

# **Solid particle erosion testing of thermal spray coatings on carbon composites**

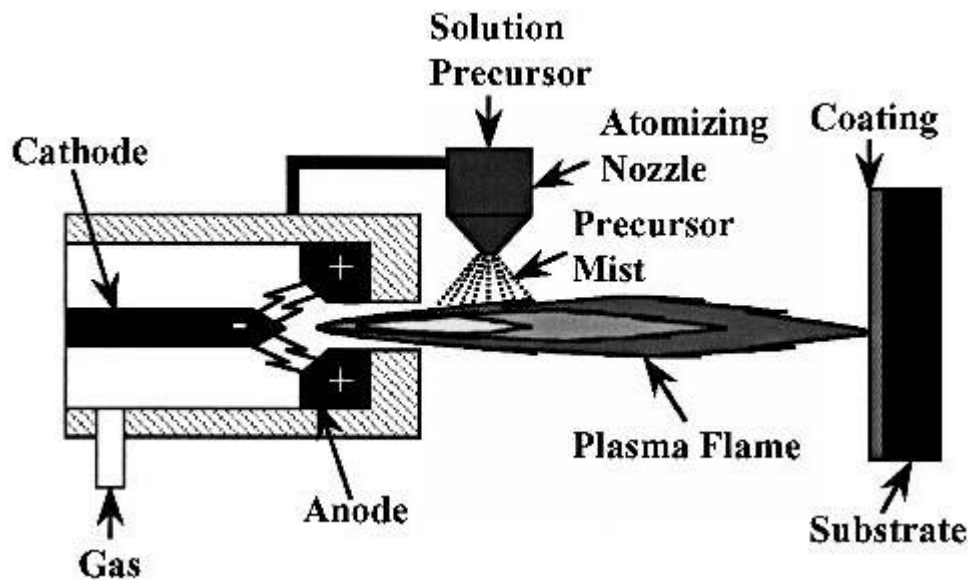
## **1.1 Introduction**

Protective ceramic coatings are used for wear and abrasion resistance and due to their superior thermal and corrosion properties, they are used in high temperature applications as well. However, the production methods for such materials are very unconventional and usually are not straightforward because of many difficulties encountered due to high melting points of the materials, poor thermal and electrical conductivity and high brittleness. Hence, ceramic coatings cannot be deposited as easily as metallic coatings. The techniques that are generally used for depositing metallic coatings are combustion spraying or electric arc process and vapor deposition techniques like chemical and physical vapor deposition. These cannot be successfully employed for ceramic coatings.

The arrival of plasma gun has overcome the difficulties that are encountered in conventional techniques. The plasma gun has much higher temperature capability and uses the raw material in powder form. Any material that does not decompose on heating can be sprayed by the plasma arc process. The advent of plasma gun has expanded the capability of coating wide range of materials and has contributed to a rapid advance in protective ceramic coatings technology. A schematic diagram of plasma spraying process is shown in figure 1 below.

Most of the coatings deposited does not exhibit properties that are similar at the time when they are in powder form. Plasma sprayed coatings behave in similar fashion. There are three types of thermally sprayed coatings:

- 1) Flame spraying: These are commonly metallic coatings deposited from the wire form using a combustion flame of oxyacetylene.
- 2) Thermo spraying: These fall into category of metallic or ceramic coatings that are generally sprayed from a powder form using a combustion flame.
- 3) Plasma spraying: Ceramics and metals sprayed in a plasma arc.



**Figure-1 Schematic diagram of Plasma Spraying process (1)**

Thermally sprayed coatings generally possess higher strength than that of bulk forms because they these coatings have interlocking microstructure. The extent of directional strength properties, anisotropic microstructure are some of its characteristics that distinguish them from bulk forms.

The adhesion of the coatings with the substrate has to be given prime importance because otherwise the strength of the coating would be compromised. This is of prime consideration in the application that demand large differences in temperature of substrate and coatings, i.e. the conditions which are prone to huge thermal gradient and thermal shock. However, there is very little information available on thermally sprayed coatings for comparison. Properties

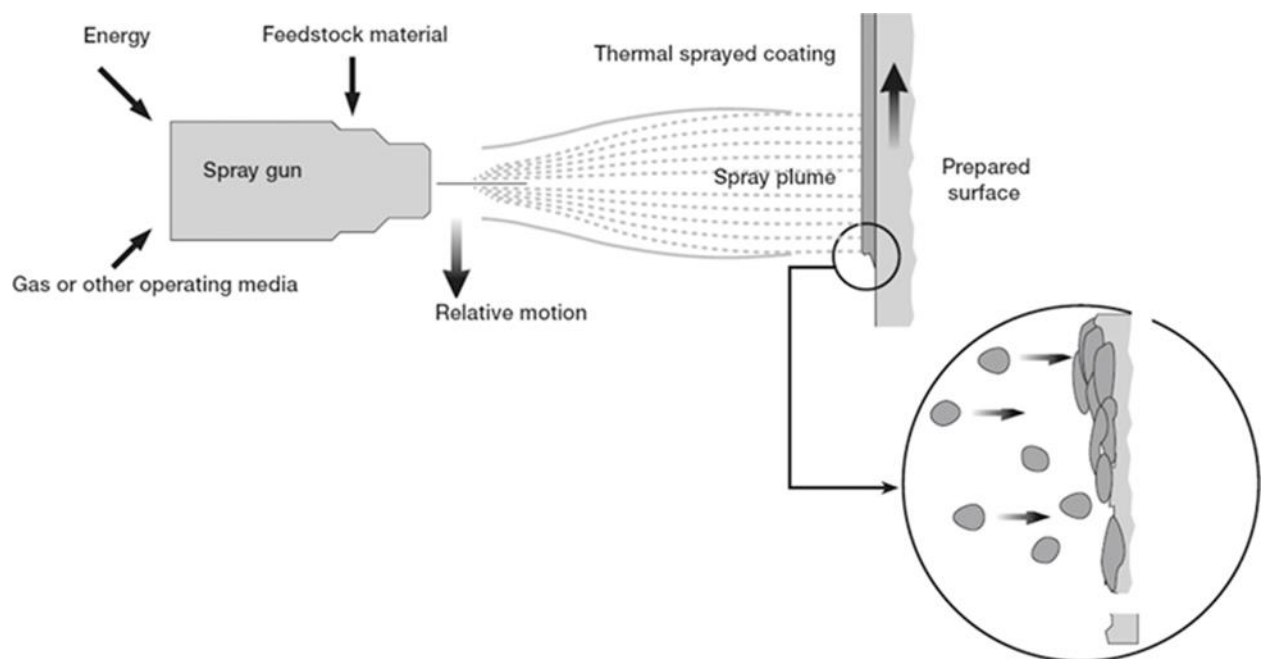
such as thermal expansion, thermal conductivity etc. related to high temperature ceramic coatings require special attention as these control up to a great extent the quality of coating. This is in turn controlled by the microstructure of the coating. The microstructure of the coating depends upon the type of spraying process used and the parameters employed to operate it. Thermal spraying of coatings introduces residual stresses within the coating especially at the coating substrate interface due to rapid cooling and differences in the expansivity of the phases. These give rise to micro cracks and sometimes peels off the coating pretty easily. Difference in thermal expansion of coating material with respect to the substrate creates lot of stresses and affects life of coatings considerably. This is motivation of many researchers to develop functionally graded materials (FGM) that compromise the large difference in thermal expansion coefficient of substrate and coating and also increase the compatibility of the coating with the substrate.

A comparison between the coatings and its counterparts in the bulk state would be an interesting feature as it would determine the cause for the difference in the coefficient of thermal expansion between the coating and the same material in the bulk state. This might be of great assistance in designing different coating materials for highly specific and customized applications.

## **1.2 Thermal Spraying process:**

This is the process where a material in powder, rod or wire form is continuously fed into a flame, or plasma depending upon the method used for spraying and the molten particles are propelled toward the substrate with the aid of high velocity gas. The different types of thermal spraying techniques are described elsewhere.(2)

The process that uses combustion of fuel gases to bring about the heating effect to melt the material produces temperature around  $3000^{\circ}\text{C}$ .<sup>(3)</sup> Hence, this method is not used for the materials that have high melting points. Almost all ceramics cannot be melted at this temperature, hence it is not used for ceramics but is used for coating low melting points materials. The electric arc process produces higher temperature than the combustion of fuel gases but is limited to conductive metals and alloy materials.<sup>(3)</sup> The schematic diagram of thermal spray process is shown below in figure 2.

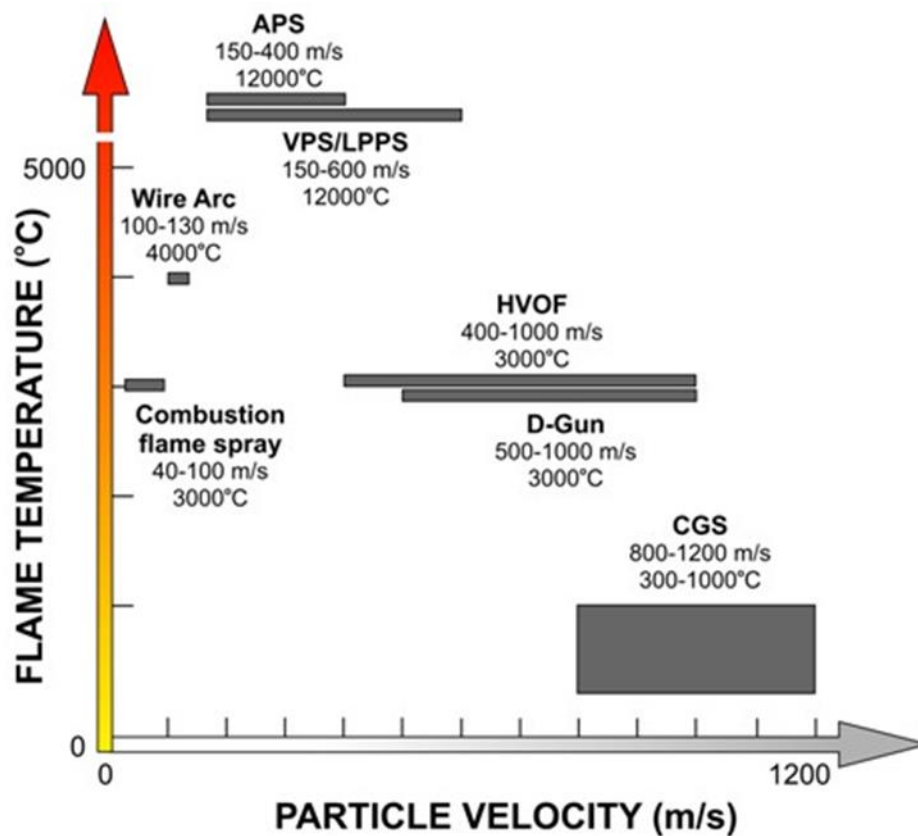


**Figure-2 Schematic of thermal spray process<sup>(4)</sup>**

Plasma arc spraying, produces the highest temperature of all the processes. This is done without damaging the specimen. Plasma is formed in the gas stabilized medium where plasma forms from a gas typically argon or helium or their mixtures. A non-transferred DC electric arc between a thoriated tungsten acting as cathode and an internally water cooled

copper nozzle which acts as anode.(5) The plasma produces temperatures in the range of 7000-10000°C. The coating material in powder form (generally) is fed into plasma with the help of an injector and plasma gas (argon, helium) carries the particles while plasma melts it. This is shown in figure 1. The molten particles as they are incident on the substrate start to solidify rapidly due to large temperature differences between the molten particles and the substrate. There is a considerable amount of mechanical interlocking between the substrate and the coating material.

As the temperatures produced by plasma are very high, there might be a problem of oxidation of the coating material (molten particles), hence the process is carried out in controlled atmosphere (low pressure or vacuum) which decrease the oxidation and also produce thick and denser coatings. Below shown is the graph of temperatures achieved by different thermal spray processes versus their particle velocities. The graph shows that plasma spraying process (APS) known to be Atmospheric Plasma Spraying achieves the highest temperature of all processes.



**Figure-3 Temperature versus particle velocity for different thermal spray processes(6)**

### 1.3 Plasma spraying parameters:

The parameters that one must maintain while thermal spraying are very critical as these control the nature of coating deposited on the substrate. The deposition efficiency that is determined by the fraction of powder injected into the plasma that is properly melted and deposited on the surface. Below is the list of thermal spray parameters:

- Spraying distance
- Powder (feed material) particle size
- Gas flow rate
- Powder injection rate

e) Entry position of powder in plasma

These are few critical factors that must be worked closely to improve the quality of coating.

For example: Spraying distance i.e. the distance between the spray gun and the specimen is of prime importance; if spraying distance is high then the molten particles would solidify before reaching the substrate specimen or if spraying distance is low then the particles would stay partially molten even though contacted by the substrate surface. These two phenomenon must be avoided and a balance must be struck to get the optimum distance.

Powder (feed material) morphology and particle size is also very important consideration in thermal spray parameters as it will determine the final coating's property. Ideally 5-50 microns particle size is used for oxides and 50-100 for metal powders. In externally fed plasma system, there is a great difficulty in feeding very fine particles because of pinch effect.

The gas flow rate controls the enthalpy of the flame which eventually increase the heat inside the chamber due to rise in the temperature.

#### **1.4 Plasma Sprayed coatings:**

When molten particles are propelled from the plasma gun toward the surface of specimen which is at relatively very low temperature compared to plasma, they under rapid solidification and get flattened. The velocity with which the molten particles impact the substrate is also very important. The particles which impact the substrate at high velocities tend to solidify with the surface geometry of the surface on which it is deposited. They have superior bonding strength with the substrate and provide excellent wear/abrasion resistance. The best example for such coatings is Tungsten Carbide with Cobalt binder (WC-Co). While other particles tend to solidify creating porosity at the coating-substrate interface.

More problems arise when arriving molten particles deposit on previously deposited coating which reduces the surface geometry effects on the solidifying layers of the coating. Oxide ceramic coatings are generally prone to this problem due to their insulating nature. This develops a thickness limitation and coatings deposited beyond a certain thickness are prone to develop surface delamination. This develops lamellar cracks in the coating and in the oxide coatings due to rapid solidification there is a possibility of shrinkage. Oxide coatings generally have higher specific heat and hence require longer dwell time in flame to get in molten state. This is sole reason behind adopting smaller particle size for oxide coatings and ceramic coatings as well. Hence, denser coatings are generally resulted with a smooth surface finish compared with metallic coatings.(7)

### **1.5 Problem of porosity:**

The first particles that impinge the substrate surface have cooling rates as high as  $10^4$ - $10^6$ °C/sec for oxides.(8) This creates thermal expansion mismatch between coating and substrate. This high solidification rates gives rise to spherical voids. Now when another layer of subsequent particles are being deposited, they do not undergo solidification rates as high as listed above as this batch of particles is deposited on the insulating surfaces which are at higher temperatures. As a result temperature gradient develops giving rise to stresses. These along with thermal expansion mismatch gives rise to interconnected pores. Also, when non uniform particles are being sprayed, it gives rise to interconnected pores. Finer powders as stated earlier produce much denser coatings and increased cohesive strength. Porosity changes with changing spray parameters and powder size distribution as well.

Pores and cracks due to shrinkage are more prevalent in oxides due to their brittle nature compared to metallic coatings. However, interconnected pores are less common in oxides because of small particle size, higher heat capacity and lower cooling rates during



solidification. Due to higher melting rates of ceramic oxides control of porosity by varying spray parameters is difficult. Compositional changes if done may improve the performance of the coating by decreasing the porosity.

### **1.6 Bonding mechanism:**

The bond strength is one of most important parameters which determines the coating's strength. Coating might be hard and wear resistant but if the bonding between the coating and substrate and between particles of the coating is not strong then the coating deposited will delaminate and will get ripped off or fall off as loose powder. Hence, bonding mechanism in the bulk of the coating and the interface between coating and substrate is an important aspect which has to be given prime importance. It is very difficult to explain and quantify the bonding mechanism in general because of the complexities of microstructure of the sprayed coatings and difficulty in producing precisely uniform conditions in the bulk of the coating. Also, the testing methods used to determine the bond strength and its mechanism are inadequate and less precise.

Hence, considering all these facts, one must adopt surface treatments of the substrate in order to increase the compatibility of the coating with the substrate. (To increase the bonding).

Surface treatments would minimize the differences between the properties of coatings and substrates over which they are coated. This would reduce the residual stresses as well.

Surface treatments such as grit blasting is done to increase the surface roughness. Surface roughness brings about mechanical interlocking with the deposited coating that increase the bond strength between the two. Grit blasting is generally adopted just before the application of coating. Application of bond coats also helps in increasing the compatibility of coating

with the substrate. Such techniques minimize the residual as well as externally applied stresses and are helpful especially when coating materials for high temperature applications.

The bond that is responsible for having such a high is mechanical. The adherence of a coating to the substrate might be due physical, chemical factors apart from mechanical. The physical bonding is due to secondary and van der Waal's forces. However, as this bonding is very weak, it is easily destroyed and its contribution toward the bond strength is negligible as preheating and pre-roughening of the surface is essential as stated in above paragraph to increase the compatibility through mechanical interlocking. If the surface of the substrate is very smooth and shiny, the coating will not form a strong mechanical bond with it as coating needs some kind of grooves or roughness to get interlocked with. Hence, surface preparation is an important process for strong adherence of the coating. While, many methods are undertaken for the surface preparation but, grit blasting with alumina, Silicon carbide is most economical and simplistic way to roughen the surface. The main aim of the surface preparation treatment is to expose the bare metal and remove some oxide layer from the surface. Through blasting, surface becomes rough and anchors the coating to the substrate which results in strong mechanical bond.

Vadivasov (9) links mechanical interlocking to the shape of the surface micro-irregularities generated by grit blasting which promotes the wedging of the particles being deposited. The effect of surface roughness on shear bond strength on alumina was evaluated by Grisaffe. (10) Bond strength increases with increase in surface roughness but up to a certain limit. It decreases again for highly rough surfaces.

Apart from physical, chemical and mechanical bonds, metallurgical bonds also contribute toward the bond strength of the coating. This had led to the development of "exothermic coatings" which by the heat of fusion of the constituents adhere to the substrate and form

strong metallurgical bond. Hence, it can be said that higher substrate temperatures usually result in improve bonding as diffusion and metallurgical reactions are favored at higher temperatures resulting in more cohesive bond. But of all the bonds stated, mechanical bonding contributes the most in the strong bonding of the coating with the substrate. The cohesive bonding of the particles must also be considered and depends upon the spray parameters. Variation in spray parameters control the properties of the molten particles before impinging on the substrate. Viscosity of the molten particle at the time of impact must also be considered. There is a critical velocity of the impact of the particles that must be maintained, if otherwise exceeded the result will be excessive fragmentation of the particles and will eventually reduce the mechanical strength of the coating.(11)

Some of the hard and wear resistant materials which when coated result in poor coating because the particle-particle bonding is very poor. Particle-particle bonding has been given very less importance compared with coating-substrate adhesion phenomenon. However most ceramic oxides are believed to have higher bond strength due to mechanical interlocking, but certain ceramic mixtures such as alumina with 5-25% of fine titania particles bonded to their surface are claimed to produce chemically reacted interfacial layers which when flame sprayed produce much denser coatings. (11)

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surface are claimed to produce chemically reacted interfacial layers which when flame sprayed produce much denser coatings. (11)

## **2.1 Introduction**

Polymer matrix composites (PMCs) are very competitive materials for aerospace applications, because of their high strength to weight ratio compared to metals. High reliability is a fundamental requirement for aerospace applications, and this makes it somewhat difficult for making more use of PMCs. Strength, stiffness, fatigue strength and corrosion resistance of PMCs can meet the requirement, but the key factor that restrict PMCs from being more widely used are their heat resistance and erosion resistance. The surface properties of the polymer matrix composites are very weak, i.e. they get easily abraded and wear down upon impact of solid particles (for example: sand) in air. Especially in the southeastern areas (hot and arid) where sand concentration in the air is very high and the flow with which sand flows in the air is high as well.

Most of the aerospace applications demand high strength and lower weight materials hence, carbon composites popularly known as carbon fiber reinforced plastics/composites are used extensively in the manufacture of aircraft, helicopters, etc.

Carbon Composites are the materials that are composed of carbon fibers as reinforcement (dispersed phase) and a polymer as a matrix phase. The reinforcing dispersed phase may be in form of either continuous or discontinuous carbon fibers of diameter about 0.0004” commonly woven into a cloth.

Polymer resin serving the purpose of the matrix phase can be of various types: Thermoset resin such as an epoxy or thermoplastic material such as nylon, polyester or vinyl ester etc.

Also, the carbon fibers can be of various types categorized according to their types, method of manufacturing and applications.

The properties of the final CFRP product can also be affected by the type of additives introduced to the binding matrix (the resin). The most frequent additive is silica, but other additives such as rubber and carbon nanotubes can be used. In CFRP the reinforcement is carbon fiber, which provides the strength. The matrix is usually a polymer resin, such as epoxy, to bind the reinforcements together. Because CFRP consists of two distinct elements, the material properties depend on these two elements.

Unlike metals, alloys and other conventional materials, CFRP has properties that are dependent on the direction/orientation of the reinforcement phase (carbon fiber in our case) i.e. it is an anisotropic material. Also the properties depend upon the proportion of the carbon fibers relative to the polymer matrix phase.

Generally the carbon fibers (reinforcing phase) provides strength and rigidity while the polymer matrix phase that binds the carbon fibers provides the toughness to absorb the energy/shock acted on the composite material. The strength and stiffness is usually measured by stress and modulus of elasticity

There are many applications of Carbon Fibers reinforced composite materials including Aerospace, automotive, medical, civil structures, etc. The thesis focuses on the testing of different thermal spray coatings on carbon composites for their use in helicopter blades. Hence, the advantages and disadvantages mentioned below are limited to aerospace applications.

#### **Advantages: [2]**

##### **1) Lighter:**

As measured by density, steel is 4.4 times heavier, aluminum is 1.6 times heavier and titanium is 2.5 times heavier than carbon fiber composites.

2) **Stiffer:**

Carbon fiber composites are 33% less resistant to deformation than steel but are 88% and 29% more resistant to deformation than aluminum and titanium respectively.

3) **Stronger:**

Carbon fiber composites are 3.8 times stronger than steel, 4.5 times stronger than aluminum alloys and 7.4 times stronger than titanium.

4) **Fatigue Resistance:**

Carbon fiber composites are inherently much more resistant to fatigue than traditional metals. Fatigue is caused by the propagation of micro cracks due to cyclic loading. In traditional metals those micro cracks tend to propagate at a much faster rate than in carbon fiber composites. The reason being is that carbon fiber composites are made of millions of individual strands of carbon fiber and micro cracks tend to stay isolated at the individual strands and not propagate like they do in metals.

5) **Corrosion Resistance:**

Moisture in the air will over time react with iron to create an iron oxide otherwise known as rust. Given sufficient time, oxygen, and water, any iron mass will eventually convert entirely to rust and disintegrate. Carbon fiber composites don't contain any iron and therefore do not rust.

**Disadvantages:**

1) **Low wear/abrasion resistance of surface:**

Though very strong and hard in itself, but the surface properties of carbon composite are very poor. It can be easily abraded when impacted by sand particles or other solid medium in air.

2) **Low working temperature:**

Carbon Composite cannot be used at very high temperatures. Here, the carbon is not the problem but the matrix which is made of some polymeric resin degrades at high temperature. Due to this, the bonding between the carbon fibers weakens and eventually the composite fails.

### **2.1.1 Surface protection of Carbon Composites:**

As a surface modification technology, thermal spray has gained great success in the protection of metal parts. Several attempts have been made to use these technologies to produce protective coatings for PMCs. The method by which the surface of carbon composites can be protected is through application of coatings.

Surfaces of the solid substances degrade with time as they are in constant contact with the environment. Environmental degradation of the surface phase over time can be caused by wear, corrosion, fatigue and creep. Surfaces can be engineered to enhance their performance either mechanically or physically through various processes. They are as follows:

- 1) Molten process: Thermal Spraying and welding
- 2) Gaseous process: Chemical Vapor Deposition and Physical Vapor Deposition
- 3) Solution process: Electro-less or electrolytic plating, resin bonding
- 4) Solid process: Friction stir welding, roll cladding etc.

Out of these methods thermal spraying technique is the best suited because of the following advantages:(12)

- Variety of the materials that can be applied is very large in case of thermal spray technique. Cermet, ceramic, metals, alloys, polymer etc. can be applied as a form of coating.
- Virtually any material that does not decompose upon heating can be applied as a coating.
- Thermal spraying has the capacity to form barrier and functional coatings on a wide range of substrates.
- Thick coatings can be applied at considerably high deposition rates compared to any other coating
- Coating does not need to fuse with the substrate and hence, the technique applies coatings on the substrate without much heat input. Hence, high melting point metals can also be applied pretty easily.

Hence, thermal spray coatings were applied on carbon composite substrates to know their validity in harsh sandy environment and sand particle erosion testing of coatings was carried out with sand blasting machine set-up.

American Society of Material's Handbook of thermal spray technology states that, "Thermal Spray coating is a generic term for a group of coating processes used to apply metallic or non-metallic coatings." Various heating sources are used to heat the coating material which is generally in the form of powder, wire or rod to a molten state and is accelerated toward the substrate with high velocity so that it creates a strong bond between the semi-molten or molten coating and the substrate surface. The quality of the bond depends on various process parameters and conditions.



### **2.1.2 Materials used for thermal spray process(13)**

#### **1) Single phase materials:**

- **Metals & Alloys**

Most pure metals have been used extensively for thermal spray processes. Metals such as Tungsten, Molybdenum, Nickel based super alloys, Cobalt based stellites, various trialloys, NiCrBSi based self-fluxing alloys, etc. have been utilized for various applications. These provide many advantages such as high strength, wear and corrosion resistance, and also their fatigue resistance. These have excellent bonding characteristics with the base substrate and form mechanically strong bond which have advantages of its own.

- **Intermetallics**

Almost all intermetallics are prone to oxidation at high temperatures and also reactive at that temperature; hence, inert atmosphere must be maintained in order to get the maximum resource efficiency out of the process. Inter-metallics such as TiAl, Ti<sub>3</sub>Al, NiAl, Ni<sub>3</sub>Al, etc. have been used for thermal spraying.

- **Polymers**

Thermoplastic and thermosetting polymers can be thermally sprayed provided they are available in particulate form. Polymers such as Polyurethanes, Polytetrafluoroethylene

(PTFE), Polymethylmethacrylate (PMMA), polycarbonate, etc. are thermal sprayed depending upon the applications.

- **Ceramics**

Wide range of ceramics can be thermally sprayed to get wear and abrasion resistance.

Ceramics are very hard in nature, provide thermal insulation/thermal barrier, conductivity, corrosion resistance etc. Ceramics include metal oxides such as Alumina ( $\text{Al}_2\text{O}_3$ ), Zirconia ( $\text{ZrO}_2$ ), Chromium Oxide ( $\text{Cr}_2\text{O}_3$ ), Magnesium Oxide ( $\text{MgO}$ ), etc. Various carbides include Tungsten Carbide ( $\text{WC}$ ), Silicon Carbide ( $\text{SiC}$ ), Titanium Carbide ( $\text{TiC}$ ), etc. These are generally sprayed by plasma spray that has high energy capable of melting such high melting point materials.

## **2) Composite materials:**

Composite materials are used in the applications where one requires the synergetic effect of ductile/tough metal matrix phase and hard/strong reinforcing phase. The ductile phase acts as a binder which holds the hard reinforcing particles. Wear resistant cermet coatings such as  $\text{WC/Co}$ ,  $\text{Cr}_3\text{C}_2/\text{NiCr}$ ,  $\text{TiC/NiCr}$ , etc. are been extensively used in wide areas of applications.

## **3) Layered or graded materials:**

These are generally known as functionally graded materials (FGM). These are useful in the production of thermal barrier coatings where one needs to minimize the intensity of thermal

stresses developed due to the large difference in coefficient of thermal expansion between the substrate and the coatings. It has following advantages:(14)

- FGM increases the bond strength between two incompatible materials
- FGM interface layer if applied, it reduces the residual stresses and thermal stresses as well
- Reduces the crack propagation through the interface

It is generally used for the production process of:

- Materials used in the application process where they are subjected to a large thermal gradient.
- Low cost clad materials for corrosion and wear resistance applications.
- Energy conversion applications

## **2.2 Literature review**

Limited work has been done till now in surface protection of Carbon composites using thermal spray technology. These are very attractive option for their use in aerospace applications because of their high strength to weight ratio.

Miyoshi et al conducted an experiment to investigate the erosion behavior of uncoated and coated Polymer Matrix Composite (PMC) specimens subjected to solid particle impingement. Bond coats were applied prior to applying the main coating of WC-Co to improve the adhesion of the coating and the substrate. Erosion were conducted using Arizona road dust equipment at impingement angles of 20°-90°

on both uncoated and two layer coated PMC at an air flow velocity of 220 m/s and at a temperature of 366K. (15)

The results obtained in the investigation carried out by Miyoshi et al. indicated that two layer (WC/Co top coat and metal bond coat) on PMC's remarkably reduced the erosion volume loss by a factor of approximately 10.

Vasquez et al studied the effect of surface modification on adhesion of metal bond coat polyimide composites. This study examined the effect Silica layer has on the adhesion of the proprietary metal bond coat to PMR-15 and PMR-II-50 composite surfaces. The surfaces of PMR-15 and PMR-II-50 composite were modified by following methods: Grit blasting, RF oxygen plasma etch, PECVD of silica, ultra violet ozone etch, UVCVD of Silica. Pull off adhesion test were carried out and fracture surfaces were examined using optical microscope and electron microscopy. (16)

PMR-15 samples with PECVD silica showed a significant increase in adhesion when compared with both UVCVD silica and grit blasting only. PMR-II-50 showed no significant difference in the adhesion strength for any surface modification method. The poor surface quality of PMR-II-50 composites is believed to have a major effect on bond coat adhesion strength.

Rezzoug et al investigated the effect of metallic coating for carbon fiber reinforced polymer matrix composite substrate. In this experiment they used copper and steel powder known as filler powders that will reduce the impact of thermal spray particles which eventually protect the carbon fibers from getting ruptured by thermal spray particles. Further, they showed through microstructure analysis that zinc coating will improve the erosion resistance of the PMC's and also has a good compatibility with the substrate. (17)

Aiguo Liu et al conducted an experiment where they used arc spray to form an erosion-resistant coating for PMC. A cored wire composed of steel skin and Ni–Cr–B–Si as filler material was used as the coating material. Influence of the pretreatment on the shear bond strength, thermal fatigue resistance, and erosion resistance of the coatings were also investigated. They showed that thin Zinc layer as a bond coat could improve the thermal fatigue resistance of the coating.

They proved that the mass loss of the coated PMC in erosion testing was half of that of the uncoated one, and the arc-sprayed coatings could provide good protection of the PMC from erosion.(18)

Drexel University, supported by NASA Glenn Research Center, investigated high velocity oxy-fuel (HVOF) sprayed functionally graded coatings for polymeric substrates (19), in order to solve the problem of thermal expansion coefficient mismatch between the substrate and the protection coating.

The functionally graded coating structures were based on a polyimide matrix filled with varying volume fractions of WC–Co. They found that the pre-heat temperature of the substrate had a great influence on the bond strength of the coating. Anyway, the bond strength was not very high, 5–6 MPa only. The commonly used pre-treatment, grit blasting, would cause substrate damage, and resulted in low bond strength.

### **2.3 Motivation of work: (Part-I)**

The main focus was on to improve the low erosion resistance of the surface of carbon composites as that is the primary reason which limits its use in the applications of the aerospace where the component is subjected to hot arid sandy conditions. Sand easily abrades the surface of carbon composites.

This was the motivation of the thesis where we could protect the surface of the carbon composite components through various hard carbide coatings. This would enhance its use in aerospace manufacturing especially in the manufacture of helicopter blades that is operated in hot and arid

environment where the sand concentration in air is very high. Finally, sand particle erosion testing of specimen was carried out to check the validity of the coating.

## **2.4 Experimental**

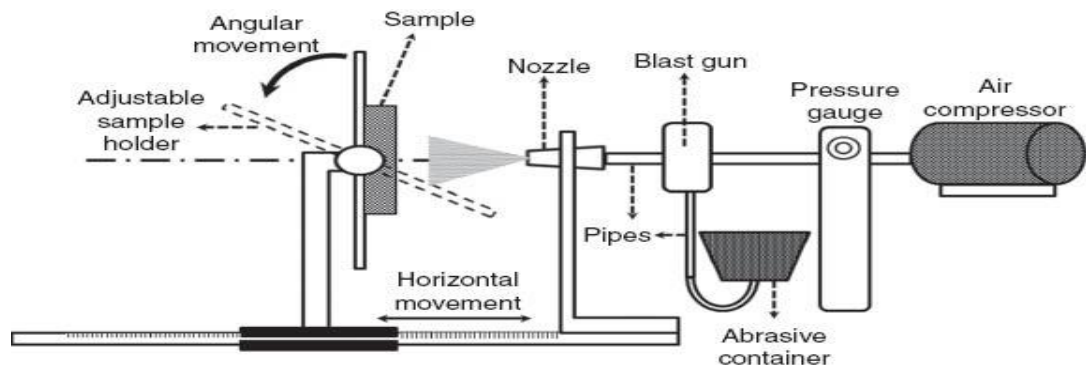
### **2.4.1 Set-up:**

The purpose of this test was to measure the sand erosion resistance of thermal spray coatings on carbon composite as substrate used in the manufacture of the helicopter rotor blades.

This test can quantify the amount of coating eroded from the substrate through mass loss. Although this test can quote the sand erosion resistance of coatings, but it alone cannot qualify the material for the rotor blade application.

Hence, other characterizations viz. adhesion test, porosity, thermal shock, corrosion test etc. are needed in order to make a decision as to which coating would be best that could serve though not all but many properties during the application. As it would take lot of details for the coating to qualify for the application, mentioning all the processes and parameters is out of scope for the thesis.

The schematic diagram of the set-up is as shown in the figure.



**Figure-4 Schematic diagram of the sand blasting machine(20)**

#### **2.4.2 Testing apparatus:**

The test apparatus is a sand blasting machine that could simulate the in-service flight conditions. Two motors were used. First one was used for the linear motion of the sand blaster and the second one was employed for the rotation of the sample.

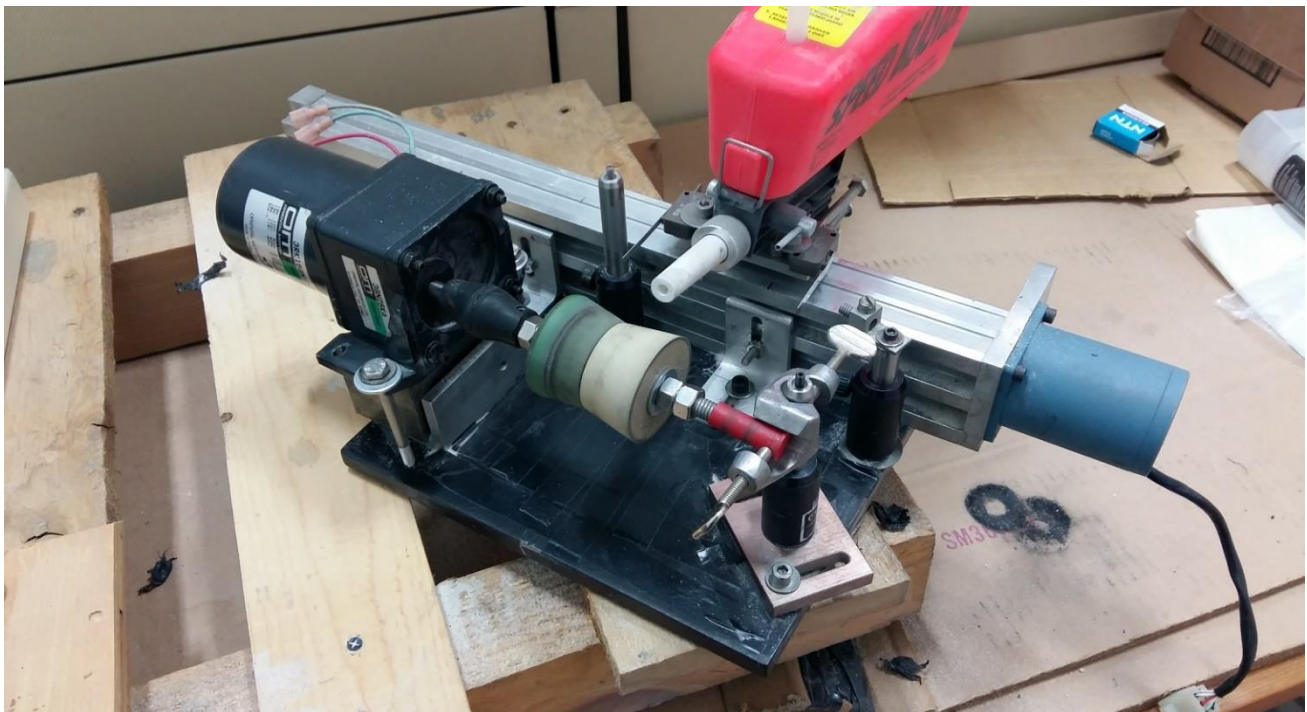
For the sand blaster, linear stage was used in order to convert the rotational motion of the shaft of the motor to linear motion. Sand blaster was placed above the linear stage as shown in the figure. The linear motion of the sand blaster was restricted beyond a certain point on both sides by limit switches as shown in the figure.

Sand blaster was loaded by the silica sand from the top through small diameter hose pipe and high pressure air jet was introduced in the blaster from the rear side through a hose connected to a pressure gauge that could regulate the pressure as needed. Pressure gauge was connected directly to the compressor hose pipe.

The shaft of the motor which run the sample was equipped with coupler to transfer the rotational motion of the shaft to the sample. The free end of the shaft of the sample was being rested on the stand in order

to prevent its wobble during the operation. In order to prevent the side motion of the sample and to fix it properly on the rotating shaft it was backed up by the rubber stoppers as shown in the figure.

Sand particles are accelerated through a small diameter nozzle and directed towards the test specimen as shown in the figure. Since, the diameter of the nozzle is smaller than the test specimen area, the test specimen is rotated continuously past the jet while the jet (sand blaster) is oscillated over the linear stage. This mechanism facilitates uniform deposition of the sand particles throughout the surface area of the test specimen.



(a)





(b)

**Figure-5 (a) and (b) Actual Set-up of the sand blasting machine**

### **2.4.3 Standard Test Conditions:**

#### **1) Erodent:**

The erodent/abrasive used was Pro-Angle White Bunker Hydrosand popularly known as golf sand. The source for this sand was R.W. Sidley Inc. located in Painesville, Ohio.

The sand here used had its grains angular in shape. The main reason behind using angular grain shape sand was that the angular shaped grains has more pronounced effect on the abrasion that the rounded shaped grains, i.e. angular shaped grains brings about more severe abrasion than the round shaped grains.

The particle size used was in the range of 240-550 microns. Sand particle size below 200 microns was unacceptable according to DOD test method standard as that was the duplication of the real flight conditions where the sand particle size is never less than 200 microns.

Also the reuse of the sand must be avoided as it would further decrease the abrading effect of the sand and we would get improper results.

## **2) Velocity of the erodent:**

The velocity is representative of the helicopter rotor tip speeds during landing and take-off conditions.

The velocity of the erodent as measured at a distance of 20 millimeters from the nozzle tip was 730 ft.

/sec

## **3) Sand particle mass load and impingement angle:**

The sand particle mass load was between 30-50 grams/cm<sup>2</sup> and the angle of the impingement was in the range of 20-90 degrees.

## **4) Nozzle diameter:**

The diameter of the nozzle used was around 4.9 millimeters. The nozzle was made of ceramic material in order to resist the abrading action of the high speed and high pressure silica sand passing through it.

## **5) Pressure & Temperature:**

The pressure at which the high speed air jet of the sand was passed through was kept around 90 PSI.

Such high pressure was kept in order to maintain the flow rate of 251.5 liters/minute. Also, here the sand has no momentum of its own unlike the real condition where in the hot arid regions the sand comes with lot of momentum and hits the rotor blades. Hence, to compromise the effect of self-momentum of the sand, the pressure of the air kept here was pretty high. The temperature at which this test carried out was normal room temperature (75°F)

#### **6) Rotational speed of the sample:**

The rotational speed of the sample was 70 RPM. It is not necessary to rotate the sample if the sample shape is other than round or cylindrical. In this test, we used a hollow tube whose dimensions are given in later sections. The goal was to test for 15 minutes, i.e. deposition time was to be 15 minutes if the sample could last under such conditions for such long. Hence, number of turns per deposition would be equal to 1050.

#### **7) Linear speed of the blaster:**

The linear speed of the blaster was kept around 7.9 cm/min. Hence, number of passes per deposition would be around 37.

### **2.4.4 Test specimen specifications**

#### **1) Type of the specimen:**

The specimen used was a hollow tube of carbon composite, over which different coatings were done.

Diameter of the hollow tube was 50 millimeters

#### **2) Size of the specimen:**

Diameter of the specimen used was 50 millimeters. The scanning length (length along which the deposition will take place) was equal to 32 millimeters. The scanning length represents the length covered by the blaster during its oscillation for one pass. Thickness of the substrate without the coating was equal to 2 millimeters.

### **2.5 Testing:**

The coefficient of thermal expansion of carbon fiber is in the range of  $-0.3 \times 10^{-6}/^{\circ}\text{C}$ – $1 \times 10^{-6}/^{\circ}\text{C}$ , and the coefficient of thermal expansion of polyimide is approximately  $50 \times 10^{-6}/^{\circ}\text{C}$ . They are very different from that of coating material, which is in the range of  $10 \times 10^{-6}/^{\circ}\text{C}$ – $12 \times 10^{-6}/^{\circ}\text{C}$ . [11], [12]

Thermal stress would be produced if the coated sample was subjected to thermal cycling, due to the great difference of coefficient of thermal expansion. So a soft bond layer was proposed between the erosion-resistant coating and the substrate to minimize the thermal stress.

### **1) Uncoated Carbon Composite:**

First test was carried out on uncoated carbon composite substrate to check the working of the test system and to know if there is considerable amount of mass loss that could be noted in terms of a proper result.

Firstly, the weight and thickness of the sample was noted as the weight and thickness of the sample before the test.

This was followed by loading the sample between the two rubber stoppers and the motor to which it was connected was switched on to make sure the sample is not wobbling due to its own weight and rotational motion. The linear stage motor was switched on to check the motion of sand blaster over the stage is proper or not.

Sand was introduced from the top of the blaster through a small funnel and a hose. A balance was struck between the sand mass deposition and its flow rate.

Lastly, the compressor valve was opened up and through the pressure regulator the pressure was adjusted to 85-90 PSI.

The test was carried out for 15 minutes. After completion of the test, sample was unloaded and properly cleaned to remove all the sand from its surface. This was followed by weighing the sample and

measuring the thickness of the specimen. This was to quantify the wear of the material after test. Results were noted in terms of mass loss and are listed in later chapters.

## **2) Zinc coating (bond coat) on carbon composite:**

The testing procedure was the same as used for the uncoated carbon composite. Results were noted in terms of mass loss and are listed in later chapters.

The test for this specimen was carried out for no more 2 minutes. The main reason behind this was that coating got ripped off from the substrate within 2 minutes of the test. Not more than 130 grams sand was used for the test.

## **3) Tungsten carbide with cobalt matrix:**

The same testing method was employed for this coating as used above. Same kind of results were obtained in case of WC-Co coating as observed for Zn bond coat, i.e. the coating got ripped off globally from the substrate within 2 minutes of the test and no more than 130 grams of sand was used to bring about the ripping effect.

## **2.6 Results**

### **1. Uncoated Carbon Composite:**

Results are as listed in the Table 1. It could be clearly inferred from the data that there is considerable mass loss after the sand particle erosion test. Although, the carbon composites are very stiff and strong but their surface is easily abradable which is an obvious decision one could make from the results.

Sample Number	Before Test						After Test		Total mass loss in %
	Diameter (mm)	Length (mm)	Thickness (mm)	Weight (g)	Sand mass (Kg)	Air pressure (PSI)	Weight (g)	Thickness (mm)	
1	50	22	2	9.333	1.46	90	3.92	1	58.0
2	50	22	1.86	9.8	1.46	90	4.86	1.36	50.4

**Table-1: Mass loss results for uncoated carbon composite sample**

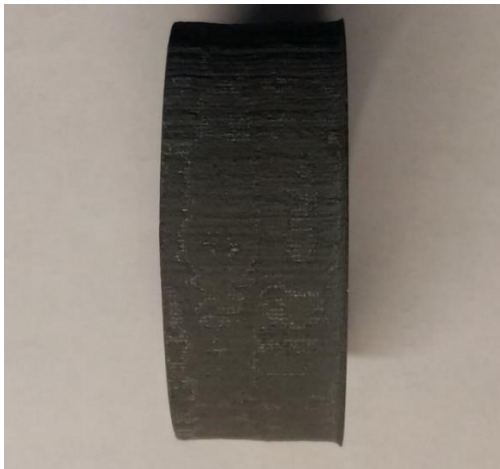
Below shown are the images of the specimen before and after the test and one can clearly see the difference in the surface of the specimen

#### **Before the test**



**Figure-6 Uncoated carbon composite specimen before the sand particle erosion test**

## After the test



**(a) Sample 1**



**(b) Sample 2**

**Figure-7 Uncoated CC after sand particle erosion test**

**1) Zinc coated carbon composite:**

Results are as listed in the Table 2. Here the test was carried out for 2 minutes as the coating started to peel off from the substrate within that time. This shows that the adhesion between the substrate and the coating is very poor and hence it could not bond properly with the substrate and got ripped off the surface pretty easily with just 130 grams of sand mass.

## Table 2

Sample Number	Before Test						After Test		Total mass loss in %
	Diameter (mm)	Length (mm)	Thickness (mm)	Weight (g)	Sand mass (Kg)	Air pressure (PSI)	Weight (g)	Thickness (mm)	

1	50	22	2.12	11.846	0.13	90	10.799	1.9	8.8
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**Table-2 Mass loss results for Zinc coated carbon composite sample**

Below shown are the images of the Zinc coated carbon composite before and after the test. One can clearly see the condition of the zinc coating on the carbon composite substrate within 2 minutes of the test and using 130 grams of the sand.



**Figure-8 Zinc coated sample before the test**



**Figure-9 Zinc coated sample after the**

**test**

**2) Tungsten Carbide with Cobalt matrix:**



Results are as shown in the Table 3. Here also the same kind of results were observed as in zinc coated carbon composite. Within 2 minutes of test the coating was seen to rip off globally from the substrate so the test had to be terminated. 130 grams of sand was used for the test.

**Table 3**

Sample Number	Before Test						After Test		Total mass loss in %
	Diameter (mm)	Length (mm)	Thickness (mm)	Weight (g)	Sand mass (Kg)	Air pressure (PSI)	Weight (g)	Thickness (mm)	
1	50	22	2.15	12.797	0.13	90	10.10	1.96	21.1

**Table-3 Mass loss results for Tungsten Carbide-Cobalt (WC-Co) coated carbon composite sample**

Below shown are the images of the WC-Co coated carbon composite before and after the test. One can clearly see the condition of the WC-Co coating on the carbon composite substrate within 2 minutes of the test and using 130 grams of the sand.



**Fig-10 WC-Co coated sample before the test    Fig-11 WC-Co sample after the test**

## **2.7 Discussion**

According to the data obtained from the experiments and testing of coatings, it can be said that either the testing equipment used was too intense for such coating material or the coating itself was not bonded properly with the substrate. The reason most likely seems to be with the coating.

Results obtained through the sand particle erosion testing clearly indicated that the coating did not bond properly with the substrate. Coating failed globally instead of failing particle-by-particle. This clearly indicates that coating and the substrate had a thermal mismatch, i.e. there was a large difference in the

coefficient of thermal expansion (CTE) between the coating and the substrate. This led to poor bonding between coating and substrate and build-up of thermal stresses. Carbon composites being very stiff and strong, has very low CTE compared to other materials which expand upon heating. This was the sole reason behind the fact that the WC-Co and Zinc coating obtained was locally deformed and cracked long before its effectiveness as a protective erosion barrier could be assessed.

This type of problem must be addressed by employing functionally graded material (FGM) coatings especially where there is large difference in CTE between coating and substrate. In contrast to traditional composites, with a uniform distribution of reinforcing phases, FGM coatings are characterized by a volumetric gradient evolving from one component to another and properties of each component can be utilized effectively without the need to compromise.(21)

According to the work published earlier on thermal spray coatings, the steel substrate best suits as a base for the coatings. Here, we can see the optimum performance of the coatings as the bond between the substrate and the coating is very strong. Hence, the coating does not peel-off from the substrate as in our case where the coating got ripped off globally from the substrate.

## **2.8 Summary & Conclusion**

- The goal of this test was to measure the sand particle erosion resistance of thermal spray coatings on carbon composites relative to uncoated carbon composite specimen for the application of manufacture of helicopter rotor blades.

- The test set-up employed for testing was a machine similar to a sand blasting machine and was an effort to simulate the in-flight or in-service test conditions where the helicopter is subjected to hot-arid environments where the sand concentration in the air is very high.
- Due to large difference in the CTE between carbon composite and coating there was a thermal mismatch that resulted in poor bonding between the two. Hence, the coated specimen obtained was deformed and cracked before it could be assessed for erosion resistance.
- Hence, the results for the erosion test obtained were pretty obvious meaning; the coating (Zn & WC-Co) could not resist the erosion test conditions and got ripped off from the substrate within 2 minutes with 130 grams of sand deposition with considerable mass loss and so the coating did not qualify for the application.