Taguchi analysis of single layer CrN coatings on AISI 304 Stainless Steel to study its erosive corrosive wear behaviour

Alok Vats

M. Tech, Production Engineering, MNIT Jaipur, Pass out 2016
Address: Plot No. 761 Mundka, New Delhi- 110041 (Near Swati Modern Public School)

***Abstract*— *The purpose of present study was to investigate the erosive corrosive wear behavior of single layer (CrN) coatings on AISI 304 Stainless Steel samples with varying coating thickness (0-200 nm) in the range of 50 nm. The slurry jet erosive test was conducted on Slurry Jet Erosion Tester in saline slurry (3.5wt% salt) under the different working conditions with varying impact velocity (10-25 m/s), impingement angle (30°-75°) and erodent discharge (160-280 gm/min). Taguchi analysis was applied to find optimum parameters for the minimization of erosion rate of various coated and uncoated samples. The results of Taguchi experiments also indicated that among all the factors, impact velocity became least significant when samples were coated with CrN whereas it was most significant for uncoated samples. Coating thickness was the second most significant factor in the case of CrN coated samples. PVD- CrN coatings reduced the wear rate by nearly 2 times.*

Keywords*— *Erosive wear, corrosive wear, single layer coating, Taguchi Orthogonal Array.

I. INTRODUCTION

Erosion–corrosion is the increase in the rate of degradation of the material caused by the combined action of electrochemical corrosion and mechanical wear processes. Corrosion is a material degradation process which occurs due to electrochemical action, while erosion is a mechanical wear process. When these two processes act together especially in the marine environments, it is known as erosion-corrosion. It is one of the major causes of failure in the nuclear power plants, chemical, petrochemical industries and marine environments where combined effect of erosion and corrosion phenomena occurs. In the marine field, this study applies to ship and boats. The study is equally beneficial in those areas where the underground salty water is supplied for household purposes, and the same has to be delivered to the overhead tank.

In the liquid- particle flow, the sand particles get impinged on steel surface which remove a layer of protective film from its surface. The chloride ions act rapidly on the exposed surface having discontinuity. In this way pure erosion and erosion enhanced corrosion are the dominant mechanisms that degrade the metal surface in erosion corrosion [1-6]. In order to improve the physical, mechanical and surface wear of metal such as steel, surface treatment such as modification of surface using silane, electroplating, Nitriding and coating etc are used [7-13]. A majority of surface modification lead to improved corrosive characteristics of the steel. Hence, they are also called corrosion inhibitors and are used on variety of steels. Neville et al. [7] studied erosion–corrosion of engineering steels by the use of chemicals on X65 pipeline steel, 13Cr martensitic stainless steel and super-duplex stainless steels. They concluded that inhibitor has a greater effect on the corrosion component of carbon steel but offers no protection on super-duplex stainless steel under the conditions tested. In another research, Hu et al.[14] assessed the effect of corrosion inhibitor on erosion–corrosion of API-5L-X65 stainless steel in multi-phase jet impingement conditions and found that corrosion inhibitor provides up to 20% protection in erosion–corrosion conditions and that oil phase reduces the erosion component by reducing the particle velocity in the flow conditions. Yao et al. [15] investigated a new method for protecting bends from erosion in gas-particle flows and reported that adding ribs on the outer-wall of the inside bend can significantly improve bend’s erosion protection ability. Surface coating technology has also been deployed to enhance the erosive corrosive characteristics of the metals. Multilayer coatings offer protection against synergistic effects on bare stainless steel surfaces.

Corrosion resistance can also be increased by providing an interlayer of a suitable material [16]. Coatings of CrN and CrCN have been commonly used in industry and research to improve surface properties of materials. However, CrCN coatings provide better corrosion resistance than CrN coated samples due to its superior mechanical properties [17]. Shan et al. [18] fabricated

CrN coatings on 316L stainless steel substrate by multi-arc ion plating system, performed Polarization tests and concluded that the multilayer structure could limit the crack propagation only to the layer and reduce cracks in the coating. However, there are various other factors besides the coating thickness that affect the erosive corrosive performance under practical conditions. These are impact velocity, impingement angle and erodent discharge respectively. The erosive corrosive performance of the material under a given combination of these factors has not been fully understood.

Hence, the aim of this work was to fabricate single layer coated AISI 304 stainless steel material, study the effect of various parameter such as coating thickness, impact velocity, erodent discharge and impingement angle on the erosive and corrosive behavior of fabricated material and finally to find different levels of factors to achieve a condition of minimum wear rate using **Taguchi's approach**.

II. EXPERIMENTAL PROCEDURE

2.1 Materials

The proposed work consists of deposition of mono-layered PVD coatings of CrN of varying thickness on AISI 304 stainless steel. AISI 304 stainless steel was purchased from Bahubali Steels, New Delhi. While Chromium powder was procured from M/s Indian Diamond Tools and M/s Scientific Instruments, Jaipur, India.

2.2 Sample Preparation and Coating Process

The samples for coating were prepared as per test standard specification with the help of a diamond cutter. Then the uncoated samples were mirror-polished using a MetaServe 250 Grinder Polisher (Buhler machine, Illinois, USA). The fine polish provides the improvement of adherence between coating and the samples. Before coating, samples were cleaned in acetone and then dried in a pre-vacuum dryer. The pellets used for coating were prepared with the help of 99.5% pure Cr powder procured from M/s Indian Diamond Tools and M/s Scientific Instruments, Jaipur. The pellet were pressed using a KBr press at a pressure of 15 tonnes.

A thermal Vapor deposition process (inside a high temperature Vacuum Box Coater -Model BC-300, Hind High Vacuum Co. (P) Ltd. Bangalore, India) (Figure.1) was used to fabricate single layer (chromium nitride) coated AISI 304 Stainless Steel with varying coatings thickness (50nm, 100nm, 150nm and 200nm). In this coating process, a sample of AISI 304 Stainless Steel was first positioned in the sample holder. Then a tungsten filament was used to hold the pellet of pure chromium. A high vacuum of 10^{-3} bar was created inside the chamber and current (50-75 A, 12 V) was supplied to the tungsten filament to evaporate pure chromium. Then, nitrogen gas was allowed to flow in the chamber which reacted with the evaporated chromium and a layer of chromium nitride was deposited on the surface of the AISI 304 Stainless Steel. Coating thickness was maintained as 50nm, 100nm, 150nm and 200nm. Four different coatings were developed. Parameters for CrN coating were taken as coating rate- 0.1 Å/sec, coating thickness-200nm, Vacuum Chamber temperature-2000°C, Vacuum Pressure- $1.2\text{x}10^{-6}$ bar, Nitrogen flow rate- 50cc/min, Voltage-12V, Current-39 Ampere.

2.3 Sample characterization

2.3.1 Slurry Jet Erosion Test

The slurry erosion behavior of single layer CrN coated and uncoated samples was evaluated using a Slurry Jet Erosion Tester (Model TR411, Ducom, India) (Figure. 2). For this test, slurry was prepared by mixing sand particles and water and taken in the hopper. Square samples of dimensions 25 × 25 mm was placed in the sample holder. Samples were eroded by flow of erodent discharge through a nozzle of 4 mm diameter. Impingement angles were changed by adjusting the sample holder at different angles (30°, 45°, 60° and 75°). The strike was applied for 10 min. The weight loss was determined by the weighing the sample before and after the slurry erosion test. The slurry erosion rate was calculated by dividing weight loss of samples with the time taken to perform an experiment i.e. 10min.

2.3.2 Design of Experiment (DOE)

Taguchi orthogonal Design of Experiment is one of the most important and highly beneficial statistical tools and is employed to examine the effect of more than one variable and their interactions on the output of the process. It involves a large number of steps in series which follow a definite sequence for the test conducted to provide an improved solution. S/N ratio is the main quantity that has to be calculated in DOE to obtain an improved solution. The S/N ratio is calculated using Eq. (1) as below:

$$S/N = 10\, log \left[\frac{1}{n}\sum_{i=1}^{n} y_i^2\right] \qquad \text{Eq. (1)}$$

Where, n is the number of tests and y_i is the value of experimental result of the i^{th} test. The characteristics of S/N ratio have been categorized into three classes, i.e. smaller-the-better, larger-the-better, and nominal-the-better. For obtaining optimal and improved performance, smaller-the-better characteristic has been selected for erosion wear. Table 1 indicates the factors selected for experimental analysis using Taguchi method. The array

https://dx.doi.org/10.24001/ijaems.3.5.10 ISSN: 2454-1311

chosen in this study was L_{16} orthogonal array design which has 16 rows corresponding to the number of variables selected.

Table.1: Levels of the variables used in the experiment

Control factors	Levels				
	1	2	3	4	Units
Coating thickness (A)	50	100	150	200	nm
Impact velocity (B)	10	15	20	25	m/s
Impingement angle (C)	30	45	60	75	degree
Erodent discharge (D)	160	200	240	280	gm/min

III. RESULTS AND DISCUSSION

3.1 Wear Characteristic analysis by Taguchi method

3.1.1 For single layer (CrN) coatings of various thicknesses (50nm, 100nm, 150nm and 200nm) on AISI 304 Stainless Steel

The experiments were performed as per Taguchi Orthogonal L_{16} array. In Table 2, seventh column represents S/N ratio of the volumetric wear rate of the coated samples. The overall mean for the S/N ratio of the specific wear rate was found to be 56.097 db for the coated samples. The analysis was performed using the software MINITAB 17 to analyze the wear rate of the coated samples. Table 4 indicated that that among various factors, erodent discharge is the most significant factor followed by coating thickness and impingement angle. Impact velocity has the least significance on erosion rate of the CrN coated 304 steel.

On the other hand, Table 3 revealed that uncoated 304 steel indicated more wear than the coated steel. The mean wear rate in Table 3 (0.0033103gm/min) was more than that in Table 2 (0.001656 gm/min). It means that PVD-CrN coating reduced the wear rate by nearly 2 times. The improvement in wear resistance due to PVD coating on AISI 304 stainless steel was also observed by Recco et al. [13]. They concluded that PVD-TiN coating reduced the wear rate by 20 times. Analysis of the results from Figure 3 leads to the conclusion that combination of factors A4, B4, C2 and D2 gives minimum erosion rate. Hence, the optimum parameter for the minimum wear rate was at coating thickness 200nm, maximum impact velocity 25m/sec, and impingement angle of 45° and erodent discharge of 280 gm/min.

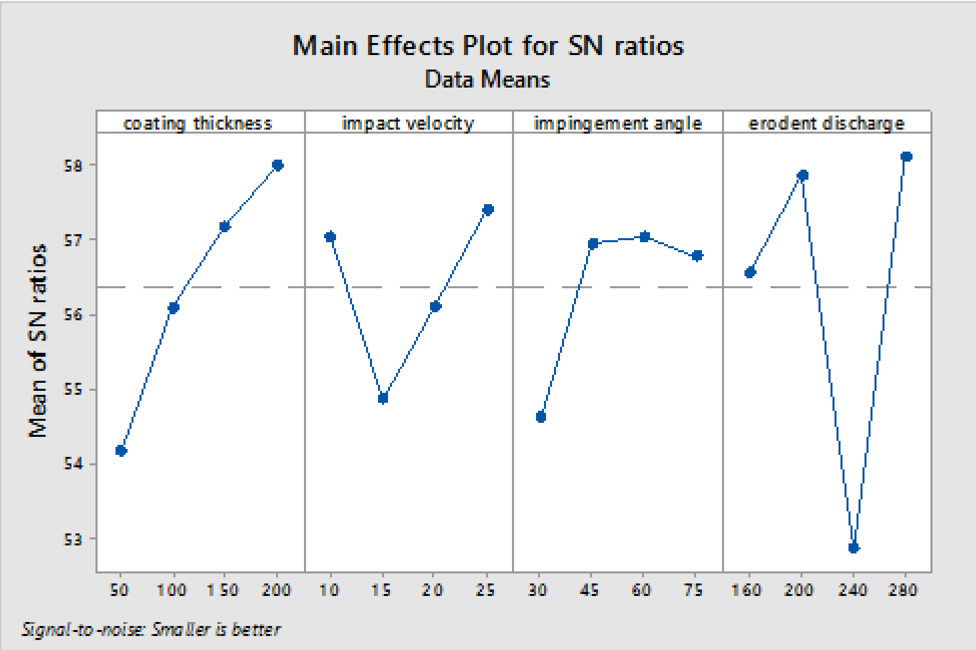

Fig.3: Influence of control factors on the Erosion rate of single layer (CrN) coatings of various thicknesses (50nm, 100nm, 150nm and 200nm) on AISI 304 Stainless Steel

3.1.2 For uncoated 304 Stainless Steel

The experimental results for uncoated 304 Stainless Steel were also analyzed by Taguchi method. The following results were obtained in which the overall mean for S/N ratio of erosion rate is 50.263. The erosion rate and S/N ratio responses were presented in Table 3 from which it can be concluded that impact velocity was the most significant factor followed by impingement angle. However, erodent discharge has indicated the least significance on erosion rate of the uncoated 304 steel. Further, Figure 4 concluded that combination of factors B4, C4 and D2 gives minimum erosion rate. Hence, optimum parameters for the minimum erosion rate of uncoated SS-304 were found to be Impact velocity of 25m/sec, impingement angle of 45° and erodent discharge of 240gm/min.

https://dx.doi.org/10.24001/ijaems.3.5.10 ISSN: 2454-1311

Table.2: Experimental design using L_{16} orthogonal array for CrN coated 304 steel

S. No.	Coating thickness (nm)	Impact velocity (m/sec)	Impingement angle (degrees)	Erodent discharge (gm/min)	Erosion rate (gm/min)	S/N Ratio (db)
1	50	10	30	160	0.0024	52.3958
2	50	15	45	200	0.0017	55.3910
3	50	20	60	240	0.0030	50.4576
4	50	25	75	280	0.0016	55.9176
5	100	10	45	240	0.0018	54.8945
6	100	15	30	280	0.0020	53.9794
7	100	20	75	160	0.0014	57.0774
8	100	25	60	200	0.0012	58.4164
9	150	10	60	280	0.0011	59.1721
10	150	15	75	240	0.0026	51.7005
11	150	20	30	200	0.0013	57.7211
12	150	25	45	160	0.0012	58.4164
13	200	10	75	200	0.0010	60.0000
14	200	15	60	160	0.0012	58.4164
15	200	20	45	280	0.0011	59.1721
16	200	25	30	240	0.0019	54.4249
Mean wear rate					0.001656	50.097

Table.3: Experimental design using L_{16} orthogonal array for AISI 304 steel

S. No.	Impact velocity (m/sec)	Impingement angle (degrees)	Erodent discharge (gm/min)	Erosion rate (gm/min)	S/N Ratio (db)
1	10	30	160	0.00400	47.9588
2	10	45	200	0.00360	48.8739
3	10	60	240	0.00370	48.6360
4	10	75	280	0.00380	48.4043
5	15	30	200	0.00340	49.3704
6	15	45	160	0.00310	50.1728
7	15	60	280	0.00330	49.6297
8	15	75	240	0.00290	50.7520
9	20	30	240	0.00280	51.0568
10	20	45	280	0.00310	50.1728
11	20	60	160	0.00260	51.7005
12	20	75	200	0.00270	51.3727
13	25	30	280	0.00300	50.4576
14	25	45	240	0.00275	51.2133
15	25	60	200	0.00240	52.3958
16	25	75	160	0.00250	52.0412
Mean wear rate				0.0033103	50.2630

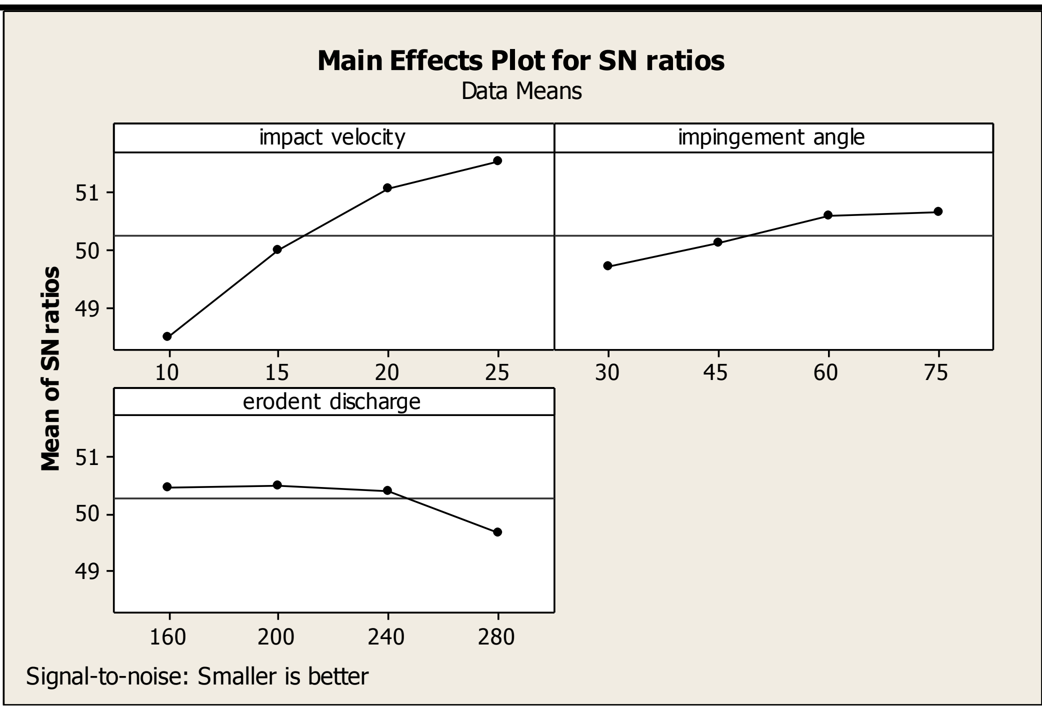

Fig.4: Effect of control factors on the Erosion rate uncoated AISI 304 Stainless Steel

3.2 ANOVA Analysis

Analysis of variance (ANOVA) was used to investigate the influence of various input parameters like impact velocity, impingement angle and erodent discharge on the basis of experimental results. By ANOVA analysis we determined the percentage contribution of that particular variable and its dominance over other factors. Table 4 presented ANOVA results for erosion rate for single layer (CrN) coatings of various thicknesses (50nm, 100nm, 150nm and 200nm) on AISI 304 Stainless Steel the erodent discharge (46.14 %), coating thickness (33.13 %), impact velocity (7.05 %), and impingement angle (9.95 %) have an influence on the saline slurry erosion rate. Out of these factors, erodent discharge indicated the most significant factor for the saline slurry erosion wear and impact velocity indicated as the least significant factor in the case of slurry erosion rate for single layer CrN coatings of various thicknesses (50nm, 100nm, 150nm and 200nm) on AISI 304 Stainless Steel. On the other hand, Table 5 indicated ANOVA analysis for uncoated SS304 steel alloy having 3 factors, 4 levels and interaction of those factors. It can be revealed that the impact velocity (81.22 %), impingement angle (8.4764 %) and erodent discharge (7.01%) have an influence on the saline slurry erosion rate. Out of these factors, impact velocity was considered as the most significant factor for the saline slurry erosion of the uncoated 304 steel samples and erodent discharge is the least significant factor in the case of slurry erosion rate for uncoated 304 steel. This result was in agreement with Gautam et al. [19] who obtained impact velocity (79.85%) as the most significant factor in case of uncoated granite powder reinforced composites. Therefore, from both the Table 4 and 5 it can be seen that for uncoated sample impact velocity has most significant effect on erosion rate whereas it has least significant effect for CrN coated AISI 304 Stainless Steel. Hence, it can be concluded that for the application where velocity of slurry and water is high, thicker the single layer coating of CrN on AISI 304 Stainless Steel the more effective it will be.

Table.4: ANOVA table for Erosion rate of single layer (CrN) coatings of various thicknesses (50nm, 100nm, 150nm and 200nm) on AISI 304 Stainless Steel

Source	DF	AdjSS	Adj MS	F-Value	P-Value
coating thickness	3	42.399	14.133	8.91	0.053
impact velocity	3	9.023	3.008	1.90	0.306
impingement angle	3	12.742	4.247	2.68	0.220
erodent discharge	3	59.048	19.683	12.41	0.034
Error	3	4.760	1.587		
Total	15	127.972			

https://dx.doi.org/10.24001/ijaems.3.5.10 ISSN: 2454-1311

Table.5: ANOVA table for uncoated AISI 304 stainless steel

Source	DF	Seq SS	Adj SS	Adj MS	F	P
impact velocity	3	22.2345	22.2345	7.4115	49.28	0.000
impingement angle	3	2.3204	2.3204	0.7735	5.14	0.043
erodent discharge	3	1.9165	1.9165	0.6388	4.25	0.063
Error	6	0.9025	0.9025	0.1504		
Total	15	27.3738				

IV. CONCLUSIONS

The following conclusions can be drawn on the basis of erosive corrosive wear of the single layer (CrN) coatings of various thicknesses (50nm, 100nm, 150nm and 200nm) on AISI 304 Stainless Steel and uncoated 304 steel by setting the various parameters such as coating thickness, impact velocity, impingement angle and erodent discharge at significant levels:

1. Impact velocity was the most significant factor for uncoated AISI 304 steel sample whereas it indicated least significance for single layer CrN coated AISI 304 steel samples. However, for coated material, erodent discharge was the most significant factor, followed by coating thickness in the erosive corrosive wear of the CrN coated samples.
2. Analysis of the results for CrN coated 304 steel samples leads to the conclusion that combination of factors A4, B4, C2 and D2 gives minimum erosion rate. Hence, the optimum parameter for the minimum wear rate was at coating thickness 200nm, maximum impact velocity 25m/sec, and impingement angle of 45° and erodent discharge of 280 gm/min.
3. Combination of factors B4, C4 and D2 gives minimum erosion rate in case of uncoated steel. Hence, optimum parameters for the minimum erosion rate of uncoated SS-304 were found to be Impact velocity of 25m/sec, impingement angle of 45° and erodent discharge of 240gm/min. Erodent discharge has indicated the least significance on erosion rate of the uncoated 304 steel
4. Results also revealed that uncoated 304 steel indicated more wear than the coated steel. The mean wear rate in uncoated 304 steel (0.0033103gm/min) was more than that in CrN coated 304 steel (0.001656 gm/min). It means that PVD- CrN coating reduced the wear rate by nearly 2 times. Hence, for the application having high flow of water coated samples should be preferred over uncoated samples.

Fig.2: Test setup of Slurry Jet Erosion Tester

Fig.1: Thermal/E-beam PVD Coating Unit

REFERENCES

[1] Zhang, G. A., Xu, L. Y., Cheng, Y. F. (2009) Investigation of erosion–corrosion of 3003 aluminum alloy in ethylene glycol–water solution by impingement jet system, Corros. Sci. 51, pp 283–290.

[2] Shrestha, S., Hodgkiess, T., Neville, A. (2005) Erosion-corrosion behavior of high-velocity oxy-fuel Ni—Cr—Mo—Si—B coatings under high-velocity seawater jet impingement, Wear 259, pp 208–218

[3] Saha, G. C., Khan, T. I. G. A. Zhang, (2011), Erosion–corrosion resistance of microcrystalline and near-nanocrystalline WC–17Co high velocity oxy-fuel thermal spray coatings, Corros. Sci. 53, pp 2106–2114.

[4] Hong, S., Wu, Y., Zhang, J., Zheng, Y., Qin, Y., Lin, J. (2015), Effect of ultrasonic cavitation erosion on corrosion behavior of high-velocity oxygen-fuel (HVOF) sprayed near-nanostructured WC-10Co-4Cr coating, Ultrason. Sonochem. 27, pp 374–378.

[5] Zhao, Y., Zhou, F., Yao, J., Dong, S., Li, N., (2015), Erosion–corrosion behaviour and corrosion resistance of AISI 316 stainless steel in flow jet impingement, Wear 328-329, pp 464–474.

[6] Lin, C. K., Hsu, C. H., Cheng, Y. H., Liang, O. K., Lee, S. L. (2015), A study on the corrosion and erosion behavior of electroless nickel and TiAlN/ZrN duplex coatings on ductile iron, Appl. Surf. Sci. 324, pp 13–19.

[7] Neville, A., Wang, C. (2009), Erosion–corrosion of engineering steels—Can it be managed by use of chemicals, Wear 267, pp 2018–2026.

[8] Pantoja, M., Abenojar, J., Martínez, M. A., Velasco, F. (2016), Silane pretreatment of electrogalvanized steels: Effect on adhesive properties, International Journal of Adhesion and Adhesives 65, pp 54-62.

[9] Pantoja, M., Martínez, M. A., Abenojar, J., Encinas, N., Ballesteros, Y. (2011), Effect of EtOH/H2O ratio and pH on bis-sulfur silane solutions for electrogalvanized steel joints based on anaerobic adhesives, The Journal of Adhesion 87 (7-8), pp 688-708.

[10] Martinez, M. A., Calabrés, R., Abenojar, J., Velasco, F., (2008), Sintered High Carbon Steels: Effect of Thermomechanical Treatments on their Mechanical and Wear Performance, Materials Science Forum 591, pp 271-276.

[11] Pantoja, M., Martínez, M. A., Abenojar, J., Velasco, F., Real, J. C. (2010), Structural and mechanical characterization of γ-methacryloxypropyltrimethoxysilane (MPS) on Zn-electrocoated steel Journal of Adhesion Science and Technology 24 (11-12), pp 1885-1901.

[12] Pantoja, M., Velasco, F., Broekema, D., Abenojar, J., Real, J. C. (2011), The influence of pH on the hydrolysis process of γ-methacryloxypropyltrimethoxysilane, analyzed by FT-IR, and the silanization of electrogalvanized

steel, Journal of Adhesion Science and Technology 24(6), pp 1131-1143.

[13] Recco, A. A., Lopez, C. D., Bevilacqua, A. F., da Silva, F., Tschiptschin, A. P. (2007),

[14] Improvement of the slurry erosion resistance of an austenitic stainless steel with combinations of surface treatments: Nitriding and TiN coating, Surf. Coat. Tech. 202, pp 993–997.

[15] Hu, X., Alzawai, K., Gnanavelu, A., Neville, A., Wang, C., Crossland, A., Martin, J. (2011), Assessing the effect of corrosion inhibitor on erosion–corrosion of API-5L-X65 in multi-phase jet impingement conditions, Wear 271, pp 1432– 1437.

[16] Yao, J., Zhang, B. Z., Fan, J. R. (2000), Investigation of a new protection method for protecting tube from erosion in gas-particle flows, J. Therm. Sci. 9(2), pp 158-162.

[17] Wood, R. J. K. (2007) Tribo-corrosion of coatings: a review, J. Phys. D. Appl. Phys. 40(18), pp 5502-5521.

[18] Ye, Y., Wang, Y., Chen, H., Li, J., Yao, Y. (2015) Doping carbon to improve the tribological performance of CrN coatings in seawater, Tribol. Int. 90, pp 362–371.

[19] Shan, L., Wang, Y., Li, J., Jiang, X., Chen, J. (2015) Improving tribological performance of CrN coatings in seawater by structure design, Tribol. Int. 8278–88.

[20] Gautam, V., Patnaik, A., Bhat, I. K. (2016) Microstructure and Wear Behavior of Single layer (CrN) and Multilayered (SiN/CrN) Coatings on Particulate Filled Aluminum Alloy Composites, Silicon DOI 10.1007/s12633-015-9357-9.