# Protective coatings on superalloys and C/SiC composites for efficient gas turbine components



By Hafiz Muhammad Ijaz Tahir Reg#171299 Session 2016-18 Supervised by Prof. Dr. Zuhair Subhani Khan

A Thesis Submitted to the U.S Pakistan Center for Advanced Studies in Energy in partial fulfillment of the requirements for the degree of MASTERS of SCIENCE in ENERGY SYSTEMS ENGINEERING

US-Pakistan Center for Advanced Studies in Energy (USPCAS-E) National University of Sciences and Technology (NUST) H-12, Islamabad 44000, Pakistan 18<sup>th</sup>, August 2020

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#### THESIS ACCEPTANCE CERTIFICATE

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## Certificate

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#### ABSTRACT

Energy is one of basic need of any human entity. Pakistan is 6<sup>th</sup> largest state in the world on the basis of population. It requires huge amount of energy to fulfill their demands. Pakistan producing approximately 65% of the total electricity from thermal power plants. Gas turbines are the essential part of thermal power sources. Objective of this research is to make turbine components more durable and sustainable under severe environmental conditions. There are two ways to meet the required energy demand, either to improve the process efficiency or to develop renewable energy source. Gas turbines used at thermal power plants are operated at temperature i-e normally 700-1000 °C in a CHP loop while gas turbines used in aerospace application operated at very high temperature i-e 1400-1500 °C. At this elevated temperature, the components of gas turbines are corroded and we must protect turbine components like Inlet section, Compressor, Blades and combustion chamber from heat, corrosion and wear. Under this research project, we used YSZ coating to protect superalloy from wear and silicon slurry optimization for C/SiC substrate. Firstly, we select the substrates, one is Monel-400 from superalloy category while other is C/SiC from ceramic matrix composites. The salient reasons behind the selection of these substrates are (i) Super alloys are used for the manufacturing of hot sections of gas turbines. (ii) C/SiC having high strength to weight ratio used in jet engine. (iii) Both having excellent mechanical and thermal properties. Silicon slurry was optimized by varying the concentration of silicon powder, solvent and dispersant, coating process deposit this slurry via dip coater on C/SiC substrate. On Monel-400 we used bilayer concept and deposit aluminum as a bond coat and 7YSZ as top coat via thermal spray process. After deposition of bond layer, we performed thermal annealing at 500 °C to form nickel-aluminum bonding. This thing strengthens the bond layer and provide excellent surface for top coat. Then characterized the coated coupon with different tools like XRD, SEM, EDX and especially via tribometer to check the wear rate and compare it with non-coated sample. It was observed that coated sample having much less wear rate i-e 1.38% than non-coated sample under 5N load condition and this coating definitely enhance the efficiency of turbine components against wear and tear.

Key words: Thermal spray coatings, 7YSZ, Slurry optimization, Gas turbine hot sections.

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#### Dedication

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#### Chapter #01

# Introduction

#### 1 Energy:

Energy is basic need of any human entity. Today world is facing energy issues in almost every field of life like environment, economic, and development. Some fields are directly affected by energy issues and some are indirectly affected. For global progress and prosperity, efficient, clean, consistent and affordable energy services are indispensable. Energy issues are become more severe for developing countries. These developing nations must need to meet the reliable and cheap sources of energy in order to improve the health of their citizens and to reduce their poverty level. For instance, in the absence of efficient energy services, both health clinics and education sector didn't function properly. So, if a country with limited access to energy is unable to fulfill the basic needs of their population. Without effective pumping capacity, we are unable to access clean water and effective sanitation system. Food supply chain and security is also adversely affected by energy issues. Still World is heavily dependent on conventional fossil fuels like oil and natural gas and coal etc. in order to fulfill their energy needs. But these sources of energy production are adversely effects on environmental conditions locally and globally as well. CO2, NOx and SO<sub>x</sub> emission from these sources are major responsible to damage ozone layer and land and water acidification. In long term the key climate goal is to minimize the carbon intensity of power generation, i-e amount of carbon released against consumption of per unit of energy. It is within the subject of united nation Advisory Group on Energy and Climate Change (AGECC) to address the multiple challenges of meeting global energy needs for development while minimizing the emission of GHG's [1]. Approximately three billion peoples totally dependent on traditional biomass sources for heating and cooking purposes and about one and half billion having no access to electricity globally. On the other hand, one billion peoples depend on unreliable sources of electricity. The energy poor region suffers from serious

health issues due to inefficient burning of coal and wood in poor ventilated buildings, and their revenue generating activities are also badly affected by shortage of power or electricity. If we are able to develop an efficient system for generating electricity by usage of modern technologies and modern schemes than it would definitely improve the living standards of poorest few billion peoples on planet. According to World Bank study that countries may loss 1-2 percent of growth rate if they didn't have sustainable and modern energy production system [2].

#### 1.1 World energy scenario:

World electricity generation grew by 2.8% in 2017. Major part of this growth rate is come from developing nations. Organization for Economic Cooperation and Development (OECD) demand for power is slightly raised due to variation in economic growth, but in past decade the demand was pretty flat. Coal remains the world's dominant source of power, with a share of 38.1% in 2017, almost as much as natural gas (23.3%) and hydroelectricity (15.9%) combined. Natural gas and hydro power are at 2<sup>nd</sup> and 3<sup>rd</sup> place for electricity generation in world. Renewable energy sources having only just 8% share in world's electricity production in 2017, and only 6.1% increases from 2007-17. While in this same span, nuclear power generation decreased by 3.4 percent and coal lost only 3.1 percent. In 1998, the coal share was 38% in world's electricity, same as in 2017. In the start of this century, a little increase in coal power generation due to China's heavily dependency on coal. In 2016, the Global installed electricity capacity was 6474 Giga watts (GW), while in 2010 it was 5047 GW and annual growth rate is 4.1%. The world's electricity significantly depends on thermal sources. The major electricity generating countries are China, India, Russia, US, Canada and Japan. All these nations having huge reserves of coal [3]. The leading countries which are generating power from coal are China and India, but china is now shifted towards renewables and biomass sources with excellent acceleration. The reasons behind shifting towards renewables are, the insecurity of conventional fuel supply, minimizing the dependence on other countries and serious

environmental issues. Thermal power is still rank number one source for power generation. In 2030, the thermal power installed capacity reaches up to 5318GW with compound annual growth rate of 2.1%. Wind energy having highest share in world's renewable power sector of about 7.6% in 2016 and solar is only up to 4.6%. The renewable share will be increased up to 23.4% in 2025. In general, the world's energy generation trend is shifted from conventional fossil fuels to clean renewable sources, but it takes few decades for complete transformation [4, 5].



Figure 1: (a) World's energy shift towards renewables, (b) World's power generation by source and (c) World's power generation by region [4,5]

#### **1.2 Pakistan's energy scenario:**

Electrification in Pakistan is done by two authorities, Water and Power Development Authority (WAPDA) and Karachi-electric. WAPDA supplied electricity in all over the Pakistan except Karachi. Karachi electric provides electricity only for Karachi region. These two authorities are vertically integrated with each other. Both these authorities are responsible for power generation, its transmission, Distribution and retail supplied to their customers. Besides these two authorities, there are forty-two independent power producers (IPPs) that share a handsome amount of power generation in Pakistan. These IPPs generate electricity mostly from oil and gas fuel and sold it to the WAPDA and Kelectric. Pakistan has been facing a persistent unprecedented energy crisis owing to ever increasing demand and supply gap. The country's current energy needs are reliant on oil and gas and the demand far exceeds its domestic supplies. Thus, its primary energy supplies are heavily dependent upon imported crude oil and imported petroleum products due to which the country's oil import bill has risen to approximate US\$15 billion in fiscal year 2013, which is a huge burden on the economy and its forex reserves. Two conventional fossil fuels like oil and gas are key components in Pakistan energy mix. Approximately 75-80% power generation by these two sources in fiscal year 2012, While power generation through coal and nuclear was just only 7 and 2 percent respectively. In fiscal year 2012, the growth of power generation is just about 0.32%. On the other hand the average growth of net energy supply is 1.3%, which is consistent for about last five years. In 2012, the net energy consumption is increased by 3.1% as compared to previous fiscal year. So, In Pakistan there are four significant power generation authorities: WAPDA, Pakistan Atomic Energy Commission (PAEC), K-electric and IPP [6, 7, 8].

- Electricity, Net Installed capacity: about 25,000MW (2017).
  - ➤ Nuclear source: 1330 MW, (3-5%)
  - ➢ Hydro power generation: 6610 MW (25-30%)
  - ➤ Thermal power sources: 14, 633 MW (65-70%)

- $\blacktriangleright$  Renewables and others: 0.5-1%
- ➢ Average demand: 17,000 MW



Figure 2: Energy scenario in Pakistan [HDIP]

#### 1.2.1 Hydro power sources in Pakistan:

Electricity from hydel source is socio-economic uprising of any country in the world. It is cheapest way to extract the electricity from water. Pakistan has huge amount of hydro power potential. The explore potential is about 40,000MW and Pakistan extract only 15-16% of that potential, which is 6595MW. The reasons behind to this much low extraction are: lack of technological advancement and not much effective policies. If we properly utilized this cheap source of power generation then we will be soon able to overcome the energy crisis. The hydro sources are spread in almost all provinces. Most of hydro sources are in Northern areas, Azad Jammu and Kashmir, KPK and Punjab region. In a hydro power plant water is stored at some elevation from turbine. Then intake gate was open and water runs down, strike with turbine blades and moves away from

discharge point. Turbine is coupled with generator, which produced electricity and sent to the grid. Figure-3 shows a schematic diagram of hydro power generation.



Figure 03: Hydel power plant [6]

In Pakistan, currently the highest hydro-power source is Tarbela dam with capacity of 3478MW, located in Haripur district of province KPK. The 2<sup>nd</sup> and 3<sup>rd</sup> largest hydro units are Ghazi Barotha and Mangla dam with capacity of 1450 and 1000MW respectively. Electricity obtained from hydro source is much cheaper than other sources besides their other agricultural benefits. Energy extraction from hydro sources are required a huge amount of capital to build a hydro power source. Following table 1.1 depicts a comparative summary of the hydel projects in various regions of Pakistan.

Name of the province	Projects in Operation (MW)	Public sector Projects (MW)	Private sector projects (MW)
NWFP	3767.2	635	84
Punjab	1698	96	NIL
AJK	1036	973.8	828.7
Northern Areas	93.7	18	NIL
Sindh	NIL	NIL	NIL
Baluchistan	NIL	NIL	NIL
Total	6595.032	1722.8	912.7
Sr. No	Name of Project		Installed Capacity (MW)
01	Tarbela		3478
02	Ghazi Barotha		1450
03	Mangla		1000
04	Warsak		240
05	Chashma		184
06	Malakand		19.6
07	Dargai		20
08	Rasul		22
09	Shadiwal		13.5
10	Chichoki Malian		13.2
Total			6440.2

 Table 1.1: Hydel power potential in Pakistan [8]

#### **1.2.2** Nuclear power sources in Pakistan:

Nuclear power plants are also utilized for power generation purposes. In nuclear power plants electricity is produced by steam generation through boiling of water. Then this steam is utilized for power generation. In nuclear power plants, electricity is produced through a process called fission. Enriched uranium is commonly used fuel in nuclear power plants. The first nuclear power plant constructed in Pakistan in 1966 under the name of KANUPP at Karachi with the help of Canadian General Electric Company. Its power generation capacity is 137 MW, and in 1971 KANUPP was linked with national grid. In 1980, PAEC took the charge of this nuclear power plant and starts to produce its fuel indigenously[9]. The construction of second nuclear power plant in Pakistan was started in 1993. The second nuclear power plant is C-1 with the help of China National Nuclear Cooperation, with the gross capacity of 325MW. The 3<sup>rd</sup> nuclear power plant is C-2 having generation capacity of 330MW and 4<sup>th</sup> one is in pipeline. Pakistan generating just 2-3% of its total power generation from nuclear sources [10, 11].



Figure 04: Sketch of nuclear power plant [Nuclear energy.net.Printerset]

Power Station	Location	Generation capacity (MW)	status		
KANUPP-1	Karachi, Sindh	85	Operational since 1972		
CHASNUPP-1	Mianwali, Punjab	320	Operational since 2000		
CHASNUPP-2	Mianwali, Punjab	320	Operational since 2011		
CHASNUPP-3	Mianwali, Punjab	340	Operational since 2016		
CHASNUPP-4	Mianwali, Punjab	340	Operational since 2017		
KANUPP-2	Karachi, Sindh	1161	To be operational in 2021		
KANUPP-3	Karachi, Sindh	1161	To be operational in 2021		
CHASNUPP-5 Mianwali, Punjab		1161	In feasibility stage		

Table 1.2. Nuclear power stations in Lakistan [11	Table	1.2: N	uclear	power	stations	in	Pak	cistan	[11	1
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### 1.3 Renewable energy sources in Pakistan:

Renewable energy source is one in which the parent source is replaced or reused naturally. Pakistan is blessed with numerous renewable energy sources. Renewable energy sources play a significant role for the economy of any nation in the world. Besides hydro power potential, Pakistan is blessed with following major renewables:

- ➢ Wind power source
- Solar energy system
- Biomass energy source

There are countless advantages for using of renewable sources instead of conventional fossil fuels for power generation purposes. The major advantage of solar energy system is that it emits only 0.07-0.2 pounds of carbon dioxide per KW of power while Natural gas release 0.2-6 pounds of CO<sub>2</sub> and coal emits 1.4-3.6 pounds of CO<sub>2</sub> per KW of power generation. This is the biggest advantage of solar system that our environment is secure from pollution and average rise in temperature of earth is in controlled range. Pakistan is loaded with huge solar energy potential and it can generate approximately 2.324 million MW electricity per year. The average sunshine time in Pakistan is 8-10 hours per day except few cloudy days. The average solar radiation intensity is greater than 205 W/m<sup>2</sup> in most of the areas, which is highly suitable for power generation purposes. Pakistan's first largest solar park is located in Bahawalpur region under the name of Quaid e Azam solar park with capacity of 100MW. The major limitation of renewable systems is the low efficiency of system as compared to fossil fuels power generation system at large scale.



Figure 05: Solar energy potential in Pakistan [12]

#### 1.3.1 Wind energy scenario in Pakistan:

Wind source of energy production is also considered as renewable source. Two provinces of Pakistan, Baluchistan and Sindh have capability to generate power from wind source. There are several places in both provinces which are suitable for wind energy, like District Thatta in Sindh and District Makran in Baluchistan. In Sindh province, the most suitable place for wind energy is Ghoro and Keti Bandar in District Thatta. Jhimpir wind power plant with the capacity of 50MW is also installed at District Thatta, few other are also in operational phase with limited capacity and few are in pipeline under CPEC agreement. In Baluchistan, the most suitable places for wind energy are coastal areas like Pasni and Gawadar in District Makran. Currently five wind projects having total capacity of 256 MW are in operational phase. Pakistan having almost 50000MW wind energy potential, but we harness only few hundred MW's. For optimum wind turbine rotation, the wind speed of 3 to 4 meter per second, but Pakistan having 6-7 m/s wind speed[12].



Figure 06: Wind power plants and potential areas in Pakistan [12]

#### 1.3.2 Biomass energy scenario in Pakistan:

Harvesting energy from organic sources is known as biomass energy. Biomass energy has the potential to play a substantial role in combating the expanding energy crisis in Pakistan owing to the massive and diversified biomass potential of the country. Biomass is counted as a clean energy source since it recycles carbon dioxide through photosynthesis during biomass growth. Utilizing energy from Biomass is oldest way of harnessing energy. Pakistan is an agricultural country and there is a huge potential for generating power from biomass resources. In Pakistan, more than 55% of total population is directly or indirectly involved with agriculture sector. The major biomass resources are crops residue, municipal waste and animal manure etc. Majority of the cotton sticks are used as domestic fuel in rural areas so only one-fourth of the total may be considered as biomass energy resource. Harvesting energy from sugarcane was laid under the first generation of biofuels or bioenergy. Pakistan cultivates a huge amount of sugarcane in every year. From this cultivation we can meet our sugar requirements and also get sufficient amount of bagasse for power generation purposes. Pakistan can generate power up to 9476 Giga watt per year from sugarcane residue. In second generation of biofuels or bioenergy, we can extract energy from vegetable and fruit waste. Pakistan is ranked 4<sup>th</sup> in world for the production of milk, and huge number of animals are present. From these animals, we get solid and manure waste to generate a sufficient amount of biogas which is used for power generation as well as heating and cooking purposes. In addition to nonwoody agricultural residues, the woody portion also contributes a great share to the production of energy. According to World Bank report, Pakistan also generates 23000 GWh power from animal manure, if properly managed. According to United Nations Food Agriculture Organization (FAO), Pakistan ranked 11<sup>th</sup> in the world for the production of rice paddy, with capacity of about 10 Metric ton for the year 2009-10. Some other crops are also having high potential for bioenergy like cotton sticks, rice husk, wheat straw and tobacco sticks[13].



Figure 07: Biomass potential in Pakistan [13]

#### **1.4 Thermal power sources in Pakistan:**

Pakistan electricity sector is a developing market. For years, the matter of balancing the country's supply against the demand for electricity had remained a largely unresolved matter. The country faced significant challenges in revamping its network responsible for the supply of electricity. Electricity generators were seeking a parity in returns for both domestic and foreign investors indicating it to be one of the key issues in overseeing a surge in electricity generation when the country was facing growing shortages. Other problems included lack of efficiency, rising demands for energy, and political instability. In a thermal power station electricity or electrical energy is produced from heat energy. In a thermal power system, firstly we produce steam through the boiling of water. Then this mechanical work is converted into electrical energy through generator. For achieving high efficiency in a thermal power system, we must require high pressure and high temperature steam. Almost all thermal power sources are operated on conventional fossil fuels like Oil or natural gas. Pakistan majorly relies on thermal power plants in order to fulfill their energy requirements.



Figure 08: Thermal power share in Pakistan's power generation sector [8]

#### **1.4.1 Gas turbine power plants in Pakistan:**

Gas turbine is essential part of any thermal power generation. As Pakistan producing over 60% of its electricity from thermal sources, so gas turbine plays a significant role in our power generation sector. Gas turbine is also known as internal combustion device which generates work by the action of fluid. In history, firstly the gas turbine used for power generation at Neuchatel, Switzerland in 1939 was developed by Brown Boveri Company[14]. The major components of gas turbine are inlet, Compressor, Shaft, Combustion chamber or burner, Turbine and nozzle as shown in following figure.



Figure 09: Gas turbine components [14]

# **1.4.2 Gas Turbine Components:** Inlet:

Inlet is the starting area of turbine from which air is entered. The coming fluid either air or gas must be pretty clean in order to avoid from corrosion, erosion and any mechanical damage. Several parameters like inlet temperature, atmospheric conditions like dust, salt and any other foreign elements must be in consideration while designing inlet section of turbine.

#### **Compressor:**

Compressor is a salient part of gas turbine assembly. The basic purpose of compressor is to provide high pressure fluid to the downstream components of turbine. It also enhances the temperature of working fluid either air or gas. Generally, a compressor is consisting of rotor blade and vanes. Gaseous energy is produced from compressor rotor blades through mechanical work. This conversion of energy mainly increases the total pressure of the fluid. Several types of compressors are used like centrifugal compressor, axial flow compressor and mixed compressor. In centrifugal compressor, basically centrifugal force is used to enhance the pressure of coming fluid and is mostly used for small applications because of its size and weight, compare to axial flow compressor. In axial flow compressor, fluid is compressed axially to the direction of engine. Axial flow compressor consisting of several stages and having high compression ratio as compared to the centrifugal compressor[15].

#### **Burner:**

Burner is also known as combustion chamber and fuel is provided in this section. This combustion chamber also consisting of some igniting source and fuel nozzle. In this chamber air and fuel are mixed and controlled burning occurred. This section delivers the products of controlled combustion to the turbine within the design

temperature range. Fuel is mostly provided by the top side of the burner in a sprinkle or highly atomized spraying method. Several types of fuel nozzles were used for fuel injection depending upon requirements and nature of fuel. There are two types of air is used in combustion chamber i-e primary and secondary. The purpose of primary air is to control the flame length and temperature of combustion chamber while secondary air is used for cooling purposes of combustion chamber walls. Some turbines used for aerospace applications, where temperature of combustion chamber reaches up to 2000  $^{0}$ C and secondary air plays a significant role to maintain the temperature of combustion chamber within design limit.

#### **Turbine:**

The high energy gases entering to the turbine section is converted to the shaft power. Then this shaft power is used for multiple purposes i-e to drive compressor and other support systems. In power generation application, turbine is connected to electric generator which converts mechanical energy into electrical energy. Turbines are either connected in a single cycle or may be connected in a combined loop arrangement for higher efficiencies. In a combined cycle system, we can extract heat from hot exhaust gases of turbine and generate steam, which is also used for more power generation.

#### Exhaust:

The gas after passing through the turbine is sent to the exhaust chamber. In this chamber special arrangements were made to extract more heat from exhaust gases, which improves the overall efficiency of the process. The power generation from gas turbine combined loop is shown in Figure.



Figure 10: Gas turbine in a combined loop [14]

#### 1.4.3 Working principle of gas turbine:

Gas turbine usually operates in an open and close cycles. Air at ambient conditions enter from inlet section after removal of necessary cleaning and drying, reaches at compressor section where it's both temperature and pressure raised. Then this highpressure air is entering into combustion chamber where fuel is burnt at a constant pressure. Then this high-pressure gas enters into turbine and generates shaft work. After that the generator converts mechanical work into electrical energy and the exhaust gases release to atmosphere after sufficient recovery of heat. Generally, gas turbine follows the Brayton cycle. Brayton cycle was firstly invented by American scientist George Brayton in 19<sup>th</sup> century, he describes working of heat engine at constant pressure[16]. Ideal Baryton cycle consist of four stages as follow,

- i. Isentropic Compression
- ii. Isobaric heat addition
- iii. Isentropic expansion
- iv. Isobaric heat rejection

Actual Baryton cycle is bit different from ideal cycle in such a way that compression and expansion stages are adiabatic not isentropic. So, the actual Baryton cycle having following four stages:

- i. Adiabatic Compression
- ii. Isobaric heat addition
- iii. Adiabatic expansion
- iv. Isobaric heat rejection

Pressure-Volume and Temperature-Entropy diagram of ideal Baryton cycle is shown in following figure.



Figure 11: P-V and T-S diagram of ideal Brayton cycle [17]

In Pakistan all gas turbine power plants are operated in a combined loop fashion and major plants are listed below:

Power plant	Location	Capacity (MW)	
Guddo Thermal Power Plant	Guddu, Sindh	2402	
Foundation Power Company (Daharki) Limited	Daharki, Sindh	185	
Uch-I, II Power Plant	Dera Murad Jamali, Balochistan	1000	
Nandipur Power Project	Gujranwala, Punjab	425	
HabibullahCoastal Power (Pvt) Company	Quetta, Balochistan	140	
WAPDA Quetta Thermal Power Station	Quetta, Balochistan	35	
Liberty Power Project	Daharki, Sindh	235	
Rousch (Pakistan) Power Plant	Kabirwala, Punjab	425	
Engro Powergen Qadirpur Limited	Ghotki, Sindh	227	
Halmore Power Generation Company Limited	Sheikhupura, Punjab	225	
Orient Power Company Limited	Kasur, Punjab	229	
Saif Power Limited	Sahiwal, Punjab	229	
Haveli Bahadur Shah Power Plant	Jhang, Punjab	1230	
Bhikki RLNG-based Power Project	Sheikhupura, Punjab	1180	
Balloki Power Plant	Balloki, Punjab	1223	
Sindh Nooriabad Power Company Pvt Ltd	Jamshoro, Sindh	100	

 Table 1.3: Gas turbine power plants in Pakistan [Thermal power. energy]

#### **1.4.4 Steam turbine for power generation:**

In Pakistan, mostly all thermal power plants are usually operated in a combined loop fashion. In a steam turbine the working fluid is steam, which is produced by constantly heating of water. This steam is utilized for mechanical work to rotate the shaft. Then this mechanical work is converted to electrical output through generator. Sir Charles Parsons

invented the modern form of steam turbine in 1884. In USA, steam turbines are extensively used for power generation in the last decade of 20<sup>th</sup> century. The steam turbine is also a form of heat engine and requires improvements in thermodynamic efficiencies because it has multiple stages for the expansion of steam. Generally, the ideal steam turbine system is followed isentropic process. In isentropic process, the entropy of the entering steam is equal to the entropy of the leaving steam. In other words, we can say that entropy remains constant in an ideal steam turbine engine. In actual, there is no truly isentropic steam turbine is available or in operation but the typical isentropic efficiencies are in the range of 40-65% depending upon the nature of its application. A steam turbine is generally consisting of several numbers of blades and buckets. These blades are present in the form of sets on a rotating shaft. Some blades are connected to the casing, they are called stationary blades. Some blades are connected with shaft, they are known as rotating blades. There are several factors which affects the efficiency of the steam turbine are turbine size, load condition, friction losses, moisture content in steam and friction losses etc. For better efficiency steam must work in a number of stages and how the energy is extracted from steam in different sets of blades. Turbines are either impulse or reaction type. The high pressure stages are impulse type while low pressure stages are reaction type and over all turbines uses both types[17].



Figure 12: Steam turbine used for power generation [18]

#### **1.5 Gas turbine for aerospace propulsion:**

Gas turbine is used in aerospace application to achieve better efficiency. It provides power or thrust to the engine. In aerospace application the shaft power of gas turbine is utilized in turboprop system in order to move propellers and rotors. In the start of 20<sup>th</sup> century, Wright brothers attempt first effort to fly with powered engine. The first use of gas turbine in turbo engine is in 1918 during WWI by Germen Electric Division. In this turbo engine turbine wheel was run through the pistons exhaust gas. In 1930, Frank Whittle submitted a patent application for using gas turbine in jet propulsion. His design was successfully tested in 1937 at pilot scale, which consist of single stage centrifugal compressor connected with turbine[18]. In 1939, a company having name Power jet Ltd. Get first contract from Air Ministry to design a gas turbine-based flight engine. In May 1941, the WI engine starts its first flight on Gloster Model E28/39 aircraft. This aircraft achieved excellent speed with 1000 pounds of thrust force. After that gas turbine become essential part of any propulsion system. In recent gas turbine engine, a nozzle is used that produced high kinetic thrust stream. In modern turbo engine, the thermodynamic efficiency limit is almost achieved and huge advancement in research is required in materials or in cooling systems in order to enhance the capability of hot sections of gas turbine[19].



Figure 13: Gas turbine in Aerospace engine [19]

#### Summary

In this chapter, firstly discuss the need of energy because energy is the basic need of any human entity. In world energy scenario, the trend for generation of electric power is shifted towards renewable sector from conventional fuels. But full dependence on renewables require several decades. Electricity in Pakistan is provided by two major authorities, i-e WAPDA and K-electric. K-electric is responsible for generating and supply of power to the Karachi and its surroundings, while WAPDA providing electricity throughout the Pakistan. Major power production sources in Pakistan are hydro and thermal, from which we can generate over 80% of its total energy. In Pakistan, the renewable energy share is very small in power generation although we have huge renewable potential like biomass, solar and wind source etc. Gas turbine play significant role in power generation sector. Gas turbine is widely used for both power generation and propulsion purposes. From previous century, gas turbine was introduced in aerospace application for better efficiency discussed at latter part of this chapter.


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## Chapter #02

# Gas turbine materials and coatings

## 2.1 Efficiency of gas turbine:

Gas turbines are devices for converting fuel energy into electric power (via electric generators) or mechanical power. They normally use the Brayton Cycle, which is a thermodynamic cycle that involves compression and expansion of a gaseous medium. An ideal cycle may offer 100% performance, but a real gas turbine will always have a certain level of losses and friction. The evolution of gas turbines has often been in lockstep with market dynamics. As power producers have increasingly turned to natural gas as their fuel of choice, turbine manufacturers have pressed to keep pace, knowing generators want reliability and availability along with reductions in carbon emissions. General Electric in December announced its largest 9HA gas turbine (Figure 13) is now available at 64% net efficiency; the company says the turbine is its "most advanced gas turbine technology." The milestone comes 18 months after GE was recognized by Guinness World Records for achieving 62.22% efficiency with an HA turbine at an EDF-operated combined cycle plant in Bouchain, France.



Figure 14: Gas turbine with highest efficiency (9HA) [1,2]

Thermal efficiency of actual Baryton cycle is always less than ideal one. The reason behind this lack of efficiency is the drop-in pressure during heat addition and rejection. This phenomenon decreases the pressure ratio, which have direct relation with efficiency of turbine. Thermal efficiency of gas turbine may increase by either increasing the turbine inlet temperature or extract heat from exhaust gases [1, 2]. The recovered heat is used for steam generation in a closed loop. The thermal efficiency of gas turbine is as follow,

The energy balance for a steady-flow process can be expressed, on a unit-mass basis, as

$$(q_{\rm in} - q_{\rm out}) + (w_{\rm in} - w_{\rm out}) = h_{\rm exit} - h_{\rm inlet}$$

The heat transfers to and from the working fluid are:

$$q_{\text{in}} = h_3 - h_2 = c_p(T_3 - T_2)$$
$$q_{\text{out}} = h_4 - h_1 = c_p(T_4 - T_1)$$



$$\frac{T_2}{T_1} = \left(\frac{P_2}{P_1}\right)^{(k-1)/k} = \left(\frac{P_3}{P_4}\right)^{(k-1)/k} = \frac{T_3}{T_4}$$

The thermal efficiency of the ideal Brayton cycle,

$$\eta_{\text{th,Brayton}} = \frac{w_{\text{net}}}{q_{\text{in}}} = 1 - \frac{q_{\text{out}}}{q_{\text{in}}} = 1 - \frac{c_p(T_4 - T_1)}{c_p(T_3 - T_2)} = 1 - \frac{T_1(T_4/T_1 - 1)}{T_2(T_3/T_2 - 1)}$$
$$\eta_{\text{th,Brayton}} = 1 - \frac{1}{\frac{1}{r_1(k-1)/k}} \quad \text{Constant specific heats}$$

where 
$$r_p = \frac{P_2}{P_1}$$
 is the **pressure ratio**.

We may enhance the efficiency of ideal gas turbine by following two factors:

- i. Increase in pressure ratio
- ii. Increase in specific heat ratio (k)

## 2.2 How to improve gas turbine efficiency:

Corrosion in gas turbine is very common phenomenon. There are several reasons for the occurrence of this corrosion. We must have tried to overcome this corrosion in order to enhance the thermal efficiency of turbine. The necessity of prevention of corrosion from gas turbine is due to the reason that it saved a huge amount of money in the form of material saving. There are several techniques or methods are used for the protection of corrosion and some of the important ways are given below:

- i. Selection of suitable materials for manufacturing
- ii. Used anti-corrosion inhibitors in fuel
- iii. Via a protective coating
- iv. Used ultra clean fuel
- v. Preventive maintenance
- vi. Used drying agents to minimize moisture content

Above mention methods are commonly used for prevention of corrosion in gas turbine as per requirements. Therefore, one or any combination of above two methods are used because all these methods needs a handsome amount of investment. Generally, we preferred to purchase more clean fuel for gas turbine and produced ceramic coating at hot components of gas turbine[3]. Some of the important and commonly used methods are also shown in following Fig.



Figure 15: Different ways to improve gas turbine efficiency [3]

#### 2.2.1 Different coating strategies:

In past several types of coatings are used for the protection of different structural materials from wear, corrosion and severe environmental conditions. Different materials and coating strategies are applied on different substrates totally depends on the nature of application. If our substrate is operated in a high temperature environment then thermal barrier coatings will be preferred. All thermal barrier materials are suitable for this application in order to provide thermal insulation<sup>[4]</sup>. In TBC application, we have a long list of suitable materials but the final selection of a particular material is depends on several other factors like available deposition technique, compatibility with substrate and economics of the process. In the early stages of coatings, only alumina and silica are used for the protection of substrate from severe environmental conditions and that's why these coatings are known as Environmental barrier coatings. But with the passage of time, people require such coatings which provide thermal insulation as well as protection from environment. Then dual layer coating concept is used. In this technique, firstly we deposit a thin layer of suitable bond coat which provides protection against oxidation and then develop second layer known as top layer. The material used in top layer must be compatible with bond layer and must having enough thermo-mechanical strength to stand

in a consistent high temperature environment[5]. Currently following types of coating strategies are used:

- i. MCrAlY type in which M is any metal element, Cr is chromium, Al is aluminum and Y is Yttrium.
- ii. Aluminide coatings like heavy metal penetration in aluminum coating pool.
- iii. Ceramic coatings like YSZ, usually for the protection of super alloys
- iv. Rear earth silicates are also emerging materials for current TBC schemes.

#### 2.2.2 High performance material for gas turbine:

The current gas turbine engine that are either use in propulsion applications or in energy production system, made of several different categories of materials, may faces different stresses and severe environmental conditions. The selection of material for manufacturing of gas turbine components is very critical thing. There are several factors which must kept in mind before the selection of materials like operational temperature of component, vibration level, strength to stand against oxidation and thermo-mechanical properties etc. There is a very high temperature at burning stage of fuel, so the selected material must possess enough strength to stand in a high temperature environment. For power generation, the temperature of hot sections of gas turbine reaches up to 1000 °C and for propulsion system the operating temperature of hot sections may reach up to 1500 °C. So, there is a huge amount of thermal stress may appear in hot sections besides consistent high temperature environment. However, by considering all above factors super alloys and ceramic matrix composites are suitable candidates for the manufacturing of hot sections of gas turbine. In super alloy category, a long list of materials is available

which are used for the manufacturing of hot sections of gas turbine[6]. In the early history of gas turbine Nimonic super alloy is used for manufacturing of gas turbine components. It is nickel based super alloys and used in early jet turbine engine. This alloy contains major portion of Nickel, Chromium and Cobalt. Then in late 60's, the super alloys were made from two new techniques like directional solidification and single crystal method in order to improve the properties of alloy. Then for further improvements was made and coatings of several materials was done on super alloys for better efficiency. In 1970s, the first thermal barrier coating on super alloy used for the manufacturing of jet components was made and it was modified aluminide coating. These coatings have capability to enhance the thermal resistance of material. In last few decades, for the manufacturing of jet engine turbine ceramic matrix composites are also used. They have capability to operate at a temperature of 1500 °C. The major advantage of ceramic matrix composites over super alloys is their light weight and strong resistance against elevated temperature. For low pressure turbine for F414 jet engine, German Electric aviation wing have successfully demonstrated CMC's instead of conventional super alloys [7][8][9]. Gas turbine hot sections are usually composed of super alloys or ceramic matrix composites. The selection of these two categories of materials for manufacturing gas turbine components was due to their excellent thermo-mechanical properties. In this project we select two substrates, one from the category of super alloy which is Monel-400 and the second one is from category of ceramic composites i-e Silicon Carbide composites (C/SiC). The selection of these two substrates is due to the reason that both are used in manufacturing of hot sections of gas turbine. Monel-400 is a Nickel-Chromium super alloy containing about 67% Nickel, 23% Chromium and small amount of silicon, carbon, manganese, iron and sulfur are present.

Alloy	Composition, weight percent									
167	Cr	Co	w	Мо	Re	Та	AI	Ti	Hf	Others
				Con	ventio	nally C	ast			
IN738LC	16	8.2	2.6	1.7		1.8	3.5	3.5		0.11C;0.01B;0.03Zr
IN792	13	9	3.9	2		3.9	3.2	4.2		0.21D(0.02B)0.1Zr
IN939	23	19	1.9			1.3	1.9	3.7		1.1Nb;0.15C; 01B;0.1Zr
				Direct	ionall	y Solid	lified			
MARM27LC	9	10	10	0.6		3.0	5.5	1.0	1.5	0.15C;0.15Bl9,93Zr
GTD-111	14	9.5	3.8	1.5		2.8	з	4.9	0.15	0.1C;0.01B;0.15Hf
ExA17"	12	9	3.5	1.8	2-3	4	3.4	3.9		
TMD103*	3	12	6	2	5	6	6		0.1	0;0.0158
				s	ingle	Crysta	1			
PWAI480 (1" gen)	10	5	4			12	5	1.5		
PWA1483 (1" gen)	12.8	9	3.8	1.9		4	3.6	4		
René N4 (1 <sup>#</sup> gen)	9	8	6	2		4	3.7	4.2		0.5Nb
SC16 (1" gen)	16			3		3.5	3.5	35		
PWA1484 *2nd gen)	5	10	6	1.9	3		5.6		0.1	8.7Nb
René N5 (2 <sup>se</sup> gen)	7	8	5	2	3	7	6.2		0.2	
CMSX-4 (2 <sup>nr</sup> gen)	6.5	10	6	0.6	3	6	5.6	1.0	0.1	
CMSX-186 (2 <sup>nd</sup> gen) <sup>b</sup>	6	9.3	8.4	0.5	2.9	3.4	5.7	0.7	1.4	
CMSX-486 (2 <sup>nd</sup> gen) <sup>±</sup>	5	9.3	8.6	0.7	3	4.5	5.7	0.7	1.2	
René N6 (3º gen)	4.2	12.5	6	1.4	5.4		5.8		0.15	7.2Nb;0.05C;0.004B;0.01Y
CMSX-10 (3 <sup>rd</sup> gen)	2	3	5	D.4	6	8	5.7	0.2	0.03	0.1Nb
TMS75 (3 <sup>rd</sup> gen)	з	12	6	2	5	6	6		0.1	
MX4/PWA1497 ("4"" gen)	2	16.5	6	2	6	8.3	5.6		0.05	3Ru;0.03;0.04B;0.01Y
TMS139 ("4*" gen)	2.9	5.8	5.8	2.9	4.9	5.5	5.8		0.1	3lr
TMS162 ("5"" gen)	2.9	5.8	5.8	3.9	4.9	5.6	5.8		0.1	6Ru
TMS196 ("5"" gen)	4.6	5.6	5.0	2.4	6.4	5.6	5.6		0.1	5Ru

 Table 2.1: Materials used for gas turbine components [7,8]

Monel-400 is generally used at sever environmental conditions because of its excellent mechanical strength and resistance against corrosion. Monel-400 is suitable material for marine applications, power generation equipment's and for aerospace applications [10]. Some of the major properties of alloy 400 are given below:

Property	Value	Units
Density	8.8*10 <sup>-3</sup>	Kg/m <sup>3</sup>
Thermal expansion (20°C)	13.9*10 <sup>-6</sup>	°C <sup>-1</sup>
Thermal conductivity	21.8	W/m*K
Specific heat capacity	427	J/kg*K
Electric resistivity	54.7*10 <sup>-6</sup>	Ohm*m
Tensile strength	550	MPa

 Table 2.2: Properties of Monel-400 [10]

Ceramic Matrix Composites (CMC's) is one of the most important class of materials and Silicon carbide belongs to the same category. Ceramic Matrix Composites consist of carbon fibers embedded in ceramic matrix. Conventionally, they are produced from Isothermal Chemical Vapor Infiltration process (CVI) but now a day it is manufactured form Liquid Silicon Infiltration (LSI) technique and Liquid Polymer Infiltration (LPI). From LPI process the end product obtain is C/SiC, while in LSI technique the end product is C/C-SiC. Both silicon carbide materials used for corrosion resistance and high temperature applications, because silicon carbide show excellent thermo-mechanical characteristics[11][12]. The major areas of applications of C/SiC are:

- i. Automotive Industry
- ii. Power plants Components
- iii. Jet engine
- iv. Grinding media with excellent hardness
- v. Manufacturing anti-corrosion and heat resistant equipment's
- vi. Electronics industry (Heat resistant diode)

Sr.	Property name	Value	Unit
INO			
1	Specific Density	2.31	g/cm <sup>3</sup>
2	Bulk Modulus	176	GPa
3	Thermal Expansion (2000 <sup>0</sup> C)	5.8×10 <sup>-6</sup>	<sup>0</sup> C <sup>-1</sup>
4	Specific Heat Capacity	1.26	J/g. <sup>0</sup> C
5	Thermal conductivity $(20^0 \& 1000^0 C)$	0.41& 0.21	W/cm. <sup>0</sup> C
6	Melting point	2700	<sup>0</sup> C
7	Young's Modulus	137	GPa
8	Hardness	9-10	Mohs Scale
9	Compressive strength	3359	MPa
10	Tensile strength	336	MPa
11	Sher Modulus	179.8	GPa

 Table 2.3: Properties of Silicon Carbide [11, 12]

## 2.3 Material issues of gas turbine:

For the manufacturing of gas turbine engine, the selection of suitable material is also a key factor. Material issues play a significant role for turbine efficiency. With the passage of time degradation may occur in manufacturing material due to oxidation, wear, erosion and high temperature environment. During the manufacturing of gas turbine engine, the major cost is associated with its hot section components. So, almost 50 to 60% of total cost of gas turbine engine is utilized in hot sections of gas turbine. The above mention degradation issues are also occur in turbine shaft, casing, compressor and blades but little less magnitude as compared to hot sections. The oxidation and wear problems are frequently occur at burning chamber, where fuel is burnt. The reason behind this phenomenon is that fuel may contain some sort of impurities which are not burned and with the passage of time a layer of non-degradable material is formed, which causes wear and oxidation of chamber's material<sup>[13]</sup>. Several different mechanisms are also responsible for degradation like thermal stresses, metal embrittlement and cracking. Environmental attack is caused by oxidation and hot corrosion, and results in the loss of the surface profile. Environmental attack can lead to cracking of the metal in combination with fatigue and creep. Coating degradation, which is a very important degradation mode, is considered as a form of environmental attack, since this degradation is caused by this attack. Creep is the gradual distortion or elongation of the component, and can lead to cracking or wear. Metallurgical embrittlement is the loss of mechanical properties because of metallurgical changes within the metal. Wear processes, especially for combustor hardware, are important for surfaces in contact with each other [14, 15]. Creep is occurring due to thermal stresses and metal elongation occur which causes degradation; this thing is commonly appear in turbine blades. Cobalt based super alloys are preferable for turbine blades and shaft. When nickel is mixed with majority of cobalt element then it gains face centered cubic structure, which is more ductile than hexagonal close packed structure of normal cobalt element. This property of cobalt super alloy makes it suitable candidate for turbine manufacturing material. In order to address the oxidation resistance of materials, nickel based super alloys are used within their limitation. Majorly, those nickel alloys which are formed through solid solidification process are suitable for turbine manufacturing because they have enough strength to tackle material issues during turbine operation. Hastelloy X, Nimonic 263, Inconal and Haynes 230 lies in this nickel based super alloy category, suited for turbine manufacturing materials. Wear is the phenomenon where two disparate surfaces are moving against each other. Wear can result in the removal of the surface metal and in the formation of cracks in the surfaces. It is a widely studied phenomenon, with many forms and sub-forms identified, and with its own terminology. The subject of wear is sometimes called tribology. For hot section components, wear is most important for combustion hardware[16]. The contacting surfaces of combustion liners and transitions are continually rubbing against each other due to combustor pulsations[17]. During the start-stop operation of the engine, these surfaces can undergo large relative motion. Depending upon the fit up of the components, binding can be created which is detrimental to the wear of the components[18]. Generally, high temperature attacks are of two types, oxidation attack and hot corrosion attack. High temperature hot corrosion and Low temperature hot corrosion are further two sub division of hot corrosion attack. Sometimes low temperature hot corrosion and high temperature hot corrosion are also known as type I corrosion and type II corrosion respectively. The temperature range of these two attacks and their severity of attack are shown below:



Figure 16: Material problems in gas turbine [16,18]

## 2.4 Coatings for gas turbine:

Coating is a necessary part of any equipment used at severe environmental conditions. There are several types of coatings available to protect our precious material and enhance the process efficiency. The type of coating and coating material depends upon the type of application. Basically, coating is a thin enough layer deposit on to the surface of the object. The basic purpose of applying the coating is to be protective, decorative, optical or adhesive and enhance turbine process efficiency up to 30% as turbine inlet temperature increases up to 1250 °C. Mostly, coating is used for protective purposes when our substrate is dealing with high temperature environment. There are several ways to apply the required coating on substrate depends upon their application. Sometimes we require thick layer of coating and sometimes we require thin enough layer to preserve our substrate. The most common procedures used for the application of required coating are Slurry method, Thermal Spray system, Sputtering mechanism, Thermal evaporation, CVD, PVD, Plasma Spray system, High velocity oxy fuel technique and electroplating. Each technique having its own advantages and limitations[5]. Despite all different mechanisms and applications, the basic purpose of the coating is to secure and sustain our substrate. Coating on any substrate is not a new phenomenon. In the first quarter of 20<sup>th</sup> century, thin film is developed on a laboratory scale and is become very popular among various scientific fields for multiple purposes. According to American Society of Material (ASM) international, Dr. Max Ulrick from Switzerland in 1911 invented and developed plasma spray process on a proto scale. And soon He becomes successful for developing coatings from molten and powder materials. Few years later, they become successful to develop an instrument for metal spraying which is in the form of wire by using extremely high electric voltage[18]. In late 40s, Organic polymers were sprayed on to metallic substrate. Dr. Erwin, a German scientist developed a technique for deposition of thermosetting powder coatings on different substrates and filed a patent in 1955. The idea to apply a protective layer on Ni- based alloy was first practiced in 60's. TBC was first successfully tested in a research turbine engine in 70's and then this field entered into revenue services in aerospace applications[19]. In 90's a new class of coating ETBC was introduced to protect Si-based ceramic and composites. TBC coating passes through several stages during its evaluation period i-e low density, EBPVD, multilayer, low conductivity and finally reaches to ETBC. Environmental thermal barrier coating is very advance technique used advance ceramic materials to protect several surfaces like gas turbine hot components and they are in the range of 100µm to 2mm.

#### 2.4.1 Literature review:

Monel-400 is a nickel based super alloy having excellent strength against a range of high temperature environment. Monel-400 alloy reveals excellent thermo-mechanical properties in corrosive environment and suitable candidate for turbine manufacturing material. In its composition, the majority portion of alloy-400 consist of nickel and copper while other elements like carbon, silicon, manganese and sulfur may present in traces[10]. Monel is used in a variety of application like Marine engineering, chemical processing equipment's, de-aerated still, Heat exchangers and crude oil distillation tower etc. Nickel based super alloys are used for the manufacturing materials of those equipment's used for temperature at up to 1000 °C. At higher temperature mostly, material is loss due to erosion problem which occur due to mechanical interaction between surface and a fluid or solid particle. In past, titanium and its compounds are used for coating to provide protection against erosion. For wear and corrosion protection, generally we used transition metal carbide like WC, TiC and TaC. They are usually applied by APS or HVOF technique. For good adhesion, we generally prefer Al<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub> matrix system for coating up to temperature 800 °C. This type of coating is composed of TiO<sub>2</sub> reinforced into Al<sub>2</sub>O<sub>3</sub> matrix. Another system of coating consist of WC-17Co powder is used for the protection of turbine components from wear resistance. This system is thermally sprayed providing dense coating having thickness up to 462 µm and bond strength is 36MPa. This system provides satisfactory results regarding wear resistance. Nanostructured wear resistance coating provides superior hardness and toughness. In recent past diffusion Aluminide coatings were also used to protect the super alloy substrate from aggressive oxidation attack. This thing is achieved either by pack cementation process or by CVD techniques. Sometimes we used modified aluminide coating for particular purposes and elements like platinum, palladium, rare earth elements, Hf and Zr are used[20]. Another super alloy protection system consists of oxidation resistant bond coat and ceramic top coat. The ceramic top coat is  $ZrO_2$  stabilized with MgO instead of  $Y_2O_3$ . The ceramic top

coat provides excellent heat resistance. The purpose of stabilizing ZrO2 with MgO is due to the reason that MgO is much cheaper than Y<sub>2</sub>O<sub>3</sub>. Pure zirconia is transform from tetragonal to monoclinic phase and thus volumetric changes associated with this transformation. This system of coating is only stable up to temperature of 750 °C[20]. Now in recent days, ceramic top coat along with metallic interlayer as a bond coat is preferred because of its extremely efficient results under temperature of 1000 °C. The advances in TBC mainly focus on improving durability and efficiency. To increase the efficiency through higher operating temperature, materials having low thermal conductivities are studied. Adding several rare earth ions as a co-stabilizer in YSZ didn't provide satisfactory results. Some stabilizer oxides like Gd<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub> and Sm<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub> shows low thermal conductivity but they not thermodynamically compatible with alumina interlayers[21]. Ceramic matrix composites are used in the manufacturing of advance turbine engine components and shows excellent advantages over conventional super alloys in their performance. The primarily benefit of ceramic matrix composites are displaying higher temperature capability up to 1200-1400 °C, with good tolerance against damage, low weight and density. In past, several systems of coatings are used for the protection of matrix composites. The first generation of EBC was proposed in late 80's and consisted of 3:2 Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> (Mullite) for SiC substrate. Silica was volatilized from mullite leaving behind alumina layer when exposed to high temperature for long time. Crack formations occur during annealing at 1000 <sup>0</sup>C due to silica volatilization and can't provide desirable protection. In the second generation of EBC's, rare earth silicates were found excellent coating material due to its numerous benefits. They provide excellent thermo mechanical properties at elevated temperature. In past, another system of protecting SiC is Y<sub>2</sub>O<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> for high temperature environment. Thermally sprayed YAS system along with Si bond coat. The coating was amorphous with low conductivity value and didn't change with temperature. The only hindrance for this system is that, YAS system is capable only up to 900 °C. Efforts were made at NASA

Glen Research Center under the program of Ultra Efficient Technology (UEET) to focus on protecting the turbine components from high thermal environment in 2001. The current state-of-the-art EBC system under development in the UEET system consist of CMC as a substrate, silicon as a bond coat and Barium Strontium Aluminum Silicate (BSAS) as EBC top layer. Silicon based ceramic are used for high temperature engine applications. They shows low silica formation at elevated temperature but degradation in moisture and alkali environment may create hindrance for its utilization over 1300°C. Another technique used to safeguard the ceramic matrix composites by the addition of liquid aluminum into SiC particles to make complex compounds which shows excellent thermal resistance at a temperature of 900 °C. Aluminum metal having great capability to form multiple complex compounds with several other atoms. These complex compounds are having high significance due to their specificity and excellent working capability than pure parent atoms. At interface alumino-silicate compound is formed for good oxidation resistance and alumina is top coat. High temperature coating trend is completely shifted towards the rare earth silicates and zirconates. Rare earth mono silicate like Yb<sub>2</sub>SiO<sub>5</sub>, Lu<sub>2</sub>SiO<sub>5</sub> and Sc<sub>2</sub>SiO<sub>5</sub> are commonly used in advanced thermal barrier coating due to their excellent high temperature durability. The most commonly used rare earth elements are ytterbium, lutetium, scandium, yttrium and their compounds. The utilization of these elements is depending upon the type of substrate and required application. Generally preferable materials are mono and di-silicates of ytterbium due to their excellent thermo-mechanical properties at a temperature up to 1500 °C[22]. Ytteria stabilized hafnia is also used as a top coat for a temperature of 1300°C along BSAS intermediate layer. Sometimes we also used a tri-layer combination of coating for a particular application. This technique is also used in recent past for the safeguarding of ceramic matrix composites in which a combination of Si/Mullite/RE mono silicate is used. For the usage of tri-layer concept we must have a good control on several parameters like CTE, coating thickness, working environment and nature of substrate.

## 2.5 Thermal and Environmental barrier coating materials:

Environmental and thermal barrier coatings are advanced system of materials usually applied on hot sections of gas turbine. These turbine engines are used in power generation application as well as in propulsion systems. Thermal barrier coatings are in the range of microns, depending upon the nature of technique which is used and application. These new advance materials are able to insulate substrate from high temperature environment like 1000 °C and 1500 °C. These advance materials having high melting points and excellent thermo-mechanical properties. Generally, complex compounds are preferred for thermal barrier coatings because a single element didn't have all capabilities or properties which are necessary for any TBC material. Sometimes thermal barrier coatings allow us to operate our substrate above from its melting point, because TBC material provides necessary shield from thermal degradation [23, 24]. Due to increasing demand for higher engine operation (efficiency increases at higher temperatures), better durability/lifetime, and thinner coatings to reduce parasitic weight for rotating/moving components, there is significant motivation to develop new and advanced TBCs. Advanced TBC materials are given below:

- i. Alumina
- ii. Mullite
- iii. Rear earth zirconates
- iv. Rear earth oxides
- v. Metal glass composites
- vi. Yttria stabilized zirconia
- vii. Zirconium with different stabilizers
- viii. Silicon
- ix. Alloys of Nickel and Chromium



Figure 17: TBC and EBC coating structure [23]

Thermal Barrier Coatings (TBC)	Environmental Barrier Coatings
	(EBC)
Applied and used in hot gas engine, their role is to protect the engine components from high heat flux.	Used on CMC's in order to protect the substrate from environmental effects like oxidation and corrosion.
An aero TBC is YSZ on a metallic bond coat over super alloy.	An EBC has zirconia or hafnia top coat over a silicate ceramic environmental coating to protect the substrate from water vapor attack.
Because of continuous high heat environment, a TGO layer is formed between bond coat and ceramic top coat. This TGO act as a oxidation barrier to the top coat.	Mostly silicon bond coat is used between substrate and EBC ceramic top coat. EBC provides protection from environmental assault.
TBC are for metallic substrate and its top coat is in compression.	EBC top coat is in tension and cracks are easily formed.
Compared with EBC, TBC's have better strain tolerance.	EBC's used for less temperature as compared to TBC's.
The major failures of TBC's are: Thermal stresses and oxidation of bond coat.	Their major failures are: Chemical attack, Degradation and spallation

Table 2.4:	Comparison	between	TBC and	EBC	[24]
1 abic 2.1.	Comparison	between		LDC	[ <b>-</b> · ]

### **2.6 Focus of this research and objectives:**

The major focus of this research is to find out new ETBC coating for gas turbine. We develop a novel coating scheme in order to minimize the wear rate of gas turbine due to severe environmental conditions. Our more than 70% of electricity is generated from thermal power plants and gas turbines are widely used on those plants. Turbines are corroded due to several factors like air or steam inlet system, water or coolant used, chemicals used to control the NOx emission like ammonia and dual fuel utilization method. Corrosion occur majorly due to contamination present in hydrocarbon based fuel used causes sulfur deposition and oxidation of surface at extreme temperature. On the other hand, safeguarding from corrosion and surface oxidation are not well established fields in our country as per our requirements and we depend a lot on exports. So in this regard this project is highly suitable for current industrial need. There are several thermal power stations installed and few were in pipeline in Pakistan under CPEC treaty. It very clear that, in near future Pakistan fulfill their energy needs through thermal power sources. So, this research is very much compatible with our national goals. Moreover, the research on such a novel topic also enhances my technical skills.

#### 2.6.1 Objectives of this research:

- To safeguard the Monel-400 super alloy at 1000°C: aluminum interlayer and YSZ top coat will be made by thermal wire spray and thermal powder spray respectively. The approximate coating thickness of each layer will be 130 μm.
- Aluminide Ni<sub>3</sub>Al formation of aluminum interlayer coating through heat treatment at a temperature of 500°C prior to YSZ top coat is planned because it provides good oxidation resistance and excellent surface for ceramic top coat.
- > To check the bond and top coat thickness via cross sectional analysis.
- To check interface morphology, structure and purity of bond and top coat performed XRD, SEM and EDX analysis.

- To investigate the wear volume and wear rate of coated Monel-400 coupon via digital tribometer.
- > To optimized the silicon slurry.
- > Deposition of slurry through digital dip coater.

## **Schematics of Research Strategy**



## **Summary**

In this chapter, firstly we discussed the efficiency of gas turbine and how we calculate the efficiency of the gas turbine thermodynamically. Then we discussed different techniques in order to improve the efficiency of gas turbine. Different coating strategies and high-performance materials are discussed regarding gas turbine efficiency. Material issues like corrosion, wear and oxidation of gas turbine are also discussed. A detailed literature review of gas turbine coatings is discussed at the middle section of this chapter. The major focus of this research is to find out new ETBC coating for gas turbine. We develop a novel coating scheme in order to minimize the wear rate of gas turbine due to severe environmental conditions. At the end we discussed advanced ETBC materials and objectives of this research.

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## Chapter #03

# **Experimental and characterization tools**

In order to develop a novel coating system thermal spraying technique are used to enhance the efficiency of gas turbine. These processes play significant role in ETBC's due to higher deposition rate and dealing with variety of substances.

## **3.1 TBC processes:**

Thermal spray coatings are used in a wide variety of applications which can secure huge material loss from severe environmental conditions. There are several different processes used for implementation of TBC coatings on different surfaces, but the most common methods are given below[1]:

- i. Powder flame spray system
- ii. Wire Arc spray system
- iii. High velocity Oxy-fuel system
- iv. Plasma spray Technique
- v. Cold spray system

#### **3.1.1** Powder flame spray system and its operational procedure:

In this technique, our coating material is in powder form and energy require for melting powder is taken from gaseous fuel. Generally, two gasses Oxygen and any other fuel gas like Acetylene or Methane is used to generate flame. This flame melts the feedstock powder and then deposits on substrate. A carrier gas is also used to provide sufficient kinetic energy to the melted material, which can easily deposit on require substrate. Carrier gas may be oxygen, compressed air or nitrogen. In this technique we achieve high porosity, relatively low cohesive strength and typical spray rate is 0.5 to 9 kg/h (1 to 20 lb/h)[2]. Typical powder flame spray mechanism is shown in following.



Figure 18: Powder flame spray Process [2]

## **Operational procedure for SX - 6016 subsonic Powder spray equipment:**

- Turn on power supply for compressor from main power cabinet
- Check the voltages for compressor; it must be in the range of 390-410V and monitoring any abnormal sound.
- Plug-in power supply for dryer and check the level of drying gasses indicated on gauge.
- $\blacktriangleright$  Push start button from compressor controller.
- Check leakages from all bending or coupling involved.
- Drain out the moisture through valve from bottom of the storage tank.
- $\blacktriangleright$  Push green button to start the dryer.
- > Open the compressed dried air supply to the powder spray equipment.

- Ensure the smooth availability of gasses with desired pressure from sources.
- Open the supply of gases (Oxygen and Acetylene) from cylinders to the control cabinet of powder spray equipment.
- Adjust the required parameters i-e Pressure and flow rates of gasses.
- Insert the required powder (must be fully dried and free of contamination) into the bottle mounted on gun.
- Turn-on gases from control cabinet and generating the flame by providing external spark source.
- After that, stabilize the flame by regulating the parameters.
- > Then turn on the powder and start the coating on required sample.

#### 3.1.2 Wire arc spray system and its operational procedure:

In this type of flame spray, the feedstock is in wire form and is melted by the means of high electric current. In this technique, compressed air is used to atomize the melted substance onto required surface. Compressed air also provides cooling to the spray gun. Wire feed speed is controlled with the help of speed controller place at control cabinet and adjust according to the nature of substrate, nature of wire and required coating thickness. The potential difference between two wires is generally in the range of 15-50V. The mechanism of wire arc spray system is shown in following fig. In wire arc spray system, generally wires are in the range of 0.2- 3mm diameter depends on required application. The two major limitations of this technique are wires must be electrically conductive and high voltage power supply is required to melt the wires[3].





#### **Operational procedure for SX-400 wire arc spray system:**

The operational procedure consists of compressor and then followed by wire arc spray system.

#### Air Compressor Unit:

- Screw air compressor
- LG series oil injection screw air compressor is a kind of double axis capacity rotating air compressor.
- > Air inlet port is at the top of the machine and discharge port is at the bottom.
- There are two high precision rotors fixed parallel and horizontally inside the machine.
- > One is main rotor and other is subsidiary rotor.
- > There are 5 teeth in main rotor while subsidiary rotor contains 6 teeth.

Before starting the air compressor, two things mustly checked, First is the lubricant oil level and second is make sure any physical damage or leakages due to insects or environmental conditions.

#### Working principle of Screw compressor:

The working mechanism of Screw compressor (Model: LG-3/8) consists of three parts, Compressor, Storage tank and a dryer. The screw compressor consists of following four strokes:

- Suction Stroke
- Sealing and Transit Stroke
- Compressing and oil injection stroke
- Discharge Stroke

In suction stroke, a regulating valve controls the air inlet. When teeth-ditch the space of the two rotors, rotate to the air inlet end the outside air be sucked into vacuum teeth-ditch space. When the space is full of air the rotor's air-inlet end will rotate away and seal the space. This is called course of suction and suction stroke is completed. In Sealing and Transit stroke, the teeth peak part of the rotor will sealing with machine cover to avoid the outflow of air. This is called course of sealing. The two rotors go on rotating their teeth ditch and teeth peak will integrate at the suction end and the matching surface will rotate towards the discharge end gradually. This is called transit course. In 3<sup>rd</sup> stroke, the air between matching surface and the discharge port being compressed gradually and the pressure rise up quickly. This is called compressing two duties, lubrication as well as cooling. In final discharge stroke, the rotor's discharge port end connects with the machine cover, the compressed air begin to be discharge till the matching surface move away from the discharge end. This is called

discharge stroke and at the same time the distance between matching surface and the inlet port is the longest. This compressed air goes to storage tank having capacity 8 liters. Then this compressed air moves towards dryer where R-404A refrigerant is used to dry the compressed gas before reaching to the equipment. Some of the important parameters of screw compressor are given below:

Parameters	Value and Unit
Air flow rate	3m <sup>3</sup> /min
Compressed air	0.5MPa,
pressure	
Revaluation	3240 R/min
Power	18.5KW
Weight	650kg

 Table 3.1: Compressor parameters

#### Air Tank:

- > Open the valve to drain out any entrapped air or moisture.
- > Note down the temperature and pressure of the compressor unit.
- Check the voltage reading it should be in the range of 390-410V. If it is higher than compressor must be switch off.
- ➢ Air pressure in compressor set at 0.5MPa.

#### **Refrigerant in dryer:**

Refrigerant, a substance used for refrigeration. It may be a single component or mixture. Refrigerants are available in any phase depends upon the type of application. Refrigerant must contain following properties

Excellent thermodynamic properties

- Non Corrosive
- Non Toxic
- Non Flammable

Refrigerants are generally classified into three categories as follow,

Class 1: It includes refrigerants that cool by phase change, so Latent heat is involved.

**Class 2:** It includes refrigerants that cool by temperature changes, so sensible heat is involved.

**Class 3:** This group consists of solutions that contain absorbed vapors of liquefiable agents or refrigerating media.

Generally used following refrigerants in dryer for cooling purposes,

### R 404A, R 22, R 134a, R 407.

- R 400 series made up of zeotropic blends (No intersection of bubble and dew curve).
- R 500 series consist of azeotropic blends. (Bubble and dew curve intersect each other).
- ▶ R 700 series contain non-organic blends.

## Wire Feeding in Spraying Gun:

Two metallic wires as the coating feedstock will feed in electric arc spray gun as par following procedure:

- Ensure Main Circuit breaker from Main Electrical DB is OFF.
- > Cut the safe length of wire and feed it to insulated loop of wire attach with gun.
- > Feed wire in Loops till feeding gears set in gun on both sides.
- Loose up the gears assembly and adjust the wires in front tip of gun hat.
- Cover loop with suitable insulator.

- Now Ensure Power A (Black) & Power B (Red) cables are not attached at front side of the control cabinet.
- On main circuit breaker from main electrical distribution board (DB) and then attach the power cables at front side of the machine.
- On the power switch present at the back of equipment. It will supply the electricity to equipment but it does not start the machine.
- Press the start button present on supersonic arc spray equipment machine on front panel top right side having green color.
- Turn the spraying voltage controller at 1 (36V), its values indicate on spraying volt meter (placed at top left corner).
- Rotate the knob of wire speed controller to adjust the desired wire feed voltage (clockwise to increase and anti-clockwise to decreases).
- > Now operate wire feeding button on gun handle.
- When both wires equally came out from front of the gun nozzle and joint at a point, stop feeding.
- > Shutdown main power unit and then cutoff wires ahead of jointing point.

## Wire spray operation:

- > Ensure enough feedstock of both wires are available with electric arc spray gun.
- > On main circuit breaker from main electrical distribution board.
- On the Power Switch present at the back of equipment. It will supply the electricity to equipment.
- > Turn on the power switch present at the front side of equipment.
- Rotate the spraying voltage controller at number 1=36V, 2=42.5V & 3=48V as per requirements, its values indicate on spraying volt meter.
- Now turn on the electricity from button available on gun and flame is start, check the flame stability and spraying intensity.

Spray on the required coupon.

#### 3.1.3 High velocity oxy-fuel (HVOF):

HVOF is modified form of powder flame spray technique in which we get high particle velocity for excellent adhesion. In this technique oxygen and acetylene gasses are used to generate the flame. The particle velocity in powder flame spray technique is 50-70 m/s while in HVOF system the particle speed reaches up to 420-450 m/s. To achieve this high speed HVOF guns having convergent-divergent nozzle. We can achieve an excellent deposition rate through this technique, i-e 2.3-14 kg/h. There are several key benefits of using this technique like fast deposition rate, good coating density and minimum oxide contents. The low oxide content is due to the high velocity of the system. The flame range of this technique is nearly 3000 °C which is sufficient to melt the complex compounds. The mechanism of HVOF technique is shown in following fig.



Figure 20: High velocity Oxy Fuel system [2]

#### 3.1.4 Plasma spray system:

In plasma spray technique a high voltage DC current is required to generate thermal plasma. The high frequency direct current is responsible for generating electric plasma arc. A high velocity and high flow rate inert gas like helium or argon with combination with any fuel gas like hydrogen or methane is applied along the axis of arc, so this thing is responsible to push plasma arc towards substrate. The required depositing material which is either in powder form or wire form is also supplied with injection of inert gas. The working mechanism of atmospheric plasma spray is shown in following fig.



Figure 21: Atmospheric Plasma Spray system [2]

In plasma spray system, temperature is very high so plasma spray gun is made up of copper anode and tungsten cathode. A water circulation is also provided to cool down the gun temperature. This cooling is necessary to avoid any damage due to excessive temperature at gun and also provide modest environment for operator i-e especially for manual handling. There are several types of plasma spraying besides atmospheric plasma spraying technique. The major plasma spraying categories are given below[4]:

- i. Vacuum plasma spraying
- ii. High pressure plasma spraying
- iii. Liquid stabilized plasma spraying
- iv. Inductively coupled plasma spraying
- v. Laser plasma spraying

#### 3.1.5 Cold spraying system:

In this type of spraying temperature of melted particle is much lower than other thermal spraying methods while particle velocity is very high. Generally, the temperature is in the range of 700-800 °C and particle velocity is 300-1200 m/s. To achieve higher particle velocity de-Laval nozzle is used which provides pressure of melted particles up to 4MPa with very high speed. The process gas is any fuel gas like methane or hydrogen while the carrier gas is helium. The use of helium instead of compressed air provides two major benefits, avoid oxidation of coating material/powder and also helpful for achieving higher particle velocity. The cold spraying mechanism is shown in following fig.



Figure 22: Cold Spraying Method [4]

#### **3.1.6 TBC applications:**

Thermal Barrier Coating (TBC) having a wide range of applications in different engineering fields. Several mechanical or electrical appliances like turbine, boiler wall, marine engine and electrical circuits etc, require coating for their smooth and efficient operation. The purpose of coating depends on the nature of application or requirements and also on nature of coating material. Some of the major applications are given below,

i. Coating used as corrosion resistant

- ii. To control friction
- iii. To insert electrical or insulator properties
- iv. Used as wear resistant in severe environmental conditions
- v. Secure material from high heat flux
- vi. Also used as wettability resistant

## **3.2 Dip Coating Method:**

For the deposition of silicon bond coat on C/SiC substrate, automated dip coater is used. For this purpose, firstly we prepared silicon slurry as follow:

### 3.2.1 Dip Coater:

WPTL6-0.01 programmable dip coater is designed for multi samples (06) in a single step within a controlled temperature chamber. Some of the salient Features of dip coater are given below:

- Digital display and controller (adjust pulling or dipping rate of sample and no of cycles)
- Range of pulling and dipping rates is 1-40 mm/min.
- Maximum dipping distance of sample is 80mm or 3.14 inch.
- Six 500ml glass cups are used at a time.
- Temperature of chamber is also maintained up to 98°C via a attach temperature control system as per requirements.
- > Dimensions:  $500 \times 500 \times 1180 \text{ mm} (L \times W \times H)$ .



Figure 23: Dip Coater equipment used in AEMS Lab.

## **3.2.2 Operational procedure:**

The operational procedure of automated dip coater machine is very simple and easy to use. The step wise procedure is given below:

- First of all, loading required sample (Slurry and coupon). Adjust the relative position of sample and liquid via a Manual pulley.
- Sample dipping depth should be 1 cm above from the beaker bottom.
- ➢ Close the door.
- Turn on power supply and set your required program which includes sample Up/down travel speed and no of cycles.
- > Then touch Run key to execute the program.
- > Touch pauses key if you require drying time for sample.
#### Temperature control system of Dip coater:

If we require heating of our substrate during slurry deposition, then we must well aware of its temperature control unit. A stepwise procedure for controlling the temperature is given below:

- Make sure it matches the voltages of external power (110/220 AC Volts). Close the door and then lock the handle.
- Set up desired temperature by using setting button. After that temperature starts to get up in the oven.
- When the real temperature is near the set-up temperature, the heating light is on and off. It may repeat several times and then go to constant temperature state.
- If there is difference between set and real temperature and the system is in constant temperature state then adjust the reset screw as following:
- a) When the real < set, use the screw driver to rotate the reset screw in a clockwise direction a little. Then the heating light is on to continue the job until real temperature reaches to set.</li>
- **b**) When the **real** > **set**, rotate the reset screw in anti-clockwise direction.



Maximum temperature achieved in chamber is 98°C.

Figure 24: Temperature control unit of Dip coater.

#### **3.3 Sand blasting:**

Sand blasting is a technique in which surface of substrate is cleaned or smooth with the help solid particles using high pressure compressed air. The solid particles are either grains of sand or mixed with alumina. Some sand blasters are portable and easily move from one place to other while some are cabinet type and didn't move from its installation place. Generally a sand blaster is consist of four basic components like blasting media, compressed air source, bag filter or dust catcher and controlled panel or cabinet. Firstly the substrate is strongly gripped in cabinet and high pressure air is supplied from compressor. The pressure of compressed air is adjusted as per requirements and the nature of substrate. If high pressure is adjusted for a ductile material then deformation or tiny holes may appear and surface is damage. The dust is controlled and collected with the help of bag filters and then recycles to the source. After sand blasting a smooth and clean surface is ready for further experimentation or its application. Several types of blasting Medias are also available and again it depends on nature of substrate and its application[5].



Figure 25: Sand blasting equipment [5]

## **3.4 Optical Microscopy:**

Optical microscope is also known as light microscope that uses a system of lenses and visible light to magnify the object. Optical microscopy consists of objective lens, ocular lens, reflector, stage and lens tube. Firstly, the sample is placed on sample stage and magnifies it through objective lens. After that further magnification occur through ocular lens and then a well magnified image is seen on screen. Basic components of optical microscopy are given below [6, 7].



Figure 26: Basic components of optical microscopy [6]

#### 3.4.1 Working principle:

In a compound microscope, generally two lenses are used for higher magnification and image quality. Firstly, light coming from source is reflected up through sample and inverted magnified image is created by objective lens. Then this image is further magnified by eyepiece lens and a well magnified virtual image is seen through nicked eye as shown in following fig [8].



Figure 27: Principle of optical microscopy [8]

## **3.4.2 Operational Procedure:**

In order to operate optical microscopy in a safe mode and get desired result, a detailed stepwise procedure is given below:

- First of all, check all hardware and make sure that there is no physical damage is visible. Then turn on the power supply and lamp.
- After providing the power, then start to adjust the brightness with the help of knob as shown in fig. By rotating the knob in a clockwise direction, we can increase the voltage and hence our brightness will be strengthened and vice versa.



- Then place the sample on sample stage and be sure that sample surface is parallel to the board and it should be placed properly i-e eliminate the shaking effect of the sample.
- Generally, use the slide for placing un-even surfaces on sample stage. Then use the portrait and lateral adjustment knob to move the specimen onto the required position.
- Now start to adjust the focusing right area on surface with the help of Diopter.
- With the help of ND filter knob, we can select the light path either it is Brightfield (BF) or Dark field (DF).
- After adjusting the light path on focused area, we can ready to take desired image with suitable magnification lens.
- After taking desired image, use suitable filter to process the image i-e marking dimensions, highlight focused area and color adjustment.
- If you are working in a short distance between objective lens and sample then be sure that, there is no physical contact between sample and lens during the changing or rotating the magnification lens.



Figure 28: (a) Sample Stage, (b) Diopter and (c) Lever selector

#### **3.5 SEM and EDX:**

Scanning electron microscopy is an imaging technique used for analyzing the topography, structural crystallinity and orientation of materials making up the sample. Generally, an electron beam is emitted from an electron gun having tungsten filament, which act as a cathode source. Tungsten filament is used because it has highest melting point and very low vapor pressure among all metals. Several other electron emitters are also used like lanthanum hexaboride (LaB<sub>6</sub>) and Zirconium oxide, depending upon the requirements and type of electron gun is used. Electron beam having energy in the range of 0.2 keV to 40 keV is directed towards specimen with the help of condenser lenses to a tiny spot having diameter up to 0.4-5 mm. This beam passes through scanning coils and deflector plates, so it will propagate in x, y direction. This thing provides a suitable environment to beam in order to scan our specimen in a roster fashion [9]. All these emitting signals are captured by specialized detector and display on screen. Each emitting signal contains some specific information about sample. Backscattered electron provides information about atomic number and phase differences of the sample, while secondary electron is provided information about sample's topographical information and x-rays reveals information about sample's constituents[10].



Figure 29: Working mechanism of SEM [10]

#### 3.5.1 Raster Scanning:

In a raster scanning, a highly focused beam of electron is swept from top to bottom in a row by row fashion. In this pattern, scanning starts from left to right and covering whole area of specimen systematically. In this operation, an image is divided into several strips which is known as scan line. Then each scan line is transmitted in the form of analog signals and reads it from video source then further divided into several pixel for processing in attach computer system. This ordering of pixel is known as roster order or roster scan order. Refresh buffer memory or Frame buffer memory can store picture definition coming from video controller. Then these stored values painted on digital screen one row at a time. In this technique, sample is scanned horizontal as well as vertical scanning[11].

 Table 3.2: Benefits and Limitations of Raster scanning [11]

Benefits	Limitations
Shows more realistic picture	Low resolution
Provide more control	So tedious when picture was too small
	than screen
Shadow screens are conceivable	Expensive

#### **3.5.2** Types of electron guns:

There are three types of electron guns which are commonly used as a source of electron beam given below:

- ➤ Thermal emission electron gun
- ➢ Field emission electron gun
- Schottky emission electron gun



Figure 30: (a) Schottky Emission E-gun, (b) Thermal Emission E-gun, (c) Field emission E-gun [11]

In thermal emission and field emission guns, they generally used filament of Tungsten (W) or Lanthanum hexaboride (LaB<sub>6</sub>). In thermal emission gun, electrons are emitted from outer shell of filament source with the help of external energy provided by any thermal source. But, in case of field emission gun, strong electric field is applied so that outer shell electrons tunnel through the barrier. A single crystal tungsten filament is used, which is coupled with tungsten wire and the tip of the crystal is in curvature shape. A very high vacuum i-e 10<sup>-6</sup> to 10<sup>-8</sup> Pa is required, depending upon the requirements, filament used and type of gun used. In Schottky emission electron gun, a beam of electron is produced due to Schottky effect. In this effect, a very high electric field is applied across the filament which decreases work function for outer shell electrons. In this gun, a single crystal tungsten filament is used coated with zirconium oxide. A very high vacuum of 10<sup>-7</sup> is required for Schottky emission electron gun. The parametric comparison of these three guns are given below[12]:

Characteristic	W	LaB <sub>6</sub>	Cold FE	Thermal	Schottky
Type of Emission	Thermoionic	Thermoionic	Field emission	Field emission	Field emission
Vacuum (Pa)	10-4	10-5	10-7	10-7	10-6
Electron source size	30µm	10µm	5nm	5nm	20nm
Cathode Temp. K	2,800	1,800	293	1,800	1,800
Energy spread eV	2	1.5	0.2	0.3-1	0.5-1
Brightness (A.cm <sup>-3</sup> .sr)	106	107	109	108	108
Stability %	1	1	5	5	2
Life hours	50-100	200-1,000	2,000	2,000	6,000
Application	Standard SEM. VP SEM. EDS WDS		High Resolution SEM. EDS EBSP		HR-SEM EDS,WDS CL EBSP

 Table 3.3: Comparison of electron sources [12]

#### **3.5.3 Energy Dispersive X-ray spectroscopy:**

When a beam of electron is interacted with sample, several emissions take place along with X-ray emission. A special EDX detector is used to capture characteristics xrays and generates its energy spectrum. Then this energy spectrum is used to analyze the elemental composition of the sample. Generally, EDS system is attached with Scanning electron microscopy (SEM) in which a special detector is used for capturing x-rays usually made up of Si(Li) crystal. This detector is placed inside the sample chamber. At the end we get our result in portrayed graphical form, where graph is drawn between x-rays (Counts) on abscissa and energy (keV) on ordinate. A typical EDS spectrum and micrograph of Aluminum is shown below:



Figure 31: EDS analysis of pure Aluminum

## **3.6 X-ray Diffraction:**

X-rays were discovered by Wilhelm Conrad Roentgen, a German physicist in last decade of 19<sup>th</sup> century. He discovered x-rays while performing experiments on cathode ray tube. X- Rays are generally electromagnetic waves having wavelength of 1A°, which is also the size of an atom. They occur in the region between gamma and ultraviolet on spectrum. There are two methods for x-rays production. In first method, when high energy negative particles strike on a metal target then x-rays are produced. This is usually done in a tube. In second technique, high energy electrons are captured in a storage ring, this method is known as synchrotron. When they are moving in a circular path, electrons are accelerated towards the center of the ring and thus emitting electromagnetic rays. X-ray diffraction is normally used for identification of crystalline phases of various materials. Each crystalline substance having unique structure just like fingerprint of any human. X-ray diffraction technique requires a very small amount of sample and characterizes it without any damage.

#### 3.6.1 Working Principle:

In working principle, firstly sample is placed in analyzing chamber and then x-rays are generated from any suitable available facility. These rays are pointed towards the sample surface. Some rays are diffracted and some are transmitted depending upon the nature of sample. Generally, x-ray diffraction phenomenon is based on constructive interference by following Brag's law. In this principle, some monochromatic x-rays are incident on crystalline material, atoms in parallel layers act as a source of scattering the rays of same wavelength. When x-rays are scattered from any crystalline substance, several peaks with different intensities are observed which correspond to the following Brag's conditions[13]:

- i. Angles made by incident ray and scattered ray must be equal.
- ii. The path difference must be integral multiple of wavelength.



 $n\lambda = 2d'\sin\theta$ 

Figure 32: Sketch of Brag's principle [13]

## 3.6.2 Major components of XRD equipment:

- **X-ray tube:** Used for the generation of x-rays
- > Collimator: To provide the specific direction to the scattered radiation
- Detector: Used to captured diffracted rays and convert it into readable signals.
- **Goniometer:** It is used to rotate and hold the specimen for analysis.
- Monochromator: It allows only a specific wavelength of rays over a wide availability



Figure 33: XRD machine and its major components [13]

#### 3.6.3 Areas of applications:

- i. Crystal structure
- ii. Size of particles
- iii. Differentiate between Cis-Trans isomers
- iv. Find out linkage isomerism
- v. Low angle scattering
- vi. Different phases in material
- vii. Find out the atomic arrangement

## 3.7 Tribometer:

Tribometer is an instrument used to measure several quantities like wear rate, coefficient of friction and wear volume of two surfaces that are relative in motion. These tribological properties play a significant role for the selection of suitable material for any particular project. Its working was very simple, generally we insert our desired parameters to a calibrated system and then receive results in graphical form. Its working largely depends on relative motion of two surfaces. A typical Pin and Disc tribometer is shown below:



Figure 34: Tribometer equipment used at IST Tribometer Lab

#### 3.7.1 Types of tribometer:

There are several types of tribometers are available now a day. But, their usage mainly depends on requirements and the nature of the sample to be analyzed. Some of the major types are given below:

- Ball and Disc Tribometer
- Pin and Disc Tribometer
- Bouncing Ball Tribometer
- Block on ring Tribometer
- Twin Disc Tribometer
- > Static friction Tribometer

#### **3.7.2 Applications:**

- Materials tribology (Wear rate, Wear volume)
- Automotive Industry (Lubricant test on different materials, Gears and Breaks)
- Medical field (Bio tribology especially in artificial cardiovascular devices)
- Mechanical equipment's (Gas turbine components, Heat exchanger and Boiler)
- Aviation field

## **Summary**

In this chapter, firstly we discussed all thermal spray methods like wire arc spray, powder spray system, HVOF and cold spraying etc in details along with their schematic diagram. Then described the dip coating method along with slurry preparation and slurry optimization. After that, discussed the sand blasting equipment in detail. This equipment is usually used for the preparation of substrate surface for good adhesion. Then discussed the characterization tools like Optical microscopy, SEM, EDX, XRD and Tribometer in detail. The working principle of these equipment's, their types, operational procedure and applications were discussed. These characterization tools having high importance in order to extract required results.

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## Chapter #04

## **Experimentation**

## 4.1 Surface preparation via sand blasting:

Sand blasting was performed in order to smooth the surface of substrate for upcoming coating. In this research work, we have a super alloy (Monel 400) as a substrate having dimensions  $15 \times 12 \text{ mm}$  (L  $\times$  W). We used a sandblaster manufactured by Guangzhou Sanxian Metal S & T Co limited manually operated. We clutch substrate with one hand and sand blasting gun is firmly grasp with other hand in a blasting machine. Adjust the parameters like blasting pressure, compressed air pressure and stopwatch timer. The power to start the gun is controlled from foot. Start the gun and tried to develop blasting in a shape of number eight (8) for surface homogeneity. There are several factors which affects the sandblasting process like time, blasting pressure and carrier gas pressure. In this work, Compressed air is used as a carrier gas. Blasting process. The blasting pressure depends on nature of the substrate, soft material requires low blasting pressure and vice versa. Similarly, if we provide more blasting time a thick layer of sand is formed at surface which is undesirable. The process parameters are given below:

Compressed air Pressure = 0.5MPa, Time = 15 sec, Blasting Pressure = 0.3 MP



Figure 35: Sand blaster

Sand blasting is salient equipment for preparation of substrate surface. Step wise procedure of sand blasting is given below:

- First of all, provide power supply.
- Check air pressure (must be less than 0.5Mpa)
- ➤ Grasp the substrate with wise clip.
- > Adjust the blasting pressure suitable for required substrate.
- Now start the sand blasting on substrate in the shape of spiral or 8.
- At the end must ensure uniform and smooth coating on substrate.
- Then wash the substrate with ethanol to remove the unadhere sand particles.



Figure 36: Salient parts of sand blaster

#### 4.2 Aluminum deposition via wire arc spray method:

After sand blasting on super alloy (Monel 400) substrate, we want to develop aluminum bond coat via wire arc spray system. Pure aluminum having diameter 2mm is used. Aluminum is suitable candidate for bond coat due to its excellent surface for top coat and also creates hindrance form oxidation. We used a state-of-the-art technology (wire arc spray system) for bond coat due to the reasons that it provides good adhesion, fast deposition rate and uniformity of the thickness. The thickness we achieved by this method with adjusted parameters is 180 µm, confirmed by cross sectional analysis. For deposition of bond coat, we used machine manufactured by Guangzhou Sanxian Metal S & T Co Limited having model name SX-400 supersonic arc spray system operated manually. A high voltage of power is used to melt the aluminum and then compressed dry air is used to provide sufficient velocity for deposition. In this technique we controlled coating thickness with the help of wire feed voltages, distance between gun and substrate, compressed air pressure and time. Following parameters are adjusted to achieve desirable coating thickness:

Wire feed voltage = 5 V, Air pressure = 0.5 MPa, Time = 8 sec Coating thickness =  $180 \mu m$ , Distance between gun and substrate = 8cm



Figure 37: Wire Arc spray System

## **4.3** Aluminide formation via thermal annealing:

Aluminide formation plays significant role against severe environmental conditions. After successful deposition of aluminum coating via wire arc spray system, nickelaluminum bond formation occurs via thermal annealing. Thermal annealing is performed at 500°C for three hours. During this span Ni-Al bond formation occurs and this bond plays salient role against oxidation. This thermal annealing is done due to the reason that, it will definitely enhance the properties of bond coat. Nickel aluminide (Ni<sub>3</sub>Al) shows similar properties of ceramic and metal because this compound is considered as intermetallic alloy. An ordered phase formation occurs at a certain temperature between two metallic substances, known as intermetallic alloy. Generally, bonding starts between nickel and aluminum from 350°C. In this particular case, where aluminum is deposited on Monel-400 alloy, the bonding between nickel and aluminum also starts at 350°C and content of nickel is increased as we raise the temperature. The ordered structure of intermetallic alloy shows superior properties against oxidation and also provides excellent barrier to the diffusion of heat. These intermetallic alloys are possessing high mechanical strength and low density, make it suitable for high temperature applications like jet engine and gas turbine blade coatings.



Figure 38: Nickel-Aluminide formation via thermal annealing

## 4.4 Deposition of silicon on silicon carbide via Dip coating:

For the deposition of silicon bond coat on C/SiC substrate, automated dip coater is

used. For this purpose, firstly we prepared silicon slurry as follow:

#### **Chemical required:**

Silicon powder = 40 gram, Ethyl Alcohol = 20 gram (which act as a solvent) Polyvinyl-butyral = 0.8 gram (which act as a Binder) Phosphate Ester = 0.12 gram (which act as a dispersant).

## **Procedure:**

First of all, mix ethyl alcohol with phosphate ester and magnetically stirred for one hour. Then added Silicon powder and solution is stirred for 5 hrs. Finally, PVB is added and again stirred for 24 hrs. After preparation of slurry, substrate is vertically dipped in well stirred slurry via dip coater using defined parameters, like dipping speed and number of cycles etc.



**Figure 39: Silicon slurry preparation** 

#### 4.4.1 Silicon Slurry optimization:

We can collect optimized results from silicon slurry deposition by varying the amount of solvent, dispersant and dipping speed. When we used more solvent and dispersant then we obtain excellent uniform coating. The dipping and pulling speed of substrate also affects the end product. Following parameters were adjusted for slurry optimization:

Chemical/Parameter	Experiment -01	Experiment -02	
	(gm)	(gm)	
Silicon Powder	15	20	
Ethyl Alcohol	20	25	
Polyvinyl Butyral	0.8	0.8	
Phosphate Ester	0.12	0.15	
Dipping speed	25	20	
Pulling speed	25	20	
Number of cycles	02	02	

**Table 4.1: Silicon slurry optimization parameters** 

#### **4.5 Deposition of YSZ top coat via Powder Spray Method:**

After deposition of aluminum bond coat via wire arc spray system. Then we deposit a ceramic (7YSZ) top coat to safeguard the substrate from continuous high temperature environment i-e 1000  $^{0}$ C. A very fine 7YSZ powder is deposited via thermal powder spray system. Yttria stabilized zirconia having excellent thermo-mechanical properties at elevated temperature. We can achieve fast deposition rate, good adhesion and effectively handle variety of substrates via powder spray system. In this method, Acetylene (C<sub>2</sub>H<sub>2</sub>) and Oxygen (O<sub>2</sub>) are used to generate the flame which melts the powder. During whole deposition process a great care about leakages of gases must be taken to ensure a safe and smooth process. The temperature of the flame goes above  $3000 \, {}^{0}$ C and safety of operating personal must be very necessary because the operation was performed manually. Compressed air is also used which provide enough velocity to melted material to be deposited on required substrate. There are several parameters which affects the coating thickness like gas pressure, flow rate, pressure of compressed air, time and distance between gun and substrate. The thickness we achieved by adjusted parameters is 144  $\mu$ m confirmed by optical microscopy. Following parameters are adjusted to achieve desire coating:

Oxygen  $\longrightarrow$  0.2MP. Oxygen  $\longrightarrow$  1 m<sup>3</sup>/h, Acetylene  $\longrightarrow$  0.4MP Acetylene  $\longrightarrow$  0.9 m<sup>3</sup>/h, Compressed air  $\longrightarrow$  0.4 MP.



Figure 40: Powder Spray system for top coat deposition

## 4.6 Wear rate via tribometer:

At the end we can use ball and pin tribometer in order to calculate the wear rate of coated and non-coated sample. We used different loads like 3N and 5N, in both cases the coated sample shows far less wear rate and co-officient of friction than non-coated one. We can measure tribological quantities like wear volume, coefficient of friction and wear volume from a particular instrument called tribometer. Wear rate is calculated when two opposite surfaces moving each other with some adjusted parameters. Tribometer was firstly used by a Dutch scientist Musschenbroke, in 18<sup>th</sup> century. This equipment is used to characterize the substrate and provides the suitable data which is graphically presented and analyzed. Generally, tribometers are very much specific in their operation and fabricated as per requirements. Some of salient feature of experiment is given following table:

Element	Property
Disc Material	Steel
Ball/Pin	Steel
Lubricant	Nil
Ball diameter	3.0mm
Speed	100 RPM
Weight	3 and 5 Newton
Distance	10m

**Table 4.2: Parameters of tribometer** 

## Summary

First of all, substrate surface was prepared via a sand blasting on Monel-400 super alloy. Sand blasting was performed in order to smooth the surface of substrate for upcoming coating. There are several factors which affects the sandblasting process like time, blasting pressure and carrier gas pressure. There are several factors which affects the sandblasting process like time, blasting process like time, blasting process like time, blasting process like time, blasting on super alloy (Monel 400) substrate, we want to develop aluminum bond coat via wire arc spray system. We used a state-of-the-art technology (wire arc spray system) for bond coat due to the reasons that it provides good adhesion, fast deposition rate and uniformity of the thickness. In this technique we controlled coating thickness with the help of wire feed voltages, distance between gun and substrate, compressed air pressure and time. Aluminide formation occurs through thermal annealing. Then deposit YSZ top coat via powder spray method. At the end, wear rate was determined via ball and pin tribometer.

## Chapter #05

## **Results and Discussion**

After successful deposition of both (bond and top) layers via thermal spray techniques, we characterize these layers to generate results with the help of characterization tools.

## 5.1 XRD analysis of Aluminum deposition:

After deposition of aluminum bond coat via thermal spray technique, XRD analysis is performed. During analysis the value of angle  $(2 \theta)$  was adjusted from 20-80 and peak intensity was measured in Counts. All major silicon peaks are laying in the above given 2 Theta range, confirmed form literature and that's the reason I would like to adjust this range of value for analysis. After XRD testing with desired parameters the final result was analyzed with Jade software then plotted in origin software and it is pretty clear that pure aluminum was deposited, no oxygen was found and this thing shows that there is no oxidation occurs during deposition process. A single PDF card (JCPDS# 85-1327) is match with all silicon peaks and following figure confirmed it. If oxygen peak was detected, then surely oxidation of aluminum occurs and eventually it will form Al<sub>2</sub>O<sub>3</sub>.



Figure 41: XRD pattern of aluminum deposition

#### **5.2 SEM and EDX analysis of bond coat:**

After XRD analysis, the coated sample was analyzed by Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDX) in order to study the morphology and composition of coating. Firstly, Monel sample coated with wire sprayed aluminum was analyzed through SEM under different magnification i-e 50x, 275x and 345x. As magnification increases over 200x several very fine pores were seen on coated areas. The presence of these pores is due to irregular movement of manually operating coating gun and distance between the gun and substrate. These wholes appeared due to excessive burning of aluminum. In following figure, we can clearly see that coating is well adherent and uniform besides few little pores. Secondly, this coated sample was analyzed under state of the art EDX machine in order to confirm the composition of the coating. In following figure, the EDX spectrum clearly shows that there are no impurities like oxygen, carbon and Sulphur etc. involved in this coating deposition. If any impurity is involved in this process, it must appear on digital EDS spectrum[1].



Figure 42: SEM analysis (a) 50x, (b)275x, (c)345x, (d) EDX analysis of Aluminum deposition

## 5.3 Cross sectional analysis of bond coat:

After confirmation of pure deposition of bond coat, its thickness is measured via cross sectional analysis. In order to measure the thickness of bond layer we used organic epoxy resin and hardener. The approximate quantity of epoxy was 16 grams and 10 drops of hardener, depending upon the dimensions of the sample. After placing our sample in a suitable quantity of epoxy solution then remain this whole system for a while to dry the sample (dipped in solution) at room temperature. After the completion of drying, the epoxy sample was manually polished with different grades of sand papers, starting from sand paper number 120 and reaches up to number 1200 (a very fine). Then this polished sample was analyzed in optical microscopy and reveals that our bond coat thickness was 180  $\mu$ m. The coating thickness was in the range which we require and expected before the experiment[2,3].



Figure 43: Cross sectional analysis of Aluminum deposition

## 5.4 Ni-Al bond formation via thermal annealing:

After successful deposition of aluminum bond coat on Monel-400 sample thermal annealing is performed at 500 <sup>o</sup>C for 3 hours. The reason for this heat treatment is the formation of Ni-Al bond, which strengthen our bond coat and provides excellent safety against oxidation. The selection of temperature 500 <sup>o</sup>C is related to literature, so that at this temperature Nickel-aluminum bonding was formed. As temperature increases the amount of Nickel in compound with aluminum is also increases, but we require only Ni-Al so this thing is achieved at 500 <sup>o</sup>C. After thermal annealing, the sample was analyzed under XRD machine, which reveals the confirmation of Ni-Al bonding at this selected temperature 500 <sup>o</sup>C, which is taken from nickel-aluminide phase diagram. Monel substrate contains majority part of nickel element and during thermal annealing the nickel molecules excited and move towards surface and hence combine with aluminum to form nickel-aluminide[4].



Figure 44: (a) XRD analysis of Thermal annealing, (b) Reference card, (c) Phase diagram of Ni-Al bonding [4]

#### 5.5 XRD analysis of Silicon deposition and slurry optimized results:

Silicon was deposited via slurry deposition technique on silicon carbide substrate by using digital dip coater. This layer of silicon is act as a bond coat, which reveals excellent protection against oxidation and provides good surface for top coat. After successful deposition of silicon, XRD analysis was performed. During this analysis the angle (2 Theta) was adjusted between 10-70 and intensity was measured in counts. The reason for adjusting above range of angle is that, all major peaks of silicon were laying in this range confirmed from literature. Then XRD data was analyzed in Jade and respective graph was plot in origin. In Jade analysis, it is pretty clear that all major peaks of silicon were found as well. Silicon slurry was optimized by variation in concentration of different components like solvent, Dispersant and Dipping & Pulling speed of dip coater. The optimized result of silicon slurry was shown in following fig.





Figure 45: XRD analysis of Silicon deposition and silicon slurry optimization.

#### 5.6 XRD, SEM and EDS analysis of YSZ top coat:

After successful deposition of aluminum bond coat, we deposit YSZ top coat via thermal powder spray technique. After deposition of top coat, the sample was analyzed under various characterization tools like XRD, SEM and EDX in order to understand the surface morphology of coating and elaboration of impurities if involved. Firstly, we analyze our sample under XRD analysis and it reveals that only zirconium and its major compounds are identified. Our top coat material consists of yttria stabilized zirconia and XRD image shows that zirconium oxide and yttrium along with zirconium was found. In SEM testing of coating, a well adherent and smooth coating was analyzed. At higher magnification few agglomerations were seen, the reason behind this cluster formation is that YSZ having melting point of 2700 <sup>0</sup>C and partial melting of YSZ powder occurs along with other errors like spraying distance, powder feed rate and efficiency of equipment. EDX analysis reveals that, there is no impurities involved during deposition process and pure YSZ powder was deposited[5]. YSZ shows excellent thermo-mechanical properties that's why this material is used for thermal insulation purposes [6].



Figure 46: Top Coat (a) XRD analysis, (b) SEM image, (c) EDX analysis and (d) Coating thickness

#### 5.7 Wear rate via tribometer:

After successful deposition and surface characterization of both layers, final testing was done via tribometer. From tribometer, we can calculate the wear rate of both coated and non-coated sample. In tribometer, we can collect data regarding wear rate under different load implementation like 3 N (Newton) & 5 N. A ball and pin tribometer used for this experiment and both coated and non-coated surfaces were analyzed under same parameters. From this core characterization experiment we can collect excellent wear rate results. In first case, where coated and non-coated samples were analyzed under 5N load condition and calculate wear rate graph. The wear rate of coated sample was much less than non-coated sample. For how much percent of wear rate was reduce for coated sample, select three different points like 1m, 2m and 3m. On point 1m, the value of wear rate of non-coated and coated sample was  $7.6 \times 10^{-7}$  mm<sup>3</sup>/m and  $1.19 \times 10^{-8}$  mm<sup>3</sup>/m respectively. The difference between these two values was  $7.48 \times 10^{-7}$ . With reference to the coated sample, the percentage of wear rate was reduced by 1.5% [%age reduction= (C/NC) ×100]. Similar calculation was done on points 2m and 3m, the percentage reduction on these points was 1.6% and 1.1% respectively.



Figure 47: Wear rate of coated and non-coated sample at 5N load conditions.

Then we took average percentage on these points, we can say that wear rate of coated sample was reduced by 1.38% averagely from non-coated sample under same load condition of 5N.

Then we analyze the coated sample under little less load condition like 3N. We can see from the following graph that the wear rate was lies in the range of  $10^{-8}$  for whole experiment, which was significantly low from non-coated sample under 5N load condition [7, 8].



Figure 48: Wear rate of coated sample under 3N load condition

## **Summary**

In this chapter, firstly we discussed the results of Aluminum bond layer taken form characterization tools like XRD, SEM and EDX. From these tools it is clear that a well adherent and a uniform coating was formed. Then cross-sectional analysis was performed to measure the coating thickness. Then thermal annealing was done at 500 <sup>o</sup>C for 3 hours in order to make aluminide formation, which is confirmed by XRD analysis. The morphology of YSZ top coat was characterized with the help of XRD, SEM and EDX. The top coat was purely deposited, well adherent and uniform through the whole surface. At the end, the core result was taken from tribometer in which relevant graphs clearly shows that coated sample having much less wear rate than un-coated one.

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Chapter #05

# **Conclusion and Recommendations**

Energy is basic need for any human entity. Pakistan is 6<sup>th</sup> largest state in the world by population and it requires enormous amount of energy to fulfill their needs. Gas turbine plays a significant role for the generation of electricity in Pakistan, where 65% of total electricity is generated form thermal power sources. We face a lot of problems in gas turbine like wear and tear, corrosion and erosion due to severe environmental conditions and chemicals. In this research work a silicon bond coat was deposited via slurry deposition on C/SiC composites. Moreover, a bi-layer coating system (Nickel-Aluminide and YSZ) on Monel-400 superalloy in order to safeguard the turbine components and following salient results are drawn:

- For Monel-400 super alloy, after deposition of both layers, Tribometer analysis was performed at different load conditions like 5N and 3N for coated sample while non-coated sample examine only under 5N. Tribometer analysis shows that wear rate of coated sample was reduced by 1.38% averagely under 5N load condition.
- Cross sectional analysis was performed to confirm the coating thickness, which is 180µm for bond coat and 145µm for top coat.
- For C/SiC substrate, Silicon slurry was optimized by enhancing the quantity of solvent (Ethanol), dispersant (Polyethylene imine) and lowering the dipping & pulling speed of dip coater. Dip coater results depict that a well adhere and smooth layer of silicon was achieved.
## **Recommendations:**

Some of the salient recommendations are given below:

- Use of different bond coat instead of aluminum like titanium while top coat will remain same like YSZ on Monel-400 superalloy and then characterize the final product via tribometer.
- Try other processes to deposit top coat like slurry method and spin coater etc. instead of thermal spray techniques.
- Sample dimensions were more than 15mm for more efficient results of ball and pin tribometer.
- Try to prepare at least 3 coated coupons for tribometer test under different load conditions like 5N, 7N and 10N etc.
- ➤ Use of E-beam and PVD deposition for top coat, because in thermal spray methods the coating thickness was around 150-200µm, which may be considered as thick layer.

# Dedication

This work is dedicated to my teachers, whose perseverance with work will always be a source of inspiration.

Poster presented at International workshop on energy at COMSTECH Islamabad 2018.



# To investigate wear rate of ETBC for the protection of Monel-400 super alloy used for manufacturing of hot sections of gas turbine.

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Environmental Thermal barrier coating (ETBC) systems are generally composed of metallic bond coat and ceramic top coat. Metallic bond coat provides excellent resistance against oxidation and wear, while ceramic top coat provides thermal barrier. We want to develop a novel scheme of ETBCs which is used for the protection of hot sections of gas turbine used for power generation as well as vehicular propulsion. Deposition of such protective coating surely enhance the efficiency of turbine as well as secure our material from severe environment. For the protection of Monel-400 super alloy, bi-layer concept is used in which aluminum is act as a bond coat and 7YSZ used for top coat. Both layers are deposited via spraying technique. Aluminum is deposit by Wire Arc Spray System and 7YSZ top coat is deposited via powder spray technique. For analysis of each layer of coating XRD, SEM and EDX tests are performed to analyze the coating. After deposition of both layers, wear test is performed via tribometer and it will reveal that service life of coated coupon is far better than uncoated. Yttria Stabilized Zirconia reveals excellent thermomechanical properties and suitable candidate for ETBC against severe environmental conditions.

**Key words:** Environmental Thermal Barrier Coating, Gas Turbine Protection, Thermal Spray Coating.

## 1. INTRODUCTION:

Energy is one of basic need of any human entity. Pakistan is 5<sup>th</sup> largest state in the world on the basis of population. It requires huge amount of energy to fulfill their demands. Pakistan producing approximately 70% of the total electricity from thermal power plants. Gas turbines are the essential part of thermal power sources and the turbine corrosion is due to oxidation and heat is a very common phenomenon. Mostly turbine corrosion occurs due to usage of contaminated fuel, Chemicals used for NOx prevention like ammonia, dual fuel used and water used for cooling purposes. We want to made turbine components more durable and sustainable under severe environmental conditions. At elevated temperature several types of corrosion may occur at metallic and alloy surfaces, important two are: High temperature hot corrosion and low temperature hot corrosion. High temperature hot corrosion occurs in the range of 815-955°C and significantly appeared at metallic surfaces. On the other hand, low temperature hot corrosion occurs at a temperature range of 595-815 and significantly appeared at alloy surfaces. There are two ways to meet the required energy demand, either to improve the process efficiency or to develop renewable energy source. Gas turbines used at

thermal power plants are operated at temperature i-e normally 700-1000 °C in a CHP loop while gas turbines used in aerospace application operated at very high temperature i-e 1400-1500 °C. At this elevated temperature, the components of gas turbines are corroded and we must protect turbine components like Inlet section, Compressor, Blades and combustion chamber from heat and corrosion George Y Lai [1]. Monel-400 is a nickel-copper alloy having excellent strength over a wide temperature range. In its composition, the majority portion of alloy-400 consist of nickel and copper while other elements like carbon, silicon, manganese and sulfur may present in traces. Monel is used in a variety of application like Marine engineering, chemical processing equipment's, de-aerated still, Heat exchangers and crude oil distillation tower etc. Nickel based super alloys are commonly used for the manufacturing materials of those equipment's used for temperature at up to 1000 <sup>0</sup>C. Several coating systems and coating techniques were used to protect turbine components like  $Al_2O_3$ -TiO<sub>2</sub> and modified aluminum coating for temperature up to 800°C. Another system of coating consist of WC-17Co powder is used for the protection of turbine components from wear resistance, M. S. Sahith [2]. This system is thermally sprayed providing dense coating having thickness up to 462 µm and bond strength is 36MPa. This system provides satisfactorv results regarding wear resistance. Nanostructured wear resistance coating provide superior hardness and toughness, R. Rajendran [3]. Several combinations of materials were also used for protection purposes in order to minimize corrosion and erosion phenomenon gas turbine like super Co in 605(Cobalt based alloy), Ti-6Al-4V(Titanium based alloy) and MDN121(Iron based alloy) N. D. **Prasanna** [4]. Another super alloy protection system consists of oxidation resistant bond coat and ceramic top coat. The ceramic top coat is ZrO<sub>2</sub> stabilized with MgO instead of  $Y_2O_3$ . The ceramic top coat provides excellent heat resistance. The purpose of stabilizing ZrO2 with MgO is due to the reason that MgO is much cheaper than  $Y_2O_3$ . Pure zirconia is transform from tetragonal to monoclinic phase thus and volumetric changes associated with this transformation. This system is only stable up to temperature of 750 <sup>o</sup>C A. N. Khan [5]. In Monel-400 super alloy, we deposit an interlayer of pure aluminum from Wire arc spray mechanism. Then perform XRD, SEM and EDX analysis of the coated coupon and found that there is no oxide formation on the surface of

coupon and pure aluminum is deposited. Then we perform some heat treatment of the coated coupon to form aluminide formation at temperature of 500 °C for three hours in a box furnace. Aluminide formation prior to YSZ top coat is necessary due to the reason that it provides good oxidation resistance as well as excellent surface for ceramic top coat. At a temperature of 500°C NiAl compound is formed E. G. Colgan [6]. This treatment creates bonding between nickel and aluminum which is more stable against oxidation and also provide excellent surface for top coat that. deposition. After deposit ceramic top coat(YSZ), which protect our equipment from heat, corrosion and oxidation. There are several benefits for the selection of thermal spraying deposition: wide range of coating materials and substrates are handled: Fast deposition rate is achieved and enhance the service life of our equipment's. Tribometer is extensively used to characterize coating which is used for high temperature or severe environment G. Darut [7], J. W. Murray [8], A. Shriram [9].

2. METHODOLOGY:

Both bond and top coat were deposited via thermal spray techniques. Firstly, Aluminum is deposited by wire arc spray system. Sample is in cylindrical shape having dimension 10×12mm  $(L \times W)$ . Aluminum wire having diameter 1.2mm was spraved with wire feed voltage 5V, air pressure is 0.5MPa and deposition time is 7seconds. By applying these parameters, we achieve a smooth and uniform coating having thickness 180µm. The selection of this technique for bond coat deposition is due to several salient engenders: high deposition rate, low porosity level, high bonding strength and arc spraying requires no combustion deposition gases. After of Aluminum, coupon was thermally annealed at 500°C for three hours to made Nickel-Aluminum bonding, which act as a oxidation resistant. Then deposit top coat YSZ via powder spray technique. In this method Nano size YSZ powder was deposited where flame spraying speed was 370 m/s, particle velocity was 180-240 m/s and a measured quantity i-e 25gm was deposited. During powder spray deposition following parameters were adjusted to achieve a uniform and well adherent coating: flow rates of oxygen and acetylene were 0.2 and 0.4 m<sup>3</sup>/h respectively. Distance between gun and substrate is 9cm. While the pressure of oxygen, Acetylene and compressed air were 0.1MPa, 0.5MPa and 0.2MPa respectively. Compressed air is used for both transport media as well as cooling purposes of the spray gun. Powder spray coating gun having diameter 3.5mm. The selection of powder spray technique for top coat is due to the reasons that our top coat YSZ material is present in powder form and YSZ melting point is 2700°C which is much difficult to deposit via arc spray system. After deposition of required coating we used XRD, SEM, EDX, OM and Tribometer equipment's for characterization purposes.

#### 3. RESULTS AND DISCUSSION:

After deposition of aluminum via wire arc spray technique, Energy Dispersive X-ray spectroscopy is used to analyze the coating.



Fig 1: EDX image of Al deposition From Fig-1, it is pretty much clear that pure aluminum is deposited and no oxidation occur during deposition on our sample. If impurities are involved, then they must appear in our spectrum. Our requirement is also the same that pure aluminum should be deposited.



Fig 2: XRD image of Al deposition X-ray Diffraction also reveals that pure aluminum deposited as shown in Fig-2. Scanning Electron Microscopy image shows excellent adherent with surface as well as uniformity (Fig-3).



Fig 3: SEM image of Al deposition In SEM image few cracks were also found which are probably due to extensive temperature of manual operation of deposition. But overall, it's a well adherent and uniform coating having thickness up to 180µm confirmed via cross sectional analysis. Then this sample was thermally annealed up to 500°C for

three hours to made nickelaluminum bonding at interface between sample and bond coat. After formation of Ni-Al bonding, we deposit ceramic top coat (7YSZ) via a powder spray technique. This top coat shields our sample from corrosion and wear in a high environment. The temperature thickness of top coat is 147µm. SEM image of YSZ deposition shows that top layer was deposited uniformly and few grains were seen at high resolution.



Fig 4: SEM image of YSZ deposition

The presence of grains was due to following reasons; powder was not completely burnt and agglomeration occur at surface. XRD and SEM images of top coat were attached.



# Fig 5: XRD image of YSZ deposition.

Then perform tribometer analysis of coated and uncoated sample at different load i-e 3 & 5 Newtons. The obtained graphical results (Fig 6) clearly show that coated sample having much less wear rate than uncoated sample due to the reason that YSZ having excellent thermomechanical properties.



Fig: 6 Comparison of Wear rate

### 4. CONCLUSION:

Aluminum and YSZ (7wt%) are successfully deposited via a flame spraying method. After thermal annealing aluminum is bonded with nickel to form Ni-Al compound, which

act as a bond coat. This bond coat will protect our sample from oxidation and provides excellent surface for top coat adhesion. Then deposit Nano size YSZ (7wt%) by using powder spray technique for thermal insulation of Monel-400 coupon. These coatings were analyzed by using XRD, SEM and EDX tools. Then tribometer perform analysis and comparison of graphs clearly show that coated sample having much higher efficiency regarding wear rate than uncoated sample.

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