 3. A stain of red corrosion was evident after 24 h for the untreated sample whereas it first appeared after 240 h for the treated samples. It should be noted that the dimension of test samples obviously would affect the corrosion area and consequently change the weight loss results. The weight of the untreated sample before salt spray test was 43.06 g and the sample were kept inside the salt spray chamber. After the salt spray test, the weight of the sample was reduced to 40.91 g. The weight loss was 2.15 g, showing the effect of corrosion. For sample treated for 1200 s, the initial weight before salt spray test was 52.16 g and the final weight after salt spray test for 240 h was 51.92 g (Figure 3). As evident in Figure 2, the weight loss for the treated sample is 0.24 g. It is important to note that the corrosion resistance of a material is related to the weight loss. The lower the weight loss after salt spray test, the better the corrosion resistance. The treated 304 SS samples experienced lower weight loss after salt spray test. Table 2 shows the summary of the salt spray test for untreated and treated 304 SS samples after different times of exposure. After 240 h of treatment, compared with the treated samples with no change in colour and sign of red dust, a stain of red dust was observed along the edge of the untreated sample.

*Table 2. Results of salt spray test for untreated and treated 304 SS samples after different times of exposure.*

Treatment Time (min)	24	48	52	Time (h) 96	120	168	240
0	Stains	Stains	Stains	Stains	Stains	Stains	Stains
5	No change	No change	No change	No change	No change	No change	Stains
10	No change	No change	No change	No change	No change	No change	Stains
20	No change	No change	No change	No change	No change	No change	Stains

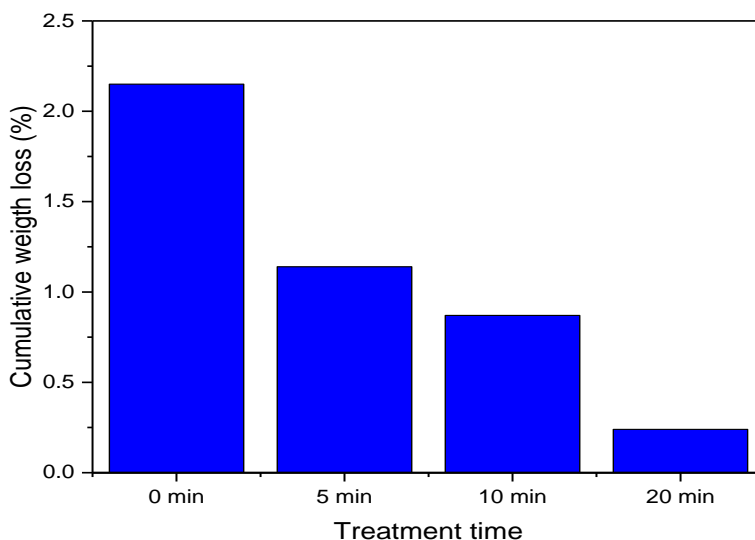


Figure 2. The cumulative weight losses for untreated and those 304 stainless steel samples treated for 5, 10, and 20 mins, after salt spray test for 240 h.

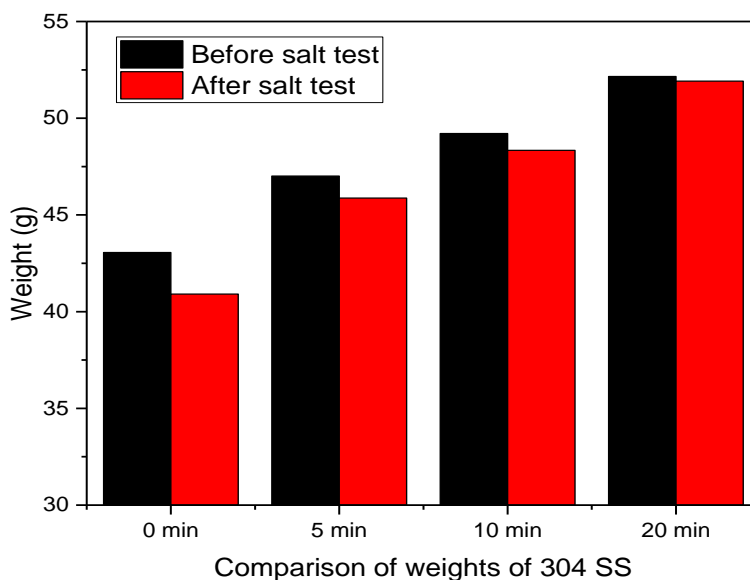
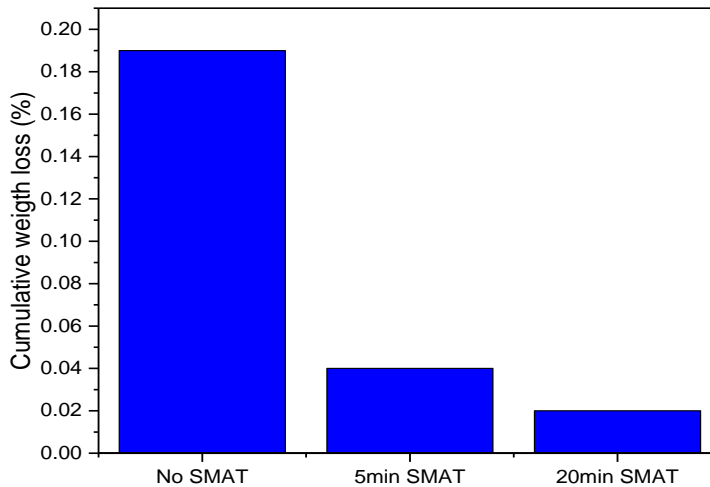


Figure 3. Comparison of weights of untreated and treated 304 SS samples, before and after salt spray test, at the end of 240 h.

### 3.2 Corrosion rate for 301 SS samples

Figure 4 shows the cumulative weight losses for untreated and treated 301 SS samples after salt spray test while Figure 5 shows the comparison of weights of untreated and treated 301 SS samples before and after the salt spray test at the end of 240 h. The first sign of red rust on the untreated sample was evident after 24 h.

The weight before salt spray test was 43.51 g and the sample were kept inside the salt spray chamber. After the salt spray test, the weight of the untreated sample has reduced to 43.32 g, indicating a weight loss of 0.19 g and shows the effect of corrosion. As shown in Figure 5, for 301 SS sample treated for 300 s, the initial weight before salt spray test was 43.23 g and the final weight after salt spray test for 240 h was 43.19 g, with a weight loss of 0.04 g while the weight loss for the treated 301 SS sample for 1200 s was 0.02 g (Figure 4)..



*Figure 4. Salt spray test results showing the cumulative weight losses for untreated and treated 301 SS samples, at the end of 240 h.*

The treated samples experienced a lower weight loss compared to the untreated one, denoting a lower corrosion rate.

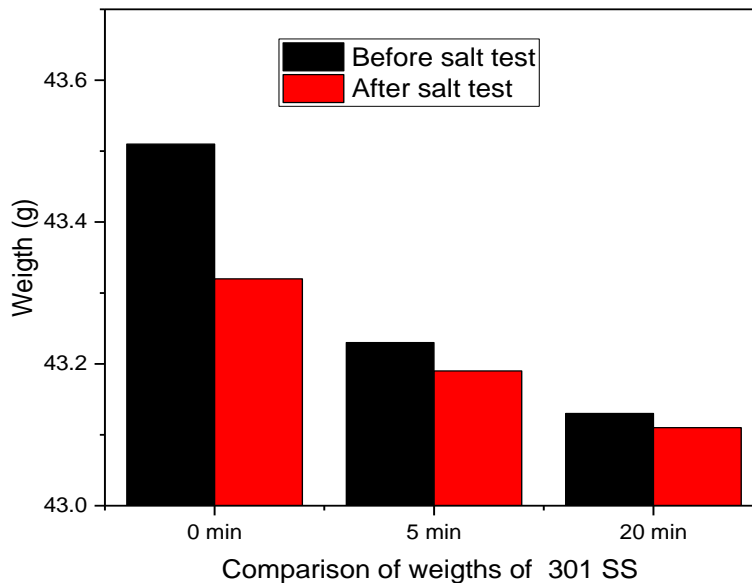


Figure 5. Comparison of weights of untreated and treated 301 SS samples before and after salt spray test, at the end of 240 h.

Table 3 summarizes the results of salt spray test results for untreated and treated 301 SS samples after different times of exposure. The salt spray test was performed for 240 h at 24 h interval. At 96 h, for the untreated sample, a point of red corrosion along the edge was spotted and abundant red corrosion was observed after 168 h up to 240 h of testing. For samples treated for 5 mins, points of red corrosion along the edge were observed after 96 h and red corrosion after 240 h of testing. Similar results were obtained when the samples are treated for 1200 s.

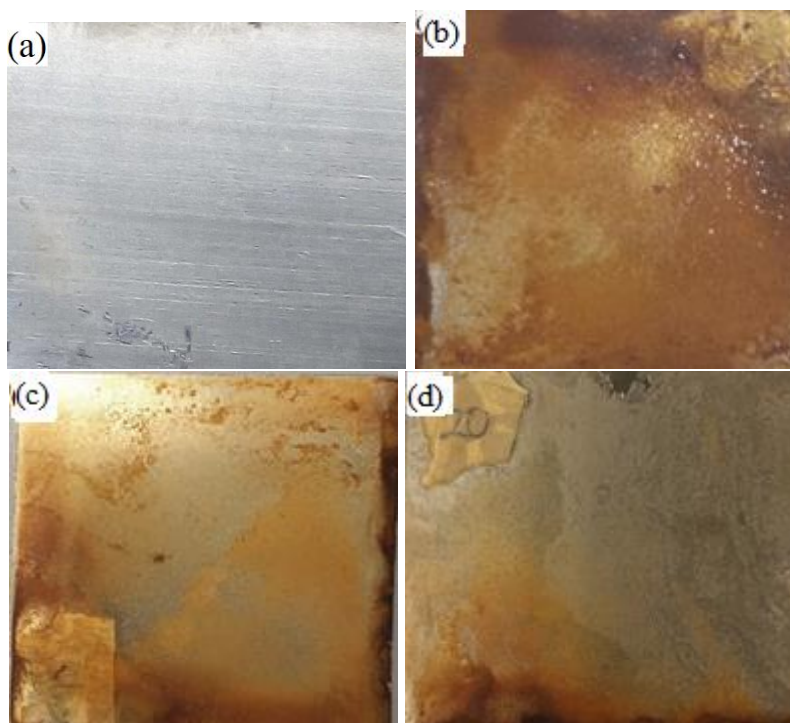
Table 3. Results of salt spray test for 301 SS after different times of exposure.

Treatment Time (min)	24	48	52	Time (h)	96	120	168	240
0	Subtle staining	Evident staining	No change	Point of red corrosion along the edge	Red corrosion	Abundant red corrosion	Abundant red corrosion	Abundant red corrosion
5	No change	Point of red corrosion along the edge	Points of red corrosion in the lower regions	Points of red corrosion along the edge	Red corrosion	Red corrosion	Red corrosion	Red corrosion
20	No change	Point of red corrosion along the edge	Points of red corrosion in the lower regions	Points of red corrosion	Red corrosion	Red corrosion	Red corrosion	Red corrosion

Figure 6 shows the surface condition of untreated and treated 304 SS samples before and after exposure to the salt spray test for 240 h. Compared to the untreated sample (Figure 6a), the treated samples (Figures 6b-d) experienced more corrosion attack.

The surface condition of untreated and treated 301 SS samples before and after exposure to the salt spray test for 240 h is shown in Figure 7. Compared to the treated samples, the untreated sample experienced more corrosion attack.

Several factors could influence the corrosion rate and consequently the weight loss after salt spray test including the extent of deformation, degree of exposure to heat treatment or corrosive environment [22-24].

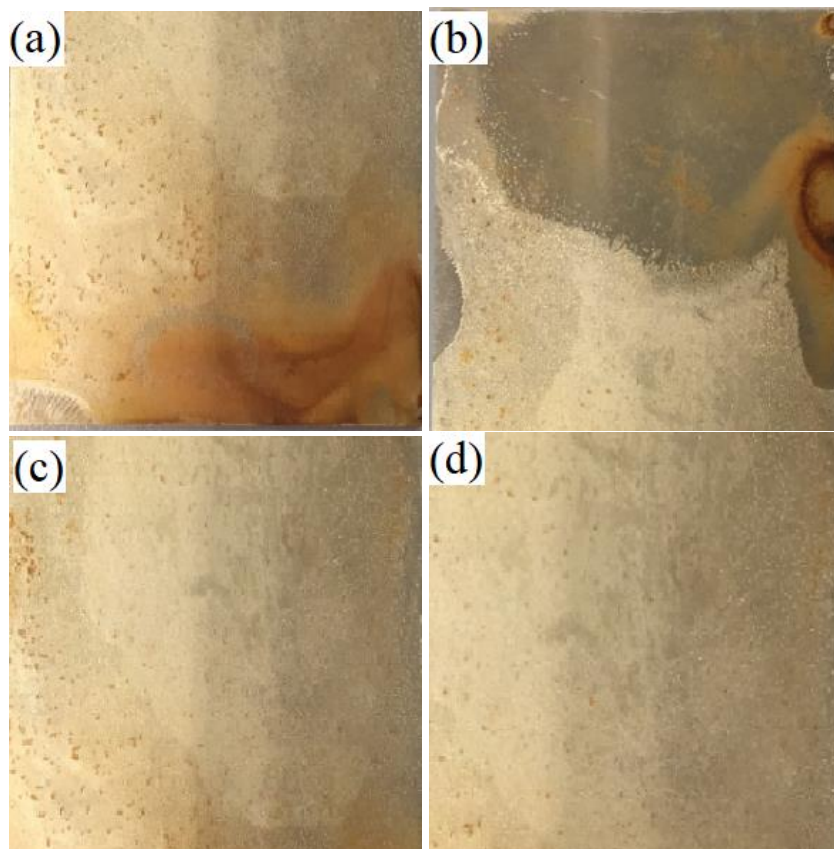


*Figure 6. Surface condition of 301 stainless steel samples after exposure to the salt spray test for 240 h; (a) untreated sample without salt test, (b) untreated sample with salt test, (c) treated sample for 5min with salt test, (d) treated sample for 20min with salt test.*

As obvious in Figure 7, the corrosion of test samples seems to be severer in the edges, which may indicate that the processing quality of sample edges has a significant effect on corrosion and might affect the weight loss result.

In addition, the dimension of test samples obviously would affect the corrosion area and consequently influence the weight loss results.





*Figure 7. Surface condition of 304 stainless steel samples after exposure to the salt spray test for 240 h; (a) Untreated sample without salt test (b) Untreated sample with salt test (c) treated sample for 5min with salt test, (d) treated sample for 20min with salt test.*

In the present study, an enhanced corrosion resistance of treated 301 and 304 stainless steels using salt spray corrosion test (5% concentration) was achieved (Figure 2, Figure 3, Figure 4, Figure 5, Table 2, Table 3). Similar improvement in the corrosion resistance was achieved when ceramic coatings (zirconia ceria powder and yttria-stabilized zirconia) were deposited on 304 steel. The samples with ceria powder coating exhibited more corrosion resistance than the yttria zirconia coating. If properly deposited in right proportions, coatings have the tendency of improving the fracture and corrosion resistance of materials [25-26]. In addition, Esfahani et al. [17] reported an increase in corrosion resistance of normal steel panel as compared with galvanized steel. In another study [16], the corrosion resistance of both AZ91D and AM50 alloys decreased with an increase in chloride concentration. After coatings/electrodeposition [27] and surface modifications by rolling [28], machining and moulding, mechanical treatment [29], an improvement in the electrochemical properties of 316 steel [30-33], 301 steel [4], 17-4PH steel [34], 304 steel [35], and mild steel was also reported. This was attributed to the passivation ability of the coating and nanostructured layers generated on the sample surfaces which protect the sample outer surface from corrosion especially in aggressive conditions. The surface-conditioned 301 and 304 SS with improved corrosion resistance will find applications in many manufacturing, aerospace and petroleum industries where excellent corrosion resistance over time is needed.



## 4. CONCLUSION

The effect of mechanical attrition treatment on corrosion resistance of 301 and 304 stainless steels (SS) was investigated in this work under salt spraying test in a controlled corrosive medium using 5 % concentration salt solution. The salt spray test and the corrosion rate of untreated and treated 301 and 304 stainless steel samples was determined by the weight loss after the salt spray test. The treatment method decreased the weight loss of 301 and 304 SS from 0.19 g to 0.02 g and from 0.15 g to 0.01 g after salt spraying test, respectively. In addition, the treated 304 SS samples showed the best result on exposure to salt spray, where the first spot of corrosion along the edge occurred at 240 h. Meanwhile, the untreated sample showed changes after 24 h of exposure. In addition, the first spot of red corrosion on the surface was noticed at 24 h and abundant red corrosion after 240 h for the untreated 301 SS sample.

Compared with the untreated 301 and 304 SS samples, the treated samples exhibited a lower weight loss, which denotes a low corrosion rate, hence an improved corrosion resistance.

## DISCLOSURE STATEMENT

The authors declare no conflict of interest.

## ACKNOWLEDGEMENTS

Many thanks to the members of Centre for Advanced Structural Materials (CASM), City University of Hong Kong, Hong Kong SAR as regards the surface treatment.

## REFERENCES

- [1] Zhang, Y., C. Yang, L. Zhao, and J. Zhang. 2021. Study on the Electrochemical Corrosion Behavior of 304 Stainless Steel in Chloride Ion Solutions. *International Journal of Electrochemical Science* 16:210251.
- [2] Tian, W, Du, S. Li, S. Chen, and Q. Wu. 2014. Metastable pitting corrosion of 304 stainless steel in 3.5% NaCl solution. *Corrosion Science* 85:372–379.
- [3] Pan, C., L. Liu, Y. Li, and F. Wang. 2013. Pitting corrosion of 304SS nanocrystalline thin film. *Corros. Sci.* 73:2–43.
- [4] Olugbade, T., C. Liu, and J. Lu. 2019. Enhanced passivation layer by Cr diffusion of 301 Stainless Steel facilitated by SMAT. *Adv. Eng. Mater.* 21: 1900125.
- [5] Anderson, A. 2017. Corrosion resistance of ceramic coatings sprayed on stainless steel substrates. *International Journal of Ambient Energy.* 38(3):320-322.
- [6] Bao, Z.B., Q.M. Wang, W.Z. Li, J. Gong, T.Y. Xiong, and C. Sun. 2008. Corrosion Behaviour of AIP NiCoCrAlYSiB Coating in Salt Spray Tests. *Corrosion Science* 50: 847–855.
- [7] Rogerio, M.A., S.O. Rogero, M. Terada, E.F. Pieretti, and I. Costa. 2014. Localized Corrosion Resistance and Cytotoxicity Evaluation of Ferritic Stainless Steels for Use in Implantable Dental Devices with Magnetic Connections. *Int. J. Electrochem. Sci.* 9:1340 – 1354
- [8] Islak, S., S. Buytoz. 2011. Microstructure Properties of ZrO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub>-% 13TiO<sub>2</sub> Composite Coating Produced with Plasma Spray Method on AISI 304 Stainless Steel. 6th International Advanced Technologies Symposium (IATS'11), Elazığ; May 16–18.
- [9] Zhang, J., A. Kobayashi. 2005. Corrosion Resistance of Al<sub>2</sub>O<sub>3</sub> + ZrO<sub>2</sub> Composite Coatings Sprayed on Stainless Steel Substrates. *Transactions of JWRI.* 34 (2):17–22.

- [10] Mert, S, S. Mert, and S. Saridemir S. 2018. An investigation of  $\text{Al}_2\text{O}_3\text{-ZrO}_2$  ceramic composite-coated engine parts using plasma spray method on a diesel engine. *International Journal of Ambient Energy*. DOI: 10.1080/01430750.2018.1501748.2014; 9: 1340–1354.
- [11] Liu, Z.Z., Y. Chu, Y. Dong, X. Yang, X. Chen, Y.D. Kong. 2013. The Effect of Metallic Bonding Layer on the Corrosion Behavior of Plasma Sprayed  $\text{Al}_2\text{O}_3$  Ceramic Coatings in Simulated Seawater. *Vacuum* 101:6–9.
- [12] Sathish, S., M. Geetha. 2015. Comparative Study on Corrosion Behavior of Plasma Sprayed  $\text{Al}_2\text{O}_3$ ,  $\text{ZrO}_2$ ,  $\text{Al}_2\text{O}_3/\text{ZrO}_2$  and  $\text{ZrO}_2/\text{Al}_2\text{O}_3$  Coatings. *Transactions of Nonferrous Metals Society of China* 26:1336–1344.
- [13] Limarga, A.M., W. Sujanto, H.Y. Tick. 2005. Mechanical Properties and Oxidation Resistance of Plasma-Sprayed Multilayered  $\text{Al}_2\text{O}_3/\text{ZrO}_2$  Thermal Barrier Coatings. *Surface and Coatings Technology*. 197:93–102.
- [14] Singh, V.P., A. Sil, and R. Jayaganthan. 2011. A Study on Sliding and Erosive Wear Behaviour of Atmospheric Plasma Sprayed Conventional and Nanostructured Alumina Coatings. *Materials and Design* 32:584–591.
- [15] Wang, Y., S. Jiang, M. Wang, S. Wang, T.D. Xiao, P.R. Strutt. 1999. Abrasive Wear Characteristics of Plasma Sprayed Nanostructured Alumina/Titania Coatings. *Wear*. 237: 176–185.
- [16] Mohedano, M., R. Arrabal, A. Pardo, M.C. Merino, K. Paucar, P. Casajús, E. Matykina. 2014. Salt spray corrosion behaviour of new Mg–Al alloys containing Nd or Gd. *Corrosion Engineering, Science and Technology*. 48(3):183-193.
- [17] Esfahani, S.L., Z. Ranjbar, and S. Rastegar. 2016. Comparison of corrosion protection of normal and galvanized steel coated by cathodic electrocoatings using EIS and salt spray tests. *Corrosion Engineering, Science and Technology* 51(2): 82-89.
- [18] Loveridge, M.J., H.N. McMurray, and D.A. 2006. Worsley. Chrome free pigments for corrosion protection in coil coated galvanized steels. *Corrosion Engineering, Science and Technology* 41(3):240-248.
- [19] Lu, K., and J. Lu. 1999. Surface Nanocrystallization (SNC) of Metallic Materials-Presentation of the Concept behind a New Approach. *Journal of Material Science Technology*. 15:193–197.
- [20] Lu, K., and J. Lu. 2004. Nanostructured surface layer on metallic materials induced by surface mechanical attrition treatment. *Mater. Sci. Eng., A*. 375–377: 38–45.
- [21] ASTM B117-16. 2016. Standard Practice for Operating Salt Spray (Fog) Apparatus. ASTM International. West Conshohocken, PA.
- [22] Abioye, T. E., T. O. Olugbade, and T. I. Ogedengbe. 2017. Welding of dissimilar metals using gas metal arc and laser welding techniques: a review. *Journal of Emerging Trends in Engineering and Applied Sciences (JETEAS)* 8: 225-228.
- [23] Abioye, T. E., I. S. Omotehinse, I.O. Oladele, T. O. Olugbade, and T. I. Ogedengbe. 2020. Effects of post-weld heat treatments on the microstructure, mechanical and corrosion properties of gas metal arc welded 304 stainless steel. *World Journal of Engineering* 17(1): 87–96.
- [24] Mohammed, T., T.O. Olugbade, and I Nwankwo. 2016. Determination of the effect of oil exploration on galvanized steel in Niger Delta, Nigeria, *Journal of Scientific Research and Reports* 10:1-9.
- [25] Olugbade, T.O., Ojo, O.T., Omiyale, B.O., Olutomilola, E.O., and Olorunfemi, B.J. (2021). A review on the corrosion fatigue strength of surface-modified stainless steels. *Journal of the Brazilian Society of Mechanical Sciences and Engineering* 43:421.
- [26] Olugbade, T.O. 2020. Stress corrosion cracking and precipitation strengthening mechanism in TWIP steels: progress and prospects. *Corrosion Reviews* 38: 473-488.
- [27] Olugbade, T.O., T.E. Abioye, P.K. Farayibi, N.G. Olaiya, B.O. Omiyale, T.I. Ogedengbe. 2021. Electrochemical properties of MgZnCa-based thin film metallic glasses fabricated via magnetron sputtering deposition coated on a stainless steel substrate. *Anal. Lett.* 54(10): 1588-1602

- [28] Olugbade, T.O. 2020. Electrochemical characterization of the corrosion of mild steel in saline following mechanical deformation. *Anal. Lett.* 54: 1055 - 1067.
- [29] Olugbade, T., and J. Lu. 2020. Improving the passivity and corrosion behaviour of mechanically surface-treated 301 stainless steel. *International Conference on Nanostructured Materials (NANO 2020)*, 117, Australia.
- [30] ] Olugbade, T., and J. Lu. 2019. Enhanced corrosion properties of nanostructured 316 stainless steel in 0.6 M NaCl solution. *J Bio Tribo Corros.* 5: 38.
- [31] Olugbade, T., and J Lu. 2018. Effects of materials modification on the mechanical and corrosion properties of AISI 316 stainless steel. *Twelfth international conference on fatigue damage of structural materials, Cape Cod, Hyannis, USA.*
- [32] Olugbade, T. 2019. Datasets on the corrosion behaviour of nanostructured AISI 316 stainless steel treated by SMAT. *Data-in-brief* 25: 104033.
- [33] Hao, Y., B. Deng, C. Zhong, Y. Jiang, and J. Li. 2009. Effect of surface mechanical attrition treatment on corrosion behavior of 316 Stainless Steel. *Journal of Iron and Steel Research Intl.* 16:68-72.
- [34] Olugbade, T.O., and J. Lu. 2019. Characterization of the corrosion of nanostructured 17-4 PH stainless steel by surface mechanical attrition treatment (SMAT). *Anal. Lett.* 52: 2454-2471.
- [35] Lu, J.Z., H. Qi, K.Y. Luo, M. Luo, X.N. Cheng. 2014. Corrosion behaviour of AISI 304 stainless steel subjected to massive laser shock peening impacts with different pulse energies. *Corrosion Science* 80: 53-59.