



CHARACTERISATION OF PEEK COATED SS316 L FOR BIOMEDICAL APPLICATION

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ABSTRACT

In this paper the Corrosion resistance and Micro hardness of the coating are investigated on Polyether ether Ketone (PEEK) coated SS316 L. The coatings of 5 microns thickness have been achieved in plasma spray process where the distance between the target and the substrate has maintained in the range of 2.5 to 4 inch. The uniformity of the coating and surface characteristics of the coating has been examined using Atomic force Microscope (AFM) and Scanning electron microscope (SEM). The salt spray test has been conducted for 320Hrs for Uncoated and PEEK coated SS316 L samples with sodium chloride solution and the P_H of the solution has maintained from 6.74 to 6.87. It is observed that the red rust has been formed in the substrate (SS316 L) after 300Hrs whereas the coated sample remains unchanged beyond 320Hrs. also the hardness of the material comparatively with the stainless steel substrate.

Keywords: corrosion resistance, polyether ether ketone (PEEK), micro hardness, atomic force microscope (AFM), scanning electron microscope (SEM).

Nomenclature

PEEK = Polyether ether Ketone

SS316 L = Stainless steel 316 Low carbon content

SEM = Scanning Electron Microscope

AFM = Atomic Force Microscope

Nm = Nano Meter

P_H = Potential of Hydrogen

INTRODUCTION

Polyether Ether-Ketone is a high performance thermoplastic. Peek is inert to all common organic and inorganic liquids and common solvents [1]. It has an elastic modulus similar to that of bone which makes it the perfect replacement for metal or ceramics for several bone implants in human body [2]. Biomaterials such as ceramics calcium silicate (CS), bioglass have lower elastic modulus than human bone [3]. PEEK polymer is transparent to X-rays and there are no images created in CT scan, since plastics are non-magnetic. MRI technologies can be used with patients that have received a plastic implant [3]. PEEK polymers are obtained by polymerization by the dialkylation of bisphenolate salts.

The reaction is conducted around 300 °C in polar aprotic solvents - such as diphenyl sulphone [4]. Figure-1 is showing about the organic representation of synthesis of PEEK polymer.



Figure-1. Synthesis of PEEK polymer.

The Grade SS 316L the low carbon version of 316 Table-1 shows the chemical composition of SS316 L. Thus it is extensively used in heavy gauge welded components (over about 6mm). Compared to chromium-nickel stainless steels, 316L stainless steel offers higher creep, stress to rupture and tensile strength at elevated temperatures. Stainless steel does not readily corrode, rust or stain with water as ordinary steel does. Unprotected carbon steel rusts readily when exposed to air and moisture [5].

Table-1. Chemical composition of SS316 L.

Grade	Fe	C	Cr	Ni	Mo	Mn	Si	P
%	<0.03	16-18.5%	10-14%	2-3%	<2%	<1%	<0.045%	<0.03%

EXPERIMENTAL WORK

i. Sample preparation

The samples of SS316L of 1X1 cm are being polished by automatic grinding machine which contains the emery sheet, initially to carry out the experiments. A

plain and smooth mirror finished surface is being obtained to carry out the coating process.



Figure-2. Polished SS316 L sample.

ii. Coating

The deposition of PEEK over SS316 L has been achieved through plasma spray process. In general plasma spray coating are most feasible and form rough coating surface which is more suitable for the implants. Because of higher operating temperature of the plasma it is easy to melt the PEEK and forms the coating over the substrate. An arc is formed in between 2 electrodes in a plasma forming gas, which consists of argon/hydrogen. As the plasma gas is heated by the arc, it expands and is accelerated through a shaped nozzle. In this powdered PEEK is being sprayed melted or partially melted on the SS316 L substrate. The process parameters for the coating process are discussed in Table-2.

Table-2. Parameters for plasma machining.

Parameters		Range
Volts		60-70
Powder feed		50-60 gm/min
Amps		500
Spray distance		2.5 - 4 inch
Pressure	Argon	100-120 psi
	Hydrogen	50 psi

iii. Corrosion test

In this, the specimen is sprayed in a salt solution kept in a container as shown in Figure-3 of different pH

values as listed in Table-3 for many hours for variations of result. The chamber temperature is maintained slightly variable according to the specimen and the hours of salt spraying. Initially it was done for 120 hours and no rust formation is being found over their surface. Later it was given for more 310 hours where the formation of rusting is found over the surface of the specimen.

Table-3. Parameters used in salt spray.

S. No.	Parameters	Requirement	Result
1.	pH solution	6.5 to 7.2	6.78-6.84
2.	Air pressure	12 to 18 psi	12 to 16 psi
3.	Concentration of Sodium Chloride	5% +/-1	5.2-5.4%
4.	Chamber temperature	35 +/- 2 C	34.8-35.3 C
5.	Collection of solution per hour	1~2 ml	1.2-1.5ml



Figure-3. Salt spraying chamber.

RESULTS AND DISCUSSIONS

Atomic force microscopy analysis

The AFM analysis has been done to examine the surface roughness and maximum peak of the coating. Figure-4 shows the 2D and 3D images obtained in AFM analysis.

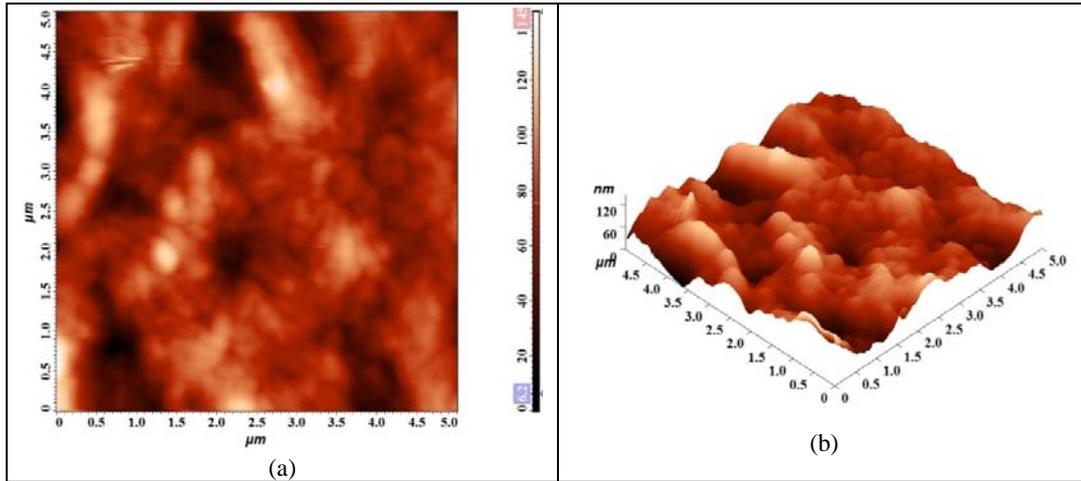


Figure-4. (a) 2-D image of the sample (b) 3-D image of the sample

The white part in the images are elevated part of the surface and the dark part are parts which are the submerged parts. where the elevated part of the AFM analysis showing that maximum peak of the coated material. Also surface roughness has increased slightly due to the coating.

the average roughness of the sample which is found out to be 70.3907 nm. Also the some of other values that has been obtained in AFM analysis are listed in Table-4.

Table-4. Roughness analysis from AFM analysis.

Parameter	Range
Amount of sampling	65536
Max	144.996 nm
Min	0 nm
Peak-to-peak, S_y	144.996 nm
Ten point height, S_z	72.8714 nm
Average	70.3907 nm
Average roughness, S_a	14.8626 nm
Surface skewness, S_{sk}	0.00137283

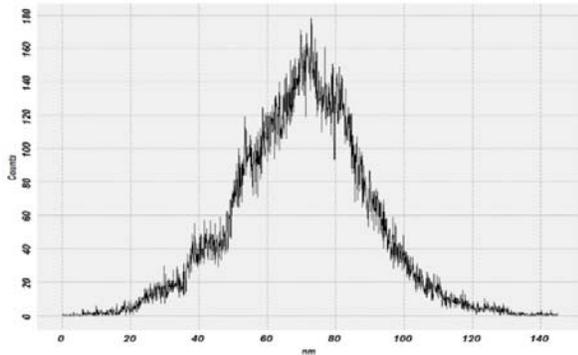


Figure-5. Histogram analysis from AFM.

Figure-5 is showing about the histogram analysis made. As we draw a line in the centre of the curve, we get

Scanning electron microscopy analysis

The SEM analysis has been done and the results are shown in Figure-6.

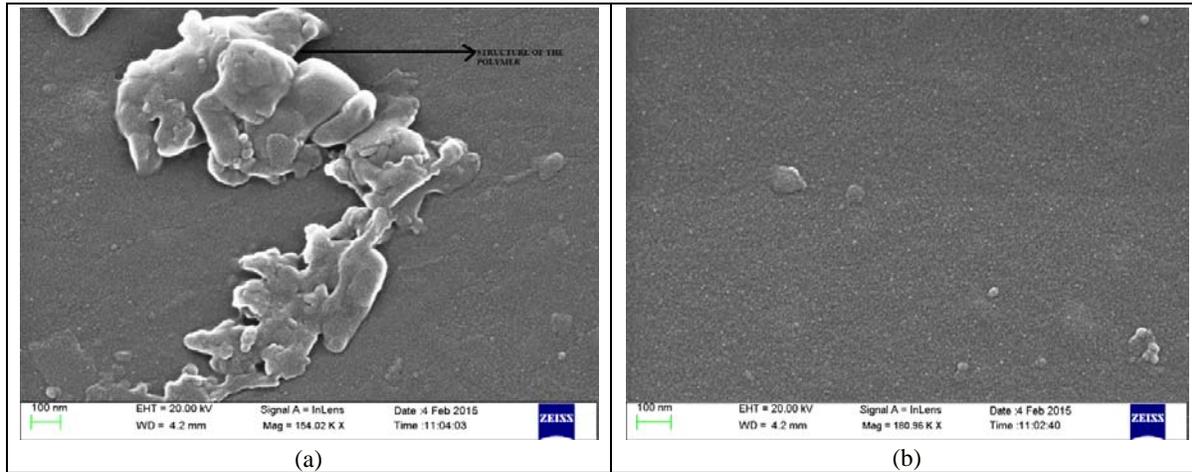


Figure-6.(a)Structure of the polymer (b)Surface of the coated surface.

The plain grey obtained in the SEM image is the surface topography of the PEEK polymer coated on the SS316 L specimen Figure-6(b). It is due the proper polishing of the sample. The structure of the polymer is obtained in the image Figure-6(a).

Micro hardness test

Vickers hardness test is being conducted on the sample. A constant load of 0.025kgf is applied on several places in the work-piece. In vicker's testing 1kgf or less than 1kgf force is applied for the hardness test and the results for the coated and uncoated samples are listed in the Table-5. Due to the coating of the sample where PEEK is a polymer the hardness value has decreased substantially compared to the uncoated (SS316 L) sample.

Table-5.Micro hardness test results.

S. No.	Load	Location	Coated Sample	Uncoated sample
1.	0.025kgf	1	50.0	175.9
2.	0.025kgf	2	63.6	177.0
3.	0.025kgf	3	59.1	174.7
4.	0.025kgf	4	61.6	176.5
5.	0.025kgf	5	52.6	178.1

Corrosion test

The coated sample is then examined for the different pH values of 6.78-6.84 of salt solution. After keeping it in a dark room chamber maintaining the temperature ranging from 34.8-35.3 C, the test is being carried out. The corrosion test conducted on the work-piece shows a little red rust formation after 310 hours.

CONCLUSIONS

With the testing being done on the work-piece, we have found out that hardness of the sample decreases substantially after the coating is being done. Uncoated sample seemed to have more hardness value. The rate of rust formed required a period of 310 hours. Hence the coating provides a slower rate of corrosion in general. PEEK is chemically stable, biocompatible and has a similar elastic modulus to that of human bone suitable for

orthopaedic implant which makes a perfect replacement of ceramics or metal implants.

PEEK is biologically inert, preventing good bonding with surrounding bone tissue after it is implanted. Surface modification helps improve the bioactivity of PEEK.

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