HIGH TEMPERATURE CORROSION BEHAVIOR OF STELLITE 6 COATED SUPERALLOY IN GAS TURBINE ENVIRONMENT

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CERTIFICATE

I hereby declare that the dissertation entitled **“HIGH TEMPERATURE CORROSION BEHAVIOR OF STELLITE 6 COATED SUPERALLOY IN GAS TURBINE ENVIRONMENT”** is an authentic record of my work carried out as requirements for the award of the degree of Master of Engineering in Production Engineering at **Thapar Institute of Engineering and Technology, Patiala** under the supervision of **Dr. Deepa Mudgal (Assistant Professor, Department of Mechanical Engineering) and Dr. Hiralal Bhowmick (Associate Professor, Department of Mechanical Engineering).** No part of the matter embodied in this thesis has been submitted to any other university or institute for the award of any degree.

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It is certified that the above mentioned statement made by the student is correct to the best of my knowledge and belief.

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ABSTRACT

Failure in components of boilers, waste incinerators, gas turbines, petrochemical installations and metallurgical furnaces often takes place due to high-temperature corrosion, erosion and oxidation. Metal substrates are usually coated with protective thermal coatings to prevent electrochemical charges present in environment from entering inside metal substrate. When corrosive environment comes in contact with protective coating, the coating forms layer of oxides that provide a barrier against corrosive species and higher the thickness of coating, the stronger is the barrier between corrosive species and metal substrate. In this way, the metal substrate stays protected from the corrosive environment. In this work, efforts have been made to enhance the life of Superni 600 by deposition of Stellite 6 coating by detonation gun thermal spray technique. The bare and coated Superni 600 samples were exposed to simulated gas turbine environment of Na_2SO_4–60%V_2O_5 upto 25 cycles to observe the hot corrosion behaviour of the samples. It was observed that formation of cobalt based oxides on coated samples helped in improving the corrosion resistance of Superni 600. Although, it seems that oxides of Stellite 6 coating in gas turbine environment get degraded with subsequent cycles but will surely enhance the life of substrate as the coating remains intact even after 25 cycles of corrosion run.

Keywords: Stellite 6 coating; D-gun; Superni 600; Corrosion; Superalloys

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NOMENCLATURE

HVOF- High Velocity Oxy Fuel

D-Gun- Detonation Gun

SEM- Scanning Electron Microscopy

EDS- Energy Dispersive Spectroscopy

XRD- X Ray Diffraction

K_p- Parabolic rate constant

Chapter 1

INTRODUCTION

1.1 CORROSION AND SIGNIFICANCE OF COATINGS

Corrosion can be defined as a physiochemical process in which metal reacts with the environment, resulting in changes in metal properties. It involves an electrochemical reaction of metallic substrate between positively charged anode and negatively charged cathode, in the presence of an electrolytic solution that causes formation of ferrous oxides, often termed as rust, when corrosion takes place on iron and steel. On steel substrate, some areas are anodic in nature while others are cathodic. On catalytically active substrate, the oxygen present on cathode forms hydroxyl ions along with the formation of peroxides, radicals or super-oxides. On anode, several corrosive reactions take place resulting in formation of ferrous oxides. The main driving force responsible for corrosion is the difference in the potential of anodes and cathodes (Fig. 1.1). Extremely localized corrosion often results in formation of small holes in metal substrate and this process is called pitting [1].

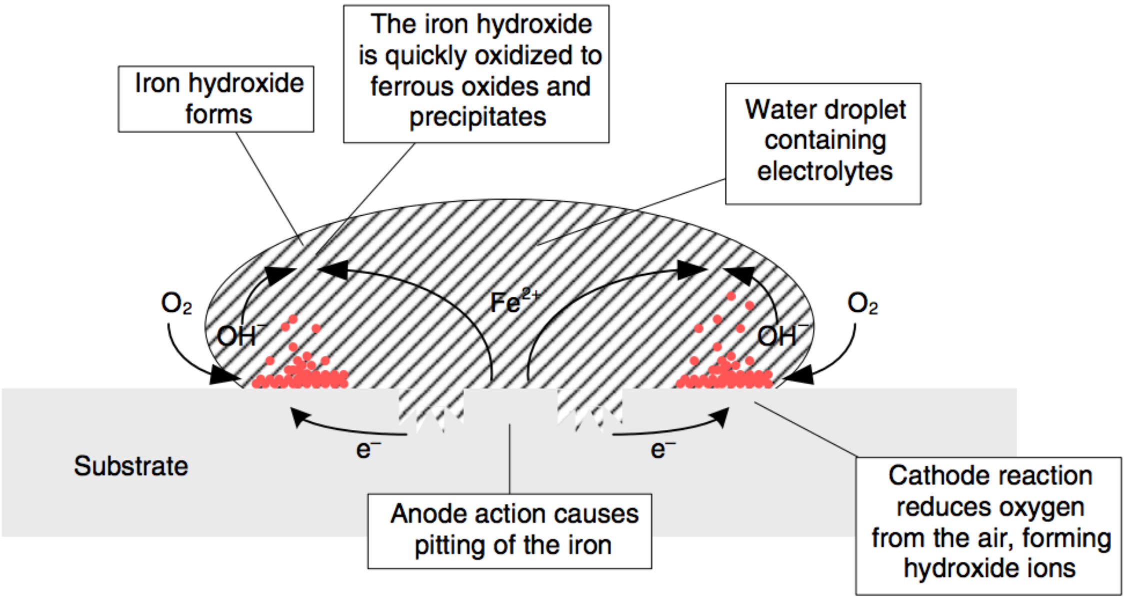


Fig. 1.1: Corrosion process [1]

At high temperatures, a portion of matter present in input material (coal) of boilers gets vapourized in the presence of heat and results in species like HCl, NaOH, SO_2 and H_2S and several other chlorine related species that cause corrosion of superheaters and other boiler equipments. Incomplete combustion of coal, lower temperatures at ash fusion and increased mineral deposition affect the corrosive behavior of boilers. Severe corrosion is responsible for reducing the lifetime of boiler tubes, that can result in unexpected failure of boilers, leading to shut down and productivity gets affected. Corrosion in boilers often takes place primarily due to two reasons: first, deposition of alkali metal sulphates that are soluble in water and result in acidic reactions and second, iron sulfide deposits that are highly insoluble in water and contain large amounts of carbon. The deposits of alkali metals are due to flame or molten slag. When the chlorine content in coal is high, then it comes out as volatile HCl present in flue gas. Chlorine related species are also responsible for corrosion of boiler tubes [2].

Failure in components of boilers, waste incinerators, gas turbines, petrochemical installations and metallurgical furnaces often takes place due to high-temperature corrosion, erosion and oxidation. Metal substrates are usually coated with protective thermal coatings to prevent electrochemical charges present in environment from entering inside metal substrate. When corrosive environment comes in contact with protective coating, the coating forms layer of oxides that provide a barrier against corrosive species and higher the thickness of coating, the stronger is the barrier between corrosive species and metal substrate. In this way, the metal substrate stays protected from the corrosive environment.

Coatings play an important role in high temperature coal fired boilers and gas turbines by performing the following functions:

a) Coatings help in lowering the temperature of metal substrate thereby increasing the lifetime of metals with lower strength.
b) Coatings help in reducing the magnitude of induced strains, thus increasing the lifetime of metal substrates that are susceptible to thermal fatigue damage.
c) Coatings help in increasing the efficiency of equipments due to the formation of barrier between corrosive environment and metal substrate [3].

1.2 CLASSIFICATION OF COATINGS FOR CORROSIVE ENVIRONMENT

For corrosive environment, four types of coatings are used:

a) Sacrificial coatings
b) Polymer coatings
c) Ceramic coatings

d) Barrier coatings

The description, examples and applications of these kinds of coatings are summarized in the form of a table (Table 1.1).

Table 1.1: Classification of coatings for corrosive environment

Type of coating	**Description**	**Examples**	**Application**
Sacrificial coating	It helps in corrosion control by application of thin layer of metal with lower electrode potential than the material to be protected. The sacrificial coating oxidizes according to anodic reactions and electrons from this coating flow towards the protected metal forming cathode and preventing corrosion.	Coating of zinc, nickel or tin on steel	Pipelines, underground tanks, refineries and water heaters
Polymer coating	It is applied on metal surface that provide separation of metal surface from electrolytic solution and other metallic parts. This coating consists of binder, solvent for control of viscosity, additives for inhibition of corrosion and pigments. They can be applied by processes of spraying, dipping and brushing.	Latex coating, polytetra-fluoroethylene coating, polyester, vinyls	Solar cell, Light emitting diodes, lithium-sulphur batteries,
Ceramic coating	It also provides corrosion resistance by separation of	Chrom oxide, aluminium	Oil pipelines,

	metal from corrosive atmosphere. It prevents air and moisture from coming in contact with the metal substrate. These coatings are produced from slurry that are applied on metal substrate and subsequently heated.	oxide, zirconium oxide	pumps, valves, ball bearings
Barrier coating	Additional coating is applied on metal substrate to protect it from corrosion. Layer of oxides are formed that provide barrier against corrosive species. If the barrier or layer of oxides get damaged then the metal substrate becomes unprotected.	Nickel based coatings, chromium carbide based coatings, tungsten carbide coating	Boiler tubes, gas turbine blades, combustion chamber, economizers, superheaters

1.3 THERMAL SPRAY COATING PROCESS

First thermal spray was based on wire feeding and heating by fuel gas with oxygen. Plasma spray, wire arc, HVOF and D-gun are some of the frequently used thermal spray coating techniques. This process is currently known as flame spraying or oxy-fuel where feed can be in the form of powder both metallic and ceramic, coating wire, detonation and HVOF. Powder spraying and plasma spraying were first developed. Since then, the basic process remains the same but powders are now being sprayed directly with high velocity.

Among all of these coating techniques, D-gun technique is highly preferred due to its high bond strength and low porosity [4]. On comparison with other thermal spraying techniques, D-gun spraying involves spraying of particles, accelerated by the detonation wave after spark plug ignition and those particles make an impact on the surface of substrate at a velocity of 800–1200 m/s [5]. Generally, it is considered that the powder gets closely attached to the surface due to high active energy that forms a layer with high hardness, strength, wear resistance and adhesion [6].High adhesion ensures the prolonged life-time of the coating and to promote good wear and corrosion resistance properties. Since this spraying method provides better adhesive and cohesive strength than other thermal-spray coating processes due to higher kinetic energy of the powder particles, it can be used for high quality coatings.

In all thermal spray processes, coatings are obtained by impacting of molten or softened particles on to a substrate and rapid solidification of small globular particles after striking the cold surface at high velocities results in formation of lamellar type granular structure. The coatings have been found to enhance the service life by 50 to 75 percent in powder, chemical, petrochemical, construction, mining, pulp and paper industries. This may also lead to cost saving. This process can also be used for rebuilding parts to provide corrosion and wear resistance.

Thermal or heat energy is essential for melting feedstock materials such as wire, powder or rod so as to deposit them on the substrate. A buffer gas is always needed to accelerate and atomize the melted particles. Hence, parameters such as gas flow and heat energy directly affect particle temperature, speed and trajectory, flame/plasma/jet temperature and velocity, and deposit temperature. Spray-pattern and particle heating are affected by these variables or factors in different ways.

The techniques having low particle velocity with high heat input always produce coatings with high porosity content, high oxide content and rounded particles. Examples of such thermal spray process are combustion-wire, combustion flame and twin-wire arc spray coatings. Process having high velocity of particles with superheated temperature always produce dense coating morphology with much finer porosity such as plasma spray coatings. The techniques such as HVOF and D-gun have very high particle temperature along with high velocity thus produce dense, compact and very low porosity coatings which cannot be achieved by any other thermal spray process.

1.4 TECHNIQUES FOR DEPOSITION OF THERMAL SPRAY COATINGS

Thermal spray coatings have been proven to provide excellent corrosion and wear resistance to metal substrates. The techniques that are mainly used for deposition of thermal spray coatings are discussed below:

a) High Velocity Oxy Fuel Coating (HVOF) Process

A mixture of methane or propane fuel and oxygen in the form of liquid or gas is allowed to undergo combustion in combustion chamber to produce a steam of high pressure gas. A spray comes out of the nozzle of combustion chamber at 1000m/s and powdered coating material is added to this jet steam to partially melt and accelerate the powder in hot gas steam. The hot jet then comes in contact with metallic substrate and coating is deposited on it with high density and adherence to the substrate [7].

b) Detonation Gun (D-Gun) Coating Process

It is a high velocity thermal spray process which consists of a gun barrel that is open at one end and closed at the other end. This gun barrel is water cooled as water continuously passes along the sides of the gun barrel. Oxygen and fuel, most commonly acetylene, are fed through inlet ports and powdered coating material is fed through another port. The gas mixture is allowed to ignite with the help of spark plug, installed at the edge of gun barrel. Due to detonation, the coating material melts and accelerates to a velocity of 600m/s. Gun barrel is flushed with nitrogen after every detonation [7].

c) Plasma Spray Coating Process

It consists of atmospheric plasma spray coating and vacuum plasma spray coating. In this process, plasma gun gives high temperature plasma at temperature of 10,000K that has the ability of melting polymers, ceramics and refractory metals. The coating materials can be powder, slurry, suspension and liquid are fed across the stream of hot plasma and high temperature melts the coating material. Due to high speed of plasma, the droplets of molten material can be easily deposited on the substrate. In vacuum plasma spraying, low temperature is used for materials that cannot undergo reactions at atmospheric conditions [7].

d) Cold Spray Coating Process

This process does not employ a heat source for melting of coating materials. Instead, it is dependent on the particles size, temperature of target material, velocity of coating material stream and its properties. Powdered coating material is fed across high velocity stream of mixture of two gases: nitrogen and helium for achieving kinetic energy. On hitting the substrate, the coating particles deform and get attached or bonded to the substrate. The temperature of the gaseous mixture can be increased to improve the process efficiency [7].

e) Arc Wire Spray Coating Process

In this process, two metallic wires (consumable) are charged with DC supply and an arc is generated between them that causes melting of the metallic wires. The molten material is then pumped out through converging nozzle on to the substrate and thus, coating is deposited. This process is limited to conductive materials and wires [7].

1.5 DETONATION GUN COATING

1.5.1 Process

Detonation Gun (D-Gun) technique uses a long gun barrel that is water cooled and has an internal diameter of 25mm. Oxygen and acetylene are fed as input to the gun barrel and coating powder is also fed through another port. Spark plug ignites mixture of oxygen and acetylene which is followed by explosion and detonation wave further accelerates the powder. To prevent the explosion of supply of fuel gas or backfiring, nitrogen (inert gas) is used between exploding mixture portions. After every detonation, gun barrel is washed with nitrogen (Fig. 1.2).

It involves the following cycles:

a) Combustion chamber of gun barrel is injected with oxygen and fuel (most commonly acetylene)
b) Backfiring is prevented by injection of nitrogen and coating material powder
c) Mixture gets ignited and powder is accelerated
d) Nitrogen is fed inside for purging of barrel

In a second, around 1 to 15 detonations take place with purges of nitrogen between each detonation [8].

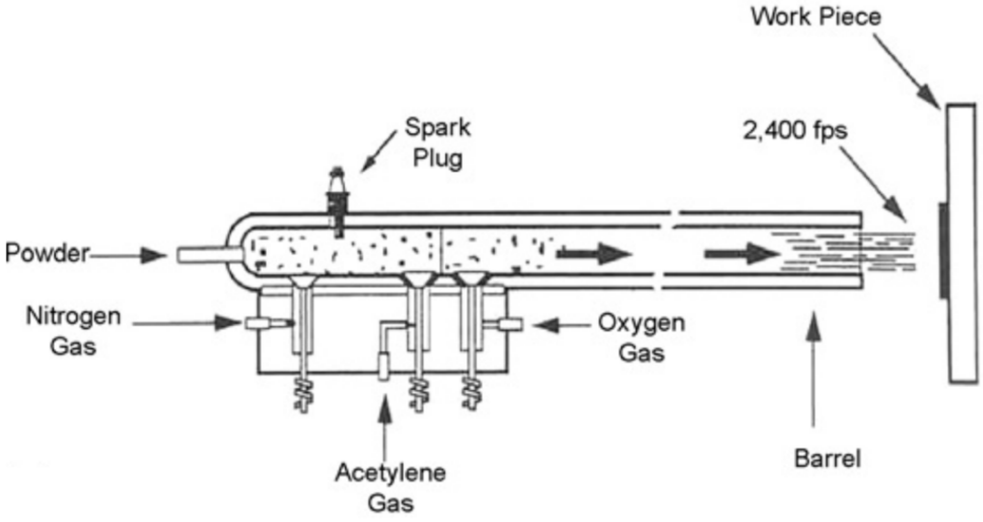


Fig. 1.2: Detonation Gun Spray Coating Process [8]

1.5.2 Process Parameters

This process depends on the following parameters:

a) Detonation wave

Detonation wave involves three aspects: composition of working gases, geometry of gun barrel and rate of firing. From the literature, it has been found that fuel gas that might be hydrogen or hydrocarbons such as butane, acetylene and propane should be used along with oxygen. Using 45% of acetylene with oxygen gave maximum temperature of 4500K. The detonation wave has been reported to reach velocity of 2930m/s, particles usually reach velocity of 750m/s and 1000m/s in super detonation gun. The length of barrel is usually in the range of 450-1350mm and diameter corresponds to 21-25mm. The rate of firing should be in the range of 1 to 15 Hz.

b) Powder

Detonation gun process usually uses tungsten carbide and chrome carbide powders possessing a variety of binders. Aluminium oxide, aluminium-titanium, aluminium-silicon carbide and cupper-aluminium coatings can also be used. The particle size should be in the range of 5 to 60μm. The most commonly used powders used this process are that of composites with reinforcements of carbide.

c) Powder injection

Powder is injected at a feed rate of 16-50g/min and oxygen or nitrogen should only be used as carrier gases.

d) Processing parameters

Spray distance for this D-gun coating technique has been reported to be 100mm and spray atmosphere should be air [9] .

1.5.3 Coating Properties

The porosity of detonation gun coatings is very small and has been reported to be 0.5% for tungsten carbide-cobalt coatings and around 2% for aluminium oxide coatings. The tensile bond strength for tungsten carbide-cobalt coatings is 83MPa and 70MPa for aluminium oxide coatings. The thickness of D-gun coatings has been reported to be less than 300μm. High bond strength and hardness can be obtained by this coating process. Coatings with low oxygen content and medium surface roughness are produced due to very high temperature and velocity attained within this process.

1.5.4 Advantages of the process

D-gun coating technique involves the following advantages:

a) Wide range of materials can be used in this process involving cemented carbides with metal matrix composition and other kinds of ceramic powders can also be used.
b) Varying coating thickness can be achieved
c) Minimal porosity less than 2%
d) Adhesion of coatings is greater than 70MPa
e) Roughness of 0.01Ra can be achieved after finishing

1.5.5 Disadvantages of the process

D-gun coating technique involves the following disadvantages:

a) Detonation guns are large in size and produce noise during operation
b) The coatings cannot be applied to malleable and expanding components
c) Under pinpoint loading, coatings tend to damage
d) It has to be placed at a specified location and requires sound proof room

1.5.6 Applications of the process

Detonation gun technique results in formation of hard and long-lasting coatings that are applicable for:

a) Machinery components involving bearings, shafts and bushes.

b) Components of engine along with blades of stator and rotor used in aviation industry
c) Gate valves, shut off valves and bushings used in oil industries
d) Electronics and radio industries
e) Tubular drills used in tooling industries
f) Shipbuilding industries
g) Plug and snap gauges plated by D-gun technique

Chapter 2

LITERATURE REVIEW

2.1 LITERATURE REVIEW

The first step was to analyze the research in the form of literature review. A wide range of literature was extracted from journals based on detonation gun coating technique applied on boiler steels and superalloys. From detonation gun coating technique process, the corrosion resistance at high temperature can be studied for materials used in coal fired boilers.

Kaur et. al. (2011) studied about the corrosion behavior of Cr_3C_2-NiCr coating applied on T22 boiler steel using D-gun coating process. The experiments were performed in boiler environment by hanging specimens in Super Thermal Power Plant of Ropar, Punjab at a temperature of 700±10°C. The XRD analysis of the coated samples had shown the formation of chromium, Cr_2O_3 and CrNi phases in strong intensity and Ni phase in medium intensity. From XRD analysis of coated and uncoated steel, it was found that coated steel had shown the presence of oxides like Cr_3C_2, Cr_2O_3, Cr_7C_3 as strong intensity phases, CrNi as medium intensity phase and Cr_3Ni_2 as a phase with weak intensity. Fe_2O_3, Al_2O_3 were found as dominant phases in uncoated specimen. The coating had successfully adhered to the surface of substrate and resulted in reduced thickness loss in comparison to the uncoated one (fig. 2.1). Chromium oxide rich oxide has been responsible for better corrosion resistance offered by coated specimens [10].

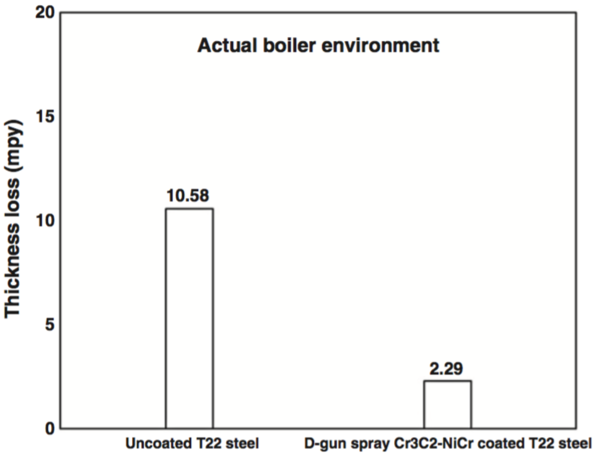


Fig. 2.1: Thickness loss obtained for coated and uncoated specimens [10]

Kaushal et. al. (2012) discussed about the performance of Ni-20Cr coating applied on TP347H with the help of detonation gun coating process. The authors preferred D-gun sprayed coating process because of the high density coating with good surface finish, adherence and corrosion resistance. The coated and uncoated specimens were hung in Super Thermal Power Plant, Ropar, Punjab at 700°C. From SEM images of coating, the coating was found to be of 1.3% porosity. The results have shown that the successful application of coating on steel helped in improvement of substrate properties at high temperature. The uncoated specimen had shown intense thickness loss which was lowered by 53% due to the application of coating. After exposure to boiler environment, the coated specimen witnessed the formation of chromium rich oxide layer that provided high corrosion and oxidation resistance [11].

Kaushal et. al. (2013) investigated about the corrosion behavior of Ni-20Cr coated T22 steel at high temperature. The coating was done using cold spray, D-gun and HVOF coating processes. The experiments were performed in Na_2SO_4-60%V_2O_5 environment and 50 oxidation cycles were performed for 50 hours. It was found that all three coating processes were successful in adherence of coating particles to the substrate and reduced corrosion rate of T22 boiler steel. Mass gain was observed to be minimum for D-gun spray followed by HVOF spray and cold spray. Out of all three processes, D-gun spray proved to be the best due to NiO, Cr_2O_3 along with $NiCr_2O_4$ oxides that provided best corrosion resistance to boiler steel [12].

Mishra et. al. (2014) researched on the high temperature corrosion resistance of Stellite 6 and stellite-21 coatings applied on SAE 431 steel used as boiler tubes at 900°C. The coatings were deposited by detonation gun technique. The hot corrosion experiments were performed in Na_2SO_4-82%$Fe_2(SO_4)_3$ for 50 oxidation cycles. From results, it was found that both coatings were successfully applied and provided corrosion resistance. During the first 10 cycles, the weight gain was negligible and increased thereafter. Higher weight gain was obtained for uncoated and stellite-21 coated specimens and lowest weight gain was obtained for Stellite 6 coated specimen. Higher weight gain in stellite-21 coated specimen might be due to formation of cracks along the edges. Stellite 6 coated steel provided better corrosion resistance due to Cr_3O_4 and $CoCr_2O_4$ oxides [13].

Mishra et. al. (2014) studied about the corrosion behavior of D-gun sprayed Al_2O_3-40TiO_2

coated nickel based superalloys. The corrosion studies were performed in Na_2SO_4-82%$Fe_2(SO_4)_3$ molten salt environment at 900°C. AE 435 and Superni-718 have been used as superalloys for corrosion studies as nickel based superalloys possess better mechanical strength along with creep resistant at high temperatures in molten salt but lack the property of corrosion resistance. It was observed that coated superalloys provided better corrosion resistance than bare samples. Due to presence of different phases in Superni-718, all imposing severe strains on surface layer resulted in spalling and cracking of oxides. So, AE 435 provided better corrosion resistance and improved corrosion resistance of superalloy by 36.8% [14].

Sharma et. al. (2014) studied about the corrosion behavior of Cr_3C_2-NiCr coated T22 boiler steel deposited by detonation gun coating process. The corrosion studies were performed by hanging of specimens at coal boiler at 900°C. Chromium and nickel were uniformly distributed in the coating and oxygen might have entered inside the coating during the spraying process. Reduced corrosion rate was observed for coated specimens than bare specimens. Due to protective oxides containing Cr, Ni and oxygen, the corrosion resistance of substrate increased and provided protection to boiler steel against corrosive species [15].

Jain et. al. (2014) researched about the corrosion resistance of Stellite 6 coated SA210 Grade 1 in coal boiler at 900°C. The coating was done using detonation gun spray process. From the observations, it was found that protection against corrosion was provided by coated boiler steel in comparison to the uncoated specimen. The corrosion resistance of steel was improved due to the presence of thick bands of Cr, Co and Mn in the oxide layer of coated specimens. These thick bands prevent oxygen from passing through the substrate, resisting corrosion after 10 corrosion cycles at high temperature. Weight gain was observed in uncoated specimens along with spalling and cracks [16].

Kamal et. al. (2015) studied about the corrosion behavior of Superfer 800H coated with Cr_3C_2-NiCr, NiCoCrAlYTa and NiCrAlY+0.4wt%CeO_2 by detonation gun coating process. The corrosion studies were performed in coal boiler at 900°C for 10 cycles, each of 100h duration. All three coatings were able to provide better corrosion resistance than uncoated specimens. Out of all the coatings, Cr_3C_2-NiCr proved to be the best coating providing highest corrosion resistance due to the formation of continuous and thin corrosion resistant layer of chromium oxide. Partial oxidation of other two coatings and deposition of ash have been responsible for reduced corrosion resistance [17].

Goyal et. al. (2017) discussed on the corrosion resistance of Ni-20Cr coated T11 steel by detonation gun coating process. The experiments were performed in Na_2SO_4-60%V_2O_5 at 900°C for 50 cycles of one-hour each. It was observed that XRD analysis of coated samples have shown the formation of chromium nickel and nickel carbide before exposing coated specimens to molten salt environment. Successful deposition of coating was obtained on boiler steel. The uncoated substrate had shown severe spalling of oxide scales. EDS and XRD analyses of coated T11 indicated Fe_3O_4, NiO and SiC as major phases. The corrosion resistance was better in the case of coated substrate due to the formation of protective oxides [18].

Rani et. al. (2017) studied about the corrosion behavior of ASTM-SA210-A1 coated with Cr_2O_3-75%Al_2O_3 by detonation gun spray technique. The corrosion studies were performed in Na_2SO_4-60%V_2O_5 at 900°C for 50 cycles. Detonation gun coating technique helped in deposition of uniform, adherent coating with dense microstructure. Bare samples had undergone intense spalling of oxide scales with deep cracks and higher corrosion rate. After application of coating, no spalling was observed with intact oxide scales that helped in reduction of corrosion rate by 95% [19].

Rani et. al. (2017) investigated on the corrosion resistance of Cr_2O_3-50%Al_2O_3 coated T22 boiler steel and Superfer 800H superalloy. The coating was performed using detonation gun coating technique and corrosion studies were done for 50 cycles of one-hour duration in Na_2SO_4-60%V_2O_5 at 900°C. Due to non-protective ferrous oxide scales, both substrates suffered from intensive spallation that led to removal of oxide scales. After coating, both substrates had shown adherence of the coating and oxides of chromium and aluminium contributed to improved corrosion resistance of the substrates. The corrosion rate of T22 boiler steel was reduced by 97% and that of superalloy was reduced by 19% due to the application of the coating [20].

Sharma et. al. (2019) studied about the behavior of Stellite 6 coated T22 boiler steel in coal boiler for 10 cycles of 100 hours each. The coating process preferred was that of detonation gun coating technique. From the results, it was found that thick bands of Cr, Co and Mn have been responsible for prevention of oxygen contact with the substrate. Severe cracks and spalling were observed on uncoated substrate along with higher weight gain due to ash deposition. Thus, coated substrate helped in enhancing corrosion resistance of the substrate [21].

Mittal et. al. (2019) studied about the hot corrosion behavior of stellite-21 and NiCr coated T91 boiler steel. The coating was performed by detonation gun spray and experiments were performed in Na_2SO_4-60%V_2O_5 at 900°C for 100 cycles of one-hour each. It was found that both coatings were successfully deposited on the substrate with no formation of cracks. The specimens' weight appeared to increase with increasing exposure temperature. Also, it was observed that stellite-21 was not able to fully protect base metal from corrosive oxides due to the formation of cracks on thermal exposure. Higher amounts of chromium were observed in Ni-Cr coating that might have led to the generation of rich Cr_2O_3layer. So, Ni-Cr provided better corrosion resistance and is more suitable for T91 boiler steel than stellite-21 coating in this type of environment [22].

Thakare et. al. (2019) investigated about the corrosion resistant properties of detonation gun coated P91 steel by subjecting it to boiler environment at 650°C for 3000 hours. The coating that was applied was 75Cr_3C_2-25NiCr. It was found that continuous uniform coating of thickness 140±5µm was obtained. The coating had shown the presence of chromium and nickel oxides with spinels of nickel and iron. The diffusion tendency of iron decreased with increase in exposure time. Maximum XRD peaks were observed for Cr_3C_2 that might have been responsible for improving corrosion resistance of the steel substrate [23].

Mittal et. al. (2020) examined the corrosion resistance properties of coated T11 boiler tubes in Na_2SO_4-60%V_2O_5 at 900°C and analysis was done for 50 cycles. The coatings preferred for this examination were Cr_3C_2-NiCr and Cr_2O_3 that were deposited on the substrate by D-Gun technique. The uncoated samples suffered from intense spallation and cracks. Cr_2O_3 coating also resulted in failure of specimen as the coating detached from the specimen during the studies. In the case of Cr_3C_2-NiCr coating, Cr_2O_3 oxide was formed as major phase and NiO oxide was formed as minor phase. Thus, it was concluded that Cr_3C_2-NiCr coating gave better corrosion resistance than Cr_2O_3 coating [24].

Saroop et. al. (2020) investigated about the corrosion resistance of WC-12%Co coated SS304 and SS314. The tungsten carbide cobalt coating was deposited by detonation gun coating process and corrosion studies were done in actual boiler at 1100°C. It was observed that coating sacrificially protected the substrates from corrosive species. It was found that SS316 suffered from reduced corrosion than SS304. In bare samples, Fe_2O_3 oxide layer was formed which was non protective towards corrosive species [25].

Sundaresan et. al. (2020) discussed about the hot corrosion behavior of NiCoCrAlY coated T91 boiler steel. The coating was deposited by detonation gun and plasma spray processes. The experiments were performed in coal ash environment of Na_2SO_4, Fe_2O_3 and K_2SO_4 at 650°C. It was observed that coating formed by detonation gun coating technique provided better corrosion resistance than that deposited by plasma spray technique. The formation of protective NiO and $NiCr_2O_4$ oxides was observed in both coating processes but detonation gun technique was more successful in improving corrosion resistance due to high strength bonding, minimum porosity and dense microstructure [26].

2.2 LITERATURE GAP & RESEARCH OBJECTIVE

From the literature, it can be found that many studies have been done based on detonation gun coating process for coating of materials. Research has been done on coating of substrates like T22, T91 and superalloy Superfer 800H. Many studies have also been done on chromium carbide, nickel chromium, chromium oxide coatings along with the studies on their corrosion resistance. However, studies have not been done on corrosion behavior of detonation gun deposited Stellite 6 and Stellite 21 coatings on superalloys at high temperatures.

Thus, the present investigation is mainly focused on studying the hot corrosion behavior of detonation gun sprayed Stellite 6 coated Superni 600 in gas turbine environment of Na_2SO_4-60%V_2O_5.

Chapter 3

EXPERIMENTAL DETAILS

3.1 SUBSTRATE MATERIAL

For the studies, nickel based Superni 600 has been used with chemical composition in table 3.1. The substrate material was obtained from Mishra Dhatu Nigam Ltd., Hyderabad, in the form of rectangular sheets.

Table 3.1: Chemical composition of substrate material

Substrate material	Fe	Cr	C	Ni	Mn	Al	Si
Superni 600	10	15.5	0.2	Bal.	0.5	0.6	-

3.2 COATING POWDER

Stellite 6, also called as CoCrWC alloy is frequently used alloy belonging to Co-based group. It contains 27-32% chromium, 0.9-1.4% carbon, 4-6% tungsten with addition of elements like iron, nickel, cobalt, manganese, silicon and molybdenum in balance amounts. It has an high corrosion and wear resistance over a wide temperature range. Wear resistance is mainly due to the presence of hard carbide phase in a marix of CoCr alloy. Spherical nature of stellite 6 coating powder can be observed in SEM micrograph (fig. 3.1).

It has high corrosion and oxidation resistance upto 1095°C. These Stellite 6 properties are used for surface coating that experience high stresses. Stellite 6 can be deposited on the surface to be coated by thermal spray coating processes. The corrosion resistance behaviour of D-Gun sprayed Stellite 6 coating were evaluated in this study.

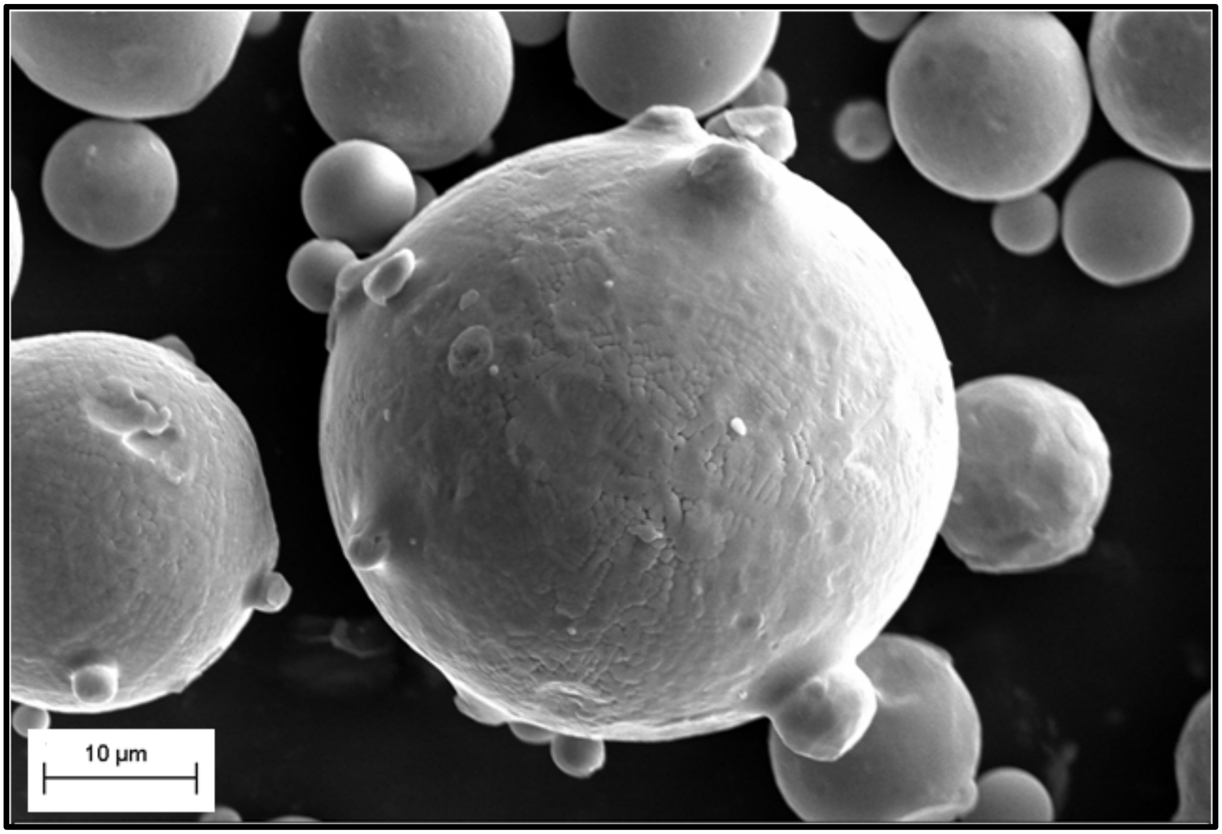


Fig. 3.1: SEM micrograph of Stellite 6 coating powder

3.3 COATING PROCESS

The samples with dimensions of 20mm×15mm×5mm (approximately) were cut from the sheet and emery sheets of 300, 400, 600 and 800 grades were used for polishing. The samples were degreased with acetone and alumina powders (grit 20) were used for grit blasting to roughen the surface of specimens prior to the deposition of the Stellite 6 coatings by D-Gun technique. Oxygen and acetylene were mixed in appropriate amounts to form a combustible mixture that is fed through barrel that is closed at one end. To prevent backfiring of mixture, nitrogen gas is flushed through the barrel for covering inlets of gas. Then, a specific amount of powdered coating is fed into the combustion chamber. Spark plug ignites for combustion of mixture. Shock waves of high pressure are formed during combustion of mixture that flow across the gas stream. The temperature of gas stream can rise to 4000°C and shock waves' velocity reaching 3500m/sec. Hot gases flow through the barrel at high velocity, plasticizing particles that accelerated at a velocity of 1200m/sec. These particles then flow out of the gun and get deposited on the surface. Dense and strong coating is formed due to kinetic energy possessed by powdered coating particles.

Parameters like powder particle size, ratio of combustion gases and distance between barrel and substrate determine the thickness of coating that can be developed on substrate in one shot. On the basis of coating thickness requirement, 1 to 15 shots of detonation gun coating process can be repeated within a second. Nitrogen gas is then flushed across the chamber that helps in removal of hot powdered particles from the gun barrel. The following parameters were set during the coating process (Table 3.2):

Table 3.2: Value of parameters for coating process

Parameters	**Value for D-gun coating process**
Ratio of flow rate of oxygen to acetylene	1:1.21
Flow rate of nitrogen gas	0.96 m^3/h
Frequency	3 shots per sec
Spot diameter	20 mm
Substrate and nozzle distance	165 mm
Flowrate of coating material	1 to 2 gram per shot

3.4 COATING THICKNESS MEASUREMENT

Back scattered electron images were obtained at the cross-section of coated samples from which coating thickness was found out. Fig. 3.1 shows the back scattered electron image of the coating and coating thickness was approximately found to be 350μm.

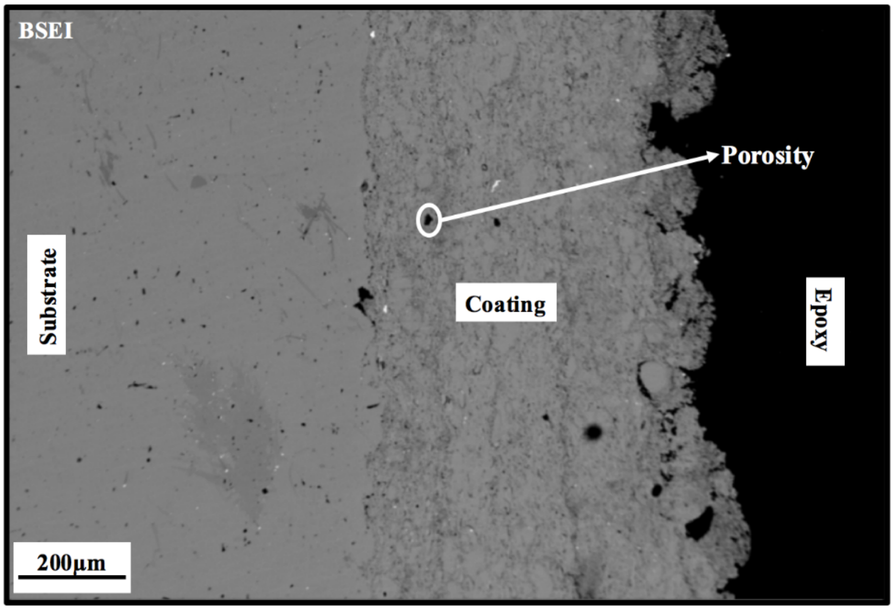


Fig. 3.2: Cross sectional analysis of D-gun sprayed Stellite 6 coated Superni 600

3.5 EXPERIMENTAL PROCESS

Hot corrosion studies under cyclic conditions were performed in simulated gas turbine environment of Na_2SO_4–60%V_2O_5 up to 25 cycles for the D-gun sprayed Stellite 6 on Superni 600 coated samples. The coated and bare samples (20mmx15mmx5mm)were preheated at 250°C for 1 hour. A uniform coating with thickness of 3–5 mg/cm^2 of Na_2SO_4–60%V_2O_5 was applied with the help of camel hairbrush on the already heated samples. Preheating helps in proper adhesion of salt coating to samples. All samples were kept in alumina boats and then placed inside the silicon carbide tube furnace. Each cycle consisted of 1 hour of heating at temperature of 900°C which was followed by 20 minutes of cooling at normal room temperature. Electronic balance with a sensitivity of 1 mg was used for measurement of weight change of samples. Weight change determination also included spalled scales. Visual observation at the end of each cycle was also performed. Weight change measurements were helpful in determination of nature of corrosion. After doing the hot corrosion studies, the samples (bare and coated) were analysed using SEM/EDAX, XRD and X ray mapping techniques.

Chapter 4

RESULTS

4.1 VISUAL ANALYSIS

With subsequent cycles, various changes were observed on the surfaces of bare and coated Superni 600 samples. Bare Superni 600 when subjected to hot corrosion at 900ºC has shown appearance of small amount of lustrous grey oxide on surface after the first cycle. Subsequently in fifth cycle this lustrous grey oxide had spread along with the formation of light grey coloured oxide. After thirteenth cycle this lustrous oxide had spread all over the surface and also light grey colour scale had increased significantly on the surface. After twentieth cycle dark grey oxide had appeared on surface. At the end of the 25 cycles, dark and light grey coloured oxide was present along with lustrous scale on it (fig. 4.1(a)). In the case of coated superalloy, colour change due to oxide formation was observed at surface top at 900ºC during corrosion. Oxide scales might have formed due to gaseous reaction between oxygen and metal in corrosive environment. The oxide layer in first cycle had green colour on the coated surface with shiny grey lustre which remained same till 20th cycle. Subsequently in 25th cycle, the oxide colour transformed to dark grey (fig. 4.1(b)).

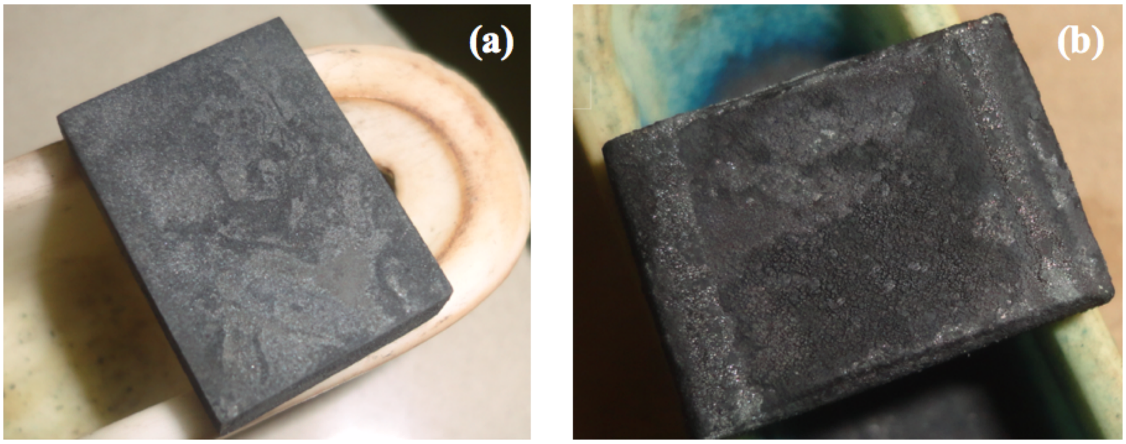


Fig. 4.1: Visual analysis for (a) bare Superni 600 (b) coated Superni 600

4.2 WEIGHT CHANGE ANALYSIS

During hot corrosion studies, the data measured related to weight change after every cycle is depicted in the form of a graph in fig. 4.2, 4.3. Weight change consists of weight gain that might be due to scale formation and weight loss that might be due to scale spalling or their dissolution along with other corrosion products. The concluding weight gain after 25 cycles for coated superalloy was found to be 71.5 mg and that for bare superalloy was found to be 5.26 mg. K_p values for bare and coated superalloys were observed to be 2.52×10^{-12} $g^2cm^{-4}s^{-1}$ and 5.08×10^{-10} $g^2cm^{-4}s^{-1}$ respectively.

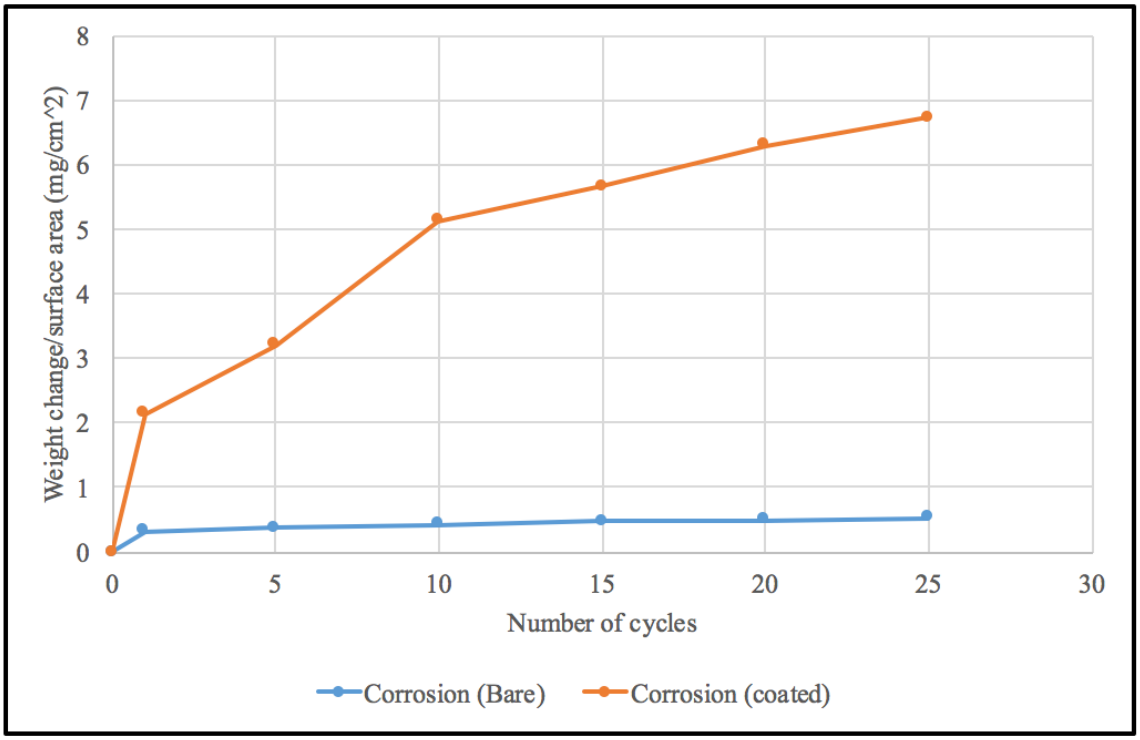


Fig. 4.2: Plot for Weight change/surface area vs number of cycles for bare and coated superalloy

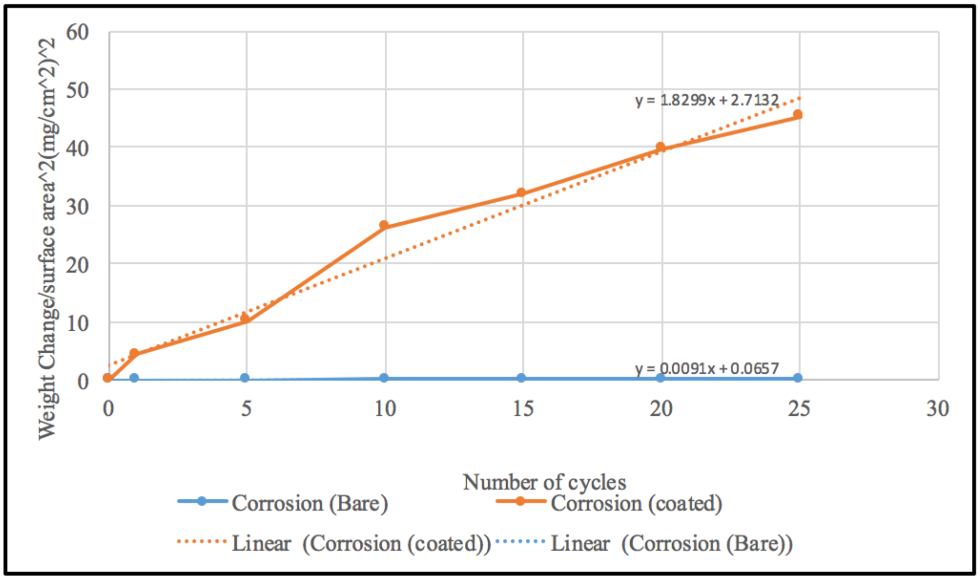


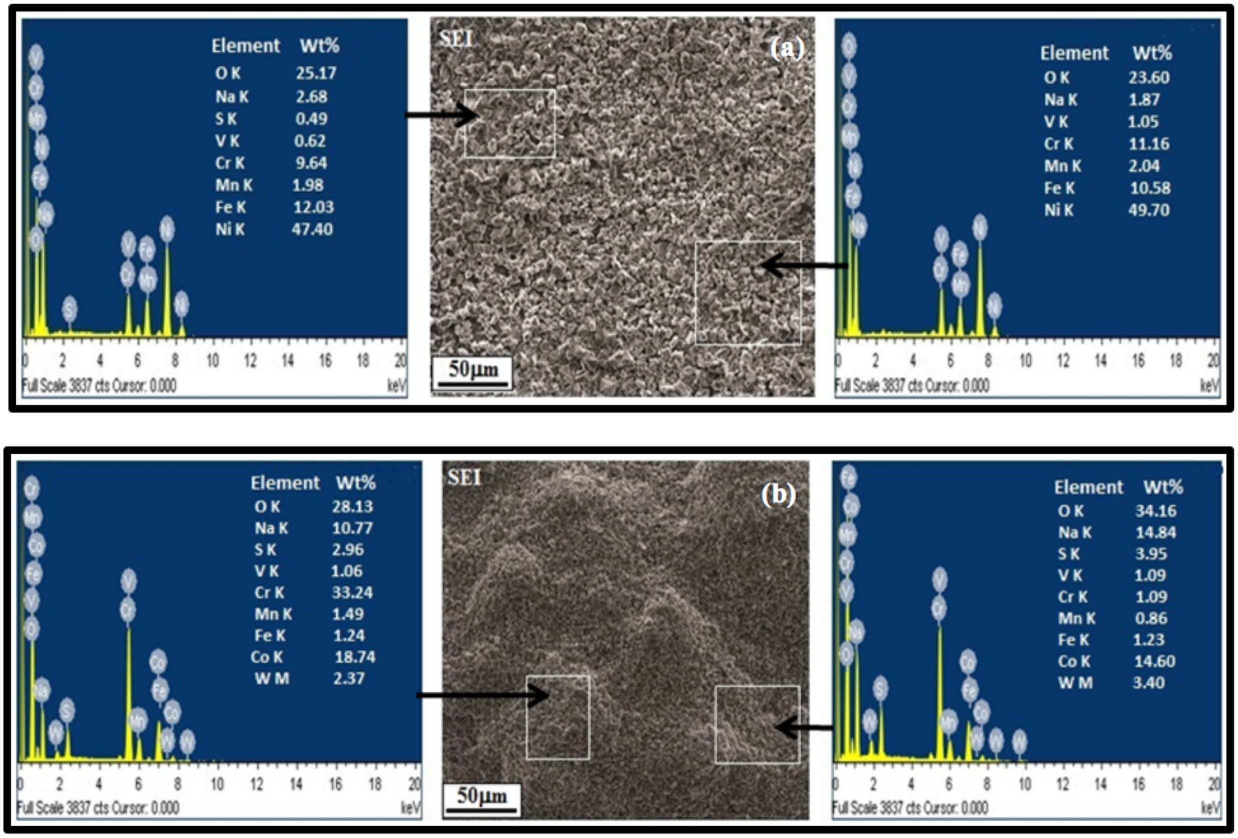
Fig. 4.3: Plot for weight change/surface area^2 vs number of cycles for bare and coated superalloy

4.3 SEM/EDS ANALYSIS

The surface morphology of hot corroded specimens after 25 cycles were studied using Field Emission Scanning Electron Microscopy (JSM 7610FPlus Schottky Field Emission SEM) with resolution of 0.8nm for accelerating voltage 15KV. Secondary electron images (SEI) were recorded for surface analysis. The specimens were scanned under the microscope and the critical areas of interest were analyzed in order to assess the compactness of the oxide, voids, cracks and the structure of the oxide formed. EDS analysis for the specimens was done using EDAX Element-C2B. The EDS software indicates the oxides present at a point along with their compositions (weight %). EDS analysis was done at the various locations to record the compositions of elements present in these areas. The SEM/EDS micrographs of bare Superni 600 and Stellite 6 coated Superni 600 after hot corrosion have been shown in fig. 4.4 (a) and (b) respectively.

From SEM analysis, it can be observed that corrosion of bare superalloy resulted in formation of oxides with high proportion of melted particles but high amount of porosity can be observed between grains that might act as a path for corrosive species to reach the substrate and result in severe corrosion attack. In the case of coated Superni 600, dense coating with no cracks has been achieved with near spherical surface morphology. Some pores and inclusions can be observed that are much lesser than those observed in SEM images of bare specimen. The grain size obtained for coated specimen appears to be lower with some proportion of unmelted particles.

From EDS spectrum, it can be observed that in case of bare Superni 600 sample which had undergone hot corrosion tests, high content of elements like Oxygen (O), Iron (Fe), Nickel (Ni) and chromium (Cr) are present. Apart from these elements, some amount of Vanadium (V), Sulphur (S) and Manganese (Mn) are also present. In the case of Stellite 6 coated Superni 600 sample which had undergone hot corrosion tests, higher amounts of elements like Oxygen (O), Chromium (Cr), Sodium (Na) and Cobalt (Co) are present. Some amount of Tungsten (W), Manganese (Mn), Sulphur (S) and Vanadium (V) are also present as observed from EDS analysis.



Element	Wt%
O K	25.17
Na K	2.68
S K	0.49
V K	0.62
Cr K	9.64
Mn K	1.98
Fe K	12.03
Ni K	47.40

Element	Wt%
O K	23.60
Na K	1.87
V K	1.05
Cr K	11.16
Mn K	2.04
Fe K	10.58
Ni K	49.70

Element	Wt%
O K	28.13
Na K	10.77
S K	2.96
V K	1.06
Cr K	33.24
Mn K	1.49
Fe K	1.24
Co K	18.74
W M	2.37

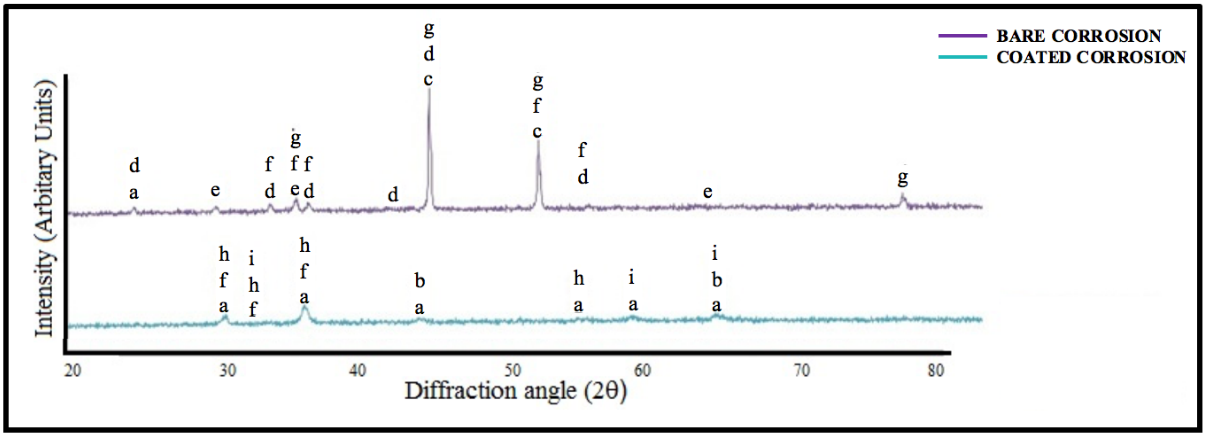
Element	Wt%
O K	34.16
Na K	14.84
S K	3.95
V K	1.09
Cr K	1.09
Mn K	0.86
Fe K	1.23
Co K	14.60
W M	3.40

Fig. 4.4: SEM images with EDS spectrum of hot corroded (a) bare Superni 600 sample (b) Stellite 6 coated Superni 600 sample

4.4 XRD ANALYSIS

For identification of different phases formed in the oxides scales of hot corroded specimens after 25 cycles of exposure simulated gas turbine environment, Bruker D8 ADVANCE

Diffractometer has been used for XRD analysis. It has energy resolution of less than 380 eV and detection modes of 0D, 1D and 2D. Specimens have been scanned in the range of 20° to 90°. The XRD patterns of hot corroded bare Superni 600 and Stellite 6 coated sample after 25 cycles are shown in Fig. 4.5. XRD patterns of bare Superni 600 hot corroded sample have shown the presence of Cr_2O_3, Ni, $NiCr_2O_4$, $NaVO_3$ and $NiCrO_4$ as main phases. Very weak phase of $NiMn_2O_4$ is also observed. While in the case of Stellite 6 coated hot corroded sample, formation of NiO, NiV_2O_6, $CoMnO_3$, $NiCr_2O_4$ and $NaVO_3$ phases can be observed.



a- $NiCr_2O_4$; b- NiO; c- Ni; d- Cr_2O_3; e- $NiMn_2O_4$; f- $NaVO_3$; g- $NiCrO_4$; h- NiV_2O_6; i- $CoMnO_3$

Fig. 4.5: XRD analysis for bare and coated Superni 600 after corrosion studies

4.5 X-RAY MAPPING

The X-ray mapping images obtained for bare Superni 600 exposed to Na_2SO_4–60%V_2O_5 environment for 25 cycles have been shown in Fig. 4.6. From X-ray images of uncoated specimen, it can be observed that the oxides formed on the surface basically consists of oxygen, nickel and chromium in large amounts. Iron and manganese have shown their presence in small amounts. The X-ray mapping images obtained for stellite 6 coated Superni 600 exposed to Na_2SO_4–60%V_2O_5 environment for 25 cycles have been shown in Fig. 4.7. From X-ray images of coated specimen, it can be observed that adherent and dense oxide scale has been formed on the substrate. Presence if huge amounts of oxygen, cobalt, tungsten, chromium and nickel can be observed. Iron and vanadium are present in lesser amounts in comparison to other elements.

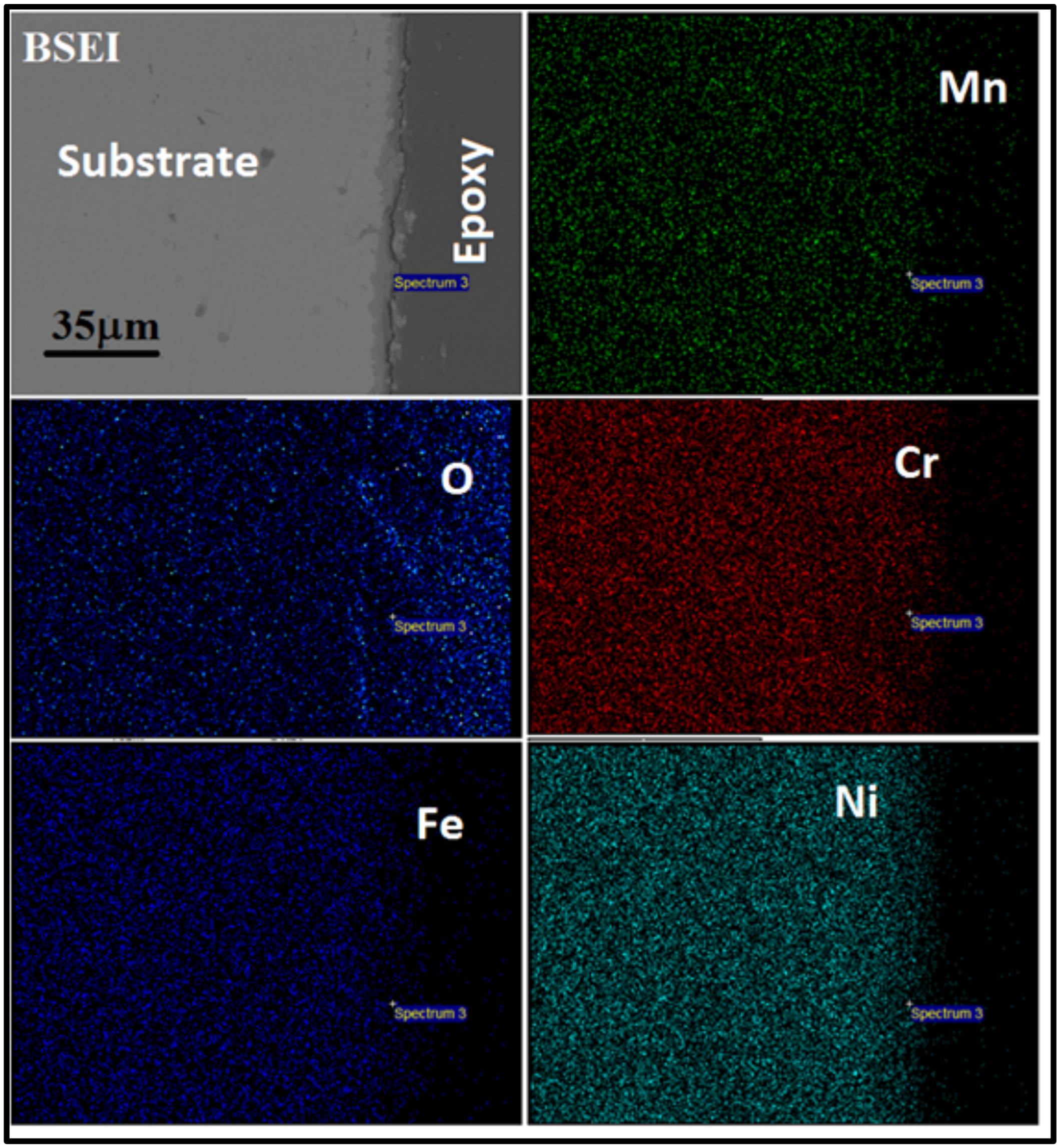


Fig. 4.6: X-ray mapping of bare Superni 600 exposed to Na_2SO_4–60%V_2O_5 environment for 25 cycles

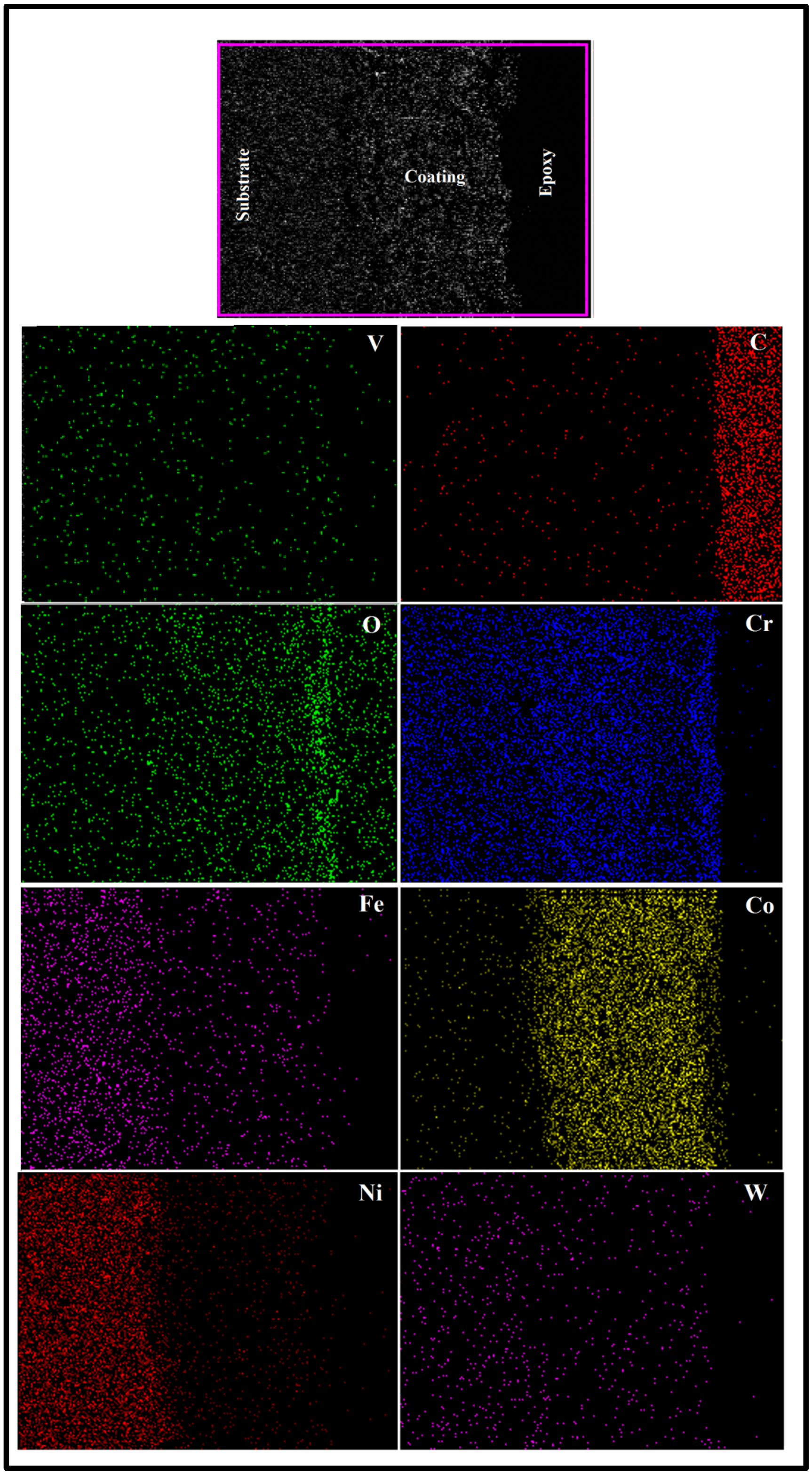


Fig. 4.7: X-ray mapping of Stellite 6 coated Superni 600 exposed to Na_2SO_4–60%V_2O_5 environment for 25 cycles

Chapter 5

DISCUSSIONS

From visual analysis of hot corrosion studies of bare and coated Superni 600 under molten salt, no spallation of oxides was observed throughout the experiment. Color of oxide scales formed on both the specimens was dark grey. Stellite 6 coating on superalloy protected the specimen from corrosion till the end of 25 cycles. From the literature, it has been observed that coating of Stellite 6 on superalloys results in the formation of dark grey oxide scale as was observed by Singh et. al. [27] after hot corrosion studies of plasma-sprayed Stellite 6 coating on superalloys.
From weight change analysis, it can be observed that bare and Stellite 6 coated Superni 600 followed parabolic behavior after hot corrosion studies. Higher weight gain in the case of coated superalloy can be observed in comparison to that of bare superalloy. Higher weight gain for stellite 6 coated nickel based superalloy was also observed by Sidhu et. al. [28] due to the formation of oxides that block corrosive species from attacking open pores of coated samples. The higher weight gain for coated specimen might be due to the formation of oxides at open pores due to entering of corrosive species. Due to this, the oxides might have completely blocked splat boundaries and pores and acted as barriers of diffusion that prevented further penetration of corrosive species [28]. From k_p values, it has been found that k_p value is lower for bare sample than the value obtained for coated sample. Higher k_p value is an indication of degradation of coating oxides due to corrosion with subsequent cycles.

From SEM images, it can be observed that bare superalloy consisted of high porosity that may allow the entry of corrosive species inside the substrate in the later cycles. Whereas, in the case of Stellite 6 coated superalloy, the porosity was very less due to the deposition of dense coating by detonation gun technique. The lower porosity helps in preventing salt species from reaching the substrate causing severe corrosion of the superalloy.

From EDS images, presence of oxygen, chromium, nickel and iron in high amounts and manganese, vanadium and sulphur in small amounts can be seen for bare superalloy. This depicts the formation of nickel and chromium oxides. Oxygen might have started reacting with these elements due to the affinity of chromium to combine with oxygen. The oxide so formed have higher melting point than 900˚C. This is the reason why vanadium is still present on the surface.

In the case of coated superalloy, higher amounts of oxygen, cobalt, chromium and sodium can be found with lower amounts of sulphur, vanadium, tungsten and manganese. From studies, it has been observed that cobalt, on reaction with oxygen and chromium, results in formation of cobalt oxides that play a major role in enhancing corrosion resistance of the coated superalloy [29]. Sassatelli et. al. [30] also obtained presence of cobalt, chromium, tungsten in large amounts and iron in small amounts on coating of AISI 304 stainless steel with stellite 6 by HVOF process. The coating obtained was dense and homogenous with low amount of porosity.

From XRD analysis, it can be observed that Cr_2O_3, Ni, $NiCr_2O_4$, $NaVO_3$ and $NiCrO_4$ as main phases and very weak phase of $NiMn_2O_4$ is also observed in the case of hot corrosion of bare superalloy. Protective oxides of Cr_2O_3, $NiCr_2O_4$ have been formed are mainly responsible for the good corrosion resistance of bare substrate. However, the oxide was porous in nature because of the reactions of sulphur, sodium and vanadium with the protective oxides that might have allowed the attack of corrosive species on the substrate. In the case of coated Superni 600, NiO, NiV_2O_6, $CoMnO_3$, $NiCr_2O_4$ and $NaVO_3$ phases can be observed. Nickel and vanadium reacts with each other to form NiV_2O_6 that acted as a protective oxide. Layers of nickel oxide along with $NiCr_2O_4$ were also responsible for providing protection to the substrate against hot corrosion. Oxides of cobalt along with manganese acted as protective oxides against corrosion [29]. Sidhu et. al.[31] also confirmed the formation of cobalt rich oxides at splat boundaries on HVOF sprayed coating of Stellite 6 on Superfer 800H. These cobalt based oxides acted as barriers and provided corrosion resistance to Fe based superalloy. In accordance with the studies of Singh et. al.[27], the protection against corrosion for plasma sprayed Stellite 6 coating on Superni 601 was provided by formation of cobalt and chromium oxides, along with spinels contaning cobalt and chromium.

From X-ray mapping images, it can be observed that oxygen, nickel and chromium were present in large amounts in oxide scales and lower amounts of iron and manganese have also been found in the oxide scales. This has also been confirmed by XRD images that have shown the presence of oxides of nickel and chromium in larger amounts in the form of Cr_2O_3, $NiCr_2O_4$ and $NiCrO_4$ oxides and weak phases of oxides of nickel and manganese were obtained. The presence of oxides containing elements like nickel, chromium and oxygen have been responsible for providing corrosion resistance to bare sample. In the case of stellite 6 coated Superni 600, mapping images have shown the presence of oxygen, nickel, chromium, tungsten and cobalt in larger amounts and iron and vanadium in lower amounts, which can be also be confirmed from XRD images that have shown the formation of NiO, NiV_2O_6, $CoMnO_3$, $NiCr_2O_4$ oxides. Oxygen reacts with chromium and nickel due to its high affinity towards these elements. The formation of protective oxides of nickel and chromium along with oxides of cobalt with manganese provided corrosion resistance to the substrate.

It can be noticed that with increasing exposure of coated superalloys, the coating does not delaminate which shows the high bonding strength present between substrate and coating. This indicates that the technique of detonation gun spray has been successful in deposition of coating. This also depicts that the coating and substrate is compatible in terms of thermal expansion which can leads to delamination of coating at this temperature. Although the k_p value for coated superalloy is high but the formation of protective cobalt oxides with chromium and manganese have been responsible for improving corrosion resistance thereby results in enhancing the life of the bare alloy. Higher k_p value suggests that the coated sample is showing lower corrosion resistance then the bare specimen till 25th cycle. From the literature, it has been observed that bare Superni 600 starts degrading after 100 cycles [32]. It might be possible that in present environment, the superalloy shows degradation after few more cycles. So, if coating is applied before the exposure of bare substrate, then the degradation of coating will take place. This is highly beneficial for gas turbine environment as it can increase the life of the super alloy. Thus, with the deposition of Stellite 6 coating on Superni 600, the life of substrate can be enhanced substantially as it does not delaminate from the substrate and also shows parabolic behaviour. Parabolic behaviour of coating indicates that the oxide so formed became protective after few cycles.

Chapter 6

CONCLUSION AND FUTURE SCOPE

6.1 CONCLUSION

The following conclusions can be made from the hot corrosion studies:

1. Stellite 6 coating has been successfully sprayed on Superni 600 using Detonation – gun technique, resulting in high strength of bond between substrate and coating.
2. The coating was dense, uniform and no formation of cracks during the hot corrosion studies.
3. Bare Superni 600 corroded in Na_2SO_4-60%V_2O_5 at 900˚C for 25 cycles had porous scale and showed weight gain which was accompanied with minor spallation whereas corroded Stellite 6 coating in the similar condition showed dense scale and overall weight gain with no spalling.
4. Nickel and vanadium reacted with each other to form NiV_2O_6 that acted as a protective oxide. Layers of nickel oxide along with $NiCr_2O_4$ were also responsible for providing protection to the substrate against hot corrosion. Oxides of cobalt along with manganese act as protective oxides against corrosion. Protective oxides have been responsible for improving corrosion resistance of Superni 600.
5. These results were also supported by the images obtained by X-ray mapping.
6. Although coating will be degraded with subsequent cycles due to higher k_p value, but with deposition of coating, the life of substrate can be enhanced substantially as it does not delaminate from the substrate and also shows parabolic behaviour.

6.2 FUTURE SCOPE

1. To study hot corrosion behaviour of coated and uncoated Superni 600 in actual coal fired boiler environment.
2. To study the hot corrosion behaviour of coated and uncoated substrate after exposing it to hot corrosive environment for 200 hours or more.
3. New coating formulation can be designed and studied in similar environment.

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