

# QUANTIFICATION OF FRETTING DAMAGE OF SELF MATED SS316L AND CHROMIUM CARBIDE COATED SURFACES UNDER CONTROLLED ENVIRONMENT CONDITIONS

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## ABSTRACT

Fretting is a surface-degradation process due to mechanical and chemical attack by small-amplitude oscillatory movement between two contacting surfaces and it is intimately related to wear, corrosion and fatigue. Stainless steel(SS316L) are often used in nuclear industry, especially in sodium-cooled nuclear power plants, because of their excellent mechanical properties under high temperature and irradiation environment, but are characterized as having relatively poor wear and galling resistance. For fast breeder reactors sodium provides a relatively benign environment for most structural materials. Sodium is a highly reactive element that tends to strip the oxide films from most metal surfaces, leaving them in ultra clean condition. This promotes adhesive wear, high friction and self-welding tendencies that are similar to those observed in a high vacuum. As part of the present study, experimental investigations were carried out for displacement amplitude varying from 50microns to 200microns with a normal load of 70N under ambient and high vacuum conditions ( $10^{-05}$ mbar) . Self-mated stainless steel (SS-316L), Chromium carbide with 25% Nickel chrome binder coatings using plasma spray and High velocity oxy-fuel (HVOF) processes on SS316-L were used for the studies. Mechanical responses in the form of variation of coefficient of friction (COF) with number of cycles and displacement amplitude were analyzed. Qualitative evaluation of damage has been carried using Scanning Electron Microscope (SEM) and quantified based on variation of wear volume with displacement amplitude. Chromium carbide with 25% Nickel a chrome binder coating using HVOF processes on SS316-L showed less fretting damage under vacuum condition and is expected to be an effective solution against fretting damage.

## INTRODUCTION

Fretting occurs whenever a small-amplitude oscillatory movement between two contacting surfaces is sustained for a large number of cycles. There are several parameters that control fretting process, among them the important mechanical variables are the normal load and slip amplitude [1]. These two parameters characterize the degradation that occurs either in the form of cracking or wear. Cracking is mainly encountered in pre-sliding condition or generally referred as partial slip condition, while wear is favored under gross sliding condition.

Material and environment conditions also play an important role in degradation mechanism. Stainless steel(SS316L) are often used in nuclear industry, especially in sodium-cooled nuclear power plants because of their excellent mechanical properties under high temperature and irradiation environment, but are characterized as having relatively poor wear and galling resistance. For fast breeder reactors sodium provides a relatively benign environment for most structural materials. However, from a tribological (wear and friction) viewpoint, sodium presents a challenging environment. Sodium is a highly reactive element that tends to strip the oxide films from most metal surfaces, leaving them in ultra clean condition. This promotes adhesive wear, high friction and self-welding tendencies that are similar to those observed in a high vacuum.

The reactor core, coolant pipes and heat exchangers can, apart from sodium pump, be regarded as relatively rigid structures in sodium cooled nuclear power plant. However, the structure consists on many tens of thousands of different components which in operation move relative to each other owing to differential thermal expansion or flow-induced vibration or during loading and unloading events [2].

Various studies show that coatings based on chromium carbide, nickel aluminide or Tribaloy 700 (a nickel-base hard facing alloy) and processes employed include detonation gun coating, diffusion coating, electro spark deposition coating and electroplating give the best performance as tribological coatings in the breeder reactor environments [3]. Mostly coatings are qualified based on the performance criteria for friction coefficients, wear rates, galling resistance and self-welding resistance in liquid sodium. But these coatings and processes are still not evaluated under fretting conditions.

Experiment results of Farwick & Johnson [3] show a great reduction of self-welding tendency of SS-316 when coated with Chromium carbide plus with 15% Nickel chrome binder even at very high temperature. Their results show that coating on any one of the wear surface to be as effective as coating both surfaces. The choices of the coating process are limited such that the substrate must be maintained in a particular metallurgical condition by limiting the substrate temperature and can be used for reactor components that are required to be maintained in 20% cold-worked condition.

Plasma spray process is one of the most sophisticated and versatile thermal spray methods, where temperature of the plasma flame is very high e.g. up to 30,000°C and can melt any material including ceramics. The process also provides a controlled atmosphere for melting and transport of the coating material, thus minimizing oxidation, and the high gas velocities produce coatings of high density. HVOF spray process also known as D-gun process represents the state of the art for thermal spray coatings. The HVOF process uses extremely high kinetic energy and controlled thermal energy output to produce very-low-porosity coatings that exhibit high bond strength, fine as-sprayed surface finish and low residual stresses [4]. In the present study one of the surfaces (flat surface) is coated with Chromium carbide plus 25% Nickel chrome binder using plasma spray and High Velocity Oxy-Fuel (HVOF) processes.

As testing under liquid sodium conditions is rather tedious, a first step in understanding the behavior of tribological pairs is to evaluate them under vacuum conditions. The results are then compared to that under ambient conditions to evaluate the effect of reduction of oxygen, as is the case under liquid sodium, on the tribological response of the mated pairs. The present work is focused to evaluate the degradation due to fretting for self-mated SS316L and chromium carbide coated surfaces under ambient and vacuum conditions. Also, the investigations are carried to evaluate the performance of the coating using different coating processes i.e. plasma spray process and High velocity oxy-fuel (HVOF) process under high vacuum condition.

## EXPERIMENT DETAILS

### Material Specifications

Experimental studies have been carried out on Stainless steel (SS-316L) and coated surfaces with Chromium carbide (with 25% Nickel chrome binder) using plasma spray and High velocity oxy-fuel (HVOF) processes. The chemical composition of SS-316L used in the present study is given in table-1. Modified marble's reagent (20g CuSO<sub>4</sub>, 50ml H<sub>2</sub>SO<sub>4</sub>, 100ml HCl and 100ml water) has been used as an etching reagent. The average grain size of the material is 10microns, measured using average intercept method. Samples of SS-316L are tempered for 3hours at 300°C to relieve the residual surface stresses due to machining process.

Average velocity of 800m/sec and average surface roughness of 6-7 microns has been obtained for plasma spray coated surfaces while average velocity of 1200m/sec and average surface roughness (Ra) of 3-4microns has been obtained for HVOF coated surfaces. The average thickness of the coating achieved is 100microns.

Table -1: Chemical composition (weight %) of SS316L.

ELEMENTS	Fe	Cr	Ni	Mo	Mn	C	Ti	Si	P	S	Co	Nb	Ti	V	W
SS316L	67.12	17.15	10.23	2.04	1.75	.03	.01	0.44	0.043	0.03	0.1	0.04	0.01	0.05	0.05

Table-2: Mechanical properties

MATERIAL	YOUNG MODULUS(GPa)	VICKERS HARDNESS
SS316L	210	284
Chromium Carbide coatings using plasma spray process	229	573.9
Chromium Carbide coatings using HVOF spray process	293	1099.73

Hardness and Young's modulus of the coatings were measured using micro-hardness tester (CSM instruments version 4.12) under normal load of 5N and are given in table-2. Before the indentation, surfaces were polished and average surface roughness (Ra) of the coated surface is maintained less than 0.2microns. Plasma coated surface and HVOF coated surface shows a depth of indentation of 7.2microns and 4.5 microns, respectively under normal load of 5N.

### Specimen design

The configuration of contact taken for the experiments is spherical contact due to ease of alignment and

other advantages associated with analysis of initiation process of cracks/damage, specifically under fretting conditions [5]. The details of the specimen for pin radius of  $R=15\text{mm}$  and flat specimen are shown in figure-1. After fabrication, polishing of the specimens has been carried out using emery paper of grit size 200, 400, 800, 1000 and 1200. The specimens are diamond polished using diamond paste of 0.1microns to achieve the required roughness. The average roughness ( $R_a$ ), measured using 3D optical Profilometer WYKO NT1100, of flat specimen is less than 0.02 microns for uncoated while for coated specimens it is less than 0.2 microns. For all the experiments the average roughness of the pin is maintained less than 0.15microns. Six experiments are performed on each flat specimen with different pins at various locations.

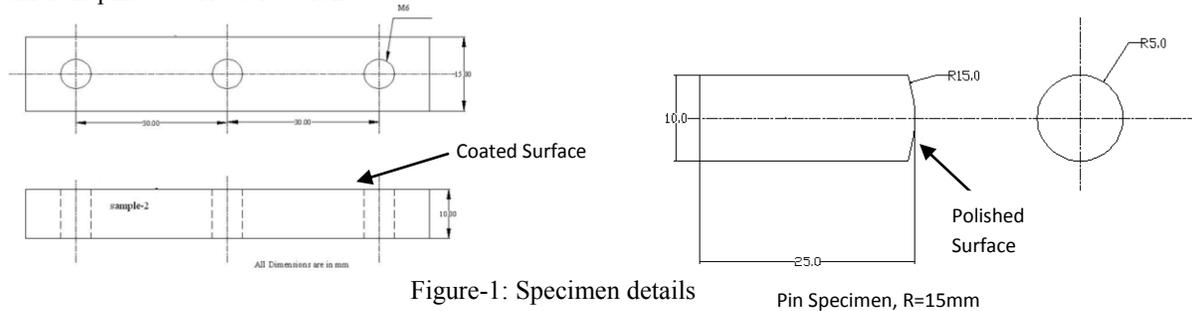


Figure-1: Specimen details

Pin Specimen,  $R=15\text{mm}$ 

### Experimental setup

A fretting wear machine has been designed and developed to carry out experiments under ambient as well as high vacuum conditions. The challenges with the fretting test rig designs are associated with the low relative displacement amplitude between the contact surfaces under constant normal load.

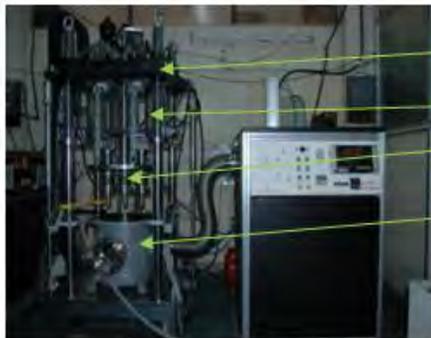


Figure-2: Experimental set-up

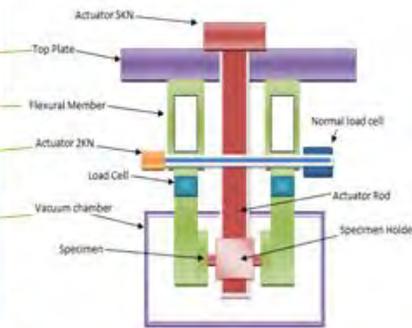


Figure-3: Schematic of experimental set-up

A 5kN double ended double acting actuator with a rated pressure of 210 bars and total stroke of 25 mm has been used for the cyclic motion to achieve amplitude from 5microns to 200microns. The control of hydraulic actuator has been made through digital encoder with a resolution of 0.1microns. Pin specimens are mounted on the actuator rod by a specimen holder while the flat specimen is mounted on the flexible rod assembly which contains flexural member. The flexural member provides low bending stiffness (100N/mm) and high axial stiffness ( $10^6\text{N/mm}$ ). The normal load is applied through the side double ended double acting actuator of 2kN and is reacted by a normal load cell mounted at specimens location. The arrangement is made in such a way that a constant normal load at contact location can be achieved. But due to offset in the contact interface and the centre line of the flexural member a variation of 5% in normal load is observed. Further, the normal load at contact location is calibrated using load cell mounted at the specimen location, shows about 95% of normal load is transferred through the contact. The correction factor for this are accounted in the experiments. Experimental set up and details of the fixture mounted on a servo-hydraulic load frame are shown in figure-2 and schematically in figure-3. Experiments were conducted under displacement controlled conditions and corresponding shear force is measured.

### Test Details

Experiments have been conducted on self-mated SS316L and chromium coated flat surface mated with SS316L pin. For self mated SS316L, under normal load of 70N, the area of the contact and the depth of indentation

have been measured using 3D optical Profilometer and responses are shown in the figure-4. Profilometer responses show the depth of indent of 0.63microns and contact radius of 177microns. The indentation profiles show negligible pile-up of the material at the periphery of the indented area.

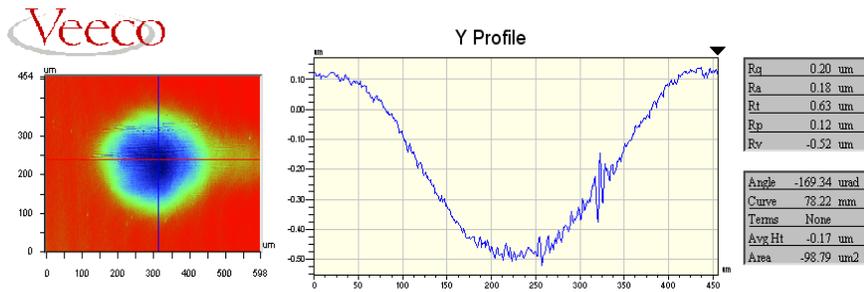


Figure-4: Profilometer responses for self mated SS316L for normal load=70N

For coated surfaces several attempts were made to find the area of contact and depth of indentation even for high normal load up to 600N, but no indents were observed except for some variations of the roughness caused due to microslip is as expected for dissimilar bodies.

Experiments under ambient condition were performed at room temperature of 25-28°C and relative humidity of 40-45%, whereas for vacuum environment a vacuum of 10<sup>-5</sup>mbar was achieved through rotary and diffusion pump. All the experiments were conducted at frequency of 4Hz and for 10,000cycles.

Qualitative evaluation of damage has been carried using Scanning Electron Microscope with EDS (Quanta 200, FEI). 3D optical Profilometer has been used to quantify the scar.

**RESULTS**

**SS Vs SS under Ambient Condition**

Coefficient of friction (COF) is an important parameter to characterize the interfacial contact conditions. Figure-5show the variations of coefficient of friction with number of cycles for SS Vs SS under ambient conditions for normal load of 70N. The steady state value of coefficient of friction is 0.75 and is found independent of the displacement amplitude.

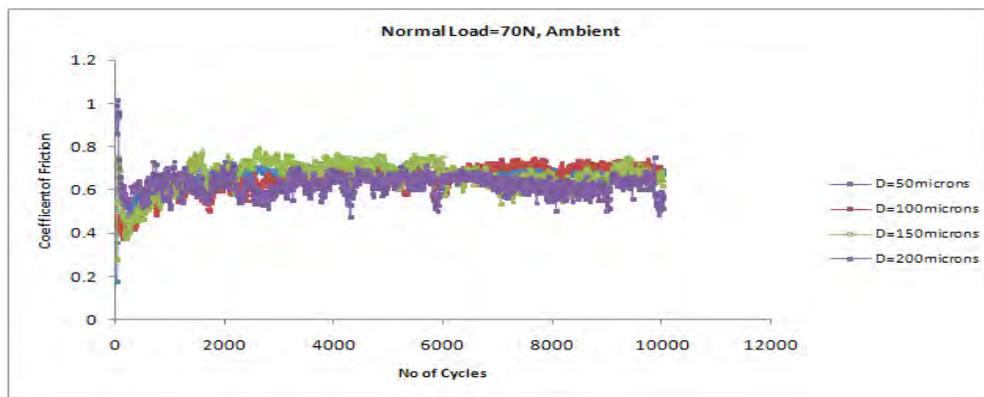


Figure-5: Variation of coefficient of friction with number of cycles for normal load of 70N under ambient conditions.

SEM micrographs of scar on flat surface and pin surface, for normal load of 70N and displacement amplitude of 50microns under ambient condition, are shown in Figure-6a&6b, respectively. Material transfer in the form of platelets from pin surface to the flat surface is quite evident from the micrograph. This form of surface degradation has been characterized under wear rather than galling, where material flows from the surfaces forming macroscopic excrecence [6].

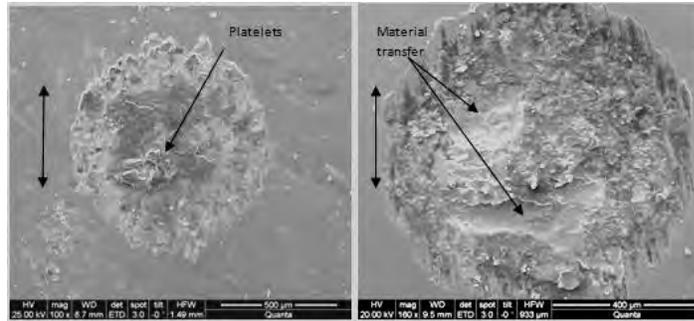


Figure-6: SEM micrographs of self mated SS316L for normal load of 70N and displacement amplitude of 50microns under ambient condition for (a) Flat (b) Pin surfaces. (Note- Arrow indicates fretting direction)

As suggested by Waterhouse [1] the sudden increase in friction is mainly due to strong adhesion immediately after mechanical action disrupts oxide films on the surface exposing clean surfaces. The exposed surface is clean and very reactive and in the presence of the atmosphere would oxidize rapidly after the disruption. The process of disruption, formation and re-disruption results in the formation of the wear debris of oxide and forms an oxide interface between the contacting bodies, resulting in the fall of frictional forces under ambient condition. Depending on the formation and rejection of wear debris, an equilibrium state is achieved and leads to a steady state condition. Further, it has been observed that at lower displacement amplitude strong adhesion prevails at the contact interface which leads to material transfer from pin to flat surface.

#### SS Vs SS under Vacuum Condition

Figure-7 shows variation in COF with number of cycles for SS Vs SS under vacuum condition for normal load of 70N. The steady state value of coefficient of friction is 1.2, shows slight variation with displacement amplitude.

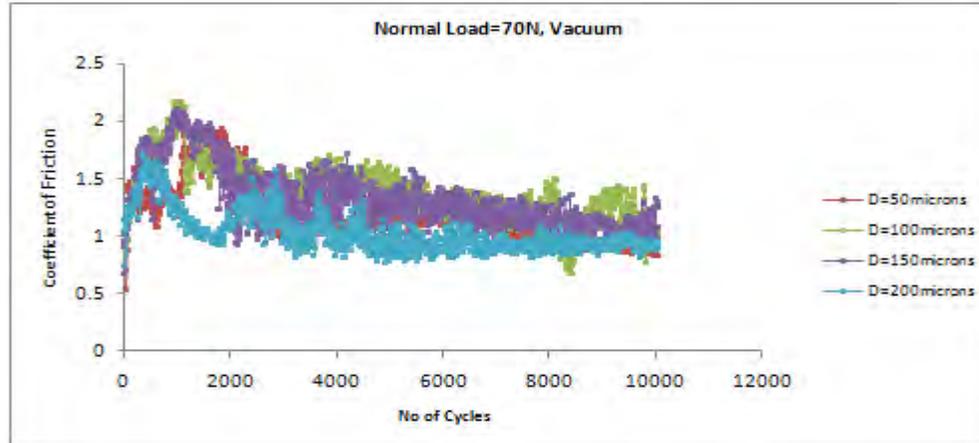


Figure-7: Variation of coefficient of friction with number of cycles for normal load of 70N under vacuum conditions.

SEM micrograph shown in Figure-8a shows surface damage on SS316L flat surface for normal load of 70N under vacuum conditions for displacements amplitude of 100microns. There is a transfer of material from pin to flat surface which can be seen from micrograph and can be further verified from the scar profile shown in figure-8b.

As discussed earlier, the sudden increase in COF is mainly due to strong adhesion, at contact interface, immediately after mechanical action disrupts oxide films on the surface exposing clean surfaces. Under vacuum environment, once the initial oxide layer is disrupted, there is no formation of the oxide layer and the metal surface remains reactive and results in high adhesion. This results in higher COF and severity of damage under vacuum condition as compared under ambient condition.

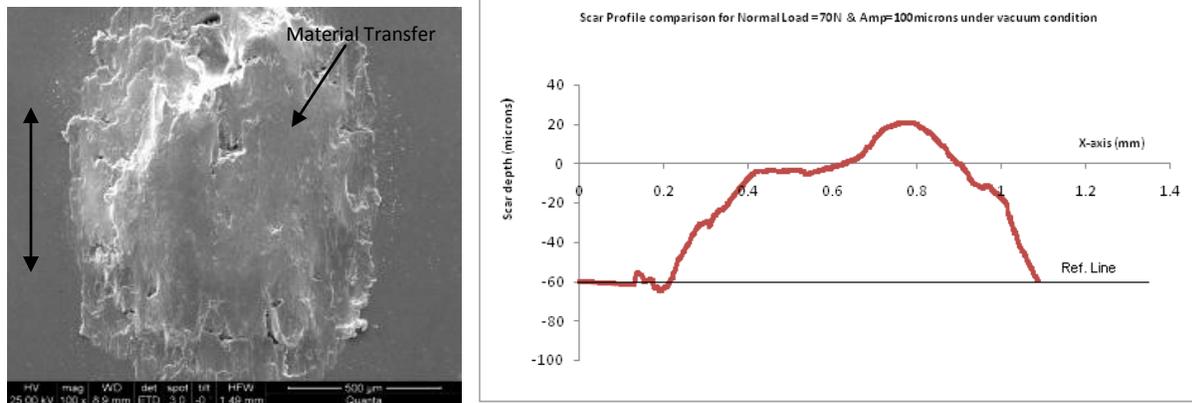


Figure-8: (a) SEM micrographs (b) Scar profile of self mated SS316L for normal load of 70N under vacuum condition for displacement amplitude of 100microns (Note- Arrow indicates fretting direction)

**Chromium carbide coated surfaces under Vacuum condition**

As can be observed, SS Vs SS shows severe damage under vacuum environment as compared under ambient conditions. Thus the performance of the coated surfaces has been evaluated under vacuum environment. Variation in coefficient of friction with number of cycles for chromium carbide coated surfaces using plasma spray and HVOF spray processes are shown in figure-9&10. The steady state value of coefficient of friction is 0.6 and 0.5 for plasma coated surface and HVOF coated surface, respectively. Slight variation in COF with displacement amplitude has been observed.

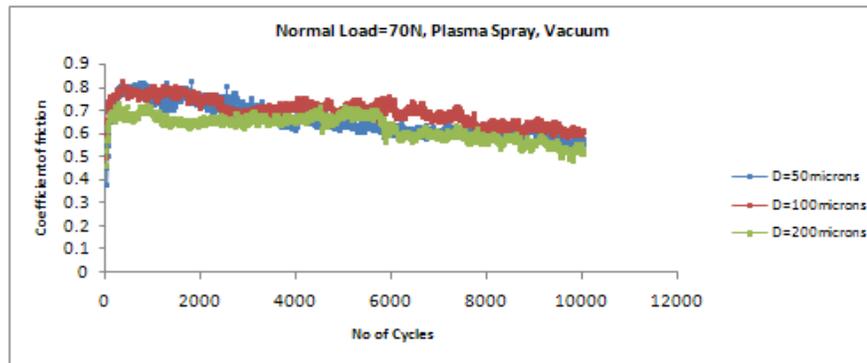


Figure-9: Variation of coefficient of friction with number of cycles under vacuum conditions for chromium carbide coated surfaces using plasma spray process.

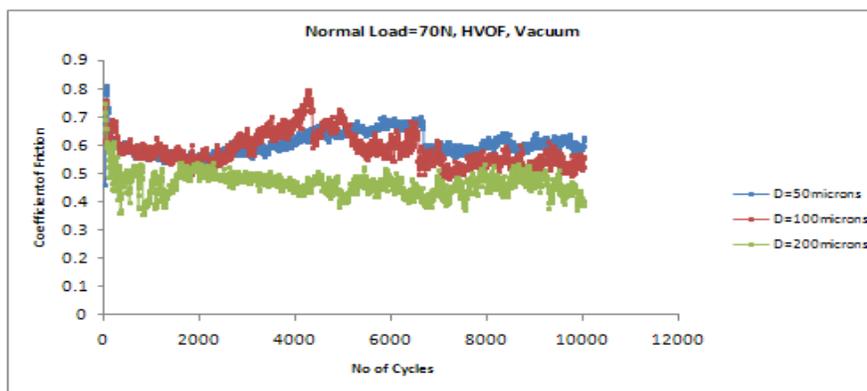


Figure-10: Variation of coefficient of friction with number of cycles under vacuum conditions for chromium carbide coated surfaces using HVOF process.

Figure-11a&b show SEM micrographs of damage on chromium carbide coated flat surface using plasma spray & HVOF process, respectively, for normal load of 70N & displacement amplitude of 100microns under vacuum condition. Plasma spray coated surface shows brittle fractures in the annular region of the contact interface and undamaged surface at the centre location with a resemblance of damage seen under partial slip condition [7]. In contrast, HVOF coated surface shows surface features originated due to gross sliding at contact interface.

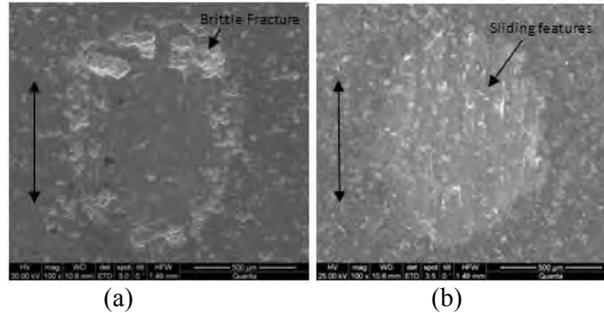


Figure-11: SEM micrographs of chromium carbide coated SS316L with normal load of 70N under vacuum condition for displacement amplitude of 100microns for (a) Plasma Coated surface (b) HVOF coated surface (Note-Arrow indicates fretting direction)

**QUANTIFICATION OF DAMAGE**

**Wear volume variation**

The wear volume has been evaluated using responses from 3D optical Profilometer in accordance with ASTM G99-05 (2010) [8]. Dissipated energy model [9] has been used to quantify fretting wear damage. According to dissipated energy model, wear volume is assumed to be proportional to dissipated energy [10] and is given by,

$$\text{Wear volume (V)} = \alpha (\Sigma E_d) \dots\dots\dots(1)$$

where,  $\alpha$  is called as energy wear factor,  $E_d$  is the dissipated energy at contact interface and  $\Sigma E_d$  gives the total energy dissipated at contact interface. Dissipated energy is given by,

$$E_d = \oint F(t) dx(t) \dots\dots\dots(2)$$

The variation of wear volume with energy dissipated for normal load of 70N under ambient condition is shown in figure-12a. Best fit line with coefficient of determination,  $R^2=0.95$  gives the energy wear factor for self mated SS316L as  $8e-03 \text{ mm}^3/\text{J}$ .

A comparison of variation of wear volume with total energy dissipated for self mated SS316L, Plasma coated surfaces and HVOF coated surfaces are shown in figure-12b. The energy wear factor for self mated SS316L, plasma coated surfaces and HVOF coated surfaces are  $5.47e-04 \text{ mm}^3/\text{J}$ ,  $3.21e-04 \text{ mm}^3/\text{J}$  and  $2.13e-04 \text{ mm}^3/\text{J}$ , respectively, obtained from the best fit line equation with coefficient of determination,  $R^2=0.95$ .

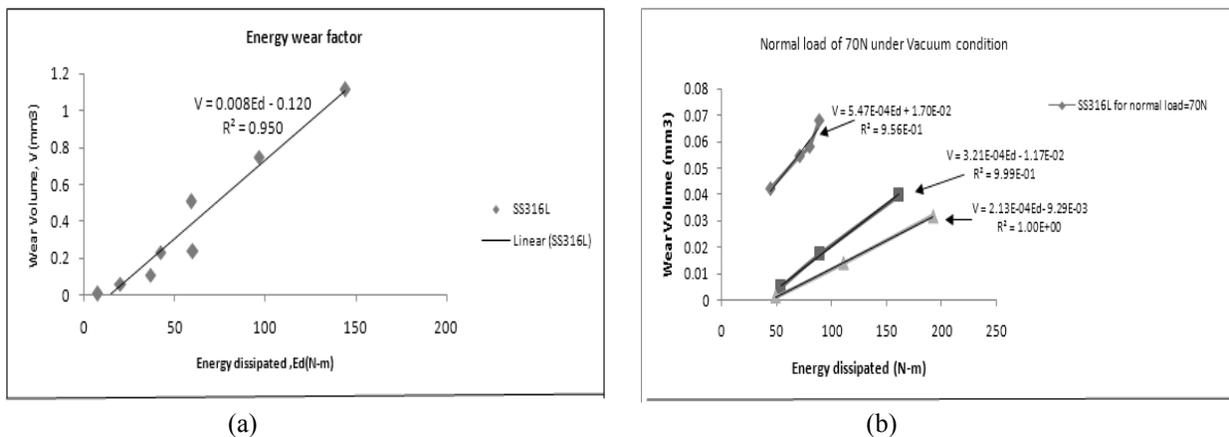


Figure-12: Wear volume variation with total energy dissipated (a) under ambient condition (b) under vacuum condition

Under vacuum condition HVOF coated samples show minimum wear factor as compared to SS Vs SS and plasma coated surfaces. It is due to the occurrence of gross sliding which results in lower surface degradation. Thus, chromium carbide plus 25% Nickel chrome binder coatings using HVOF process shows good fretting damage resistance.

## CONCLUSION

Slip amplitude in fretting is a very significant factor because it is relevant to the definition of fretting. Detailed investigations have been carried out for self-mated SS316L and chromium carbide plus 25% Nickel chrome binder coatings on SS316L under ambient and vacuum conditions for displacement amplitude varying from 50microns to 200microns. SS Vs SS and plasma coated surfaces shows increase in adhesion which further results in the increase in shear force and is responsible for the severity of damage. HVOF coated surfaces shows gross sliding due to lower adhesion at contact interface. This result in decrease of damage severity and the damage mechanism involved is abrasion rather than adhesion. Higher surface hardness of HVOF coated surfaces is believed for the change in damage mechanism.

Under vacuum condition HVOF coated samples show energy wear factor of  $2.13 \times 10^{-4} \text{ mm}^3/\text{J}$  and is lower than obtained for SS Vs SS and plasma coated surfaces. Thus, Chromium carbide plus 25% nickel chrome binder coatings using HVOF process on SS316L are expected to be an effective solution against fretting damage.

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