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EFFECTS OF AGING TREATMENT ON INCONEL SPECIMEN 600, 625, 718 COATED WITH NICKEL CHROMIUM CARBIDE (CR_3C_2-NICR) USING SALT AND ACIDIC CORROSION METHOD

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Abstract

When the material interacts with its surroundings, the deterioration of the material's important properties is referred to as corrosion. Substances used as construction material processing equipment, including the product from it, called materials. These materials can be metals and their alloys, composites, polymers, ceramics, etc., Corrosion is commonly known as rust. Generally, Iron and its alloys are the most commonly used materials opted to design different structural component, and thus are largely affected by corrosion in this aspect understanding of corrosion study plays a very vital role. Our work includes the use of the thermal spray by D-gun method on three different Inconel materials 600, 625 and 718 are coated with nickel chromium & average thicknesses of 61.34µm, 67.97µm and 60.84µm obtained. The nickel chromium coated materials then undergo acid corrosion and salt corrosion tests. In Acid Corrosion test, The Inconel coated substrates were subjected to laboratory corrosion tests utilizing glass flask and chemicals of the reagent grade. The specimen has a dimension of (15*20*6) mm. The three Inconel specimens 600, 625, 718 are soaked in three different beakers containing a capacity of 60ml HCl concentrated for 72 hours. For three different Inconel specimens 600, 625, 718 using Salt spray test rig Salt corrosion test is conducted with temperature range 35°C to 65°C and corrosion rate is calculated. Finally, after the acid corrosion test on three Inconel 625 shows a weight reduction of 0.28% and in salt Inconel 625 shows the average corrosion rate of 38.5Mpy. Hence it is concluded that Inconel 625 material has maximum coating thickness as compare to Inconel 600 & 718 coated material therefore Inconel 625 exhibited a promising physical and structural properties & also studied the surface morphology of coated Inconel substrates by Atomic force microscopy (AFM) from this it should be noted that in some areas of materials, there were indications of the existence of local crystalline structures. Cracking is clearly evident at the surface.

Keywords: Salt spray corrosion, Inconel specimen 600,625 and 718, NaCl, HCl, Surface roughness.

1. INTRODUCTION

Thermal corrosion Cycling is an innovative and cost effective process of enhancing the mechanical properties of many materials commonly used in commercial and industrial technologies. Thermal Cycling has been determined to significantly increase the corrosion properties of many ferrous alloys. The salt spray test is a standardized test method used to check corrosion resistance. Salt spray testing is an accelerated corrosion test that produces a corrosive attack to the tested samples. The appearance of corrosion products (oxides) is evaluated after a period of time. Test duration depends on the corrosion resistance of the tested material. Salt spray testing is popular because it is well standardized and reasonably repeatable.

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The correlation between the duration in salt spray test and the expected life of a material is not necessary simple to interpret as corrosion is a very complicated process and can be influenced by many external factors. Nevertheless, salt spray test is widely used in the industrial sector for the evaluation of corrosion resistance of finished surfaces.

Corrosion is a process in which a material is oxidized by substances in the environment that cause the material to lose electrons. Corrosion is Electro chemical process of a metal that withstand the deterioration and chemical breakdown that occur when the material is exposed to such an environment. Corrosion resistance is the capacity to hold the binding energy of a metal and withstand the deterioration and chemical breakdown that would otherwise occur when the material is exposed to such an environment. Corrosion of Industrial factory equipment's, Thermal Furnaces, Boiler tube applications, Internal Combustion Engine parts and many other Automobile and Industrial machines is a major problem as it results in downtime and periodic shutdowns, which in turn accounts for a significant fraction of the total operating cost of power plants and installing new parts for the heavy machinery equipment's.

Inabilities to either totally prevent the hot corrosion or its detection at an early stage has resulted in several accidents, leading to loss of life and destruction of engine hence coatings are developed with an objective to enhance the substrate life by protecting against hot corrosion in the energy conversion systems and other similar high temperature applications under simulated conditions.

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Problems with process equipment resulting from fireside corrosion have been frequently encountered in waste incinerators and biomass-fired boilers. The major problem is the complex nature of the feed (waste) as well as corrosive impurities which form low-melting point compounds with heavy and alkali metal chlorides which prevent the formation of protective oxide scales and then cause an accelerated degradation of metallic elements [1]

Corrosion occurs on the water wall tubes in the radiant zone, the passes, the screen, evaporator tubes, and super heater tubes. Heat-resistant alloys survive high temperature exposure by growing protective oxide scales. This process involves the selective oxidation of an alloy constituent and necessarily leads to a change Hot Corrosion Behavior of Detonation Gun Sprayed Al_2O_3-TiO_2 Coating on Nickel Based Super alloys at 900^0 C in alloy compositions in the subsurface regions. The hot corrosion of alloys is a complex process that typically involves both scale formation and subsurface degradation, including internal precipitation, void formation, phase formation, and phase dissolution [2]

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It has been shown that Cr in a molten chloride environment is not an effective element for corrosion resistance improvement of Fe-based alloys due to chloride salt attack [3–8]

However, on the other hand it has also been suggested that the NiCr-based alloys show better performance than Fe-base alloys. This combined effect has been established in other studies. This work addresses the corrosion resistance of Inconel 600 in NaCl & KCl mixture (1:1) at 700, 800, and 900^{0} C for 100 hours in static air. [9–14]

Thermal spraying is an effective and low cost method to apply thick coatings to change surface properties of the component. Coatings are used in wide range of applications including automotive systems, boiler components, power generation equipment, chemical process equipment, aircraft engines, pulp and paper processing equipment and ships pp.112-134 [15]

Among the commercially available thermal spray coating techniques, Detonation Gun Spray is the best choices to get hard, dense and wear resistant coating as desired [16]

Detonation Gun (D-Gun) Spraying is one of the thermal spray processes, which gives an extremely good adhesive strength, low porosity, and coating surface with compressive residual stress [17]

Detonation Gun (D-Gun) offers highest velocity (800–1200 m /s) for the sprayed powders that are unattainable by the plasma. The higher particle velocity during deposition of coating results in desirable characteristics such as lower porosity and higher hardness of the coating [18]

2. EXPERIMENTAL WORK

2.1 Materials

The materials used in this study as substrate are Inconel specimen 600, 625 & 718. These specimens are Nickel-base alloys. & Chemical composition of **Inconel 600 is** 0.15%C, 0.015%S, 14-17%Cr, 0.5%Cu, 1%Mn, 0.5%Si, 72%Ni, 6-10%Fe, **Inconel 625 is** 0.10%C, 0.15%S, 20-23%Cr, 0.5%Mn, 0.5%Si, 58%Ni, 5%Fe, 8-10%Mo, 3.15-4.15%Nb&Ta, 0.4%Ti, 0.15%P, 0.40%Al, 1%Co and **Inconel 718 is** 0.08%C, 0.015%S, 17-21%Cr, 0.30%Cu, 0.35%Mn, 0.35%Si, 50-55%Ni, 0.65-1.15%Ti, 0.015%P, 0.20-0.80&Al, 1%Co, 4.75-5.50%Cb, 0.05%Ta, 0.006%B

2.2 Coating Thickness by D-gun spray process

D-gun spray process is thermal spray process which has remarkably good adhesive strength. In this process a coating powder and mixture of oxygen (O_2) and acetylene gas (C_2H_2) is fed through a tubular barrel whose one end is closed while another end is open.

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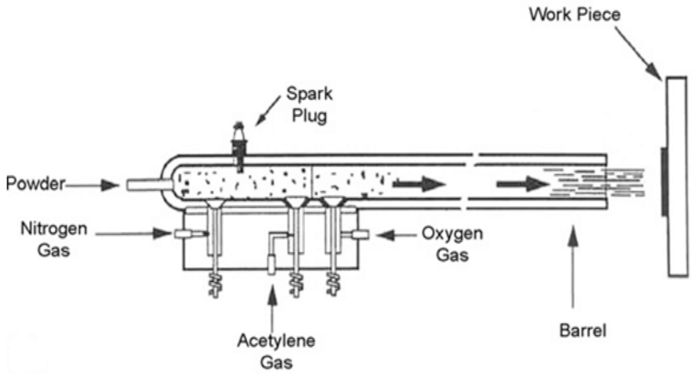
Fig 1: D-Gun Process



The Detonation-Gun process produces higher density, improved corrosion resistance, higher hardness, better wear resistance, higher bonding and cohesive strength, almost no oxidation, thicker coatings, and smoother as sprayed surfaces. It is widely used in steel industries (squeeze rollers, tension rolls), textile industries (thread guides coated with alumina titanium layer), aeronautic industries (advance coating on the vanes and driving shaft of a helicopter) and in automobile industries.

2.3 Advantages of D-Gun Method

Detonation Gun (D-Gun) is one of the thermal spray techniques in which spraying which generates a coating surface with compressive residual stress, a very low porosity, and a highly hard adhesive.

Detonation Gun (D-Gun) offers highest velocity (800–1200 m/s) for the sprayed powders that are unattainable by the plasma and HVOF condition. The higher particle velocity during deposition of coating results in desirable characteristics such as lower porosity and higher hardness of the coating.

2.4 Features of D gun coating

1) Coatings are extremely dense, durable, and clean.
2) Low compressive stresses exist in coatings.
3) Coatings have a very strong strength
4) The qualities of the coating are superb.

2.5 Coating thickness measurement

Coating thickness variation Inconel 600, 625 and 718 as measured by the electro-physic gauge has been provided in Table 1 & Two readings were taken at different location on the coated Inconel substrates to ascertain the exact thickness.

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Fig 2: Schematic representation of coating specimen Dimension and Axis of Thickness of measurement

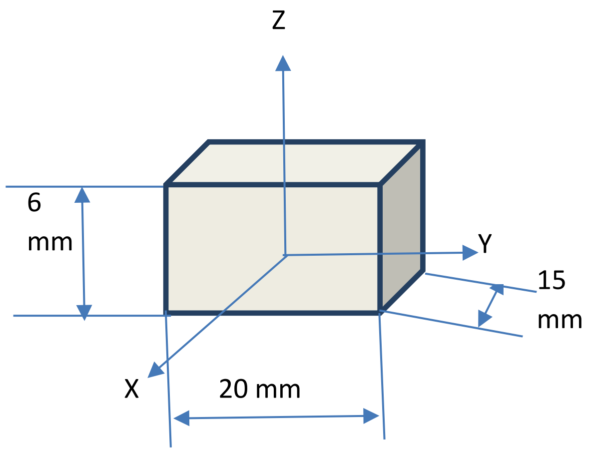

Table 1: Coating thickness on Inconel Substrates

Position	Coating Thickness in µm		
	Inconel 600	**Inconel 625**	**Inconel 718**
1	61.21	67.82	60.78
2	61.47	68.12	60.90
Avg.	61.34	67.97	60.84

3. ACID CORROSION TEST

3.1 Acidic Corrosion test of the Coated Inconel Specimens by using Concentrated HCl

Hydrochloric acid (HCl) is an important mineral acid with many uses, including the pickling of steel, acid treatment of oil wells and chemical cleaning and processing. This acid is extremely corrosive and its aggressiveness can change drastically depending on its concentration, the temperature and contamination by oxidizing impurities. One of the most commonly encountered oxidizing impurities is the ferric ion. In general, stainless steels cannot tolerate aggressive HCl solutions, hence the need to use corrosion-resistant nickel-based alloys. The Inconel coated substrates were subjected to laboratory corrosion tests utilizing glass flask and chemicals of the reagent grade. The size of specimen is 15mm*20mm*6mm.

Before the samples were weighed and tested, the surfaces of the samples were ground using 120 to 600 grit papers and then degreased using acetone. A 72-hour test was chosen, Concentrated HCl acid was the solution that was employed. Corrosion was found by weight loss method.

The Three different Inconel specimens 600, 625, 718 are soaked in three different beakers containing a capacity of 60ml of Concentrated HCl for 72 hrs

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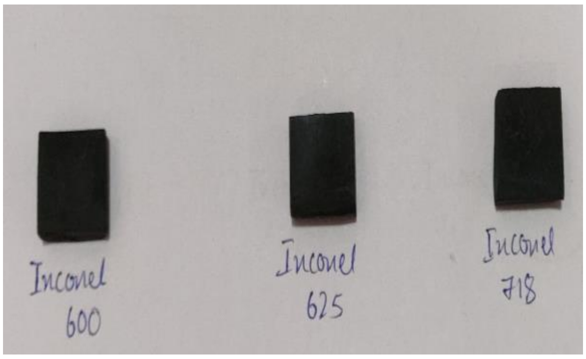
3.2 Corrosion Treatment of the Inconel Specimens 600, 625 & 718 Images

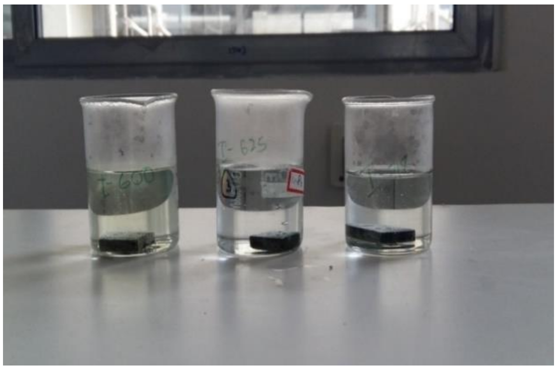
Step1: Before Corrosion Testing in Concentrated Hydrochloric Acid

Fig 3: before Acidic Corrosion Treatment



Step 2: During Corrosion Testing In Concentrated Hydrochloric Acid

Fig 4: during Acidic Corrosion Treatment



Step 3: After Corrosion Testing in Concentrated Hydrochloric Acid

Fig 5: after Acidic Corrosion Treatment

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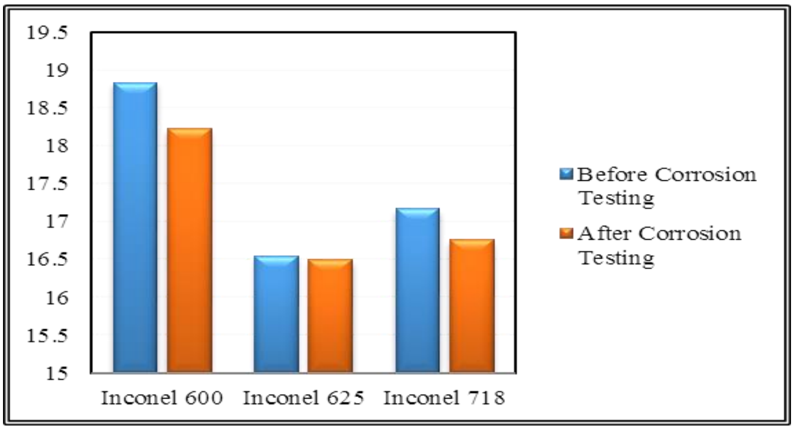
Table 2: Tabular Column for Masses of Inconel Specimens before and After Acidic Corrosion

Inconel Specimens	Weight of the Inconel Specimens (gms)		Weight in %
	Before Corrosion Testing	After Corrosion Testing	
Inconel 600	18.837	18.228	3.23
Inconel 625	16.543	16.496	**0.28**
Inconel 718	17.171	16.757	2.41

Fig.6 Coated Inconel Specimens before and After Acidic Corrosion



4. SALT CORROSION TEST

The coated substrates were studied for corrosion behavior by the standard salt spray technique. It is a popular method to check the corrosion rate of metallic and coated surface specimens. The test includes corrosion intervention on the coated samples in order to access the strength of the coating as a protecting surface. The corrosion attack is checked after a specified time, as per the experimental standards. Some specimens require less or longer period of time depending on the corrosion resistance of the coating. The salt spray test is one among the widely found corrosion tests. The internationally recognized standard test as per ASTM is performed using corrosion test chamber as shown in Fig.7 Generally, the coated specimens are opened to fine fog of salt water at 35°C temperature. The concentrated solution varies based on the material to be checked. The salt spray test is as shown in the Fig.7 an accelerated corrosion test that provides a corrosive attack to the coated samples in order to assess suitability in use as a protective finish.

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Fig 7: Salt spray test rig

The most frequently applied accelerated corrosion test is the ASTM (American Society for Testing and Materials) neutral salt spray test. Coated steel specimens were exposed to a fine-fog salt solution at an elevated temperature 35°C and the percentage of the specimen surface covered in red rust was measured as a function of exposure time & Test specification are 450ltr salt water & Temperature range is 85°C.

Table 3: Inconel substrates on corrosion rate variation with temperature

Expt. No.	Coated Substrates	Salt Chamber Temp.°C	Corrosion rate (Mpy)	Corrosion rate Avg. Value (Mpy)
01	Inconel 600	45	22	50.33
02		55	48	
03		65	89	
04		45	25	
05		55	42	
06		65	62	
07		45	30	
08		55	45	
09		65	90	
10	Inconel 625	45	28	38.5
11		55	42	
12		65	58	
13		45	18	
14		55	20	
15		65	55	
16		45	29	
17		55	20	
18		65	77	
19	Inconel 700	45	30	45.5
20		55	32	
21		65	76	
22		45	28	
23		55	40	
24		65	50	
25		45	32	
26		55	40	
27		65	78	

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4.1 Test Conditions

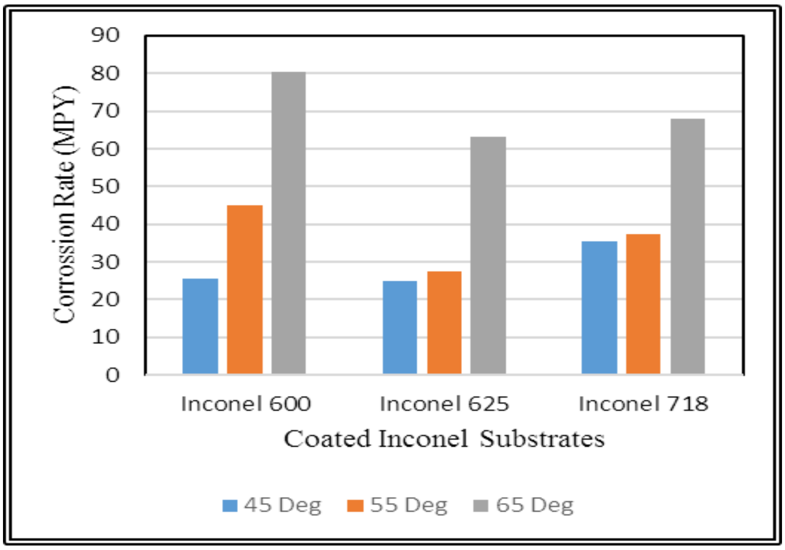
60 ltr of 5% NaCl (Sodium chloride) solution was filled into reservoir. The temperature of the chamber was maintained at 35°C. The salt solution fell on the specimens at a rate of 1.0 to 2.0 ml/hour. The fogging of the salt solution was done at the specified rate. The samples were placed at a 15^0-30^0 angle from vertical. This orientation allowed the condensation to run down the specimens and minimized condensation pooling.

Fig.8 shows the Inconel-Cr_3C_2 – NiCr coated Specimens after corrosion.

Fig 8: Cr_3C_2-NiCr coated Inconel Plates after Corrosion

The Results of the varying chamber temperature, Inconel substrates on corrosion rate are shown below for 3 trails showed in table3.

Fig 9: Corrosion Rate of coated Inconel Substrates



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5. SURFACE MORPHOLOGY (SURFACE ROUGHNESS)

The surface morphology of coated Inconel substrates was studied by Atomic force microscopy (AFM) selected images are presented as given in the Fig. (a),(b) and (c) shows the two-dimensional (2D) deflection image, the three-dimensional (3D) and (c) topography image respectively. The scan area is fairly large (2460µm^2). The deflection image, also known as the error signal image, results from inability of the electronics feedback circuit to maintain a constant force. It represents the gradient of shape change via differentiation, and is thus more sensitive to delegate spatial information such as sharp edges. Note that the Z axis is the 3D image is in a nm scale. In Fig. (a) And (b), surface cracking as well as the absence of a periodic structure are evident. The presence of random humps also supports the existence of an amorphous structure. It should be noted that in some other areas on this samples, there were indications of the existence of local crystalline structures. Cracking is clearly evident at the surface. Yet, the coating of Nickel chromium carbonate resulted in increased surface roughness and a significantly higher density of aggregates. The average roughness of the AFM images are given in the Fig.10

(Fig. a) **(Fig. b)** **(Fig. c)**

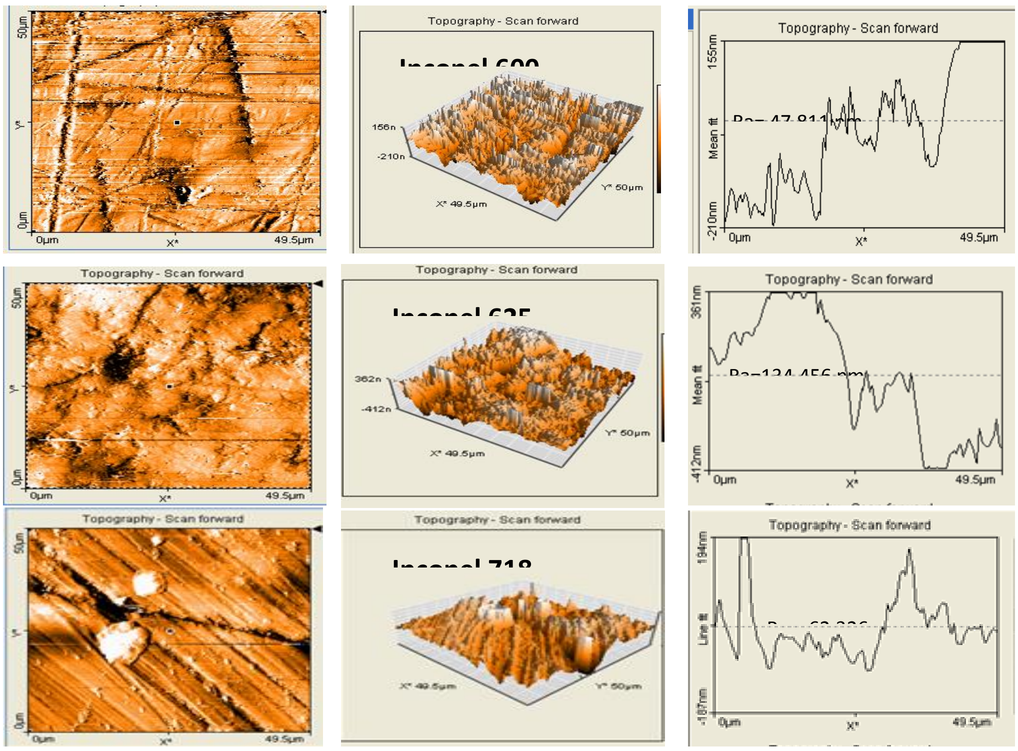

Fig. 10 (a) Atomic force deflection (2D), (b) 3D images and (c) Topography (roughness) from Inconel coatings, Inconel 600, Inconel 625 and Inconel 718

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6. RESULT & DISCUSSION

Three different INCONEL specimens 600, 625 and 718 are coated with Cr_3C_2 – NiCr and INCONEL 625 material covers maximum coating thickness 67.97µm and in acid corrosion test INCONEL 625 shows a weight reduction of 0.28% and in salt corrosion for temperature range 35^0C-65^0C, INCONEL 625shows the lesser average corrosion rate of 38.5Mpy in compare to 600(53.33Mpy) & 718(45.5Mpy) coated material. Therefore, INCONEL 625 behaves a promising metal that has good strength to withstand physical, chemical, and structural tests and can be widely used in High Temperature application in automobile like Turbine blades, piston rings etc. and also Surface roughness were observed in all coated materials and hence concluded that Inconel 625material has good roughness observed as compared to Inconel material 600 &718.

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Competing interests: The authors declare no competing financial interests.

References

1) F. J. Kohl, G. J. Santoro, C. A. Stearns, G. C. Fryburg, and D. E. Rosner, "Theoretical and experimental studies of the deposition of Na_2SO_4 from seeded combustion gases," Journal of the Electrochemical Society, vol. 126, no. 6, pp. 1054–1061, 1979.

2) D. J. Young and B. Gleeson, "Alloy phase transformations driven by high temperature corrosion processes," Corrosion Science, vol. 44, no. 2, pp. 345–357, 2002.

3) Y. Shinata and Y. Nishi, "NaCl-induced accelerated oxidation of chromium," Oxidation of Metals, vol. 26, no. 3-4, pp. 201–212, 1986.

4) N. Hiramatsu, Y. Uematsu, T. Tanaka, and M. Kinugasa, "Effects of alloying elements on NaCl-induced hot corrosion of stainless steels," Materials Science and Engineering A, vol. 120-121, no. 1, pp. 319–328, 1989.

5) Y. S. Li, M. Sanchez-Pasten, and M. Spiegel, "High temperature interaction of pure Cr with KCl," Materials Science Forum, vol. 461–464, pp. 1047–1054, 2004.

6) F. Wang and Y. Shu, "Influence of Cr content on the corrosion of Fe-Cr alloys: the synergistic effect of NaCl and water vapor," Oxidation of Metals, vol. 59, no. 3-4, pp. 201–214, 2003.

7) Y. S. Li, Y. Niu, and W. T. Wu, "Accelerated corrosion of pure Fe, Ni, Cr and several Fe-based alloys induced by $ZnCl_2$– KCl at 450^0 C in oxidizing environment," Materials Science and Engineering A, vol. 345, no. 3, pp. 964–970, 2003.

8) Y. Kawahara, "High temperature corrosion mechanisms and effect of alloying elements for materials used in waste incineration environment," Corrosion Science, vol. 44, no. 2, pp. 223–245, 2002.

9) Y. S. Li, Y. Niu, and W. T. Wu, "Accelerated corrosion of pure Fe, Ni,Cr and several Fe-based alloys induced by $ZnCl_2$-KCl at 450^0C in oxidizing environment," Materials Science and Engineering A, vol. 345, no. 1-2, pp. 64–71, 2003.

10) W. M. Lu, T. J. Pan, K. Zhang, and Y. Niu, "Accelerated corrosion of five commercial steels under a ZnCl2-KCl deposit in a reducing environment typical of waste gasification at 673–773 K," Corrosion Science, vol. 50, no. 7, pp. 1900–1906, 2008.

ISSN 1533-9211

DOI 10.5281/zenodo.7220737

11) T. J. Pan, C. L. Zeng, and Y. Niu, "Corrosion of three commercial steels under $ZnCl_2$-KCl deposits in a reducing atmosphere containing HCl and H2S at 400–500^0 C," Oxidation of Metals, vol. 67, no. 1-2, pp. 107–127, 2007.

12) K. Yamada, Y. Tomono, J. Morimoto, Y. Sasaki, and A. Ohmori, "Hot corrosion behavior of boiler tube materials in refuse incineration environment," Vacuum, vol. 65, no. 3-4, pp. 533– 540, 2002.

13) J. Porcayo-Calderon, O. Sotelo-Mazon, V. M. Salinas-Bravo, C. D. Arrieta-Gonzalez, J. J. Ramos-Hernandez, and C. CuevasArteaga, "Electrochemical performance of Ni20Cr coatings applied by combustion powder spray in ZnCl2-KCl molten salts," International Journal of Electrochemical Science, vol. 7, no. 2, pp. 1134–1148, 2012.

14) J. Porcayo-Calderon, O. Sotelo-Mazon, M. Casales-Diaz, J. A. Ascencio-Gutierrez, V. M. Salinas-Bravo, and L. MartinezGomez, "Electrochemical study of Ni20Cr coatings applied by HVOF process in ZnCl2-KCl at high temperatures," Journal of Analytical Methods in Chemistry, vol. 2014, Article ID 503618, 10 pages, 2014.

15) Gaurav Prashar, Hitesh Vasudev "Thermal spraying is an effective and low cost method to apply thick coatings to change surface properties of the component. Coatings are used in wide range of applications including automotive systems, boiler components, power generation equipment, chemical process equipment, aircraft engines, pulp and paper processing equipment and ships" pp.112-134)

16) **P. Vuoristo, in Comprehensive Materials Processing, 2014, Thermal Spray Coating Processes, Volume 4, 2014, Pages 229-276**

17) Lakhwinder Singh1*, Vikas Chawla2 , J.S. Grewal1, A Review on Detonation Gun Sprayed Coatings, Vol. 11, No.3, pp.243-265, 2012

18) **N. K. Mishra,1 Naveen Kumar,1 and S. B. Mishra1 Hot Corrosion Behaviour of Detonation Gun Sprayed Al2O3-40TiO2 Coating on Nickel Based Superalloys at 900°C, Volume 2014, Article ID 453607, 5 pages**

19) T. Burakowski and T. Wierzchon, "Surface Engineering of Metals", Principles, Equipment, Technology, CRC Press, Boca Raton, Fla, USA, 1999.

20) **James R Smith, "AFM in surface finishing: Part II. Surface roughness" pp. 171–183, Trans Inst Met Fin, 2003.**

21) P. S. Sidky and M. G. Hocking, "Review of inorganic coatings and coating processes for reducing wear and corrosion," British Corrosion Journal, vol. 34, no. 3, pp. 171–183, 1999.