3. EXPERIMENTAL PROCEDURE:

3.1 Sample Preparation:

Casting is a manufacturing process by which a liquid material is usually poured into a mold, which contains a hollow cavity of the desired shape, and then allowed to solidify. The solidified part is also known as a casting, which is ejected or broken out of the mold to complete the process.

Rings is always produced in circular shape for the market use and it is very hard to get a fresh piston rings to produce in our desired shaped because lots of finishing operation is already performed on the rings material. As per our machine Pin on Disc tribometer either we have to produce a pin of circular shape or disc of desired dimension. In our experiment we decided to produce a disc of the well know composition for the testing purpose.

A wooden pattern of the desired dimension based on the constrained of the plasma spray coating machine is prepared, for the easy removal of the casting a draft of $½^{0}$ is provided on the pattern. A sand mould is prepared with the help of press and the cavity is ready for the pouring of metal.

Molten metal is prepared of with the pure constituent powder. The powder of **Carbon (C) , Silicon (Si) , Magnesium(Mn) , Phosphorous (P) , Sulphur (P) ,Chromium (Cr) & Copper (Cu)** are used for the preparation of charge for the furnace . For the melting of this powder Induction Arc Furnace (Fig 3.1) is used at a temperature of 1540 ^{o} C.

Fig 3.1 Induction Arc Furnace Used for the Melting of Charge

Figure 3.2 Powder as charged (A) Mn Slab (B) Cu Powder (C) Si Powder (D) Cr Powder

The induction arc furnace is preferred because it provides uniform melting of the charge. Stag Casting is done for the preparation of the entire sample at one shot to get

the uniform composition of the entire slab. The composition of the slab is controlled as per the following table-3.1

Element	C (%)	Si (%)	Mn (%)	P (%)	S (%)	Cr (%)	Cu (%)
Target	3.75±0.05	2.70±0.05	0.53±0.01	0.36±0.02	.06±0.005	0.04±0.01	0.035±0.03

Table-3.1- Composition of the test sample prepared

The composition of silicon can vary on the basis of inoculation. The composition of chromium & Sulphur is very strictly controlled.

During the casting we faced problem of mould leak several times. It's also very difficult to get the casting of 280 X 90 X 15 mm thickness without bending or distortion.

After the solidification of the casting it's removed from the mould by breaking the sand mould and the sand blasting operation using SiC powder is done to clean the casting. Now slab is prepared but with irregularities and poor surface. Grinding Operation is required for the accurate dimension and clear surface.

3.2. Coating Preparation:

Coating is a covering that is applied to the surface of an object, usually referred to as the substrate. In many cases coatings are applied to improve surface properties of the substrate, such as appearance, adhesion, weldability, corrosion resistance, wear resistance, and scratch resistance. In other cases, in particular in printing processes and semiconductor device fabrication (where the substrate is a wafer), the coating forms an essential part of the finished product.

3.2.1 Plasma Arc Spray Coating

One plate made of Piston Rings material (250X90X10 mm) was plasma sprayed with a Sulzer-Metco PT F4 torch in a closed room using a robot to ensure controlled and reproducible trajectories and speeds. The powder feeding system was a single bowl apparatus and the powder feed rate was fixed at 50±5gm/min. The plates were sand blasted with Al_2O_3 powder prior to spraying. During spraying, a cooling system consisting of air jets and Venturi nozzles was applied as shown in Fig.4.1. In Table 3.2, the operating conditions are reported. A special attention was paid to the dependency of micro- structure and chemical composition of coatings on the nature of the plasma gas: Ar:H or Ar:He, the power off the plasma jet: 13–19.5 kW and the cooling device**.**

Sr. No	Process Parameter	Specification	Unit
1	Powder Port I.D.	2.2	mm
2	Water Flow Rate	4.0	Liter/min

3	Temperature of chiller	62	^{0}F
4	Distance between spray gun & mandrel	140(At gun angle 30^0)	mm
5	Argon Flow Rate	112	m^3/min
6	Hydrogen Flow Rate	13	m^3/min
7	Argon Pressure	95	psi
8	Hydrogen Pressure	80	psi
9	Powder flow Rate	50	gm./min
10	Voltage	70	volt
11	Current	460	ampere
12	Gun Feed	10	mm./min
13	Gun Angle during spray	30	degree
14	Cooling air pressure	47	kgf
15	Powder driving temperature	120	^{0}C
16	Powder mixing	90	Min.

Table:3.2- Operation condition of Air Plasma arc coating

The surface treated slab of cast iron which is cleaned with the mechanical method of Sand Blasting is used for the preparation of plasma arc spray coating. The slab is fixed on the fixture of the Plasma Arc Spray Machine as shown in the figure 3.3.

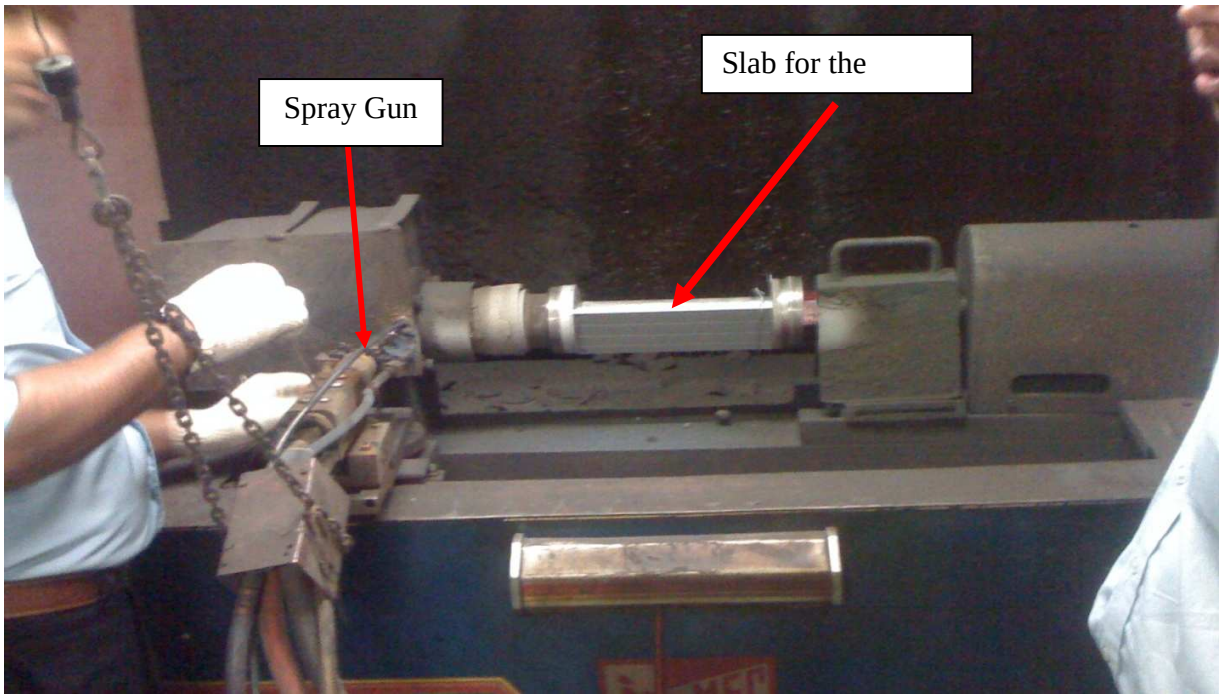


Figure 3.3 Air Plasma Arc spraying machine with the sample mounted on it for the purpose of coating

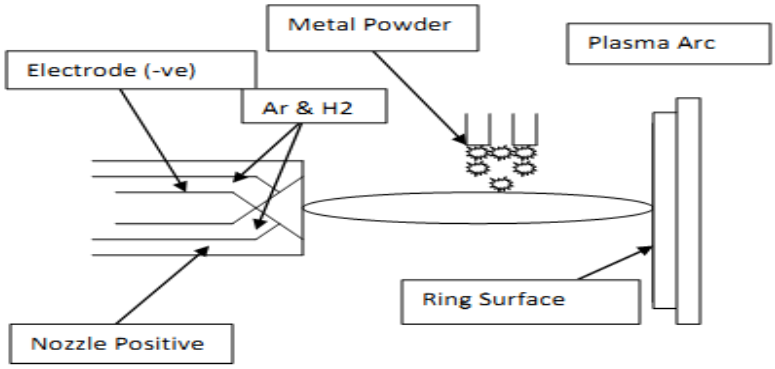


Figure 3.4 Schematic of air plasma arc spraying

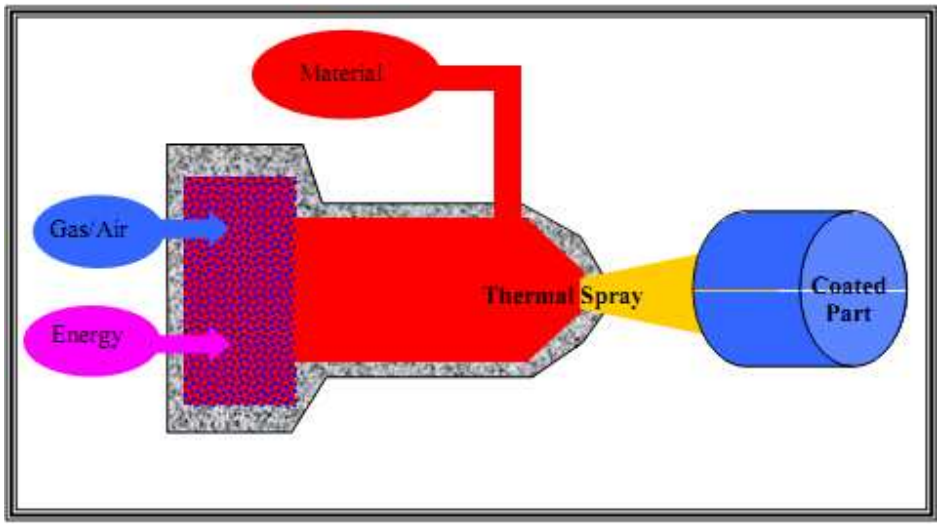


Figure 3.5 Schematic diagram of Plasma Arc coating

3.2.1(A) Principle of Plasma Spray

In this , First Primary(Argon) & Secondary(Hydrogen) Gas mix in chamber & flow inside Gun caliber then a heavy Voltage is drawn which create a mega spark by forming conduction path b/w nozzle & electrode without making contact b/w electrode & nozzle. (I.e. as spark plug in engine gives spark, by coming –ve & +ve terminal closer to each other & after that an ion exchanging starts which result as Mega Spark.) . When these hot gases pass through b/w this mega spark, it turns the gas stream into plasma.

Figure 3.6 (a)

Figure 3.6 (b)

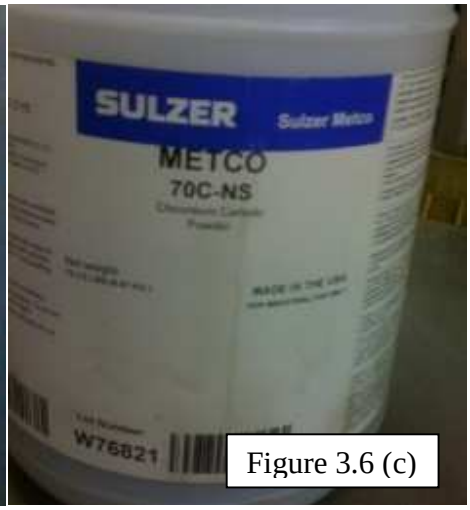


Figure 3.6 (c)

Figure 3.6 a) Plasma Spray Coating machine unit b) Arc during the operation of coating C) Coating Powder container

Moly Powder Description:-

We are using the mixture of four powders for plasma spraying. The detailed of all are given below.

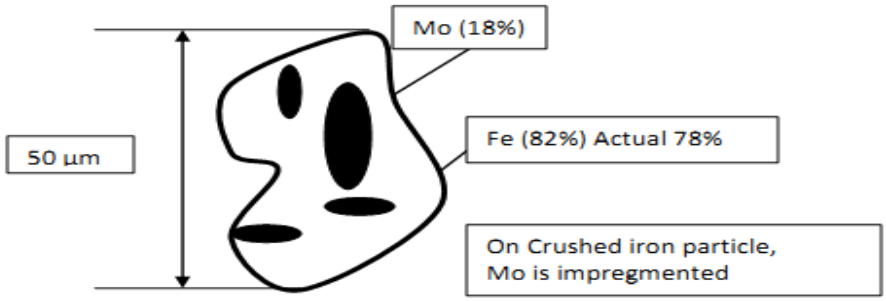
a) Metco-350

b) Metco-63

c) Metco-70

d) Metco-43F NS

Metco-350



Chemical composition of Metco-350

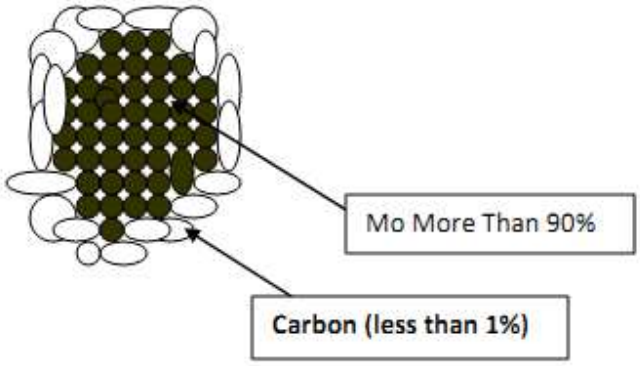
(High carbon Iron molybdenum composite powder)

Sr. No	Elements	Percentage (%)
1	Mo	**15 ~ 20 %**
2	B	**0.4 ~ 1.1 %**
3	Mn	**Less than 0.5%**
4	WC	**2 ~ 4%**

Table 3.3 Composition of Metco-350

Chemical Composition of Metco-63

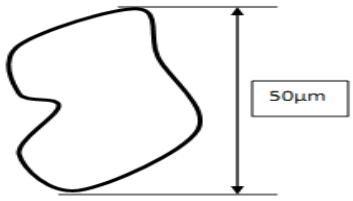
(Molybdenum powder)



- 1µm moly powder (Mo) is rolled in to a ball

Chemical Composition of Metco-70(Cr_3C_2)

(Chromium Carbide Crushed Powder

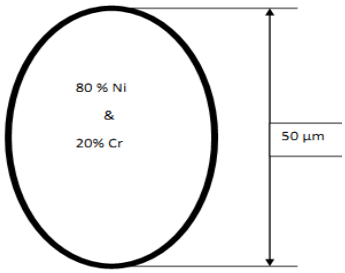


Sr. No	Elements	Percentage (%)
1	Cr	More than 86 %
2	WC	More than 13 %
3	C	Less than 0.3 % (in free form)
4	Fe	Less than 0.7 %
5	Si	Less than 0.1%

Table 3.4 Composition of Metco-70(Cr_3C_2)

Chemical Composition of Metco-43F NS (NiCr)

(Nickel Chromium Alloy



Powder Metco-43F NS (NiCr) acts as binder & helps for rapid solidification (atomizing) i.e. Quenching.

Sr. No	Elements	Percentage (%)
1	Ni	76~80%
2	Cr	18~22%
3	C	less than 0.25%
4	Mn	less than 2.5%
5	Si	less than 1.5%
6	Fe	less than 1%

Table 3.5 Composition of Metco-43F NS (NiCr)

These all the Moly powder are mixed together with Binder & Slurry. Using this mixture a lump of 50µm size is prepared. The mixture should be mixed properly to avoid the composition variation and to get the uniform property of coating after its preparation.

The approximate final composition of one charge is represented below in the table3.6. The charge amount prepared for the coating is of 6 Kg.

Sr. No	Elements	Material Type	Quantity(Kg)
1	(63)Mo	Hard material	1.2
2	(43)NiCr	binder	0.6
3	(70)Cr_3C_2	Hard material	0.6
4	(350)Fe Alloy	Base material	3.6

Table 3.6 Composition of the final mixture ready for coating

- The quantity of one lot of coating powder is 6kg.

3.2.1(B) Some remarkable point about the Plasma Spray Coating Machine

Sr. No	Parameter	Control
1	Bonding	8,000 – 9,000 PSI
2	Spray Rate	Can be increased by Increasing the Input Current
3	Choice of porosity	Porosity of deposit is higher
4	Choice of Coatings	Different metals, Combination of Metals, Ceramic can be deposited in desired quantity

5	Input	Metal in Powder form

Table 3.7 Important control parameter of air plasma spray coating

In the Plasma Arc Spray Coating the Hydrogen is used as a secondary gas because 8000℃ temperature produced by the Argon Gas (Argon temperature) is not enough to melt the high velocity powder particles. Thus to raise the temperature up to 10,000℃ to 12,000℃ for smoother melting of the mixture of po wders we used Hydrogen (H2)

.

3.2.2 Preparation of Hard Chrome Plating

Hard chromium electroplating is a common coating method that is used for coating the surfaces of industrial components. In this electrolytic process, baths containing the hexavalent chromium ion, Cr6+, is used. Hard chromium is used as a wear resistant coating in many industries. [99]. Chrome has some distinct characteristics that are advantages in resisting wear: It is very hard, slippery and it is resistant to most corrosive environments. A thick chrome deposit is of value when it provides additional wear surface and distinguishes it from a thinner decorative coating. But it may develop a pattern of tiny cracks when the stresses become greater than the strength of the coating. Cracks and the porosities due to residual stresses are the characteristics of chrome plating .These cracks often form an interlacing pattern to the base metal allowing corrosive liquid or gas to penetrate. [98]

The cast iron plate (250X90X10mm) to be coated with chromium was initially grit blasted to a roughness value (Ra) of 10µm in order to enhance the adherence while coating. Samples were prepared by conventional hard chromium plating on cast iron plate (250X90X10mm) for a thickness of 250 µm using standard acidic bath and current density with deposition parameters as practiced in industries which is provided in Table 3.8

Sr. No	Parameter	Specification	Unit
1	Bath Temrature	50-55	^0C
2	Voltage	2.5	voltz
3	Approximate current density	40	A/dm^2
4	Bath stirring method		Pneumatic Stirring
5	DC Current	200	A
6	Bath Volume	1	m^3
7	Bath Duration	4-6	h
8	Chromic Acid	240-280	g/l
9	Sulphate	0.9-1.6	g/l

Table 3.8 Hard Chrome deposition parameter

S3.2.3 Preparation of Gas Nitriding sample

It is well known that nitriding techniques are commonly used to improve the fatigue and wear resistance of metals and alloys [8]. Moine et al. [9] have also tried to increase the wear resistance of TiNi alloys by N^+ ion implantation. Gas nitriding is a case-hardening process where nitrogen is introduced into the surface of a solid ferrous alloy by holding the metal at suitable temperature in contact with nitrogenous gas, usually ammonia. The nitriding temperature for all steels is between 495^0C and 565^0C (925 and 1050 ^{0}F) .In the present study, the gas-nitriding technique has been successfully used to form a nitride layer on the piston ring material of composition C (3.75±0.05%), Si (2.70±0.05%), Mn (0.53±0.01%), P (0.36±0.02%), S (.06±0.005%), Cr (0.04±0.0), Cu (0.035±0.03%). The gas-nitriding parameters used for the nitriding the sample of 90*90*10 mm are mentioned in the table-3.9.

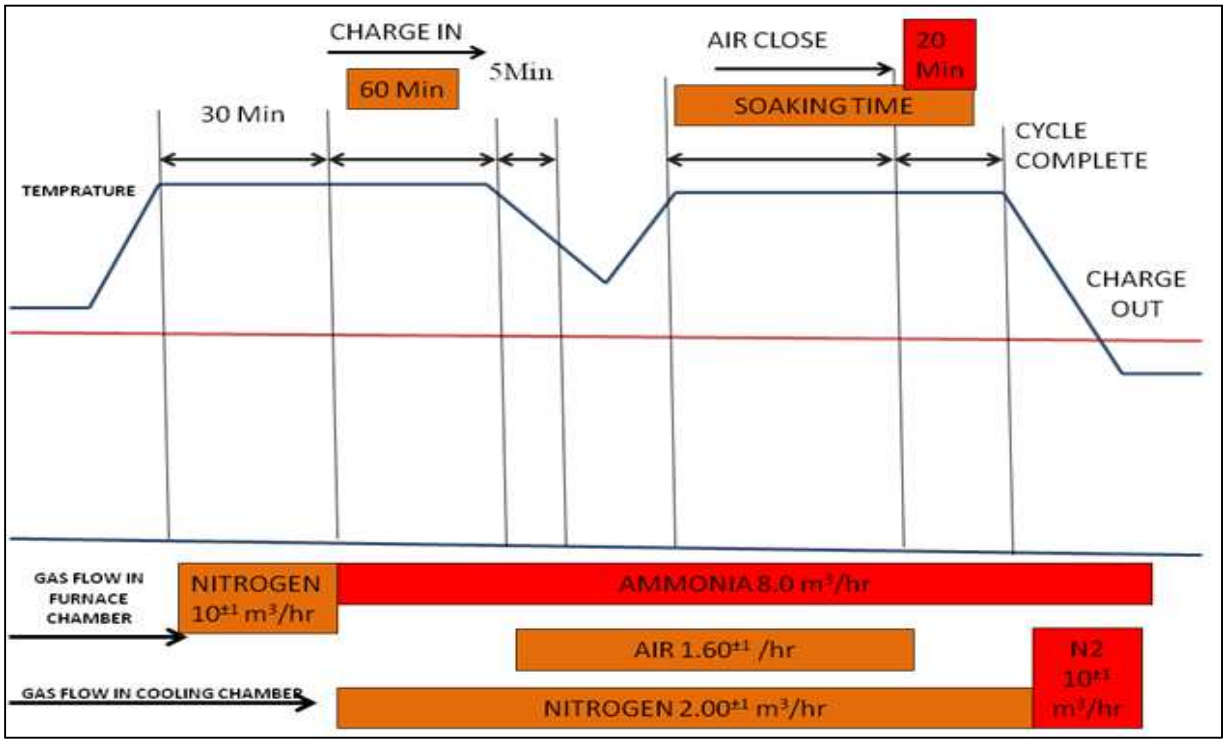


Figure 3.7 Common nitriding cycle

Sr. No	Elements	Flow Rate	Time
1	Nitrogen(In Furnace Chamber)	$10^{\pm1}$ m^3/hr	30 min
2	Ammonia(in furnace Chamber)	8.0 m^3/hr	120 min
3	Air	$1.60^{\pm1}$ /hr	80 min
4	Nitrogen(In cooling Chamber)	$2.00^{\pm1}$ m^3/hr	180min

Table-3.9 Process parameter for the nitriding cycle

3.3 DESIGN OF EXPERIMENT:

Statistical methods are commonly used to improve the quality of a product or process. Such methods enable the user to define and study the effect of every single condition possible in an experiment where numerous factors (load and sliding speed) were involved in present work to study the wear behavior of the piston rings coatings. There were one parameters (Load) which were taken into consideration to determine the wear rate and coefficient of friction. There are several methods to design the experiment but we have chosen constant sliding distance (1.2Km) & Constant RPM 500 with varying load of 30N, 40N, 50N (Table 2). To determine the response such as wear rate, coefficient of friction, microstructure, EDS analysis, and XRD analysis.

Variables	Level 1	Level 2	Level 3
Sliding speed (rpm)	500 rpm	500 rpm	500 rpm
Load (kg)	3 kg	4 kg	5 kg

Table 3.10 Variables for wear test

Coatings	Counter Body	Run	Load(N)	Sliding speed (rpm)
Plasma Coating	HCS,WC,Nikil	1,2,3	30	500
Plasma Coating	HCS,WC,Nikil	4,5,6	40	500
Plasma Coating	HCS,WC,Nikil	7,8,9	50	500

Hard Chrome	HCS,WC,Nikil	10,11,12	30	500
Hard Chrome	HCS,WC,Nikil	13,14,15	40	500
Hard Chrome	HCS,WC,Nikil	16,17,18	50	500
Gas Nitriding	HCS,WC,Nikil	19,20,21	30	500
Gas Nitriding	HCS,WC,Nikil	22,23,24	40	500
Gas Nitriding	HCS,WC,Nikil	25,26,27	50	500

Table-3.11 Design of experiment table for wear test

3.4. Pin on disc test:

Pin on disc type wear monitor with data acquisition system was used to evaluate the wear behavior of aluminum alloys against three pin of WC Nickel & En-31. Load was applied on pin by dead weight through pulley string arrangement. The system had maximum loading capacity of 200 N. The test was performed under dry unlubricated condition. The wear test can be performed on any wear tester, but for thin coatings pin on disc wear test is most commonly used (figure 3.7).

Figure 3.8- Wear and friction monitor machine for pin on disc test

Figure 3.9 Hard Chrome coating before wear test

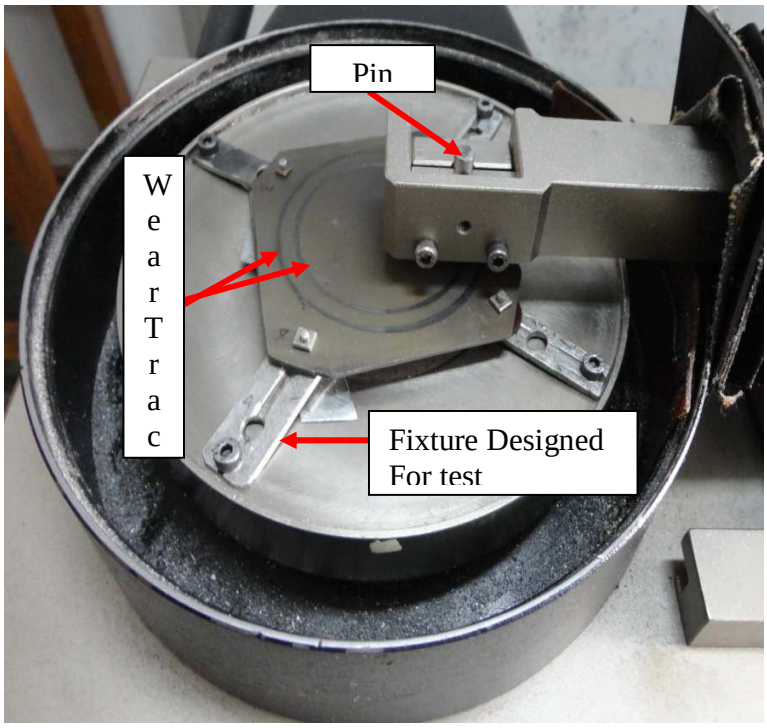


Figure 3.10 Hard Chrome coating during wear test

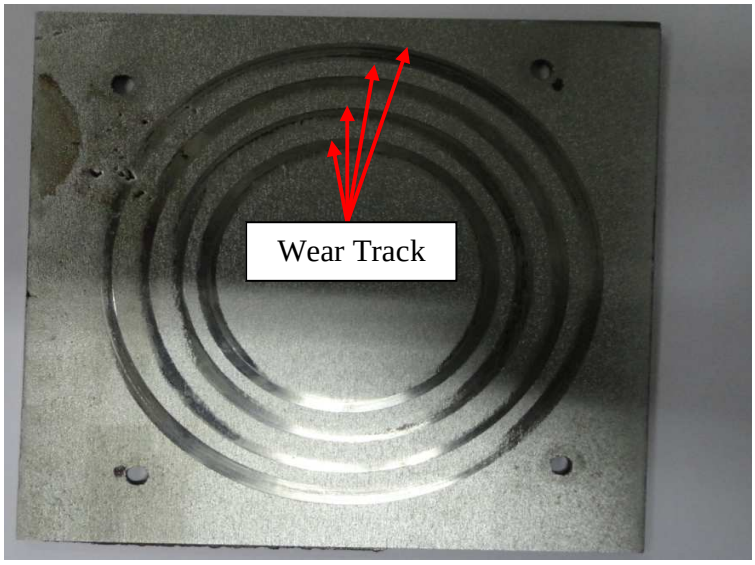


Figure 3.11 - Hard Chrome coating after wear test

The machine is attached with the computer with software WINDCUM 2008. A window is open in the software and there are options to select various loads, times, pin diameter. The machine directly gives the coefficient of friction on the selected loading and sliding conditions. In this machine basically there is a rotating disc; and a pin is fixed over stainless steel pin holder. The pin can be loaded with different loads, it can be change externally. The coating pasted disc fastened on the machine with the help of screws (figure 3.9). The load was applied on the pin through dead weight loading arrangement. The coating surface and pin was initially washed with methyl alcohol so that, moisture should not present on coating surface. Initially, the brass pin was fixed on the pin holder; the wear rate of the coatings was calculated at different loading and sliding conditions. The wear rate was calculated by weighing the disc before after the wear test in terms of grams on an electronic balance of least count 0.00001g. The load was taken as 30, 40, and 50 N respectively and the sliding speed was taken as 500 rpm & distance was taken 1.2km. The wear behaviour against three various counter pin material was analyzed that was Nickel, En-31 and WC pin. The pin of diameter 3 mm was chosen for all of the three materials. The wear test carried out at room temperature of 20°C. During the wear test some amount of material also gets deposited on the pin in the form of a tribolayer so pin was cleaned after every test. So that there was always contact between pin and the coating surface, and the wear mechanism was between pin and coating surface, and a wear track was formed on the coating (figure 3.10)

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3.5. Scanning electron microscope:

A scanning electron microscope (SEM) is a type of electron microscope that images a sample by scanning it with a high-energy beam of electrons in a raster scan pattern (figure 3.11). The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition, and other properties such as electrical conductivity.

Figure 3.12- Scanning electron microscope in DTU, Delhi

In a typical SEM, an electron beam is thermionically emitted from an electron gun fitted with a tungsten filament cathode. Tungsten is normally used in thermionic electron guns because it has the highest melting point and lowest vapour pressure of all metals, thereby allowing it to be heated for electron emission, and because of its low cost. For

conventional imaging in the SEM, specimens must be electrically conductive, at least at the surface, and electrically grounded to prevent the accumulation of electrostatic charge at the surface. Metal objects require little special preparation for SEM except for cleaning and mounting on a specimen stub. Nonconductive specimens tend to charge when scanned by the electron beam, and especially in secondary electron imaging mode, this causes scanning faults and other image artifacts. They are therefore usually coated with an ultrathin coating of electrically-conducting material, commonly gold, deposited on the sample either by low vacuum sputter coating or by high vacuum evaporation. Conductive materials in current use for specimen coating include gold, gold/palladium alloy, platinum, osmium, iridium, tungsten, chromium and graphite [47]. Coating prevents the accumulation of static electric charge on the specimen during electron irradiation. For SEM, a specimen is normally required to be completely dry, since the specimen chamber is at high vacuum. Hard, dry materials such as wood, bone, feathers, dried insects or shells can be examined with little further treatment, but living cells and tissues and whole, soft-bodied organisms usually require chemical fixation to preserve and stabilize their structure. Fixation is usually performed by incubation in a solution of a buffered chemical fixative, such as glutaraldehyde, sometimes in combination with formaldehyde [48-50]. In order to study the wear mechanism the worn surface were examined by scanning electron microscope of S-3700 series in DTU, Delhi. To see the microstructure of the wear track the coating material is coated with gold. Then it was put on job holder, the job holder was then moved inside the chamber of the scanning electron microscope. The scanning electron microscopy was used to determine the surface morphology of the wear track which

gave the wear mechanism at various loading conditions and various speeds. For SEM of samples following parameters were chosen that were Accelerating Voltage=15000 Volt, Deceleration Voltage = 0 Volt, Magnification=1000, Working Distance=12600 um, Emission Current=80000 nA.

3.6. X -Ray diffractometer:

X ray diffractometer is a measuring instrument for analyzing the structure of a material from the scattering pattern produced when a beam of radiation or particles (as X rays or neutrons) interacts with it. A typical diffractometer consists of a source of radiation, a monochromator to choose the wavelength, slits to adjust the shape of the beam, a sample and a detector (figure 3.12). In a more complicated apparatus also a Gonio meter can be used for fine adjustment of the sample and the detector positions. When an area detector is used to monitor the diffracted radiation a beam stop is usually needed to stop the intense primary beam that has not been diffracted by the sample. Otherwise the detector might be damaged. Usually the beam stop can be completely impenetrable to the X-rays or it may be semitransparent. The use of semitransparent beam stop allows the possibility to determine how much the sample absorbs the radiation using the intensity observed through the beam stop. The specimen of the worn surfaces was placed on X-ray chamber. The scanning of the specimen was done from angle 20˚ to 90˚ and the scanning speed was chosen as 2 degree/min.

Figure 3.13-X-Ray diffractometer in DTU, Delhi

3.7. Vickers micro hardness tester:

Vickers Hardness Tester is a key piece of equipment that is indispensable to metallographic research, product quality control, and the development of product certification materials.

Vickers Microhardness test procedure as per ASTM E-384, EN ISO 6507, and ASTM E-92 standard specifies making indentation with a range of loads using a diamond indenter which is then measured and converted to a hardness value. For this purpose as long as test samples are carefully and properly prepared, the Vickers Microhardness

method is considered to be very useful for testing on a wide type of materials, including metals, composites, ceramics, or applications such as testing foils, measuring surface of a part, testing individual microstructures, or measuring the depth of case hardening by sectioning a part and making a series of indentations. Two types of indenters are generally used for the Vickers test family, a square base pyramid shaped diamond for testing in a Vickers hardness tester and a narrow rhombus shaped indenter for a Knoop hardness tester.

The Vickers hardness test method requires a pyramidal diamond with square base having an angle of 136° between the opposite faces. Upon completion of indentation, the two diagonals will be measured and the average value will be considered (figure 3.13).

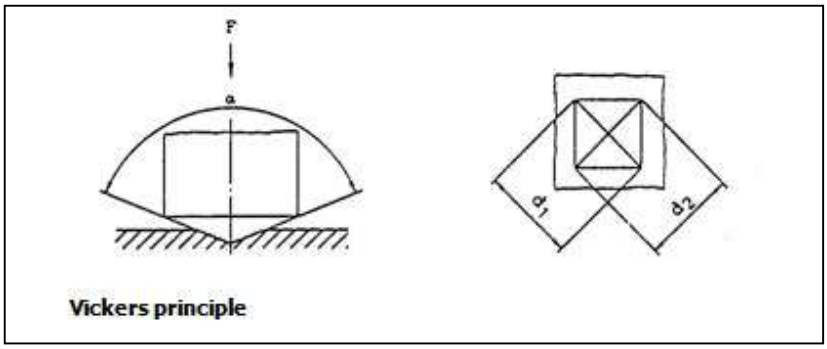

Figure 3.14 - Vickers micro hardness indentation

The loads for Micro Vickers or Knoop hardness testing methods are typically very low, ranging from a few grams to 2 kg. The load range for Macro Vickers hardness test procedure can range up to 50kgs.Normally the prepared specimens; using metallographic mounting presses are mounted in a plastic medium to facilitate the preparation and testing. In order to enhance the resolution of measurement, the

indentations should be as large as possible. The micro hardness was measured with the help of Vickers micro hardness tester. It was compatible with computer; the indentations formed by indenter can be seen. The load can be takes over the micro hardness tester was upto 100 kg. And the magnification of the indentation was 200 x and 400X. The load selected was 5 gm, because at high load the indenter would be large. The magnification chosen was 400 X, because at that low load indentation was very small.

3.8. Optical Microscope:

It is an instrument used to see objects too small for the naked eye. The science of investigating small objects using such an instrument is called microscopy. Microscopic means invisible to the eye unless aided by a microscope.

Figure 3.15 - Optical microscope

The most common type of microscope and the first invented is the optical microscope (figure 3.14). This is an optical instrument containing one or more lenses producing an enlarged image of a sample placed in the focal plane. Optical microscopes have refractive glass and occasionally of plastic or quartz, to focus light into the eye or another light detector. Mirror-based optical microscopes operate in the same manner. Typical magnification of a light microscope, assuming visible range light, is up to 1500x with a theoretical resolution limit of around 0.2 micrometres or 200 nanometers. Specialized techniques (e.g., scanning confocal microscopy, Vertico SMI) may exceed this magnification but the resolution is diffraction limited. The use of shorter wavelengths of light, such as the ultraviolet, is one way to improve the spatial resolution of the optical microscope, as are devices such as the near-field scanning optical microscope.