3. EXPERIMENTAL PROCEDURE:

3.1. Materials:

Commercial aluminum leads the production centers in varying degree of priority ranging from 99.6% down to 99.3%. The principal impurities are iron and silicon together with traces of titanium and copper [4]. Aluminium has specific gravity of 2.7 as compared with 8.89 for copper and 7.8 for iron. The thermal and electrical conductivities are high and owing to its chemical affinity for oxygen a film of oxides forms spontaneously of freshly cut surface. To this property aluminum owes its high resistant to chemical and atmospheric attack. The super pure aluminum is softer and ductile and reflects light and heat more efficiently posses high thermal conductivity and greater resistance to corrosion [2]. In general, the 3xxx series of aluminum alloy is used when moderate strength combined with high ductility and excellent corrosion resistance is required. The solid solubility of manganese in aluminum is as high as 1.82%, and magnesium from 0.8% to slightly more than 5% is widely used [58]. In present work aluminum alloy coating having aluminum 98.76%, magnesium 0.81%, and manganese 0.41% was successfully prepared by two wire arc sprays coating prepared with two wire arc sprays on the graphite substrate. The graphite substrate was used because the coating after solidification does not adhered over the graphite substrate. When graphite was used as a substrate, flat thick Al coating which could be peeled off with ease was obtained but the coating quality depended on the temperature gradient between the graphite plate and the coating and on the cooling rate. Initially, when experiments were conducted at room temperature, the Al coating did not stick to the graphite surface. It became distorted and finally cracked. Later, when some experiments were conducted by heating the graphite plate up to 120˚ C and by continuously cooling the back of it, improved results were obtained. However, when the Al alloy coating tensile specimen were tired to prepare though die, the tensile specimen were shrunken, distorted and broken due to rapid thermal gradient with metallic die and graphite plate. Commercially available Al alloy (core diameter - 2mm) was used. The spray gun was mounted on ABB-400 robot arm so that the spray process could be controlled precisely and this spray operation was performed by a Model 9000 TAFA arc spray system.

For free-standing Al alloy thermal coatings, a graphite plate (diameter 25cm and width 2.5cm) was preheated at 120 ° C and placed on experimental setup. This graphite plate was placed on angle iron table, at 4.5 cm underneath the graphite plate; an air-cooling device was mounted that had cooling rate pressure 80 Psi. After one/two pass of spray, the spray torch was off to cool down the coating (half to 2 minutes) to avoid thermal deformation and crack due to residual stresses.

Figure 16. Galvanized disc for pin on disc test without coating

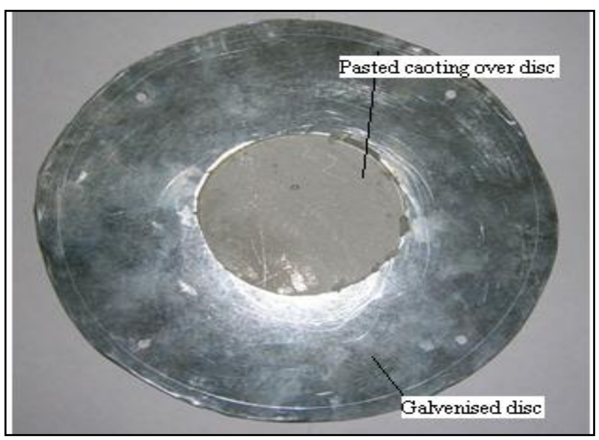

Figure 17. Coating pasted on the galvenised sheet

For wear test the coating was removed from graphite surface and after that, it was pasted on thin sheet of galvanized mild steel having 2 mm thickness and diameter 17 cm. Four holes were also drilled on the sheet so that it could be fastened on the disc of wear and friction machine. The coating thin film was pasted over the thin sheet of galvanized steel sheet (figure 16). Before fastening the coating on the steel sheet, sheet was scratched so that fabrication was strong. The coated was pasted on the sheet with the help of araldite. Squeeze out equal amount by volume, from both the tubes, mix thoroughly and then apply a thin film on both surface, clamp joint overnight. Pasted coating on the galvanized disc as shown in fig. 17.The araldite can joined metal to metal or metal to non metal.

3.2 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 sprayed aluminum coating. There were two parameters which were taken into consideration to determine the wear rate and coefficient of friction. The design of experiment was made on the software design ease 7.1. There are several methods to design the experiment in this software but we have chosen general factorial design because it can be design the experimental variable when there are more than three levels. The both of the two variables have three levels. It was sliding speed (150, 200 and 250 rpm) and loads (29.9, 44.1 and 58.8 N) (Table 2).

Variables	Level 1	Level 2	Level 3
Sliding speed (rpm)	150 rpm	200 rpm	250 rpm
Load (kg)	3 kg	4.5 kg	6 kg

Table 1.Variables for wear test

Table 1 shows the two variables used to determine the response such as wear rate, coefficient of friction, microstructure, EDS analysis, and XRD analysis. The significant variables on which the wear rate and the coefficient of friction depend were directly given by the software with the help of F-test.

Std	Run	Load(N)	Sliding speed (rpm)
7	1	29.4	250
4	2	29.4	200
6	3	58.8	200
2	4	44.1	150
1	5	29.4	150
5	6	44.1	200
3	7	58.8	150
9	8	58.8	250
8	9	44.1	250

Table 2.design of experiment table for wear test

3.3. 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 different materials. 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 18).

Figure 18. Wear and friction monitor machine for pin on disc test

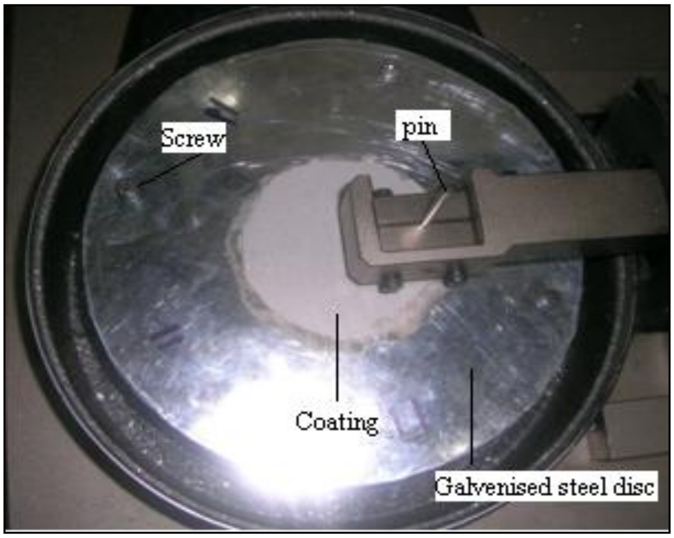

Figure 19. Thermal spray coating before wear test

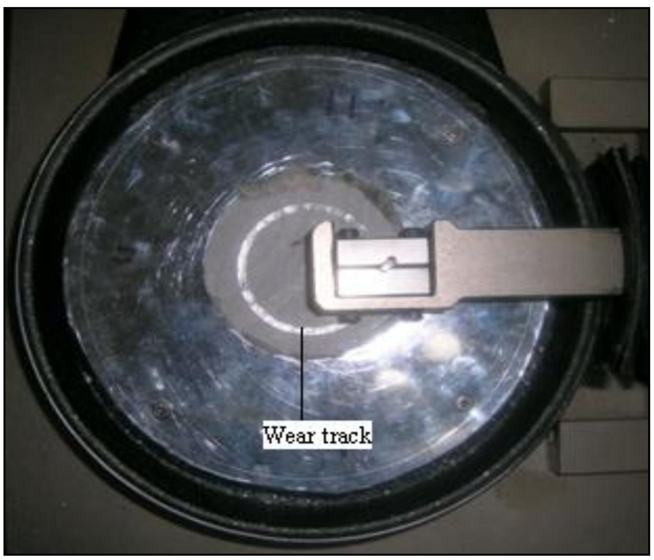

Figure 20. Thermal sprays coating during wear test

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 19). 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 thermal spray coating 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 per 74.4 m sliding distance on an electronic balance of least count 0.00001g. The load was taken as 29.4, 44.1, and 58.8 N respectively and the sliding speed was taken as 150, 200, and 250 rpm. The wear behaviour against three various counter pin material was analyzed that was brass, medium carbon steel and high carbon steel 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 brass 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 20).

3.4. 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 21). 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 21. 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.

3.5. 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 22). 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 22. X-Ray diffractometer in DTU, Delhi

3.6. 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 23).

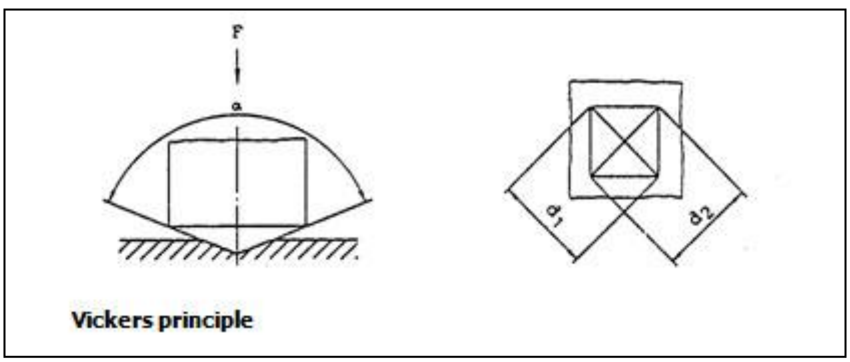

Figure 23. Vickers micro hardness indentation

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.

3.7. 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 24. Optical microscope

The most common type of microscope and the first invented is the optical microscope (figure 24). 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.