Thermal Spray Coatings for mechanical seals

3.1 Introduction

Properties of hard materials and methods of their production has been of considerable importance since many years due to their excellent hardness, mechanical properties, wear and corrosion resistance. The best example of such class of hard materials is tungsten carbide-cobalt composite (WC-Co). This comes in the class of Cermet materials. These have excellent hardness and provide superior wear and abrasion resistance. However, there are many other examples of such materials and are listed in later sections. Many different methods are used to produce such class of materials, thermal spray being one of them. Sintered carbides are one of the widely used hard materials for wear and abrasion resistance. In fact, cemented carbides were one of the first examples of successful composite materials in which the beneficial properties of two component materials are retained in the final composite. Hardness and wear resistance is provided by the carbide phase while metallic binder phase contributes strongly to ductility and toughness of the composite.

In the energy generating, aerospace, pulp and paper, automotive and many other industries, thermal spray metallic, ceramic and cermet coatings have been identified as a means to improve the operating performance of engineered surfaces. Many industrial applications such as mechanical seals, bearings, shafts, turbine blades, wear rings, thermal spray coatings are widely used to decrease the surface degradation. Also, in the aircraft and energy generating gas turbine engines, boilers, power plant machinery, thermal spray coatings are being used to mitigate the solid particle erosion and hot corrosion in high temperature components.

This part of the thesis has its objective focused on the assessment of different thermal spray coating materials for wear protection of mechanical seals used in the pump industry.

Mechanical seals are an integral part of pumps that are used for sealing or preventing leakage. It is a device used to seal the interface between a rotating shaft and a stationary housing. These are commonly employed in centrifugal pumps although, quite often used in agitators, turbines, compressors, blowers, etc. The ability of a mechanical seal to meet the requirements depend upon many factors that involves design, operating conditions and support systems and materials of seals as well.(22)

There are various types of mechanical seals depending upon the application. Also, seals can be divided into three categories of components: the seal faces, secondary sealing elements and major metal components.(22)

One of the most important part of a seal is its face as it undergoes wear, corrosion and cavitation damage in service, which reduces pump efficiency and component life and significantly increases the life cycle cost of the pump. All these degradation mechanisms are active on the surfaces of the pump components, and therefore surface coatings could be an effective way of addressing this problem. Wear of materials should be taken into prime consideration as it is one of most important parameters that controls the pump efficiency and durability.

There are types of wear that materials undergo:

a) Adhesive Wear- material pull-off between sliding surfaces
b) Abrasive Wear- loss of clearances

Mechanical seals are subject to abrasion, erosion, cavitation and sliding wear in service. Abrasion and erosion results from abrasive particles in the medium being pumped, but even in the absence of any abrasive particles sliding (or galling) wear can occur due to unintentional contact between rotating and stationery components. Moreover these wear rates in pumps are often unexpectedly high because of the synergistic relationship between wear and corrosion; even a mildly corrosive process media can significantly increase wear rates. This is often increased even more by the high flow velocities that can be found in some high-energy pumps. As the pump components in the flow path wear, efficiency critical sealing clearances increase and vane angles change, resulting in a decrease in pump efficiency over time.

There are numerous strategies to reduce the impact of wear on pump components such as specialized pump design and the use of sophisticated materials (e.g. high-alloy steels, ceramics, rubbers etc.), but surface coatings are some of the most successful and cost-effective tools for managing wear.

3.1.1 A few rules of thumb for mechanical seals [20]

Below listed are the things one must keep in mind before designing a mechanical seal:

- The materials for mechanical seals must be compatible chemically to all the fluids that are being pumped, i.e. all solvents, cleaners or steam that is generally introduced into the system to clean the lines.
- The seal faces must stay together without opening otherwise the seal will leak and penetrate all the solids and eventually damage the faces and lapped surfaces. This would deteriorate a pump's life drastically.
- Any other failure other than the wearing away of the carbon from the face of mechanical seal is considered to be seal failure and is always repairable through different techniques like application of coatings.

3.1.2 Materials used for manufacture of mechanical seals

The best seal face materials should have low friction, high hardness, good corrosion resistance (chemical resistance) and high heat conductivity, adequate wear resistance, etc. Conventionally pump manufacturers used hard martensitic stainless steels maintaining the hardness difference of 50-100 Brinell between stationary and rotary parts of the pump as experience has shown this difference to be adequate enough to prevent galling (23)

Below listed are some conventional coating materials used by the pump manufacturers for making mechanical seals.(22)

1) Carbon:

Being one of the most abundant materials on the earth, it is quite extensively used in the manufacture of faces of mechanical seals. Various forms of carbon are available for use. It takes up amorphous form to graphite and from diamond to fullerene. All forms have their own advantages and applications.

Advantages:

- It is abundant in nature
- Stable, inert, chemical resistant
- Self-lubricating
- Anti-friction properties

Carbon is not used in its original form but it’s generally impregnated with graphite and/or other resin and pitch. Metallic binders may also be added. Manufacturers use their own grades of carbon suitable for the specific application that depends upon factors such as graphite content, binder content etc.

Resin impregnated carbon has its own advantages in a way it provides good frictional resistance and chemical resistance as well. However, these lack wear resistance and hence if one operates such resin impregnated carbon faced mechanical seals in high wear and abrasive conditions it will create lot of material loss. These conditions demand metal impregnated carbon which has superior wear resistance, strength and stiffness and can be employed successfully in high pressure applications for considerable period of time. Although, these lack chemical resistance and also have low friction resistance which brings about lot of seizure or galling on the surface.

One may have to look up for the conditions in which the mechanical seal is being put and then choose the type of carbon for that application. Although there are certain advantages of carbon as a material for mechanical seal face, there is lot of wear that it has to undergo as the surface of carbon faced components is very weak and is prone to abrasion.

2) **Aluminum Oxide (Alumina):**

Alumina is a ceramic material available in different purity levels and its cost increases as the purity level increases. The most pure alumina offers best corrosion resistance but on the contrary its cost increases. Hence, a balance must be struck between the two.

Advantages:

- High stiffness
- Good chemical resistance
- Partially good wear resistance

Other than some strong acids, alumina has excellent resistance against most fluids but due to low heat conductivity it fails/fractures easily by thermal shock. Rapid heating during dry running operations is prevalent which can fracture alumina easily. Hence, alumina as a seal face material for mechanical seal is not recommended in the conditions where temperature changes are prevalent.

3) **Chrome Oxide:**

Chrome oxide is extensively been used for corrosion resistance and against other chemical attack. However, it is eaten by alkaline medium. Also, due to low heat conductivity it fails under thermal shock and its use is limited in the applications where there is a lot of heat gradient within the component. Also, it lacks hardness so cannot be employed under highly abrasive conditions. It has very low elastic modulus hence can fracture easily under high loads.

4) Silicon Carbide:

It is one of the best suited material for the faces of mechanical seals that pump manufacturers prefer to use because of following properties:

Advantages:

- Excellent hardness
- Excellent wear resistance
- High thermal conductivity
- Low coefficient of thermal expansion
- Excellent thermal shock resistance
- High endurance limit

There are two forms of silicon carbide that are used for manufacturing of mechanical seals.

Reaction bonded silicon carbide

It has high content of free silicon. Silicon carbide particles and carbon are mixed together with binding agent and pressed into desired shape. Free silicon gets affected by the chemical attack that can weaken the material and result in failure. Strong acids and high pH chemicals easily attack the free silicon hence, these limit its applications. Although, prone to caustics and chemicals, it has higher wear resistance that self-sintered silicon carbide.

Self-Sintered Silicon Carbide

Due to its high hardness, it has low mechanical strength. Unlike reaction bonded silicon carbide, self-sintered silicon carbide does not have free silicon hence, it is chemically more resistant. However, it is more brittle and prone to chipping that reaction bonded silicon carbide.

5) Tungsten Carbide:

Cobalt matrix:

It is one of the most successful seal face material used by the manufacturers and has given promising performance compared to other materials even in aggressive conditions. This material consists of hard tungsten carbide phase in the form of small particles with cobalt used as a metallic binder that acts as matrix phase. In this one can get the synergetic effect of hardness provided by hard carbide phase particles and toughness provided by the softer cobalt matrix phase.

Advantages:

- Superior hardness
- Excellent wear resistance
- Superior Strength
- High thermal conductivity
- Low coefficient of thermal expansion

However, cobalt matrix is prone to chemical attack and is eaten up by strong acids and it fails in chlorine atmosphere as well. It has low friction resistance when it is running under hot water and may cause galling.

For the applications where one needs superior corrosion and chemical resistance, nickel as a binder phase instead of cobalt has proved to be far more superior.

Nickel matrix:

Nickel as a binder phase in tungsten carbide is pretty impressive in terms of chemical resistance. Nickel has higher chemical resistance than cobalt in aggressive atmospheres.

Advantages:

- Superior chemical resistance
- High strength and toughness
- Excellent wear resistance

6) Boron Carbide:

Boron carbide is known to be the third hardest material after diamond and cubic boron nitride. It is an unconventional material that is used for coating purpose. The superior mechanical properties of Boron carbide make it very suitable as a wear resistant coating for industrial machinery and components.

Advantages:

- Excellent hardness and strength
- Superior wear resistance
- High resistance to fracture
- High resistance against chemicals (corrosion resistant)
- Anti-galling properties
- Low density
- Excellent resistance to thermal shock

However, there is one major problem associated with this material. During plasma spraying, decomposition of boron carbide powder takes place and results in the formation of BXC phase and an increase of the carbon phase.(24)

3.1.3 Coating Characteristics

In practice coatings are applied for a specific well intended application. Hence, it may possess one of the few properties:(25)

- Wear resistance
- High Hardness
- High melting point
- High corrosion resistance
- High density to protect the gases/liquid to penetrate into the surface

3.1.4 Abrasive Wear Testing

Wear is one of worst failure mechanism of the material as it involves loss of material leading to deterioration of engineering components. It can be defined as “unwanted removal and deformation of material by chemical or mechanical action of the opposite surface”.

Wear can be classified into different categories: Abrasive, fretting, adhesive, erosion, etc. Abrasive wear is predominant in most cases. Mainly the researchers have investigated the problems related to abrasive, impact, erosion and all other types of wear on uncoated specimens as separate problems.

More recently there has been a great deal of research on wear testing of coated components and regarding finding out wear testing mechanisms and standardizing it and calculating its wear resistance. Whether these techniques can be applied to thicker coatings as produced by thermal spraying is still in review. The type of wear occurring under combined impact and sliding wear has hardly been studied according to Swick et al.(26)

Apart from the different categories of wear listed above, there are other types as well that occur especially encountered by the coated surfaces. These are low and high stress abrasion, dry particle erosion, slurry erosion, sliding wear and friction etc.(25) Coatings may experience shear, tensile, or compressive stresses which may lead to deformation.(27)

3.1.5 Variables in wear testing

There are two types of variables in wear testing: One which can be easily controlled and the other one which are difficult to control. Below listed are some of the variables encountered in wear testing.

Variables that are easy to control:

- Running time
- Abrasive feed or rate
- Contact area
- Specimen configuration

Variables that are difficult to control:

- Size of the abrasive
- Shape of abrasive
- Hardness
- Toughness
- Motion of abrasive

Abrasive hardness is the most important variable that has to be considered because it may decide the severity of wear and its rate. Here one must talk about the hardness of the abrasive with respect to the surface of the material being worn.

Generally shape of the abrasive is also considered while designing the test. Because, angular shaped abrasives is more intense that similar size round abrasive. Also, angular soft particles bring about more wear than round hard particles do.

3.1.6 Wear Test Criteria (25)

Below listed factors must be considered before adopting a certain test:

1) Select a suitable test that measures the desirable properties of the material
2) Make sure the test adopted is for bulk material or for coatings
3) Presence of abrasive, its size, shape, and all the specifications must be kept in mind before incorporating that into test.
4) It is advisable to perform a test that will duplicate the actual service conditions so that we can speculate the results beforehand
5) The forces acting on the specimen must be properly calculated along with proper loading conditions.
6) If necessary, test temperature must be so adopted that it simulates the service conditions
7) Test duration is also very important factor that has to be kept in mind, otherwise hardest of the materials also wear down if kept in contact of abrasives for prolonged period.

3.1.7 Wear Testing methods(25)

In this section, some of the currently employed and previously used wear testing methods are briefly reviewed.

1) Pin-on-disc:

The mechanism consists of a stationary "pin" under an applied load in contact with a rotating disc. The pin can be of any shape but is generally taken to be spherical to simplify the contact geometry. The pin on disc test has proved useful in providing a simple wear and friction test for low friction coatings. According to Glaeser and Ruff pin on disc method is most widely used for measuring wear followed by pin on flat.(28)

Almond et al. (29) used a pin-on-disc apparatus for testing ceramics and cemented carbides on alumina discs using the pin as the test material. In a two-body abrasion test, a coated pin is pressed against a rotating abrasive paper making a spiral path to avoid overlapping (30), (31). This test process is very common for thin coatings

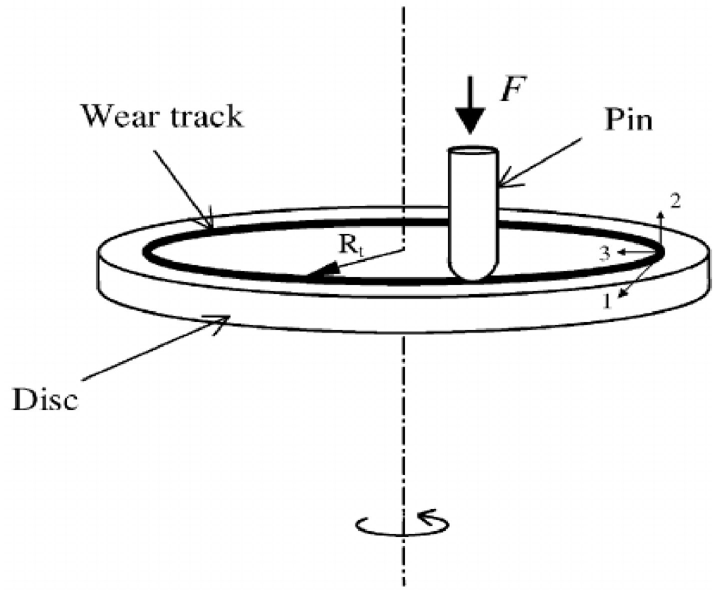


Figure: 12 Schematic of pin-on-disk wear test(32)

2) Pin-on-drum:

In this test, one end of the cylindrical pin is moved over the abrasive sheet of paper with sufficient load to crush the abrasive grains present on the sheet of the paper and in doing so the specimen will also wear down. In this mechanism pin rotates while moving; hence, it can simulate the service conditions that occur during crushing and grinding of ore.

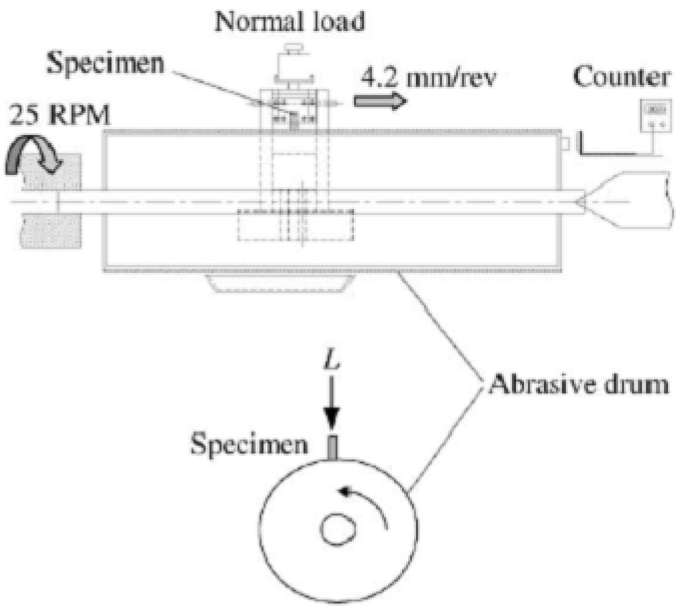


Figure 13: Schematic of pin-on-drum wear test(33)

3) Rubbing test:

This test follows an ASTM (25) standard where one cylinder rubs against other stationary cylinder at right angles. Speed of rotation is equal to 100 RPM. The volume of material loss is calculated through suitable equation. It is used for testing similar and dissimilar metals alloy systems and coatings under unlubricated conditions.

4) Block-on-ring test:

This test works according to ASTM G77-83 standard (25) where a metal ring rotates against a fixed block and makes a line contact with the surface of the block. The advantage of this test is that it allows for variation in materials, test loads, coatings, speeds, and operating atmospheres. Hence, one can tune this test as per his/her requirement.

5) Dry sand rubber wheel test:

This test follows ASTM 65-81 standard and is used to measure the abrasion resistance of materials to silica sand. Here a rubber wheel rubs against a specimen and silica particles are trapped in the rubber wheel and eventually it carries all abrasive particles with it while moving and wears down the specimen. Cerri et al. (34), using similar equipment, examined the abrasion resistance of carbide powders with several materials and coatings used for applications in abrasive environments.

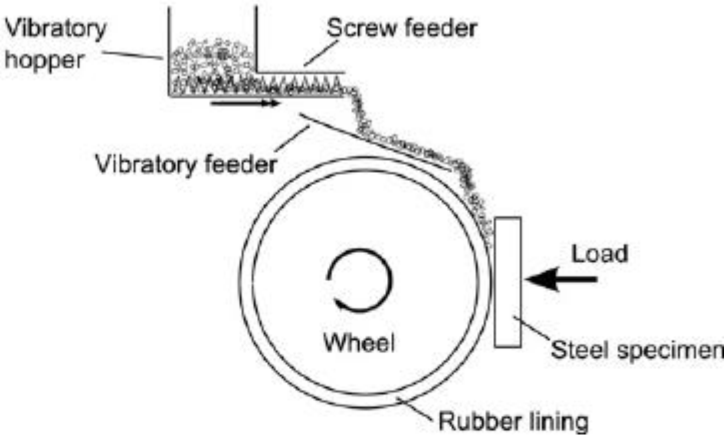


Figure 14: Schematic of dry sand rubber wheel wear test(35)

6) Alumina Slurry test:

This test follows ASTM 611 standard and uses highly abrasive alumina slurry for causing erosive wear. The mechanism incorporates a steel ring that rotates against a flat coated specimen, and the alumina slurry is constantly fed between the interface of the rotating steel ring and the coated specimen. This harsh conditions subjects the sample to a combined impact erosion.

The extent of erosive wear depends upon abrasive size, flow rate, shape of the abrasive, speed of rotation, load acting on the coated specimen, etc.

3.2 Literature Review

Mccaul and Kaufold (23) compared three different types of coatings namely cobalt-chrome, WC (Stellite), and a typical HVOF WC coating. These were sprayed through flame spray, plasma, and HVOF respectively. Coatings performance was evaluated qualitatively on the basis of change in mass and thickness.

The properties of coatings was investigated by testing their abrasive wear resistance, adhesive wear resistance and particle erosion resistance. The results were obtained with microstructure of the final specimen after every test.

Abrasive wear test results showed that tungsten carbide-cobalt coatings show consistently lower losses of both weight and thickness than nickel-chrome- boride and all other coatings. The resistance to abrasion increase as the tungsten content increases.

The results obtained for particle erosion test are consistent with abrasive wear test and showed that tungsten carbide-cobalt sprayed by HVOF method surpassed all other coatings and were more resistant to sliding and abrasive wear conditions.

For adhesive wear test, a proper way to judge a coating's quality is through values of coefficient of sliding friction, scar width, and ring weight loss. All these values must be as small as possible.

Hence, they concluded that tungsten carbide-cobalt appears to be best choice for the mechanical seals with combination of 88 percent tungsten and 12 percent Cobalt. These findings are in full agreement with those of a recently concluded program

While it was not evaluated in this program, there is a published data that states that tungsten carbide-cobalt coatings cannot be used on both mating parts as severe galling and coating failure is likely to occur.

Jari Knuuttila et al.(36) studied the wet abrasion and slurry erosion resistance of alumina and chromia coatings deposited through APS. The effect of Aluminum phosphate ($AlPO_4$) sealing treatment was studied on the wear and corrosion behavior of coatings.

Grit blasting was done prior to coating and no bond coat was used. Quartz sand was used as abrasive for dry rubber wheel abrasion tests while for wet abrasion wear tests, 28 wt% Kaolin or silica was used.

Results of the dry abrasion wear test indicated that Chromia coatings with $AlPO_4$ sealant showed very high wear resistance. However, for wet abrasion wear test, $AlPO_4$ increases the wear for alumina coatings contrary to chromia coatings.

Although, Kaolin is smaller and less hard than silica, still the wear for alumina is high because of chemical affinity between the γ-alumina and kaolin causing tribo-chemical wear.

Rong Liu (37) et al. measured the tribological behavior of Stellite 720 (high carbon Co-Cr- Mo alloy) coating deposited on 304 stainless steel under block-on-ring wear test.

Results of the block-on-ring wear test showed that wear loss increased with contact load but decreased above a certain loads (higher than 900N)

At higher loads, the enhanced friction between the two mating surfaces generates more heat resulting in oxidation of the contact surfaces.

While the oxide films were brittle and weak, they get smashed pretty easily during wear process and gets embedded in specimen surface by high contact load which enhances the hardness which eventually increases the wear resistance.

3.3 Motivation of work

Pump, throughout its lifetime is subjected to various harsh conditions, i.e. it is used in different environment depending upon the type of industry (oil & gas, petrochemical, food, automotive, paper &

pulp, etc.). All these industries have different type of conditions at which pump might be subjected to. Conditions vary from highly acidic to highly basic, highly caustic to alkaline or else neutral as well. Hence, the mechanical seals used in such pumps deteriorate and the life of pump decreases drastically.

Keeping in mind the cost saving and to reduce the downtime of the pumps, life of mechanical seals had to be increased to achieve the above objectives. This was the motivation of the work. Thermal sprayed coatings proved to be the best choice for the application due to following advantages offered:

- Coated material can be repaired easily and there is no need to replace the entire part.
- Any hard wear resistant, corrosion resistant and high melting point coating material can be applied which would protect the seal for longer time.
- It considerably reduced cost and downtime with increased life.

Coated specimen had to be tested to know whether it would be able to resist the service conditions or not. Hence, block-on-ring test mechanism was made and was an effort to duplicate the in service conditions of a pump. This test was a measure of coating's wear/abrasion resistance under load and alumina slurry as well to bring about more abrasive conditions similar to a pump when put to service.

3.4 Experimental Procedure:

3.4.1 Test set-up

This test is known as block-on-ring for measuring the wear/abrasion resistance of thermal spray coatings deposited on cold rolled steel substrate. This test can quantify the amount of coating eroded from the substrate through comparison of mass of the specimen before and after the test.

The test set-up was an effort to simulate the in service test conditions of a pump so we can compare the different coatings and could quote the coating that best resists the harsh environments successfully and increase the life of a mechanical seal.

Below shown is a schematic diagram of the test set-up.

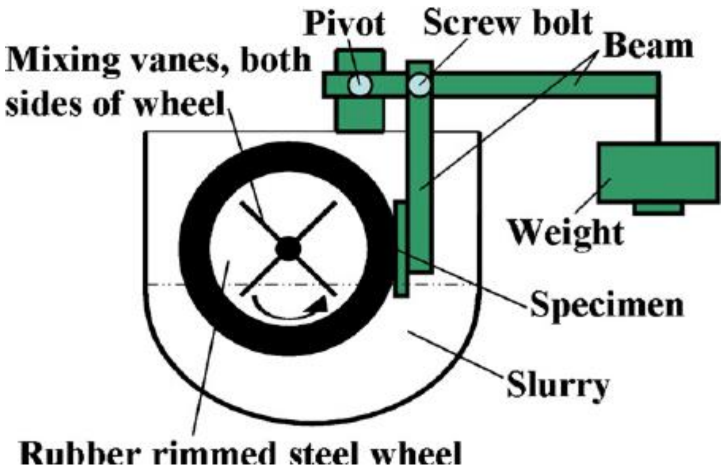


Figure 15: Schematic of test set-up (Earlier) (38)

3.4.2 Testing apparatus:

The figure below shows the actual model of the test set-up known as block-on-ring. The only difference between the actual test set-up and schematic is the way how the slurry is delivered. Earlier we followed the same test set-up as shown in schematics. The actual model of the earlier test set-up is as shown in the figure 17. The slurry delivery mechanism as shown in the schematic diagram above is rotating wheel is dipped into the alumina slurry and it takes the slurry with it to the interface between the ring and the specimen while rotating. The alumina slurry is quite thick and it usually tends to settle down in the container so it needs to be stirred continuously, either with the help of impellor blades as shown in the schematics or by keeping the slurry container over the magnetic stirrer as in our case. (Figure 17).

Unfortunately due to misalignment of the test set-up, especially the slurry delivery mechanism as described above was not giving proper results as the slurry was not mixing well. There was a problem with adjustment of the slurry container over the magnetic stirrer resulting in improper mixing of the slurry giving incorrect results.

The other problem encountered was with the size of the wheel rotating against the coated specimen. The size of the ring was so small that it could not dip properly into the pool of slurry. Hence, the height of the slurry container had to be raised by keeping some wood/plastic plank below it between its base and over the magnetic stirrer. This again reduced the magnetic stirring effect resulting in improper mixing of slurry.

Hence, considering all the problems, the test set-up had to be changed slightly in which just the slurry delivery mechanism was changed as shown in figure 18.

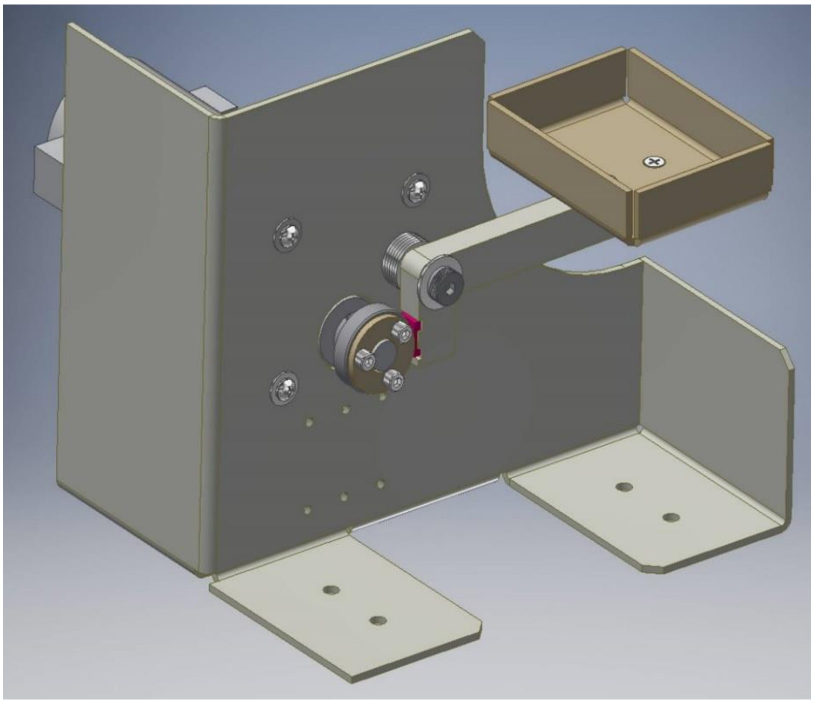


Figure 16: Actual model of the test set-up

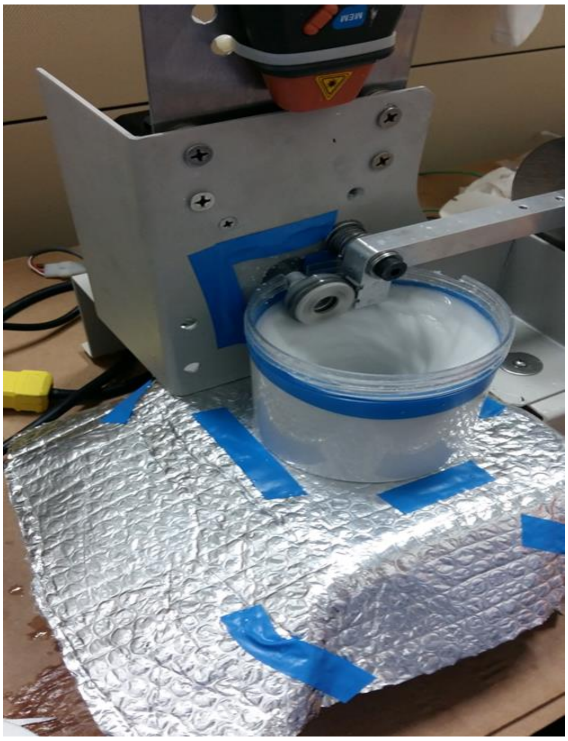


Figure 17: Actual test set-up (Earlier)

As shown in figure 15, slurry is delivered through a small hose pipe onto the interface of rotating wheel and the specimen surface. The hose pipe is attached to the slurry container which is kept over the magnetic stirrer for mixing. The slurry container must be kept exactly in center of the stirrer to get the maximum stirring effect. This is found experimentally. The magnetic stirrer is placed above a box as shown in the figure. This is to give some height to the slurry container with respect to the place where the slurry has to be delivered. This mechanism would make sure that the slurry gets the natural gradient and would flow easily without a need of any pumping mechanism.

A big container is kept below the rotating ring. This container collects the liquid (slurry) that falls from the hose. The slurry container may be refilled at regular intervals from the slurry that falls into the big container.

There is a motor mounted (attached) at the back side which rotates the ring. The ring is a bearing in our case which acts as a steel ring abrading the specimen tangentially. It is fixed against the shaft of the motor with the help of a coupler.

Figure 18: Actual test set-up (modified)

Tachometer was mounted above the shaft of the rotating wheel as shown in the figure 3. This was to measure the RPM of the wheel. Small 12×12 square of reflective adhesive tape was cut and sticked to the shaft of the motor. As the shaft rotated it could successfully measure its rotational speed (RPM) through laser.

The L shaped lever as shown in the figure 3 had a room (slit) made opposite to the rotating wheel to keep the specimen. On the other end of the lever, weights were put in order to load the specimen against the rotating wheel. This would bring about more wear/abrasion to the specimen. Obviously the amount of wear would increase as the load increases. Proper force acting on the specimen was calculated using following formula:

$$\boldsymbol{F = P \times LR}$$

Where $F = force\ acting\ on\ the\ specimen\ due\ to\ load\ applied$

$P = load\ \ (weight)\ applied\ on\ the\ end\ of\ the\ lever$

$LR = Lever\ ratio$

The space through which the shaft of the motor is coming out on the front side is covered by the plastic and taped. This is to protect the motor from getting wet by the liquid (slurry). One must make proper calculations regarding the torque acting on the motor otherwise the motor overheats which might create problems.

3.4.3 Standard Test Conditions:

1) Slurry composition:

Slurry is composed of fine alumina particles with particle size equal to 55-60 microns. 150 grams of alumina slurry is required per test & is mixed in 500 milliliters of distilled water. Hence, the abrasive weight ratio per test would be around 23 %.

2) Ring dimensions:

The rotating wheel is a roller bearing steel and has a slightly tapered geometry. The ring diameter is 35 millimeter and its width is equal to 8.7 millimeters. This rotating steel ring acts as a component that wears down the coated specimen.

3) Rotation speed of ring:

The ring is attached to a motor which rotates at a speed of 400 RPM. This speed was selected between the long ranges of speeds from 250-500 RPM which simulates the service conditions of a pump. The motor was connected to a regulator through which the speed of the motor could be changed.

4) Specimen Size:

Specimen was made of cold rolled steel whose dimensions were equal to 15mm×11mm×1/8inch.

5) Load applied:

The load applied at the end of the lever was equal to 1.45 pounds

6) Force acted on the specimen:

Force acted on the specimen by calculating the lever ratio is equal to 10 pounds. Calculation is as follows:

Here $LR\ (Lever\ ratio) =$

$perpendicular\ distance\ from\ the\ pivot\ point\ to\ applied\ load/$

$Vertical\ distance\ from\ pivot\ to\ specimen$

$$= \frac{\mathbf{147.22}}{\mathbf{21.44}} = \mathbf{6.866}$$

According to equation of force:

$$F = P \times LR$$

$$F = 1.45\ pounds \times 6.866$$

$$\boldsymbol{F = 10\ pounds}$$

7) Torque acting on the motor:

Torque acting on the motor can be calculated by the following formula:

$\tau = Force\ or\ load\ acting\ \times perpendicular\ distance\ of\ shaft\ from\ the\ specimen$

$$\tau = 10\ lbs\ \times 17.5mm$$

$$\boldsymbol{\tau = 175\ lbs.mm = \ 6.89\ lbs.in}$$

Generally, motor must be selected keeping in mind the calculations of torque applied when highest weight is applied otherwise problem of overheating might take place.

3.4.4 Materials:

Four different types of coating systems were tested by the block-on-ring test set-up. All the coating systems were deposited on cold rolled steel (15mm × 11mm × 1/8in) and tested according to the test procedure mentioned in later paragraph. The samples were provided by Nevada Thermal Spray Technologies.

- **Tungsten Carbide/Cobalt (WC/Co)**
- **Boron Carbide (B_4C)**
- **Chrome Oxide (Cr_2O_3)**
- **Alumina (Al_2O_3)**

Table 4

Type of coating system	Method of spray	Thickness (Mils)	Density (g/cc)	Porosity (%) Approx
WC/Co (WC-88% & Co-12%)	HVOF	10 to 15	13.97	1
B_4C-200	D-gun	11	2.52	5
B_4C-300	D-gun	10	2.52	5
B_4C-400	D-gun	10	2.52	5
B_4C-500 (Cermet) (B_4C-80% & Co-20%)	D-gun	8 to 9	2.52	4
Cr_2O_3	Plasma spray	10	5.22	2
Al_2O_3	Plasma spray	9 to 11	3.95	2

Table 4 Different thermal spray coating materials with the specifications and properties

3.4.5 Materials Characterization

The microstructure and macrostructure of the coatings, feedstock materials and worn surfaces was analyzed in a light optical microscope. The characterization included measurement of the thickness of the as sprayed/as received coatings. Microstructural and macrostructural images of the worn surfaces of the coatings are shown later. (Fig 31-37) and (Fig 38-44) respectively. The coatings contained different microstructures which can be distinguished through the diversification of microstructural contrast.

3.4.6 Adhesive wear test (block-on-ring) procedure:

First test was carried out on uncoated cold rolled steel 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 of the specimen was noted as the weight of the sample before the test. Here ring was getting abraded as well hence, the initial weight of the ring was measured.

The ring was attached to the coupler, and then the combined mechanism was mounted to the shaft of the motor. Proper care had to be taken in the adjustment of the ring over the shaft so that it would rotate smoothly without any wobbling effect. Otherwise it would not render proper results in terms of wear of the specimen. This was made sure by starting the motor and seeing the rotation of the ring without loading the uncoated steel specimen. Once, the rotation of the ring was checked, the specimen was loaded onto the slit made exactly to fit the specimen.

First set of tests were carried out in deionized water at room temperature. After pouring the deionized water into the smaller container, it was kept over the magnetic stirrer to maintain the optimum flow of water. Water was refilled during the test as needed. This was followed by switching on the magnetic stirrer to see its working. The hose pipe connected to the container was adjusted exactly over the interface of steel ring and the specimen as shown in the figure 15. As soon as the hose pipe is tapped down the water starts flowing. The lid of the container was closed in order to prevent the water from spilling out.

Finally the weights were attached to the end of the lever. This made the specimen to be in constant load/force while in contact with the ring. The motor was turned on and the regulator was adjusted so that the speed was maintained at around 400-425 RPM. The speed of the motor was checked at regular intervals during test by tachometer mounted over the rotating shaft as shown in figure 15.

After completion of the test, specimen and the ring was unloaded and properly cleaned with running water to remove all the traces of slurry from its surface. Proper drying of the specimen and the ring was carried out followed by measuring their weight. 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.

The same procedure was carried out for all seven coating systems.

This was followed by preparation of slurry. 500 ml of water was poured into a container in which 150 g of alumina powder was added to prepare the slurry solution. It was properly mixed before pouring it into the smaller container through which the slurry was supposed to be delivered instead of deionized water. The wear tests were done in presence of slurry as well because the aim was to duplicate the service conditions of the mechanical seals where they are subjected to abrasive slurries.

The test was carried out for 5, 10, 15, 20 minutes with 10 pounds pressure. The results for the mass loss are shown in the later chapter. Plot of pressure versus time is as shown in figure 16. This was followed by testing at 5 pounds pressure for 20 minutes to measure the effect of prolonged time with lower pressure on mass loss. The tests with alumina slurry were carried out for 10 minutes with 10 lbs pressure. Table 5 shows the testing parameters for adhesive wear tests.

Table 5

Load applied	5 lbs, 10 lbs
Testing time	5,10,15,20 minutes
Motor speed (RPM)	400-425
Temperature	Ambient room temperature
Fluid	Deionized water, alumina(slurry)
Mass loss measurements	Cleaning, drying, measurements after every test

Table 5 Testing parameters for block-on-ring tests

3.5 Results

3.5.1 Microstructure of as-received coatings

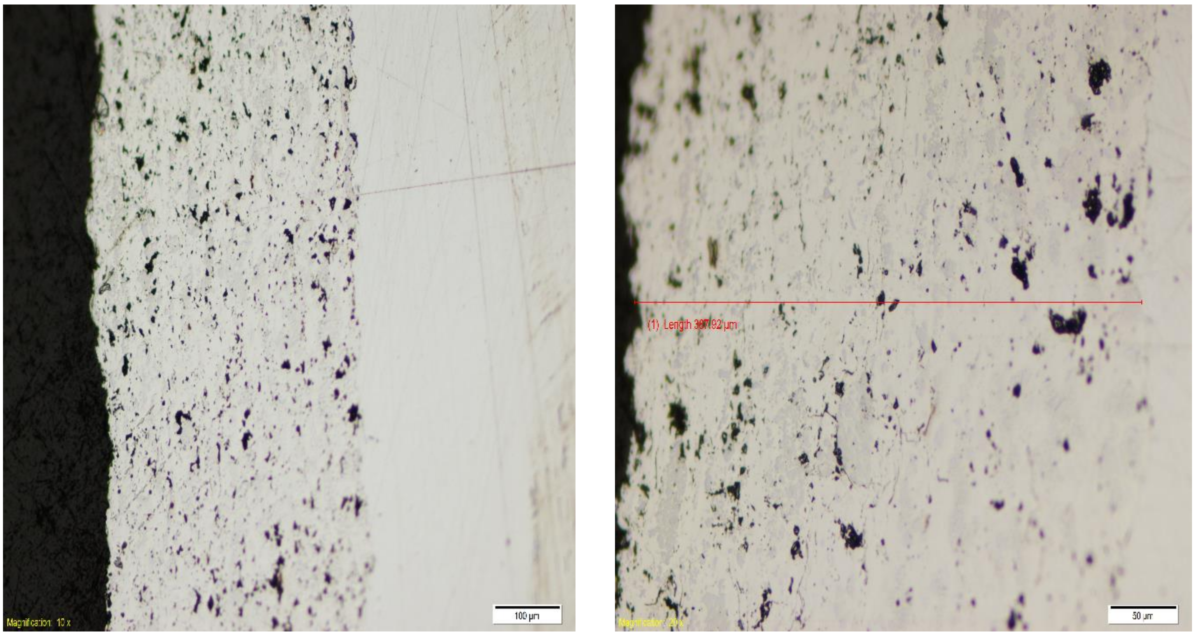


Figure 19: WC-Co Coating

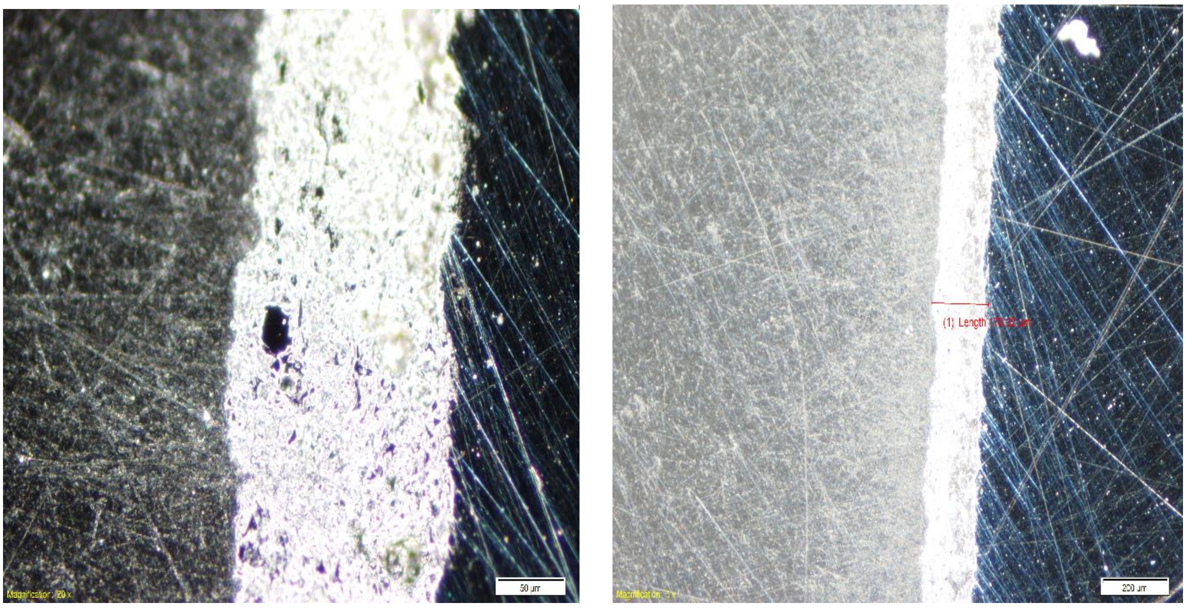


Figure 20: B_4C-200 Coating

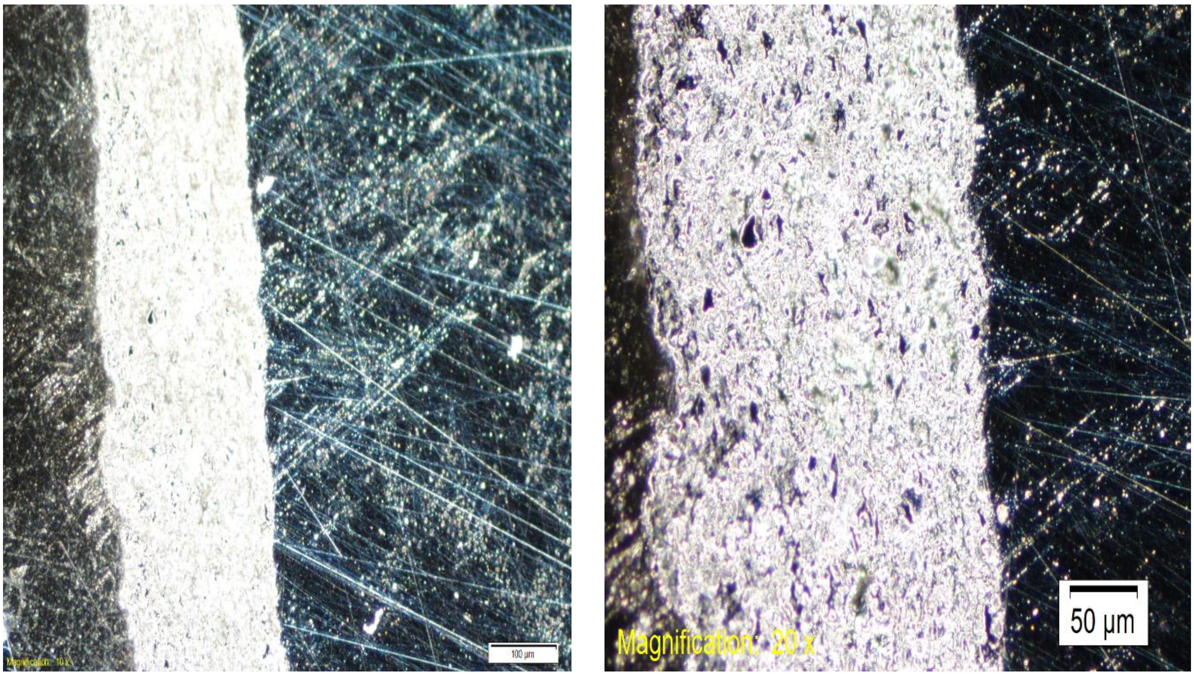


Figure 21: B_4C-300 Coating

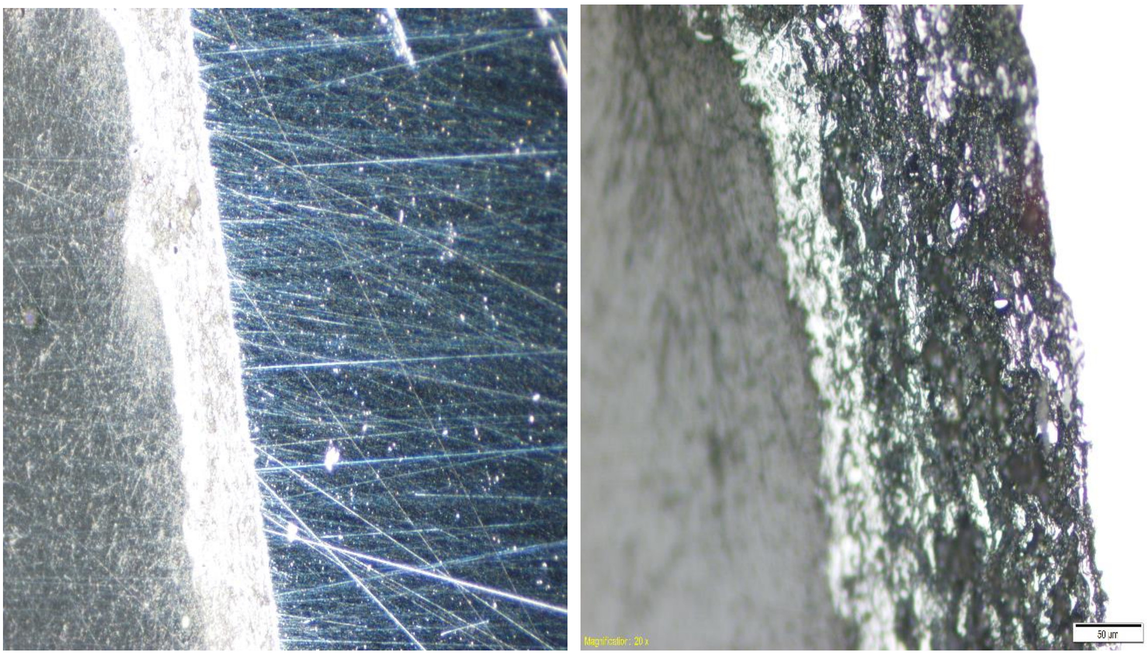


Figure 22: B_4C-400 Coating

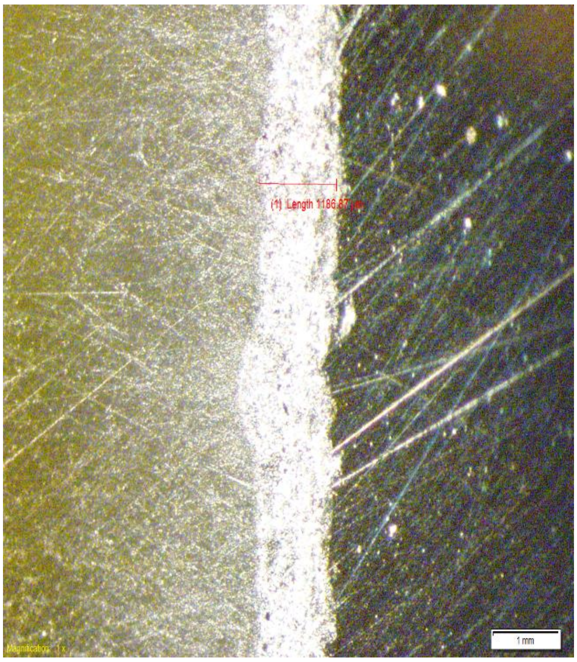


Figure 23: B_4C-500 Coating

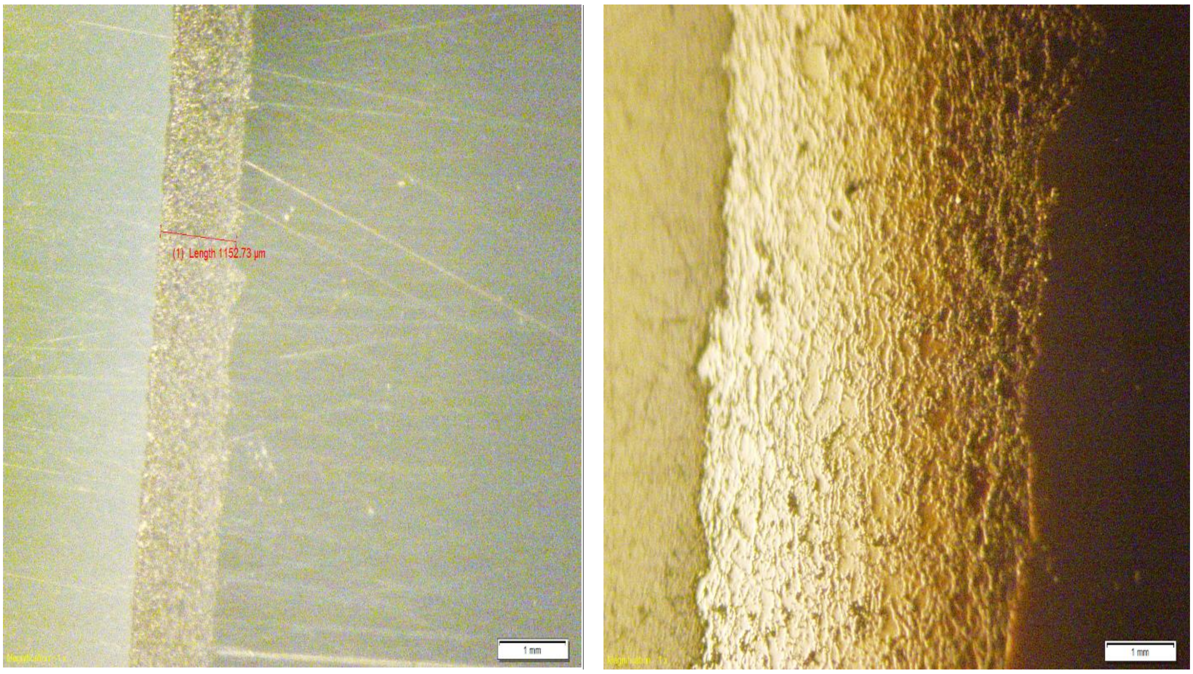


Figure 24: Cr_2O_3 Coating

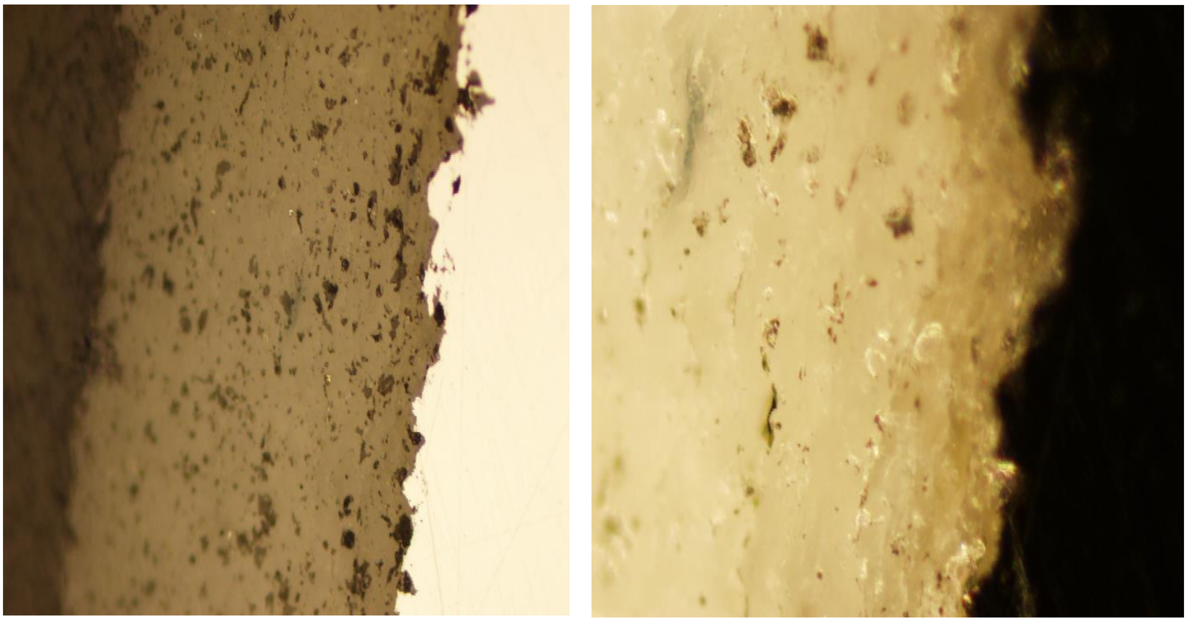


Figure 25: Al_2O_3 Coating

3.5.2 Adhesive wear test with deionized water at room temperature:

Figure 26 below shows the volume losses measured after adhesive wear test in deionized water for 10 minutes under 10 pounds load. Also, table 5 below shows the results of the tests conducted at different times with constant load of 10 pounds. The volume losses follow a linear relation with respect to time in early stages but it roughly follows the polynomial fit of second power following a quadratic approximation (Fig 29). The mass losses were converted to volume losses to get accurate wear loss data. We need to consider the density and porosity of the materials as well. Hence, volume losses would give us more accurate results rather than mass loss.

As it can be seen from the results (fig 26) tungsten carbide-Cobalt (WC-Co), showed the lowest volume loss compared to all thermal spray coatings followed by Chrome oxide (Cr_2O_3) and Alumina (Al_2O_3). B_4C-300 series showed the highest volume loss followed by B_4C-400 series amongst Boron Carbide coatings. Amongst all the four boron carbide coatings, the one with metal (Cobalt) with 20% volume showed the best results; i.e. lowest volume loss. This may be due to the presence of cobalt metal that acts as a binder that increases the bond strength of B_4C coating.

WC-Co produced the best results. It has thought that superior hardness and wear resistance are brought to the coating by high volume percentage of tungsten carbide, fine grain size of WC, and also the tough matrix phase of cobalt in comparison to other WC coatings.

It greatly depends upon the applied pressure which certainly dominated the wear rates.

For applications demanding adhesive and abrasive wear (Slurry) which is often in case of mechanical pumps, tungsten carbide/cobalt offers best wear resistance in all conditions based on testing conducted in this experiment. The sole aim was to duplicate the service conditions of a mechanical pump. This is 88% WC and 12% Co which offers the best wear resistance and has successfully performed during service according to several reports. It seems that 88-12 WC-Co performs best when run against steel but, cannot be employed if both the mating parts are made of WC-Co. This would cause high galling and coating failure. (39)

Boron Carbide coatings showed the highest volume loss and large scar width as well. Large scar width indicates that this material would deform or wear much quicker than the others. It is more fragile and less dimensionally stable under loading, implying more rapid loss of tolerances between the rotating and stationary rings in a pump.

This may be due to high porosity i.e. low bond strength between particles or very weak particle to particle bonding. According to the definition of thermal spray: “Any material can be thermally sprayed unless it decomposes after heating”. In case of Boron Carbide after heating, carbon phase falls apart from the coating in the form of loose powder. Hence, the overall coating gets too porous giving highest wear loss.

However, a reduction of porosity could probably result in an increase of wear resistance due to high hardness of boron carbide. This is showed and experimentally proven through an experiment in the current program, where the testing of B_4C-500 (Cermet) shows lower volume loss because of the presence of Cobalt metal binder.

Below shown are the results for the first run of the tests with the following parameters:

1) Rotational speed of the ring: 400-425 RPM
2) Time: 10 minutes
3) Pressure: 10 pounds
4) Temperature: Ambient room temperature
5) Slurry: None (Deionized water)

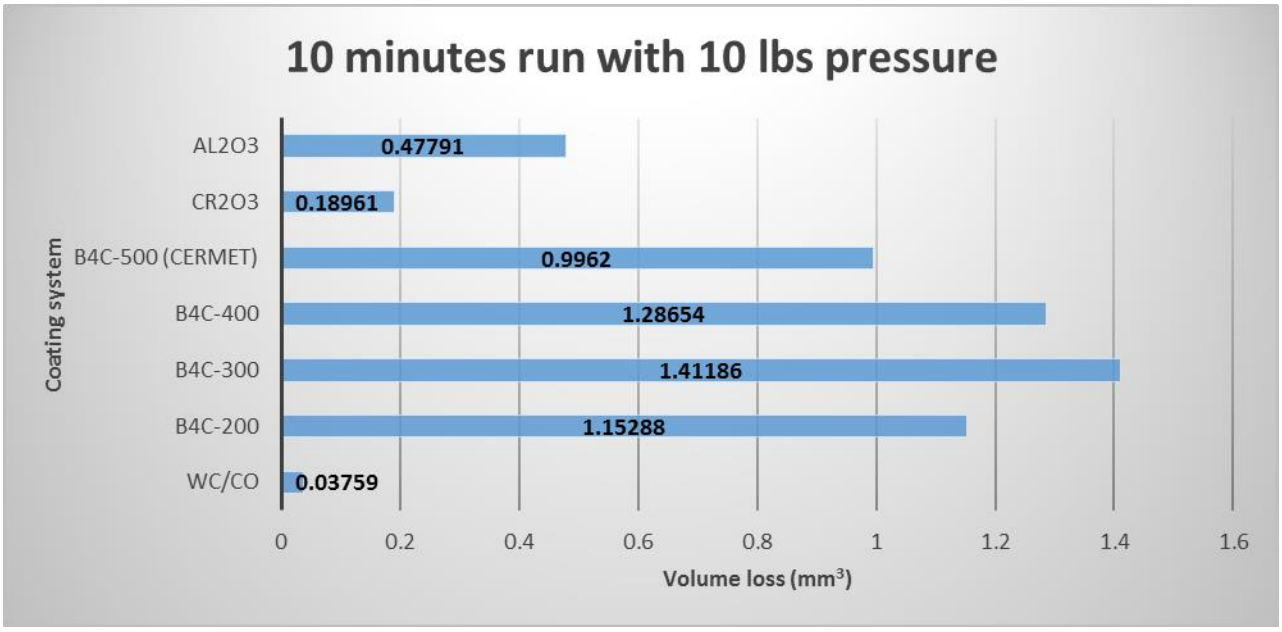


Figure: 26 Volume loss of specimen in cubic millimeter after 10 minutes with 10 pounds load

Below shown are the results for the second run of the tests with the following parameters:

1) Rotational speed of the ring: 400-425 RPM

2) Time: 20 minutes

3) Pressure: 5 pounds

4) Temperature: Ambient room temperature

5) Slurry: None (Deionized water)

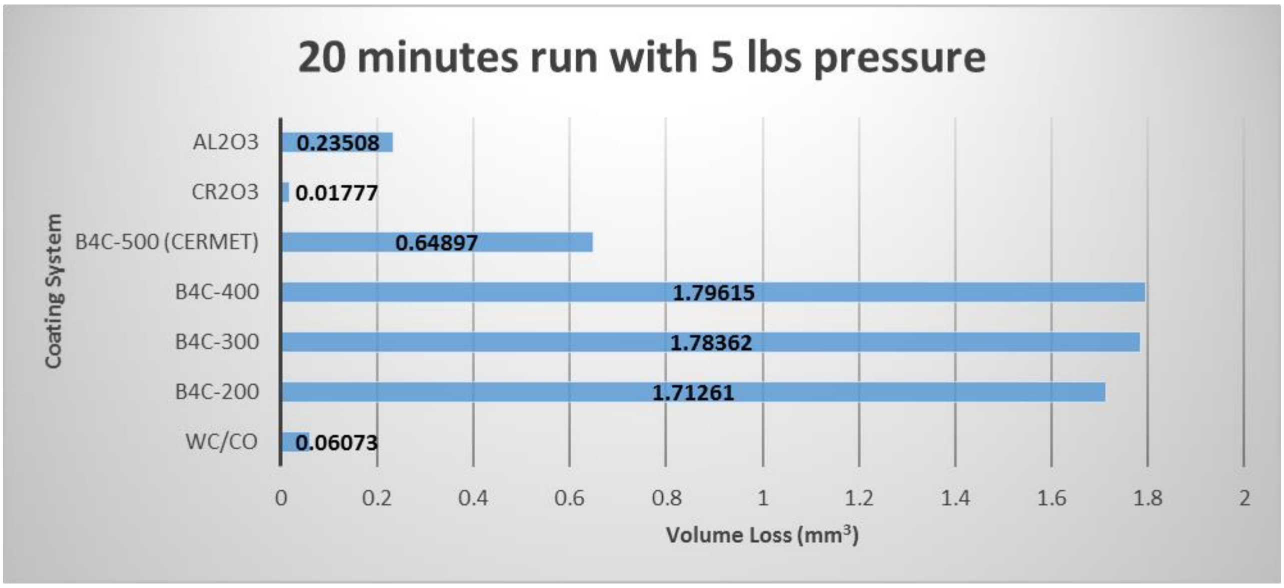


Figure: 27 Volume loss of specimen in grams after 20 minutes with 5 pounds pressure

Below shown are the results for the third run of the tests with the following parameters:

1) Rotational speed of the ring: 400-425 RPM

2) Time: 5 minutes

3) Pressure: 10 pounds

4) Temperature: Ambient room temperature

5) Slurry: None (Deionized water)

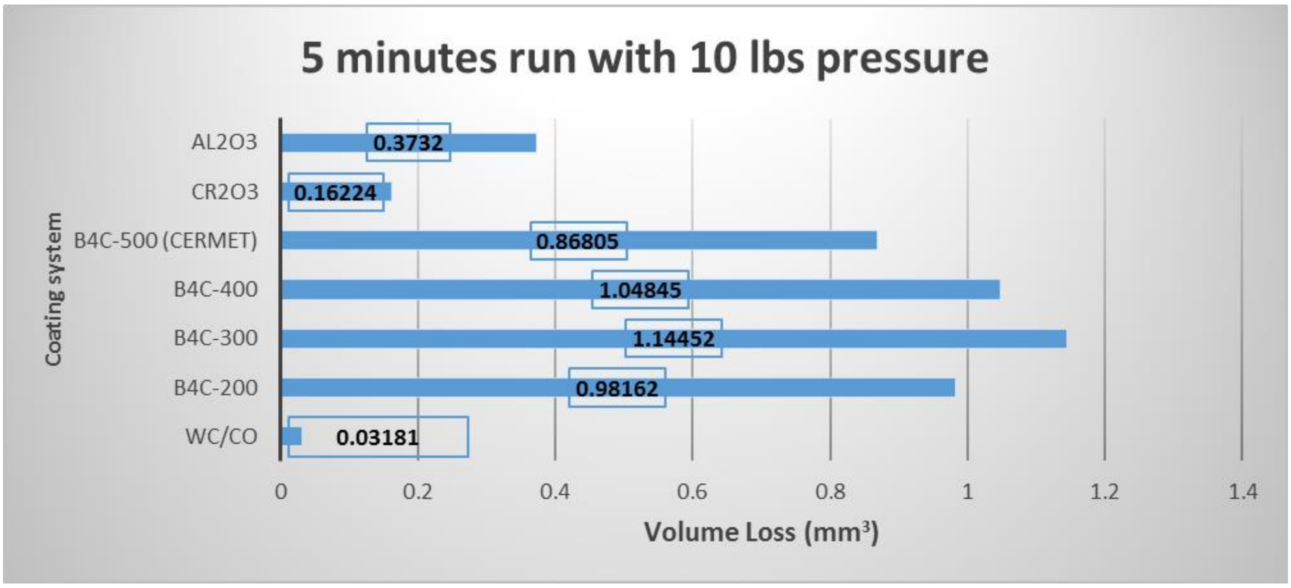


Figure: 28 Volume loss of specimen in grams after 5 minutes with 10 pounds load

Below shown are the results of volume losses over different time intervals at constant load.

Table 6

Time (Minutes)	WC/Co	B_4C- 200	B_4C- 300	B_4C- 400	B_4C- 500 (Cermet)	Cr_2O_3	Al_2O_3
5	0.03181	0.98162	1.14452	1.04845	0.86805	0.16224	0.3732
10	0.03759	1.15288	1.41186	1.28654	0.9962	0.18961	0.47791
15	0.03976	1.4035	1.59983	1.48705	1.20701	0.27367	0.56316
20	0.0535	1.80451	2.03	1.89223	1.57903	0.35968	0.84732

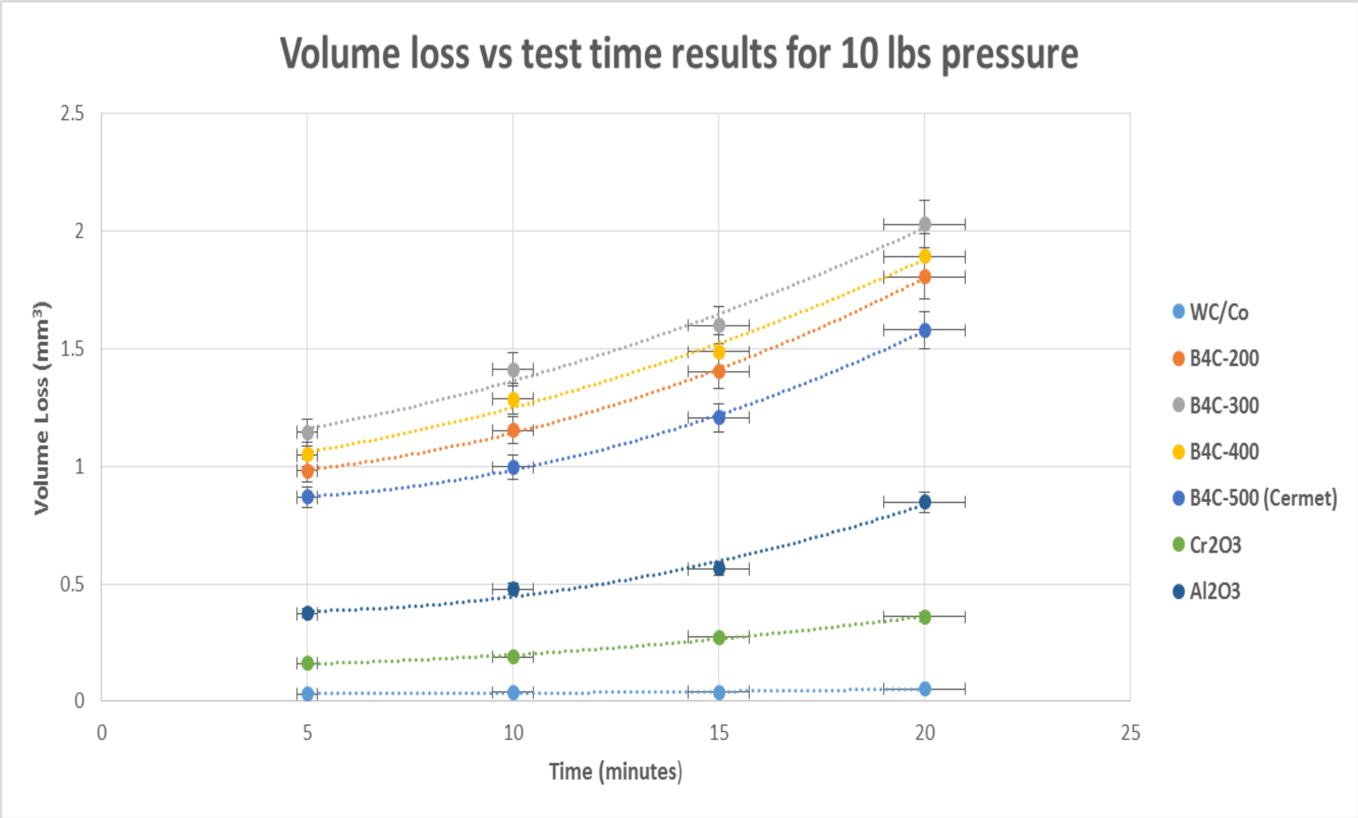


Figure 29 Volume losses in cubic millimeter with constant load (10 lbs) as a function of testing time

3.5.3 Adhesive wear test with alumina slurry at room temperature:

Alumina slurry with particle size 50-55 micron was used as abrasive medium to measure the volume losses of the coatings at 10 lbs load for 10 minutes. The concentration of slurry was 50 grams per every 150 milliliters of deionized water.

Figure 26 shows the volume losses measured after each adhesive wear test in alumina slurry. As expected, the volume losses were much higher compared to in deionized water. This clearly shows that an abrasive medium (alumina slurry) has much more damaging effect in terms of material loss compared to deionized water.

As it can be seen from the results (fig 26) tungsten carbide-Cobalt (WC-Co), showed the lowest volume loss and outperformed all the other thermal spray coatings followed by Alumina (Al_2O_3) and Chrome oxide (Cr_2O_3). Also as expected, boron carbide had the worst results in terms of mass loss/volume loss.

While it is not listed in the results, there has been research done on WC-Ni coatings instead of WC-Co where Nickel metals replaces Cobalt and gives anti-corrosive properties to the coating system. Nickel is much more chemical resistant than cobalt and hence does not get affected by the caustic, high/low pH abrasive slurries. But cobalt being an excellent binder it is extensively used with WC in pumps seals. For anti-corrosion, chemical resistance, chromium and its alloys are generally taken into consideration.

Below shown are the results of fourth run of tests with the following parameters:

1) Rotational speed of the ring: 400-425 RPM
2) Time: 5 minutes
3) Pressure: 10 pounds
4) Temperature: Ambient room temperature
5) Slurry: Alumina (55 micron)

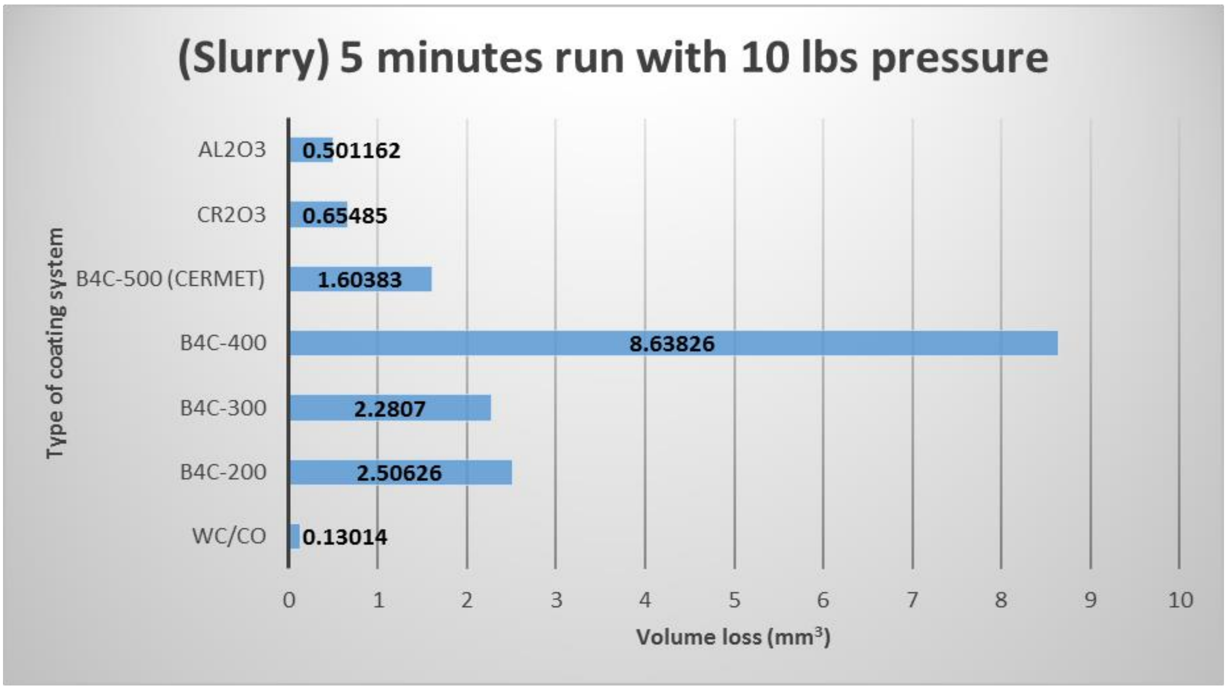
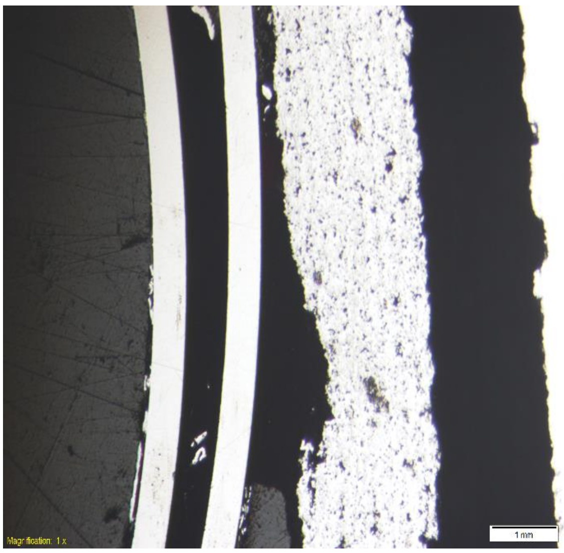


Figure: 30 Volume loss of specimen in cubic millimeter after 5 minutes under 10 pounds loads in presence of alumina slurry

3.5.4 Analysis of worn surfaces and identification of wear mechanisms in adhesive wear test with deionized water at room temperature:

3.5.4.1 Microstructural Images of worn surfaces



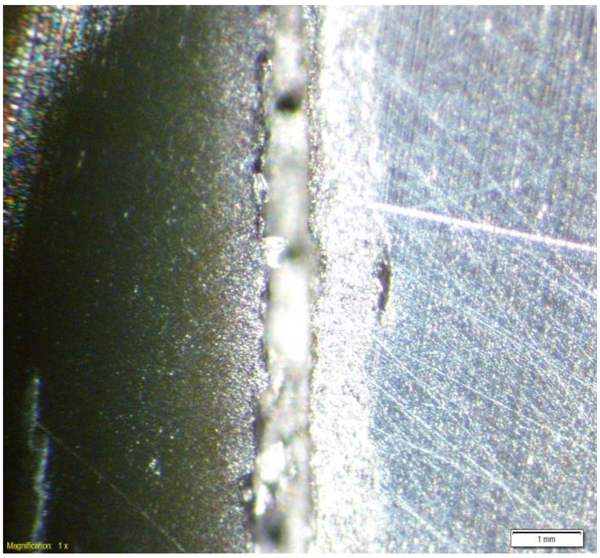
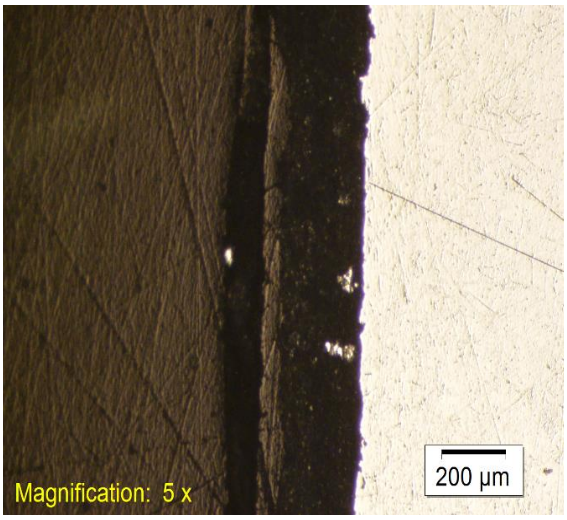


Figure 31 WC-Co Coating

Figure 32 B_4C-200 Coating



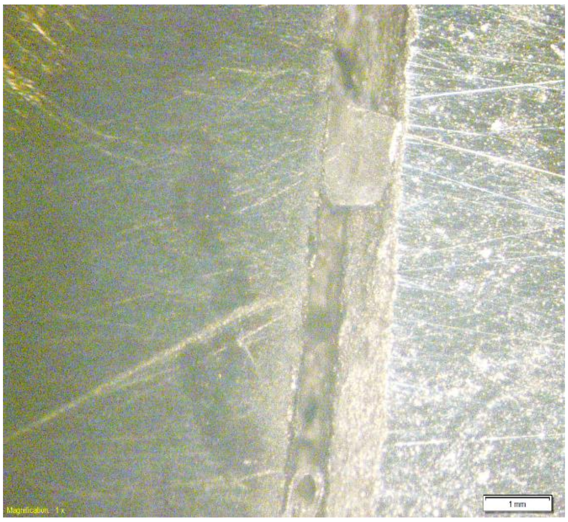


Figure 33 B_4C-300 Coating

Figure 34 B_4C-400 Coating

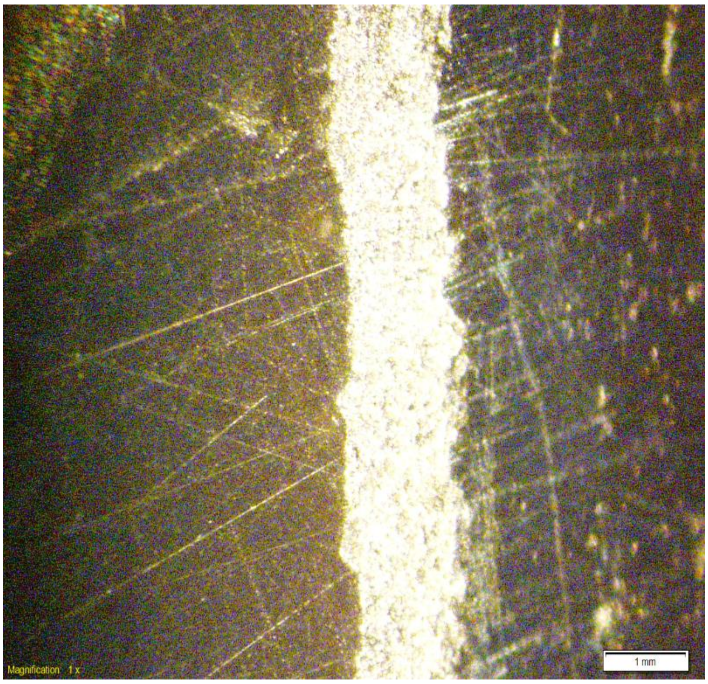
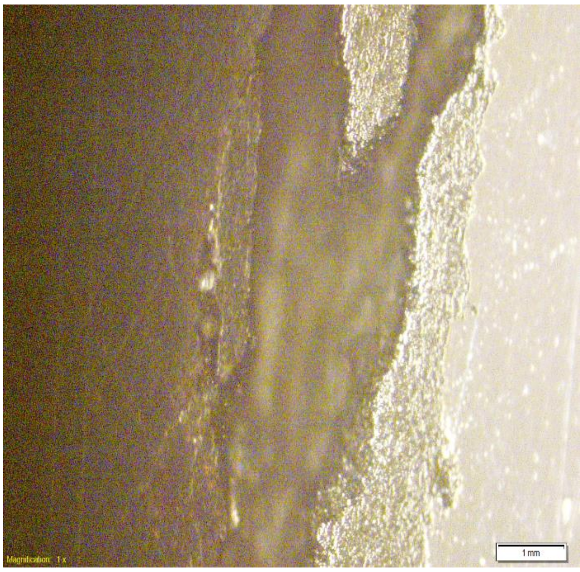


Figure 35 B_4C-500 Coating



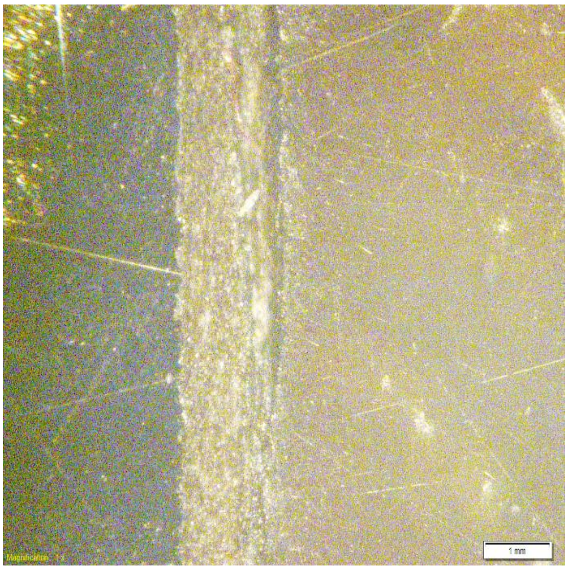


Figure 36 Cr_2O_3 Coating

Figure 37 Al_2O_3 Coating

3.5.4.2 Macrostructure images of worn surfaces

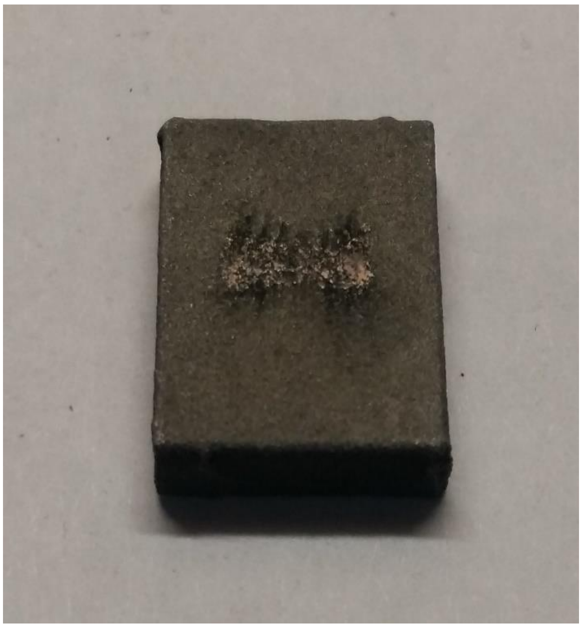


Figure 38 WC-Co Coating

Figure 39 B_4C-200 Coating

Figure 40 B_4C-300 Coating

Figure 41 B_4C-400 Coating

Figure 42 B_4C-500 Coating

Figure 43 Cr_2O_3 Coating

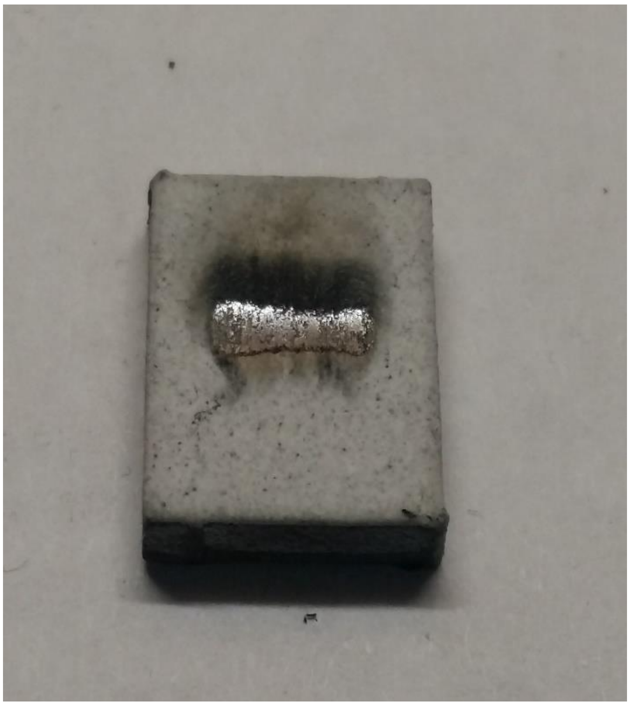


Figure 44 Al_2O_3 Coating

3.6 Discussion

According to the macroscopic test data obtained (Fig 38-44), it can be said that the scar width of the Tungsten Carbide-Cobalt (WC-Co) is smallest amongst all coatings followed by the Chrome Oxide (Cr_2O_3) and Alumina (Al_2O_3) which supports the experimentally found data of volume loss.

Although, by looking at the shiny part on the surface of the specimens (Fig 38-44) it looks like the wear scar has got down to the substrate, but the microscopic images (Fig 31-37) show that the coating has not been entirely chewed up by the wear mechanism and has not got down till the substrate.

The shiny part in the macroscopic images constitutes a very thin microscopic stainless steel layer which has been stuck on the surface of the specimen during the operation. This might be probably due to the heat produced by the friction between the mating surfaces (stainless steel ring and the specimen) during the test. This phenomenon can be seen prominently with boron carbide coatings (Fig 32-34) where one can clearly see two distinct phases in coating after the wear test.

There was a direct correlation observed between the thickness of the coatings and its wear resistance property. As it can be clearly seen from the microstructural images of boron carbide coatings (Fig 32-35), the thickness of B_4C-200 (11 mils) coating was the highest amongst all and it had highest wear resistance compared to B_4C-300 and B_4C-400. Thinner coatings are somewhat more defective, because the flattening of cermet particles impacting onto substrate surface is restrained by the deformation of the surface itself, so that the particles retain small pores. Thicker coatings, are denser because, as a new layer is deposited, the high velocity impact of the particles forces the previously deposited material with high pressure and densifies it. Also (40) new particles can flatten more efficiently, as they impact on a much harder surface (the previously deposited coating layers); therefore, the new coating layers are less defective than the first ones.

Table-5 shows that the volume losses increase linearly with time and follows the polynomial fit of second power with quadratic relation. This shows the amount of load is a prominent factor that determines the measure of volume loss. The results obtained for 5 pounds and 20 minutes (Fig 27) were unexpected and are not very definitive.

The wear test carried out in the presence of an abrasive (Alumina) (Fig 30) shows that volume losses are higher compared to the test carried out in the deionized water with all the other conditions same. Tungsten Carbide- Cobalt (WC-Co) outperforms every other coating followed by Alumina (Al_2O_3) and Chrome Oxide (Cr_2O_3). This data supports the claim that the hardness of alumina (9 on Mohs scale) is higher than Chromium oxide (8-8.5 on Mohs scale) and hence, it wears down Chromium oxide more than that of Alumina, and we get higher volume loss for chromium oxide in the presence of alumina slurry. But, the hardness of Tungsten carbide particles (9-9.5 on Mohs scale) is higher than alumina abrasive particles, hence, the wear loss is much lesser, so we get lowest volume loss for Tungsten Carbide-Cobalt (WC-Co) in presence of alumina slurry.

3.7 Summary and Conclusion

The goal of this test was to measure the abrasion/wear resistance of different systems of thermal spray coatings for the application of mechanical seals used in various pump industries.

This was done by building up a machine according to the ASTM G77 test standards (block-on-ring) test set-up that measures the adhesive/sliding wear resistance of thermal spray coatings with and without the presence of abrasive slurry (alumina in current program). This machine was built in order to duplicate the pump's service condition so we could get the perspective of the best coating in the required conditions.

The test study shows that Tungsten Carbide-Cobalt (WC-Co) with 88% WC and 12% Co offers the highest abrasive/wear resistance followed by Chrome oxide (Cr_2O_3) and Alumina (Al_2O_3) in deionized water i.e. without the presence of abrasive slurry. This data is supported by the macroscopic images of the specimens which shows that the scar width of Tungsten Carbide-Cobalt (WC-Co) is smallest followed by Chrome oxide (Cr_2O_3) and Alumina (Al_2O_3). Boron Carbide (B_4C-200,300,400) set of thermal spray coatings offers lowest wear/abrasion resistance due to high porosity (poor particle-particle bonding); which had the largest scar width. Amongst boron carbide thermal spray coatings, B_4C-500 (Cermet) comprising of 80% B_4C and 20% Co offers the highest wear resistance. This is due to presence of binder metal (Cobalt) which binds the loose boron carbide particles.

Tungsten Carbide-Cobalt (WC-Co) with 88% WC and 12% Co outperforms every other coating in presence of alumina slurry as well, followed by Alumina and chromium Oxide. Alumina offers higher wear resistance than Chrome oxide as its hardness is almost equal to the slurry and higher than chrome oxide. The volume losses follow a linear relation with respect to time in early stages but it roughly follows the polynomial fit of second power following a quadratic approximation

While, the wear does not reach up to the substrate level, it does reveal some shiny silvery part on the surface of the specimen. The silvery shiny part is revealed by optical microscopy to be a thin layer of stainless steel which is stuck on to the coatings due to heat produced by the friction between the mating surfaces.

Also, it was found through the materials characterization in light optical microscopy that thickness of the coatings has a direct correlation with their wear resistance. As the thickness of the coatings increases, their wear resistance also increase.