Morphology Evaluation of TiO2 Dip Coating on Mild Steel and Enhancing Its Surface Roughness

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***Abstract* - Now a day, steel has become an important part of our life due to its extensive applications in automotive, household appliances, business machine and heavy construction such as marine and chemical industries. TiO2 nanoparticles are odorless and nontoxic. They possess high hardness, high purity and a high melting point. It is the popular material used in the applications automotive, aerospace, sports equipment, and electronics industries. For this study, Titanium isopropoxide: (C12H28O4Ti) And Ethanol: (C2H6O) 50 ml are used as a starting material for the preparation of TiO2nanoparticles by sol gel process and size was found to be 55 nm from X-ray diffraction. Plate samples of 10x15x2 mm is the substrates were fully immersed in titanium tetrachloride solution and air dried for another 2 minutes. These steps were repeated according to the number of coatings of each substrate. The substrate then annealed at 500oC for two hours in the muffle furnace. Shows better surface roughness on coated samples at 500˚C. Coated samples are characterized by XRD, SEM and Surface Roughness.**

***keywords* - TiO2, Surface Roughness, XRD, SEM and EDX**

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1. Introduction

The interest has been stimulated by the large variety of applications in industries such as fabrication of dense ceramics, sensors, batteries, capacitors, corrosion-resistant coatings, thermal barrier coatings, solidelectrolytes for fuel cells, catalysts, cosmetics, health, automotive, bioengineering, optoelectronics, computers, and electronics etc [1].Nanomaterials exhibit unique physical and chemical properties and impart enhancements to engineered materials. There including better magnetic properties, improved mechanical activity and increased optical properties. Developments are being made to improve the properties of the materials and to find alternative precursors that can give desirable properties on the materials [2]. In this investigation a uniform TiO_2 nanoparticle coating has been applied on mild steel, using cold spray method [3]. The coating was deposited on mild steel substrate by dip coating technique [5]. The morphology and structure of the coating were analyzed using SEM, EDX and X-ray diffraction. The anticorrosion performances of the coating have been evaluated by using electrochemical techniques. Surface Properties are also evaluated by surface roughness tester [6].

2. Experimental Details

2.1 The Various Materials used in this Experimentation are as follows

- TiO_2 nanoparticles
- Mild steel plate (10x15x2 mm)
- Titanium isopropoxide($C_{12}H_{28}O_4Ti$)
- Ethanol (C_2H_6O) 50 ml
- H_2O 200 ml

2.2 Synthesis of TiO_2 Nanoparticles Using Sol Gel Process

Sol-Gel Process: It is one of the well-established approaches to prepare metal oxide. This method had good control over the surface properties of the materials. Sol-Gel method mainly undergoes in few steps to deliver the final metal oxide and those are hydrolysis, condensation, and drying process. The formation of metal oxide involve the following steps, initially the corresponding metal undergoes rapid hydrolysis to produce the metal hydroxide solution, followed by immediate condensation which leads to the formation of gels. afterward, obtained gel is subjected to drying process.

In this experimentation the Ethanol and Titanium Tetrachloride were taken into a beaker, and the solution is stirred for 30 min. During this period, the solution turns yellow. The obtained solutionand distilled water is added, therefore the solution became clear and colourless. The solution was again stirred for 30 min at room temperature and the formed gel was dried at 50oC for 24 hrs.

$$(C_{12}H_{28}O_4Ti)+C_2H_6O \rightarrow TiO_2+H_2O\uparrow+CO_2\uparrow+CO$$

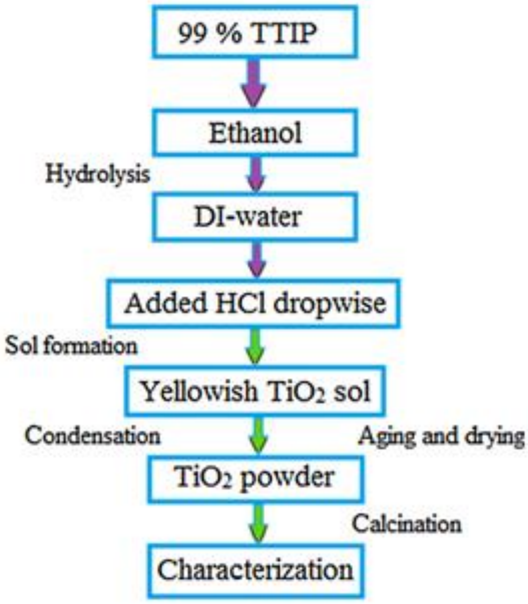

Figure 1: The above flow chart showsSequence of Procedure for Preparation of TiO_2 nanoparticles

2.3 Operations

2.3.1 Specimens Preparation: To perform the experimental test the mild steel specimen is prepared by the following steps

(i) Cutting: As per the requirement of experimental analysis the specimen is cut in the shape of square of 10x10 mm.Surface condition of specimen is considered as an important factor in this experimentation. So, it is necessary to prepare a uniform surface of the specimen.

(ii) Polishing: The specimens were polished using polish cloth and alpha alumina 1µm and 0.5µm, and then washed with distilled water. The polished specimens were dried and tested for material composition by using EDX.

2.4Experimental Procedure

The steel was mechanically cut into square shape using machining process of 10x3x10 mm. For dip-coating purpose, 1 mm hole was drilled on the top of the sample as shown in the Figure1.

Figure 2: Mild Steel Substrate

In this experimentation, for sol gel process, Ethanol and titanium tetrachloride were introduced into a beaker the solution was stirred for 30 min.Ethanol and distilled water were prepared at fixed molar ratio of1:3.2. Then acetone was added to the mixture continued to stir for another 30 minutes. Distilled water then added to continue the process for another 30 minutes.

2.5 Coating Technique

The substrates were fully immersed in titanium tetrachloride solution for 10 minute after which air dried for another 2 minutes. These steps were repeated according to the number of coatings made on each substrateas shown in the Figure 2. Once the deposition was completed, the substrates were thermally treated. During this phase, excess organic compounds found on the surface of coated substrates will evaporate. The substrate then annealed at 500_oC for two hours in the Muffle furnace.

Figure 3: Coating sequence of TiO_2 nanoparticles on mild steel

2.6 Scanning Electron Microscopy (SEM): The grain size, shape and surface properties like morphology were observed by the SEM (HITACHI S3400NS) machine R_P at different magnifications. The SEM images of TiO_2nanoparticles are prepared by solution combustion process.

2.7 Energy Dispersive X-Ray Spectroscopy (EDXS): The EDXS of TiO_2 nanoparticles was done by the SEM (HITACHI S3400NS) machine. It reveals that the composition present in the sample (in detail discussion is in 3.3 with images).

2.8 Surface Roughness: After heat treatment the samples at 400˚ C the surface roughness of the base metals are tested by using SJ-310 Portable Surface Roughness Tester. And after spraying TiO_2 nanoparticles on metals again the samples are tested for surface roughness as shown in the Table 1.

S.NO	Base metal	After heat treated at 400^0C	Deposition of TiO_2 nano particles at 500^0c
1	1.355	1.044	1.1775
2	1.764	1.649	1.882
Avg.	3.119	2.693	3.0595

Table 1: surface roughness values before and after coating TiO_2 nanoparticles

3. Results and Discussions

3.1 X-Ray Diffraction (XRD):

In the XRD pattern of the TiO_2nanoparticles, the peaks are observed at 30.577, 35.151, 37.777, 49.354, 57.501 and 63.211 (h k l) values of the peaks are (0 1 2), (1 0 4), (1 1 0), (1 1 3), (1 1 6) and (3 0 0)respectively. These results are coincided with JCPDS (Joint committee on powder Diffraction Standards database is a database of standard XRD reference patterns for various materials) card number 82-1468, and it shows that the TiO_2nanoparticle consists of spherical shaped structure. The average crystalline size is measured using Debye-Scherer's formula [4].

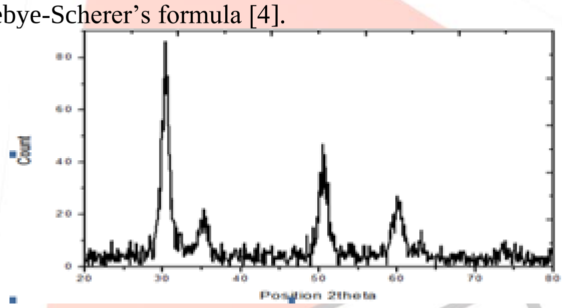

Figure 4: XRD pattern of TiO2nano particles

The formula is

$$D = \frac{0.9\lambda}{\beta\cos\theta} \text{nm}$$

Where D – Average size of the particle [nm]
λ --Wavelength of the radiation [A°]
θ –Diffraction angle [degree]
B – Full width half maximum (FWHM) of the peak [radians]
FWHM is given by the difference between the two extreme values of the two independent variable at which the dependent variable is equal to half of its maximum value.
From the above formula obtained average crystalline size is 55 nm. The lattice parameter a = b = 4.7589 A°, c = 12.9919 A°.

3.2 Scanning Electron Microscopy(SEM):

SEM images of TiO_2nanoparticles and the grain size, shape and surface properties like morphology were observed by using SEM with different magnifications. The SEM images of TiO_2nanoparticles shows respectable morphology and Grain size of TiO_2nanoparticles were nearly 163.2 nm as shown in the Figure 4.

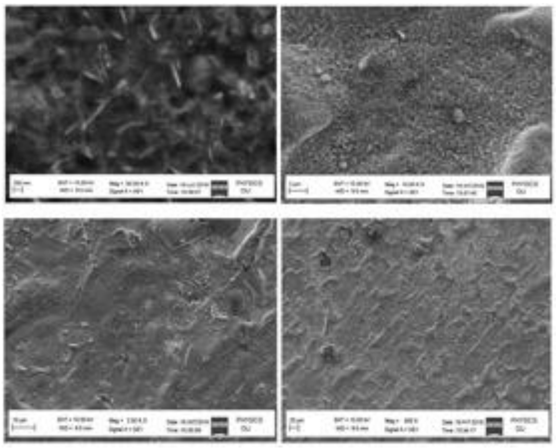

Figure 5: SEM images of TiO_2 nanoparticles

3.3 EDX of TiO_2Nanoparticles:

The EDX of the sample was done by the SEM (HITACHI S3400NS) machine. The Energy dispersive X-ray spectroscopy reveals that the required phase has Titanium (Ti) and oxygen (O) Elements present in the sample as shown in the Figure 5 and composition were clearly shown in the Table 2.

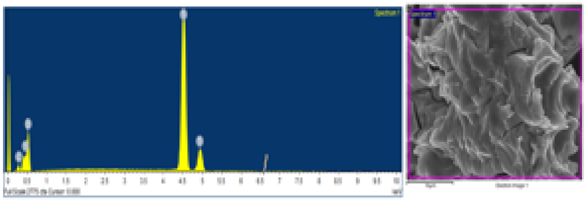

Figure 6: EDX image of TiO_2nanoparticle.

S.NO	Elements	Weight%
1	Ti	63.75
2	O	36.25

Table 2: Composition of TiO_2nanoparticle

3.4 SEM Images of the Base Metal:

SEM images of the base metal and the grain size, shape and surface properties like morphology were observed by the SEM with different magnifications. It shows coarse grain structure and size were nearly 240 nm as shown in the Figure 6.

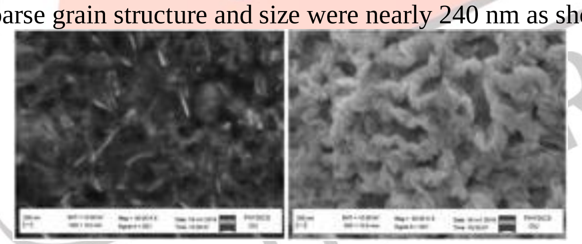

Figure 7: SEM images of mild steel

3.5 SEM Images of Coated TiO_2Nano Particles onMild Steel:

SEM images of coated Tio_2nanoparticles on mild steel at 500_o C was shown in the Figure 7. The grain size, shape and surface properties like morphology were observed by the SEM with different magnification. Coated nanoparticles on mild steel at 500_o C, shows respectable morphology when compared with heat treated sample at 400_o C. Grain size of coated sample was nearly 204 nm for 500_o C and the shape is spherical.

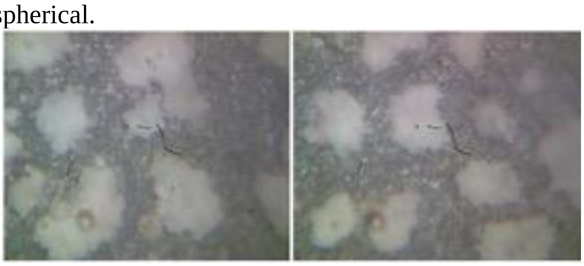

Figure 8: Coated samples at different magnification

3.6 EDX of Coated Sample:

The elemental composition of coated sample at 500_o C as shown in the Figure 8. The EDX pattern shows that the coated samples at 500_o C shows good percentage of Titanium along with Iron, Carbon and Oxygen whereas for heat treated sample at 400_o C shows less percentage of Titanium along with Iron, Carbon and Oxygen. It shows the deposition rate of Titanium is high at 500_o C when compared to heat treated sample at 400_o C as shown in Table 3.

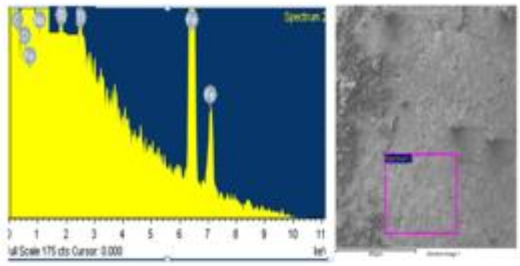

Figure 8: EDX of coated sample

S.NO	Elements	Weight%
1	C	0.94
2	O	25.08
3	Ti	1.83
4	Fe	56.63

Table 3: Composition of coated specimen

3.7 Surface Roughness:

Initially the surface roughness of the base metal is high when compared with heat treated sample at 400˚C due to the heat treatment the surface roughness of the sample is smooth. Heat treated sample at 500_{o} C shows fine surface roughness when compared with base metal and shows rough surface compared to 400_{o} C due to scale formation and high rate of deformation. Surface roughness is high after deposition of TiO_2nanoparticles on mild steel at 500˚C, due to high bond generated between TiO_2 and mild steel. Surface roughness is lower after deposition of nanoparticles on mild steel at 500_{o} C (Figure 7) when compared with untreated metal because of the bond generated during coating process. Surface roughness is lower after deposition of TiO_2nanoparticles on mild steel at 500˚C when compared with the base metal because of uniform deposition of TiO_2nanoparticles. During coating process surface roughness is low after deposition of TiO_2 nanoparticles on mild steel at 500_{o} C when compared with base metal because of uniform deposition of TiO_2 nanoparticles during coating process.

4. Conclusions

The surface properties of the steels are improved by the nanocoating process. The microstructure of the coated surfaces is studied with help of optical microscope, scanning electron microscope and EDXS. The SEM images of TiO_2 nanoparticles shows respectable morphology and Grain size of TiO_2 nanoparticles were nearly 163.2 nm. The average crystalline size of the TiO_2 nanoparticles are 55 nm. Coated nanoparticles on mild steel at 500_{o} C, shows respectable morphology when compared with base metal of the sample and grain size of coated sample at 500_{o} C were nearly 204 nm. Heat treated sample at 500_{o} C shows fine surface roughness when compared with base metal and shows rough surface compared to 400_{o} C due to scale formation and high rate of deformation. Surface roughness is high after deposition of TiO_2 nanoparticle on mild steel at 500_{o} C, due to high bond generated between TiO_2 and mild steel. Surface roughness is lower after deposition of nanoparticle on mild steel at 500_{o} C (Figure 7) when compared with untreated metal because of the bond generated during coating process. Coated nanoparticles on mild steel at 500_{o} C, shows respectable morphology when compared with heat treated sample at 400_{o} C. grain size of coated sample was nearly 204 nm for 500_{o} C and the shape is spherical

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