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ChemTech

International Journal of ChemTech Research
CODEN (USA): IJCRGG ISSN : 0974-4290
Vol.6, No.5, pp 2744-2749, Aug-Sept 2014

Performance of plasma spray coatings on Inconel 625 in Air oxidation and molten salt environment at 800 °C

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Abstract: The aim of the present work is to compare hot corrosion behavior of Al_2O_3-TiO_2 plasma spray coatings on Inconel 625 in air oxidation and molten salt environment K_2SO_4–60%NaCl environment at 800°C under cyclic conditions for 50 cycles. Al_2O_3-TiO_2 coatings were deposited on Inconel 625 superalloy with plasma spray process. The thermo-gravimetric technique was used to establish the kinetics of corrosion. The corrosion products have been analyzed using the SEM and EDS analysis. The coating performed relatively better in air oxidation against hot corrosion in the molten salt environment.

Keywords: Plasma Spray coating; Inconel 625; Al_2O_3-TiO_2; Hot corrosion.

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1. Introduction

The components operating at high temperature environments such as power plants, gas turbines and incinerators fail due to high temperature corrosion (1-3). In power plant applications, low-grade fuels with high concentrations of Sulphur, Potassium and Sodium are used in oil and coal fired power generation. During combustion, alkali metal sulfates vapors combine with other ash constituents that deposit onto the component surfaces. The boilers exposed in off-shore industrial rigs undergo hot corrosion when the Sodium chloride from the ocean breeze mixes with K_2SO_4 from the fuel and deposits on the hot-section of the components. The metal surface temperature could be such that volatile compounds in the gas stream can condense and form molten deposits. This produces aggressive conditions leading to rapid corrosion of the specimen due to oxidation, sulfidation, chloridation and even hot corrosion [4-6]. High temperature corrosion is an accelerated oxidation in the presence of molten salts environment. In general, the formation of oxides on the surface acts as a protective layer. However, the deposit of molten salts such as K_2SO_4, Na_2SO_4, KCl and NaCl lowers the melting point [4]. It results in the damage of the protective oxide layer and this phenomenon is known as hot corrosion. This cannot be completely eliminated but can be minimised by means of coating process. In the recent past, many researchers have proposed different coating processes with different coating powders to protect these alloys from hot corrosion. Many researchers [5-6] reported that thermal spray coatings are key solution to this problem and increase the life of materials considerably. Inconel 625 has been developed for high-temperature strength along with hot corrosion resistance. In the coating technologies the plasma spray process is one of the emerging technologies. The Al_2O_3-TiO_2 coatings are commonly used to protect the underlying materials from hot corrosion degradation. Jafarzadeh et al [6] deposited the Al_2O_3-TiO_2 coating with plasma spray and they found that the as-sprayed carbon steel exhibits higher performance.

Al_2O_3-TiO_2 coatings are most useful when one is seeking hot corrosion resistance for the components exposed in high temperature environment [5]. Plasma spray is one of the most common methods for applying the above mentioned coating. Many investigations have been done up to now on the microstructure, mechanical properties of Al_2O_3-TiO_2 and their wear properties. However, despite the importance of Al_2O_3-TiO_2 coatings in power plant applications, so far, very limited studies have been reported for its hot corrosion behaviour in these environments. It is found from the literature that little work has been done on plasma spray coating for high temperature applications and hence this technique is used to develop high temperature resistant Al_2O_3-TiO_2 coatings on Inconel 625. The present study is to evaluate and compare the hot corrosion behavior of Al_2O_3-TiO_2 plasma spray coated on Inconel 625 in air oxidation and K_2SO_4–40%NaCl molten salt environment at 800 °C under cyclic study.

2. Experimental procedure

2.1. Formulation of coatings

The Inconel 625 superalloy was used as substrate material in the present study and its chemical composition is listed in Table 1. Commercially available Al_2O_3- TiO_2(40%) powder was used for coatings. The alloy plate was cut into rectangular samples of size 20× 15×5mm. The specimens were polished using Silicon carbide papers up to 1200 grit and finally wheel polished with alumina powder (1μm). They were then washed with distilled water and cleaned with Acetone. Their surface area was measured and weights were determined using an electronic balance to an accuracy of 0.01mg. The coating work was carried out by a commercial firm namely Spraymet Surface Technologies Pvt Ltd, Bengaluru, India. They used 'Robotic Plasma spray 40Kw and 80Kw' plasma spray apparatus to apply the coatings. Argon mixed with hydrogen was used as powder carrying and shielding gas. All the process parameters were kept constant throughout the coating process while spraying distance was maintained in a narrow range of 80-120 mm. Ni-Cr powder was deposited as a bond coat around 40-50 μm thick before applying the final coatings. The process parameters for the plasma spray process employed for applying the coatings are listed in Table 2. Thickness of the coatings was controlled during the process of plasma spraying.

Table 1. The chemical composition of Inconel 625.

	Ni	**Cr**	**Mo**	**Fe**	**Nb+Ta**	**C**	**Mn**	**Si**	**P**	**S**
Inconel 625	58.0	20.0	8.0	5.0	4.15	0.10	0.50	0.50	0.015	0.015

Table 2. Process parameters for plasma spray process.

Coating powder	Argon Flow rate lit/min	Hydrogen Flow rate lit/min	Current (I)	Voltage (V)	Carrier gas(N 4mp)	Powder feed (gm/min)	Spray distance (mm)
Al2O3-TiO2	80-90	20-25	490	60-70	37-39	80-90	80-120
ZrO2	80-90	15-18	500	50	37-39	65-70	80-120

2.2. Hot corrosion

Oxidation and hot corrosion studies were conducted at 9000°C in a laboratory Silicon carbide tube furnace. The air oxidation and molten salt (K_2SO_4-60%NaCl) studies at 800°C were performed on plasma spray coated Inconel 625 in laboratory furnace up to 50 cycles. The plasma spray coated as well as uncoated specimens were prepared. The specimens were then heated in an oven up to 250^{o}C and a salt mixture of K_2SO_4-60%NaCl dissolved in distilled water was coated on the warm polished specimens with the help of a camel hair brush. Amount of the salt coating was kept in the range of 3.0 -5.0 mg/cm^2. The salt coated specimens as well as the alumina boats were then dried in the oven for 2 hours at 200°C and weighed before being exposed to hot corrosion tests. The uncoated as well as the coated specimens were polished down to 1μm alumina wheel cloth polishing to obtain similar condition of reaction before being subjected to corrosion run. Fig 1. The macro images of Al_2O_3- TiO_2 coated Inconel 625 before and after air oxidation and molten salt corrosion is represented in Fig 1. Physical dimensions of the specimens were then recorded carefully with Vernier calipers to evaluate their surface areas. Subsequently the specimens were washed properly with acetone and dried in hot air to remove any moisture. During experimentation the prepared specimen was kept in an alumina boat and the weight of boat and specimen was measured. The alumina boats used for the studies were pre-heated at a constant temperature of 1200°C for 24 hours and it was assumed that their weight would remain constant during the course of high temperature cyclic oxidation/corrosion study. Then the boat containing the specimen was inserted into hot zone of the furnace set at a temperature of 800°C. Holding time in the furnace was one hour in still air after which the boat with specimen was taken out and cooled at the ambient temperature for 20 minutes. Following this, weight of the boat along with specimen was measured. This process constituted one cycle of the oxidation study. Any spalled scale in the boat was also taken into consideration for the weight change measurements. Visual observations were made after the end of each cycle with respect to colour, luster or any other physical aspect of the oxide scales being formed. Surface morphology of the plasma sprayed coatings after corrosion cycles was also studied with the help of Scanning Electron Microscope (SEM). Whereas surface SEM/EDS analysis was performed to identify the elements or phases (oxides) present at a point along with their compositions.

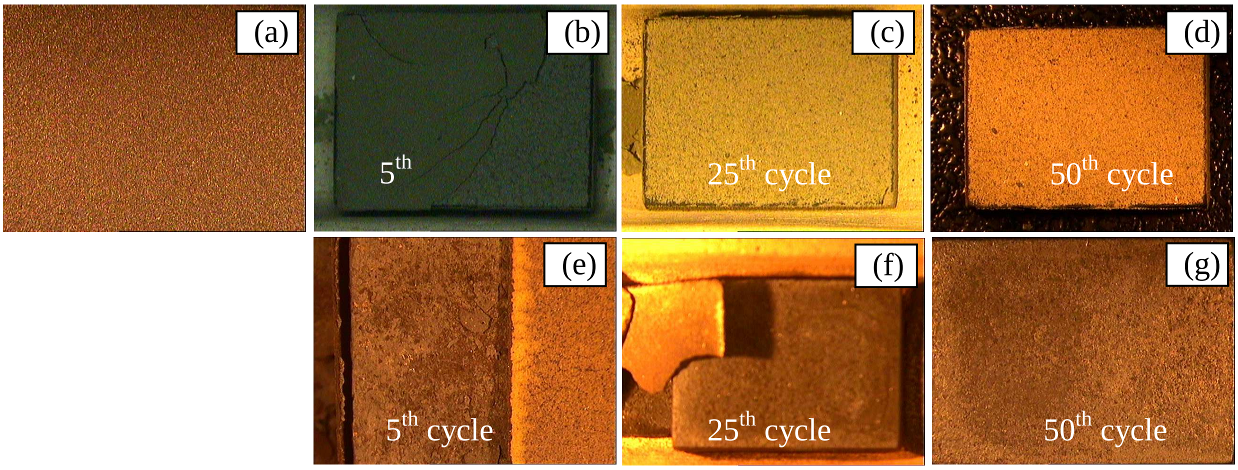

Fig 1. Macro images of Al_2O_3-TiO_2 coated Inconel 625 before and after air oxidation (a) As coated condition; b, c and d after air oxidation; e, f and g after molten salt corrosion.

3. Discussion

The Al_2O_3- TiO_2 coatings were successfully deposited on Inconel 625 plasma spray process. The coating process and powder play an important role especially for the application of high temperature aggressive environment. Sidhu Singh et al. reported that lifetime increases with increase in coating thickness and further found maximum life with 300 μm coating thickness.

It was aimed to produce thicker coatings because thicker coatings are generally required for the components of energy generation systems. But self-disintegration of thicker coatings usually restricts the thickness of the coatings. In the present study it was possible to obtain a thickness in range of 100-110 μm for the Al_2O_3-TiO_2 coating and the final coating thickness of approximately 140-160 μm. Fig 1 (a-g) shows the macro structure of hot corroded samples after 50 cycles. In the high temperature study of the uncoated Inconel 625, after completion of 35 cycles, the scale becomes thicker and most of the scale spalled out in the molten salt environment. However, the scale was found to be continuous in the case of Al_2O_3- TiO_2 coated Inconel 625 for both environments up to 50 cycles.

The Al_2O_3-TiO_2 coated Inconel 625 under study has shown less weight gain in both air oxidation and molten salt environment as compared to the uncoated alloy indicating the protective behavior of the coatings (Fig 2). Thermo-gravimetric curve for both environments shows the tendency of oscillation type reaction which could be due to changes in reaction rate as suggested by Arivazhagan et al.[7]. Further, the graph indicates that the weight gain of the uncoated alloy was found to be more during initial cycles of study. This could be attributed to the rapid oxidation during initial stages to form oxide scale of the surface elements. Arivazhagan et al. [7, 8], Devendranath Ramkumar et al. [4] had also reported the high weight gain during initial cycles of hot corrosion. In addition, the weight gain trend indicates deviation from the parabolic rate law due to spalling and sputtering of the oxide scale. The higher weight gain in the case of uncoated Inconel 625 may be attributed to the formation of Iron (Fe) rich oxide scale which is less protective in nature as compared to the oxides of Titanium (Ti) and Chromium (Cr) resulting in more spalling and sputtering (Fig 3, 4). SEM/EDAX of the after coating has revealed the top layer contains higher amount of Ti and Al as can be seen in Fig 5 and Fig 6. To provide long-term stability at elevated temperatures, it is necessary to develop diffusion-barrier coatings to minimize the inter-diffusion between the coating and the substrate. However, he further added that some inter-diffusion is necessary to give good adhesion; hence, the diffusion barriers must be tailored to limit the movements of particular problematic elements. Inter-diffusion observed in the present study is very minor and might be helpful for providing better adhesion between the substrate, bond coat and the top coat.

The SEM image indicates that the uncoated samples show a rough irregular surface as well as spalling behavior of the scale after exposure to molten salt environment (Fig 4). The basic fluxing is caused by K_2SO_4–60%NaCl at 800 °C under thermal cyclic condition. The EDAX analysis on the sub-scale reveals that scale mainly consists of the oxides of Chromium (Cr) as the major phase along with significant amounts of Iron (Fe) and Nickel (Ni). The scale formed on Al_2O_3-TiO_2 coated Inconel 625 is almost consistent and continuous when exposed in air oxidation (Fig 5). Furthermore, the EDS analysis reveals Titanium (Ti) as a principal phase along with small percentage of Aluminum (Al) and Nickel (Ni) phases. Sufficient amount of Oxygen (O) is also present to form oxides. Therefore, the surface might contain TiO_2 as the major phase along with NiO and Al_2O_3.

This study shows that the environment has been found to have pronounced effect on weight gain. Based on the magnitude of weight gain, molten salt environment found to undergo accelerated oxidation under cyclic conditions in this particular environment of K_2SO_4 - 60%NaCl as compared to air oxidation. This result is also well supported by Devendranath Ramkumar et al. (4).

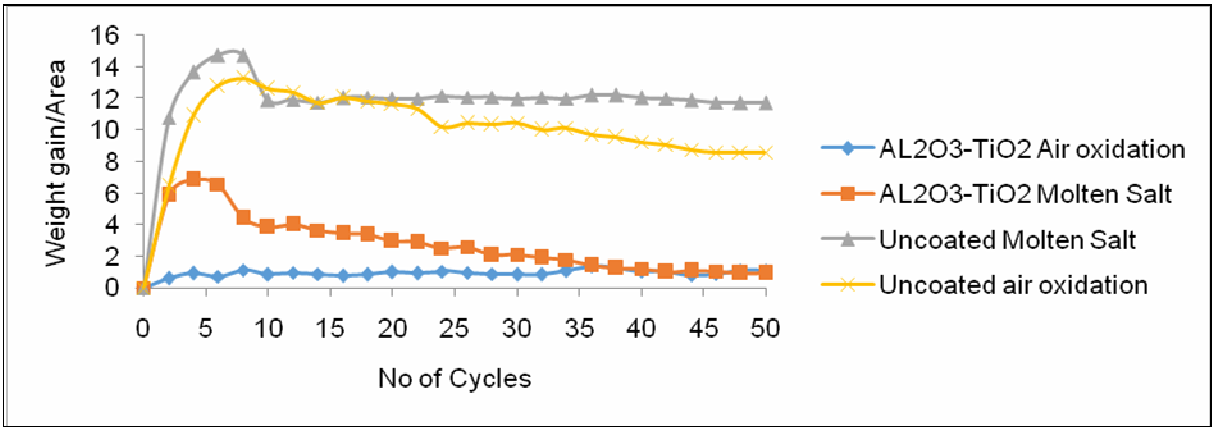

Fig 2. Thermo-gravimetric plot for uncoated and Al_2O_3-TiO_2 plasma spray coated Inconel 625 exposed in air oxidation and K_2SO_4–60%NaCl environment at 800 °C under cyclic conditions.

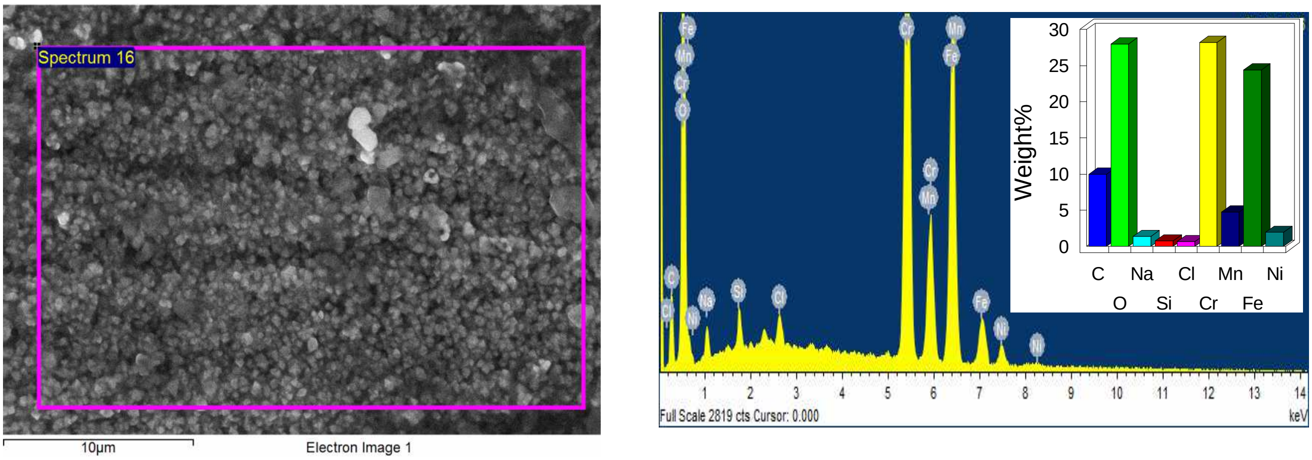

Fig 3. SEM/EDS results of uncoated Inconel-625 after exposed air oxidation at 800 ^{o}C.

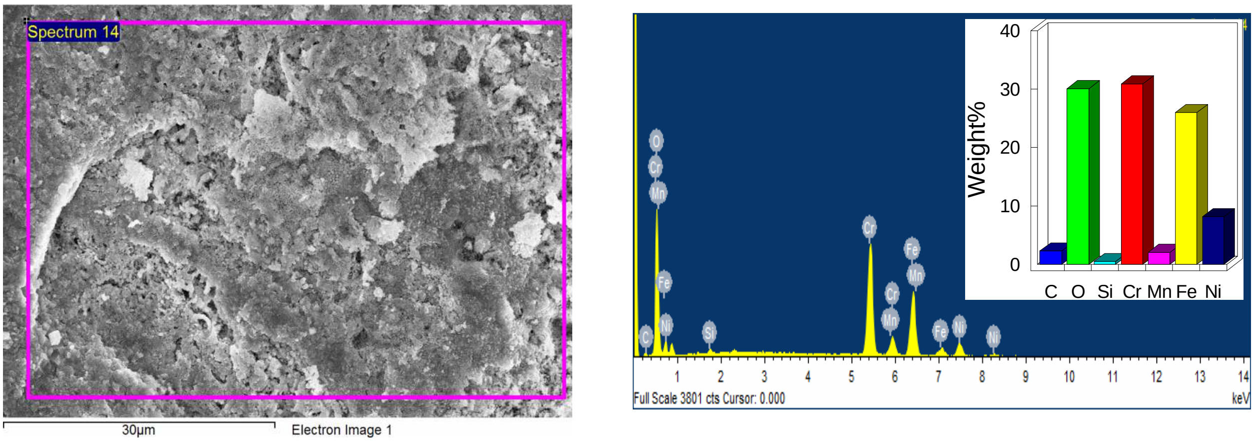

Fig 4. SEM/EDS results of uncoated Inconel-625 after exposed in K_2SO_4 – 60% NaCl at 800 C.

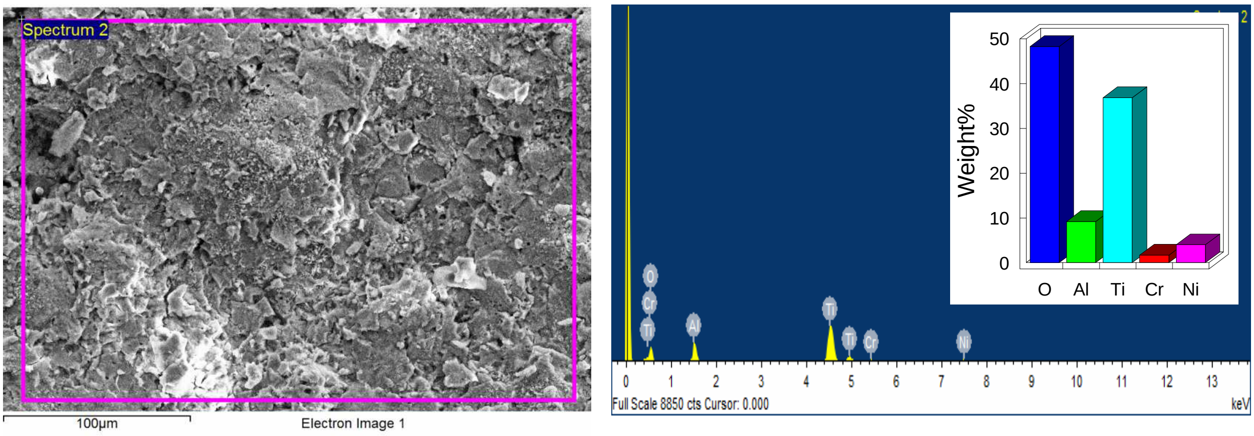

Fig 5. SEM/EDS results of Al_2O_3 -40 TiO_2 coated Inconel-625 after exposed air oxidation at 800 ºC.

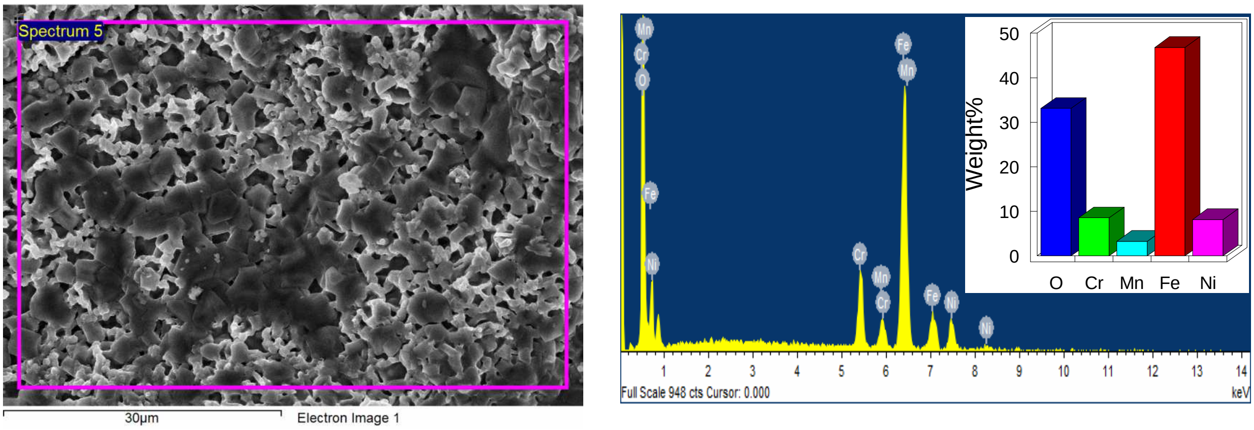

Fig 6. SEM/EDS results of Al_2O_3 -40 TiO_2 coated Inconel-625 after exposed in K_2SO_4 –60% NaCl at 800 ºC.

4. Conclusion

a) A 40-50 µm thick coating of Ni-Cr powder and 100-110 µm thick coating of Al_2O_3-TiO_2 powder were successfully deposited on Inconel 625 using plasma spray coating process.

b) The coating showed the effective hot corrosion resistance in air oxidation. However, the hot corrosion resistance in molten salt environment is relatively inferior.

c) The highest corrosion rate was noticed in the uncoated Inconel 625 in the molten salt environment followed by air oxidation.

d) The plasma sprayed Al_2O_3-TiO_2 coating protects the substrate with the formation of Cr_2O_3 scale at the top and beneath Al_2O_3 scale in K_2SO_4–60%NaCl molten salt environment at 800 °C for 50 cycles.
e) Better hot corrosion resistance of plasma sprayed Al_2O_3-TiO_2 is due to the formation of some Ni-Cr spinels oxides.
f) The weight gain of Al_2O_3-TiO_2 coated Inconel 625 is found to be considerably less in both environments due to dense coating structure obtained by the plasma spray coating.

5. References

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