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Wear and Corrosion Study of Sputtered Zirconium thin films on SS316L for Windmill Application

Arunkumar N[a], Ragul S[b]*, Harish Kumar S K[b]
[a] Professor and Head of Department, [b] Graduates
Department of Mechanical Engineering, St Joseph's College of Engineering, Chennai-119

ABSTRACT
The Aim of this study is to observe the Wear and Corrosion behavior of Zirconium coated 316L stainless steel. After polishing, SS316L was coated with Zirconium employing DC sputtering process (a technique of physical vapor deposition).Structure characterization techniques including Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD) were utilized to investigate the microstructure and crystallinity of the coating. Salt spray test was performed by spraying Sodium chloride in order to determine corrosion resistance behavior of the coated sample. Pin on disc wear test was performed by hardened and tempered EN31 steel pin in order to determine and compare the Wear resistance behavior of Coated and uncoated samples. The Objective is to recommend the zirconium coated Stainless steel SS316L can be a choice for Off-shore wind mills where the shafts undergo Wear and corrosion problems.
Keywords – Zirconium coating, Wear and corrosion study, thin film deposition, DC sputtering, SS316L coating

I. Introduction

Corrosion and Wear problems are still of great problem in wide range of Industrial application and products which result in Functional issues and sudden failure of components and systems [1]. These problems frequently occur in Production, manufacturing and processing industries affecting service life of the machineries and components.. Various technologies are used to deposit the appropriate materials in order to resist under specified condition. They are usually distinguished by the method of deposition; thin film deposition and thick film deposition. They further classified into Physical vapor deposition (PVD) and Chemical vapor deposition (CVD). Wet and Dry Coatings produced at atmospheric pressure have a thickness over 30nm, up to several millimeters are used when the functional performance depend on the protective layer thickness. Coating involves electroplating, electro less plating, hot dip galvanizing, vapor deposition, Thermal spray techniques , brazing and sol-gel methods are widely used.

This paper deals wear and corrosion resistance capability of coatings deposited by DC magnetron sputtering (a technique of physical vapor deposition comes under the thin film deposition). Sputter deposition is defined as the "a Physical vapor deposition(PVD) method of thin film deposition involves ejecting material from 'target' that is a source into a 'substrate' by the some form of energy (Direct current, plasma, Chemical Reaction etc)".

Zirconium is selected as a coating material because of its property of resistance against acids and Salt water. Aim of this study is to recommend the materials to off-shore windmill applications.

II. Coating preparation

Stainless steel SS316L samples were cut in the shape of plates of 40mmx40mm surface area from a sheet of 3mm thickness (for the structural characterization and Corrosion test) and another sample of disc shape is machined in the shape of 65mm diameter 10mm thickness and 6mm through hole at center (for pin on disc wear test) in fig 1.

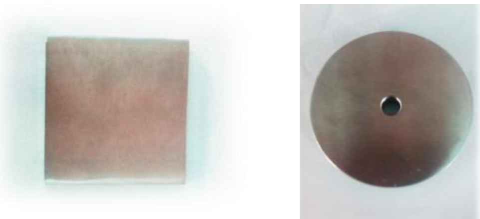


Figure 1: Substrate

Samples were subjected to mechanical polishing with a sand papers of 120 to 600 grid papers, cleaned in acetone and then dried under air pressure.

III. Experimental

Zirconium thin films were deposited by means of Direct current magnetron Sputtering system with a circular sputtering target of 76mm diameter and 1mm thickness. The target to substrate distance was 10cm. A continuously variable DC power supply of 150 W was used as a source of sputtering. The base pressure was 5×10^{-5} mbar which changed to 3×10^{-3} mbar during deposition of Zr. The Purity of the Argon gas in this work was 99.998% and controlled by a mass flow controller. Substrate for the sputtering is maintained at 250 degree Celsius. Finally, the furnace was cooled down to ambient temperature [2].

Figure 2: SS316L Plate after zirconium coating(cut into four pieces by wirecut EDM)

The Sputtered SS316L is characterized by Scanning Electron microscope (SEM), X-ray Diffraction, Pin on Disc Wear test and Salt spray corrosion test were discussed below

IV. Result and Discussion

4.1 SEM and XRD

Scanning electron microscope (SEM) is a type of microscope that produces images of a sample by scanning it with a focused beam of electrons [3]. The SEM images of the coated plate as shown in figure 3 reveals that plate consist of large number of Zirconium particles size of 71.87nm, 86.19 nm, 8062nm, 86.76nm, 77.93nm and so on. The SEM images are taken at the magnification of 25.0kx, 10.0kx and 999x.The micrographs reveal for the coated plate sticking with zirconium particles.in the figure 3,4 and 5, White dots are zirconium particle and black background is the SS316L.

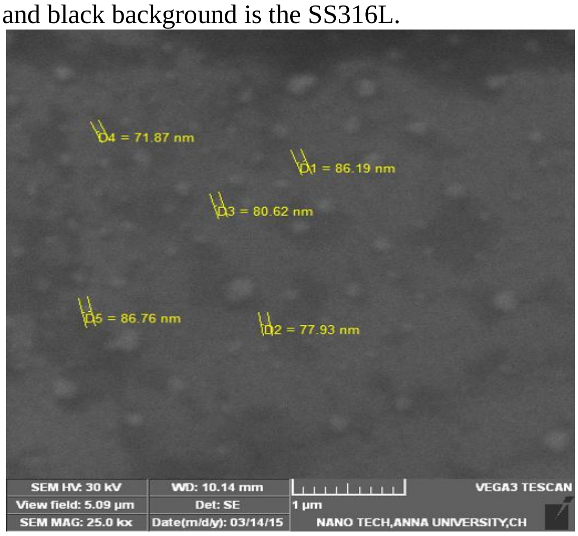


Figure 3: SEM Image at 25.0 kx

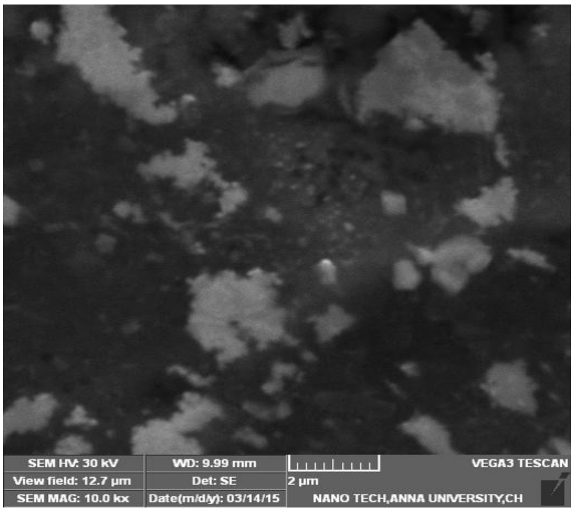


Figure 4: SEM Image at 10.0 kx

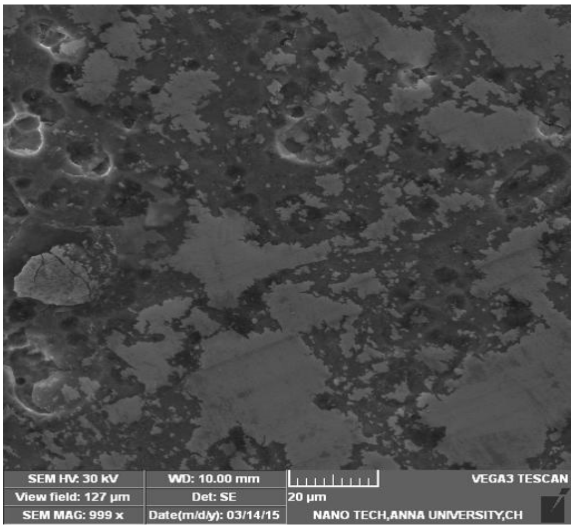


Figure 5: SEM Image at 999 x

Figure 6 shows XRD results for the Zirconium coated stainless steel substrate which are synthesized under cu-kα [4]. The diffraction peaks for the coated substrate can be attributed to the cubic phase of Fe, Cr, Si and S. The growth of Zr is observed in addition to the substrate phase of iron, Chromium, Silicon and Sulphur. The XRD results prove the product composition of zirconium shown in Table 1.

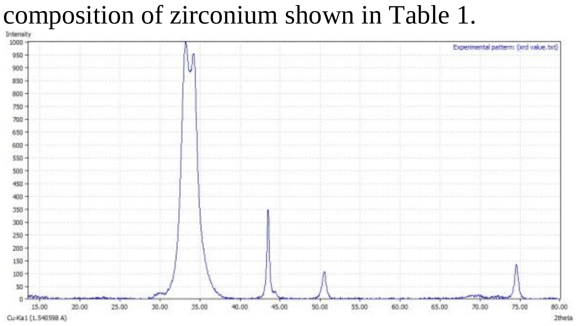


Figure 6: XRD graph shows the coated plate composition

ELEMENT	ANGLE(THETA)
Chromium(Cr)	43.52
Sulphur(S)	74.8
Silicon(Si)	37.10
Zirconium(Zr)	51.23

Table 1: XRD Element details

4.2. Pin on disc wear test

A computational approach used to study the sliding wear caused by loaded spherical pin on contacting a rotating disc widely used in tribological condition called pin on disc test shown in fig 9 [6]. This test gives understanding that, when the loaded pin contacts and slide on the rotating disc rotates at 637 RPM causes the strain growing region at the center of wear track. It is found that mass of the disc before and after coating for found to be 188.89 and 188.10 grams and for the uncoated disc is found to be 188.89 and 188.10 grams .The coated and uncoated disc is shown in fig 7.

Test parameters are

1.Load
2.Speed
3.Distance
4.Temperature
5.Atmosphere[6]

Wear rate of disc and Disc volume loss is calculated from the formula given below

$$\text{Disc volume loss} = \frac{\pi(\text{wear track radius})(\text{track width})^3}{6(\text{Sphere radius , mm})}$$

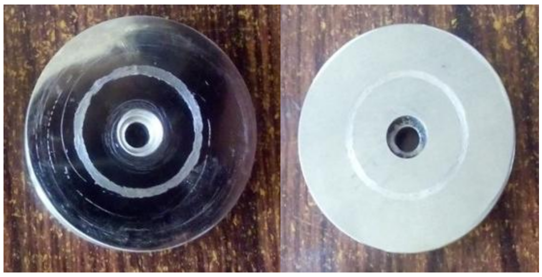
$$\text{Wear rate} = \frac{(\text{M.D.B.C} - \text{M.D.A.C})}{\text{Density X Load X Sliding distance}}$$

Where,
M.D.B.C- Mass of the Disc Before Coating
M.D.A.C- Mass of the Disc After Coating
Data obtained are:

Load	=	5N
Wear track radius	=	15mm
Track width(uncoated)	=	2mm
Track width(coated)	=	3mm
Sphere radius	=	5mm
Density of stainless steel	=	8000 kg/m^3
Sliding velocity	=	πDN/60
Density of stainless steel	=	8000 kg/m^3
Sliding velocity = πDN/60	=	(π x 0.055 x 637)/60
	=	1.83 m/s

Sliding distance = velocity x time = 1.83 x 8 x 60
Total sliding distance = 888 m

From the calculation, It is found that Disc volume loss of coated disc 42.42mm^3 is more than that of uncoated disc 12.56mm^3 and also wear rate of coated disc (2.26×10^{-11} m^3/Nm) is more than that of uncoated disc (5.44×10^{-12} m^3/Nm). Figure shows that the coated and uncoated specimen and their wear track



(a) Coated disc (b) uncoated disc
Figure 7: Wear test specimen

EN-31 pin (shown in figure8) is used to contact the rotational disc under the load of 5N. EN 31 pin is hardened at the temperature of 840 degree Celsius and tempered at 250'c after oil quenching.

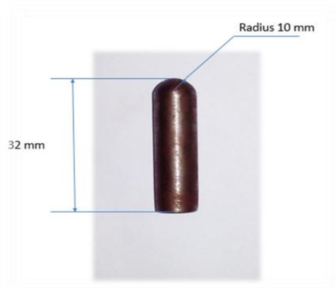
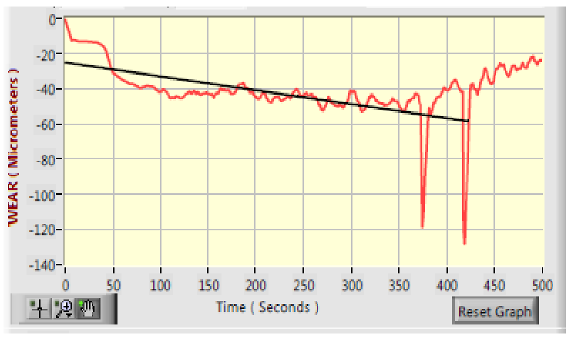
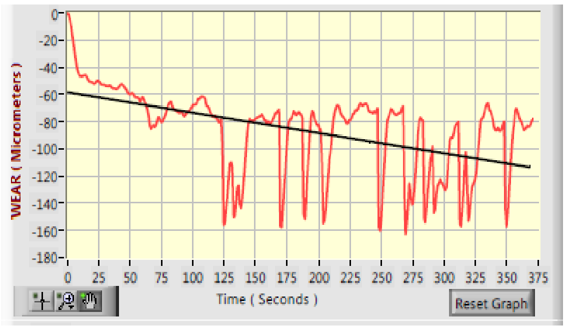


Figure 8: Hardened and Tempered EN-31 Pin

Figure 9: Pin on disc test Apparatus



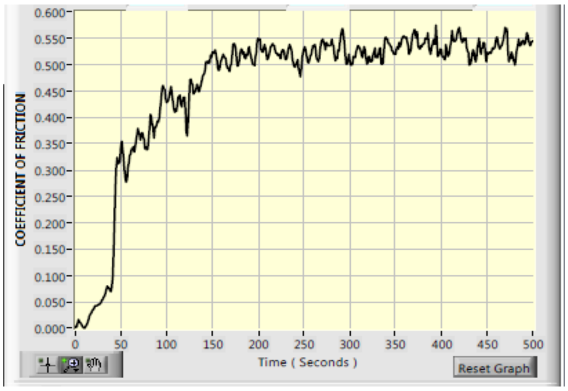
(a)Uncoated wear



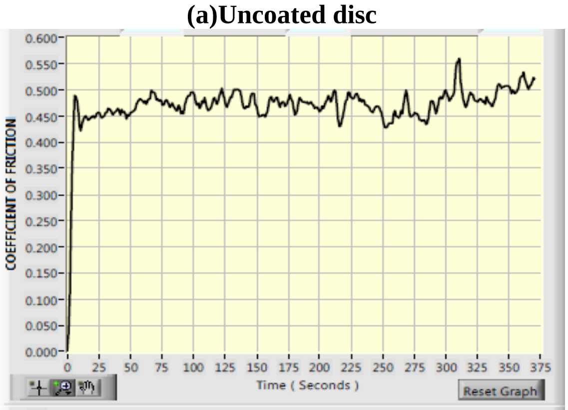
(b)Coated Wear

Figure 10 : Comparison of Wear in micrometers

Fig 10 indicate that the uncoated specimen gives the uniform wear for first 350 seconds but coated specimen gives the abnormal fluctuated wear even at 125 seconds



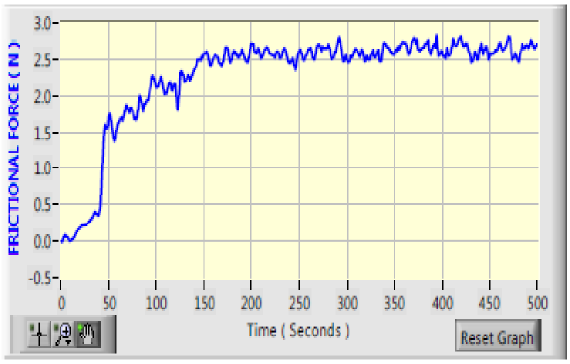
(a)Uncoated disc



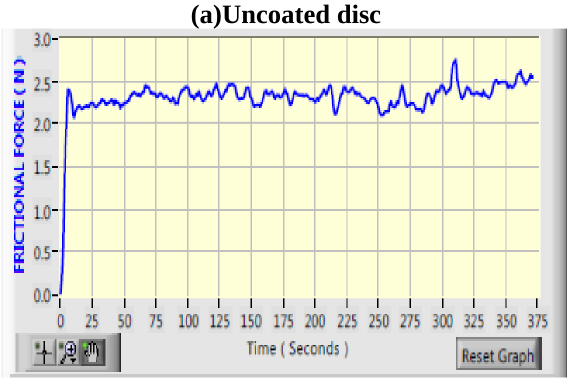
(b)Uncoated disc

Figure 11: Comparison of Coefficient of friction

Fig 11 indicates that the coated disc starts at the coefficient of friction of 0.5 more than that of the uncoated disc 0.3**.**



(a)Uncoated disc



(b)Coated disc

Figure 12: Comparison of Frictional force in Newton

In Fig 12, for the first 100 seconds, Frictional force for coated disc is more than that of uncoated disc after that it is maintained at 2.5 N.

4.3. Salt spray corrosion test

Corrosion is defined as the wearing of surface by chemical action. It is usually slow and unnoticed phenomenon [7,8]. Salt spray test is a standardized method for testing corrosion resistance property of coated plate. Stainless steel 316L can give white rust (starting of corrosion) in 96 hours but the result indicate that Zirconium coated SS316L gives no sign of corrosion up to 144 hours as shown in Table 2. pH solution, concentration of sodium chloride, Air pressure, collection of solution per hour and chamber temperature are the parameters taken from the coated plate.

Parameters	Requirement	Result
pH solution	6.5 to 7.2	6.73 – 6.98
Air pressure	12 to 18 psi	12 to 16 psi
Concentration of sodium chloride	5%+/-1	5.2 to 5.5 %
Collection of solution per hour	1-2 ml	1.2 – 1.8 ml
Chamber temperature	35+/-2'C	34.6 – 35.7 'C

Table 2: Corrosion parameters

V. Conclusion

Wear and corrosion study of Zirconium coated stainless steel 316L grade is done. Coating is done by DC magnetron sputtering. Final result shows that the Zirconium coating successfully resists the corrosion against chemical spraying for the period of 144 hours which is more than that of the stainless steel corrosion occurrence period. But, it does not resist the wear at a load of 5N which implies that wear rate and volume loss more than the uncoated disc.

Acknowledgement

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