

Influence of Mwcnt's/Zinc Oxide Nano Particles/Epoxy Resin Composite Coating on Mild Steel to Enhance Anticorrosion and Mechanical Properties



Sandeep.V.Gujjar, Nandini R Nadar, Kanaram Choudhary, Anand. M. Hunashyal

Abstract: The motive of this research is to develop a new hybrid Nano composite on the surface of mild steel to eradicate the rate of corrosion that takes place on the surface of the mild steel and to enhance mechanical properties. For this, a hybrid Nano composite of Multi-Walled Carbon Nano Tube's (MWCNT's), Zinc Oxide (ZnO) Nano particles and Epoxy resin has been used to overcome the major disadvantage of mild steel which is corrosion. The mechanical property of mild steel is also increased. Ultra-sonication method is adopted for better dispersion of ZnO Nano particles and MWCNT's. In this study, Ethanol is used for better dispersion. After applying the coating on the surface of newly developed Nano composite by using pneumatic gun spray method is used. FESEM was conducted to study the surface morphology of corroded surface of mild steel. The rate of anticorrosion and mechanical properties get improved by the application MWCNT's/ZO Nano particles/Epoxy resin.

Keywords: MWCNT's, Zinc Oxide Nano particles, Epoxy Resin, Anticorrosion, Mechanical Properties.

I. INTRODUCTION

The Coating industry is growing rapidly around the globe. Today, coating not only helps for beautification of structure but also protects the valuable metals and buildings from corrosion. Application of nanotechnology in the field of coating can improve upon the coating industry. Properties like corrosion resistance, flame resistance, UV stability, gloss retention, chemical and mechanical properties are enhanced by making use of nanoparticles. The main merits of using Nano-coating are: (1) Provides better surface appearance, (2) Offers good resistance to chemicals, (3) Increases thermal stability, (4) Surface is easy to clean,

(5) Has anti-fogging, anti-graffiti, anti-skid, and anti-fouling properties, (6) Improves electrical and thermal conductivity, (7) It is anti-reflective in nature, (8) It is Lead and chromate free, and (9) Has good adherence. The anti-corrosion hybrid coatings provide a very effective method to protect the element against corrosion. There are various types of corrosion resistant coatings available such as urethanes, latexes, epoxies, and silicone alkyd. Two major mechanisms are accountable for the breakdown of coating protection, namely, diffusion of water through the coating and dis-bond propagation between the coating and the substrate (Aglan, Allie, et al. 2007). Corrosion is degradation of materials' properties due to chemical reaction with the environment, and corrosion of most metals is unavoidable. While primarily associated with metallic materials, all types of materials are susceptible to degradation. Approaches convenient for controlling corrosion include: Application of protective coatings to metal surfaces to act as a barrier and perhaps provide votive protection, the addition of chemical species to the environment to inhibit corrosion, alteration of alloy chemistry to enhance the resistance to corrosion, and the treatment of the surface of a metal to increase its resistance to corrosion (Shaw and Kelly 2006).

Multi-Walled Carbon Nanotubes, invented by Iijima and Ichihashi, have attracted considerable interest due to their application in fabricating a new classes of advanced materials owing to their distinctive structural, mechanical and electronic properties. MWCNTs possess good mechanical properties. The tensile strength of carbon nanotubes is about 100 times greater than that of steel of the same diameter. Two factors account for this strength. The first is the strength dispensed by the interlocking carbon-to-carbon covalent bonds. The second is the fact that each carbon nanotubes is one large molecule (Yeole, Mahajan et al. 2015).

Zinc oxide (ZnO) forms colour-less hexagonal crystals or a white dusty powder. When heated, the colour changes into lemon yellow. It reverts into white again when cooled down. Zinc oxide has a density of 5.61 g/cm³. It evaporates at 1300 °C and above and sublimates as it reaches a temperature of 1800 °C. Hence, there is no (liquid) melt but a direct transition from the solid into the gaseous aggregate state. ZnO nanoparticles have attracted great attention because of their unique catalytic, electrical, electronic and optical properties as well as their low cost and vast applications in different areas (He 2004).

Epoxy resin is the most popular material in the world for anti-corrosive coating of materials. This type of materials has high resistance to chemicals and possesses attrition resistance.

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* Correspondence Author

Sandeep.V.Gujjar*, Assistant Professor, Department of Civil Engineering, Pillai HOC College of Engineering and Technology, Rasayani, Dist. Raigad - 410207, Maharashtra, India.

Dr. Nandini R Nadar, Assistant Professor, Department of Mechanical Engineering, B.M.S.Institute of Technology and management, Bangalore-64, Karnataka, India.

Kanaram Chaudhary, Civil and infrastructure engineering from Rustomjee Academy for Global Careers, Dahanu road (E) - 401602, Maharashtra, India

Dr. Anand. M. Hunashyal, Associate Professor, Department of Civil Engineering, Visvesvaraya Technological University, KLE Institute of Technology, Hubballi-580021, Karnataka, India.

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Epoxy resin has good mechanical properties and electrical properties, and also offers resistance against chemical and atmospheric attacks (Dunn 1959).

II. EXPERIMENTAL PROGRAM

1.1 Materials:

Materials required are Multi-walled carbon nanotube's (MWCNT's), Zinc Oxide Nano particles (ZO), Epoxy resin (E) (Fine coat EP 200/A) and hardener (Fine coat EP 200/B), Mild steel, Thinner (Thinner-643), Ethanol, etc.

Multi-Walled Carbon Nano Tubes were prepared by CVD method. Epoxy resin (FINECOAT- EP 200A & EP 200B) is a two- component epoxy clear lacquer, cured with polyamide hardener. It cures at room temperature (above

10°C). Multi- Walled Carbon Nano Tube's (MWCNT's) and Zinc Oxide Nano particle's (ZO) as shown in Figure.1 and 2 were purchased from "Platonic Nanotech private limited," Jharkhand, India.

Epoxy resin was purchased from the "Fine Finesh Organics Pvt.Ltd" (ISO 9001- 2008 certified company) Taloja, Navi Mumbai, India. Pneumatic spray gun was purchased from, "Burhani hardware", Dahanu road, Palghar dist., India. Mild steel was brought from "Shubh M. L. Shah and Sons Steel Pvt. Ltd." Dahanu road, Mumbai, India.

The technical specifications of Multi -Walled Carbon Nano Tubes (MWCNT's), Zinc Oxide Nano particles (ZO) and Epoxy resin are mentioned below in Table. No.1, 2, and 3 respectively.



Figure.1. Multi-Walled Carbon Nano-Tube's (MWCNT's)

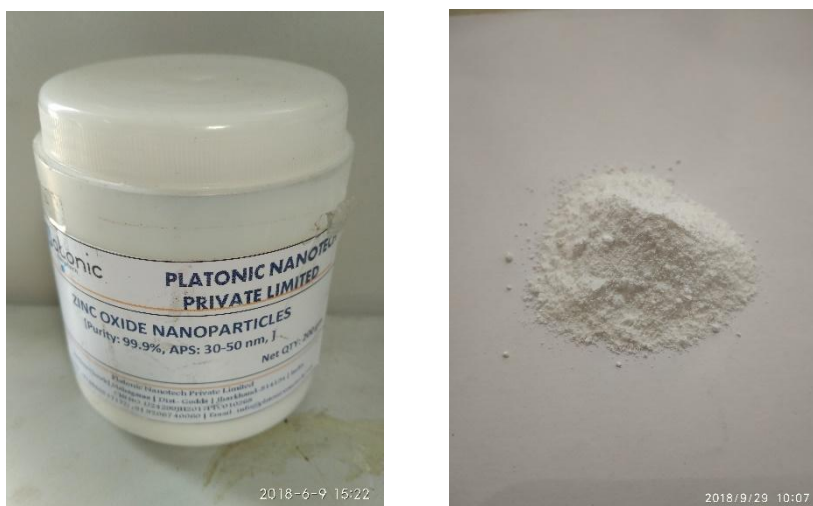


Figure.2. Zinc Oxide Nano Particles (ZO)

Table.1. Technical specification of Multi-Walled Carbon Nano-Tube's (MWCNT's)

Sl.No.	Specifications	Value
1.	Tensile Strength	30 ~ 180GPa (100 times of metal)
2.	Thermal Conductivity	6,000W/m·k (2 times of that of diamond)
3.	Electric Conductivity	6,000S/cm (1,000 times of that of copper wire)
4.	Modulus	1 ~ 2TPa (7 times of that of metal)
5.	Diameter	10 ~ 15 nm
6.	Length	2-10 microns
7.	Purity	>99 %

8.	Ash content	<.01 %
9.	Fe	<4000 mg/kg
10.	Al	<3500 mg/kg
11.	Mo	<800 mg/kg
12.	Specific Surface Area	250 ~ 270 m ² /g
13.	Bulk Density	0.06 ~ 0.09 g/cm ³
14.	Physical Form	Fluffy, Very Light Powder
15.	Color	Black
16.	Chemical Formula	C

Table.2. Technical specification of Zinc Oxide Nano Particles (ZO)

Sl. No	Specifications	Value
1.	Purity	99.90%
2.	Average Particle Size	30-50 nm
3.	Specific Surface Area	20-60 m ² /g
4.	Molecular Weight	81.408g/mol
5.	Bulk Density	0.28-0.48 g/cm ³
6.	True Density	6 g/cm ³
7.	Morphology	Spherical
8.	Molecular Formula	ZnO
9.	Physical form	Powder
10.	Color	Milky White
11.	ZnO	>99.9%
12.	Pb	<0.01%
13.	Si	<0.02%
14.	Mn	<0.02%
15.	Cu	<0.01%

Table.3. Technical specifications of epoxy resin

Sl. No.	Characteristic	Specifications
1.	Colour	Clear. Can be dyed as per the requirement
2.	Finish	Glossy
3.	Mixing Ratio	Base : Hardener = 2 : 1 (by volume)
4.	Pot life	14 - 16 Hours @30°C
5.	Theoretical covering capacity	12 sq.m. /lit @25 microns dft.
6.	Application method	Air-assisted / airless spray / brush

1.2 Preparation of Multi-Walled Carbon Nano Tube's/Zinc Oxide Nano Particles:

The composite materials i.e. MWCNTs / ZO are prepared by blending various proportions of MWCNT's (0.25%, 0.5%, 0.75% and 1% by weight of resin) and ZO (2%, 4%, 6% and 8% by weight of resin). For dispersion, both the Nano materials are Ultra-sonicated individually in ethyle alcohol (40% weight of resin) for 30 minutes as shown in Figure.5. Then, the materials are combined together for additional ultrasonication for another 30 min. After the ultra-sanitation is completed, the composite was mixed for 30 minutes at 50°C temperature by means of the magnetic stirrer to generate MWCNTs / ZO hybrid Nano composite.



Figure.5. Ultra sonication process of MWCNT's and SiO

1.3 Preparation of MWCNT's/ZO/E Composite coating:

For development of Nano composite coating, primarily, Epoxy resin (E) (Fine coat EP 200/A) of 10 g is added and mixed for an hour and then it is degassed for 20 min under the process of Ultra-sonicator. The detail regarding MWCNTs/ZO/E composite materials with various ratios is specified in Table.4. Then, hardener (Fine coat EP 200/B) is further added in MWCNTs / ZO/ E, and the composite is mixed by magnetic stirrer for 5 minutes along with the thinner. The prepared MWCNTs / ZO / E Nano composite coating was then sprayed by using pneumatic spray gun on the surface of the mild steel. The distance between the sample and spraying gun is kept at about 100 mm to 150 mm and the thickness of coating is about 120 micron to 150 µm. After the coating was applied to the surface of mild steel, the samples were dried for 48 hours at room

temperature, and then the sample were kept in the oven at 150 °C for one hour. The samples of mild steel coated with Nano composite were kept underwater containing 3.5% NaCl for 14 days (336 Hours).Corrosion rate is determined and then it is again subjected to Salt Spray Method (ASTM B-117:2016) at 34.8 to 35.1 degree centigrade, 5% NaCl, salt type. AR grade was conducted for 3 days (72 Hours) to find out the corrosion rate by the percentage of weight loss.

Samples with the same combinations were also made for testing the tensile strength Test. The specimens were tested by using fully computerized Servo Hydraulic Universal Testing Machine facility – AGIS 250kN in which Yield stress, Gauge length, Final length, Initial area, Gauge diameter, Elongation percentage, Ultimate load, initial and final thickness, yield load, Ultimate tensile Strength, and Fracture are evaluated. The scratch hardness test was also conducted to evaluate the surface hardness of the specimens.

Table.4. Detail of MWCNT's/ZO/E coating specimens

Sl. No.	Specimen Reference	Constituents	% of MWCNT's by Weight of Resins	% of Silicon oxide Nano Particles by Weight of Resins	Epoxy Resin(E) (gm.)
1	PMS	Plain Mild Steel	Nil	Nil	Nil
2	E	Epoxy	Nil	Nil	10 g
3	MZE 1	MWCNT's+ZO+E	0.25%	2%	10 g
4	MZE 2	MWCNT's+ZO+E	0.50%	4%	10 g
5	MZE 3	MWCNT's+ZO+E	0.75%	6%	10 g
6	MZE 4	MWCNT's+ZO+E	1.00%	8%	10 g

1.4 Preparation of mild steel surface

The Mild Steel specimen with 12mm diameter bar is cut to a span of 300mm (Figure.4) for Tensile test and flat specimen having (125 x 60 x 6) mm (Figure.4) for Scratch hardness test was sand blasted with Sanding paper P80 on the surface of Mild Steel and washed by acetone to clean and remove oxides present on surface of mild steel. Then, the specimen is allowed for drying by exposing to room temperature for more than an hour before application of resin coating. After the coating on mild steel specimen was dried for 48 hours at the room temperature, the specimens were kept in the oven at 150° C for one hour.

The prepared MWCNT's/ZO/E Nano composites of different ratios were sprayed by pneumatic spray gun on surface of mild steel. The distance between the spraying gun and the specimen is kept at about 100-150 mm and the thickness of coating was 160-180µm.

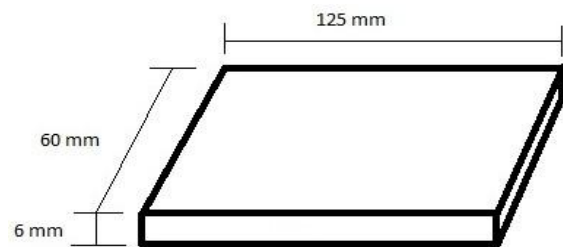


Figure.4. Specimen sample after hardness test

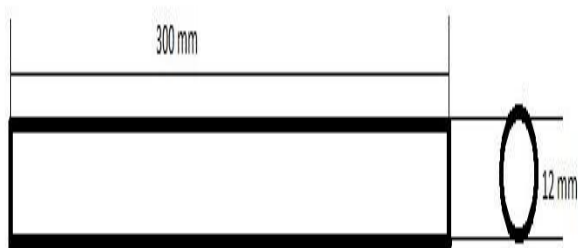


Figure.3. Tensile and Salt Spray test specimen

2.4 FESEM studies were done to study the corrosion external morphology of mild steel covered with MWCNT's/Silicon Oxide Nano Particles/Epoxy Resin coating with different combinations by field emission scanning electron microscope (FESEM). The corrosion surface is studied by gold coating to evade any charging and then images were captured at dissimilar magnification. The corroded surface using FESEM operated at an accelerating voltage of 20 kV.

1.5 Corrosion Analysis

1.6 Immersion Test:

The corrosion resistance properties of MWCNTs/Zinc Oxide Nano Particles/Epoxy Resin coated on the mild steel was analyzed by immersion of samples. The samples were immersed in the distilled water containing 3.5% NaCl for 336 hours and corrosion rate was determined by weighing technique.

The weight of the sample with resins coated is measured before and after immersion in the above mentioned solution to determine the rate of corrosion.

1.7 Salt Spray Test (ASTM B117):

In addition Corrosion resistance of mild steel coated with the MWCNTs/Zinc Oxide Nano Particles/Epoxy Resin was evaluated by means of the standard Salt Spray (Fog) test method as per ASTM B117:2016. In this examination, the mild steel specimens coated by the resins were kept at constant temperature where the specimens were open to the fine spray of salt solution having 5 percent for a definite period, and washed in water and dried. The extent of corrosion of test parts can be expressed as percent of red rust.

1.8 Mechanical Properties

1.9 Tensile Strength Test (A370:2017)

The effect of MWCNTs/Zinc Oxide Nano Particles/Epoxy Resin coated on the external surface of mild steel was studied by tensile test method on Universal Testing Machine (A370:2017). The Ultimate Tensile Strength, Yield load, Ultimate Load, Yield stress were evaluated in this experiment.

1.10 Scratch Hardness Test:

Scratch Hardness test was conducted on the flat plain mild steel sample having 120 mm length, 60 mm breadth and 6 mm thickness. Scratch Hardness testing method is conducted according to IS 101(Part-5, Sec.2):1988. The minimum load to cause failure is noted as the Hardness of the hybrid Nano coating.

III. RESULTS AND DISCUSSIONS

1.11 Corrosion properties of MWCNT's/ZO Nano Particles/Epoxy Resin coating:

The corrosion resistance properties of MWCNTs/Zinc Oxide Nano Particles/Epoxy Resin coated mild steel samples were studied by immersion method. The samples were immersed in the distilled water containing 3.5% NaCl for 336 hours by weighing method. This method was carried out to determine the percentage of loss of weight and then corrosion analysis was continued by using salt spray method for 72 hours in which the percentage of weight loss was yet again calculated. The total percentage of weight loss was considered by collective addition of both the methods mentioned above. The Comparison of corrosion protection by MWCNTs/ZO /E composite with neat Epoxy coated sample and plain mild steel in %, is shown in Figure.6 for only immersion test and Figure.7 for both immersion and salt spray test.

The samples having Epoxy resin before and after the corrosion indicates the highest percentage of protection of mild steel samples from corrosion with 65.29 %. Whereas MZE-1 indicates the second highest value about 32.57% as shown in Figure.13 and 14 before and after the corrosion respectively. The MZE-3, MZE-2 and MZE-4 show 16.28%, 12.63%, and 8.06% of anti corrosion respectively compared to plain mild steel sample without coating shown in Figure.16 and 17 (before and after corrosion). The percentage of protection by Nano composite against corrosion is mentioned in Table.5. (Immersion test) and Table.6. (Immersion and salt spray test).

Table.5. % of protection by coating against corrosion by immersion test

Sl. No.	Specimen	% of protection by resin against corrosion after 336 hours (Immersion test)
1	PMS	0
2	E	96.95
3	MZE 1	61.79
4	MZE 2	45.96
5	MZE 3	49.77
6	MZE 4	44.14

Table.6. % of protection by coating against corrosion by immersion test and Salt Spray Test

Sl. No.	Specimen	% of protection by resin against corrosion after 408 hours (Immersion and Salt Spray Test)
1	PMS	0
2	E	65.29
3	MZE 1	32.57
4	MZE 2	12.63
5	MZE 3	16.28
6	MZE 4	8.06

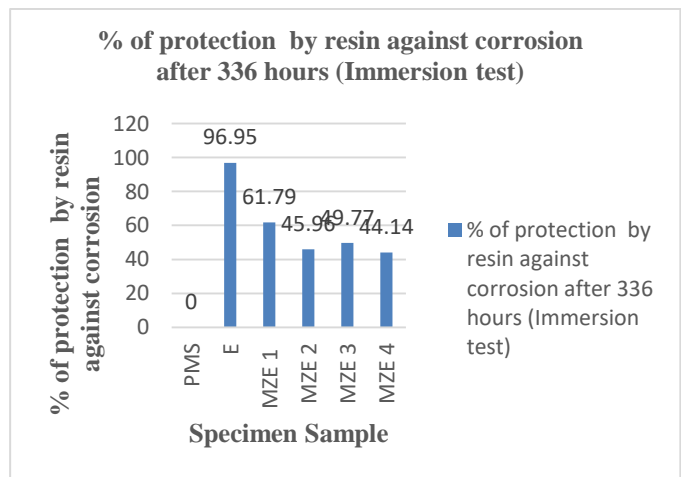


Figure. 6. Comparison of corrosion protection by MWCNT's/ZO/E in % by immersion test.

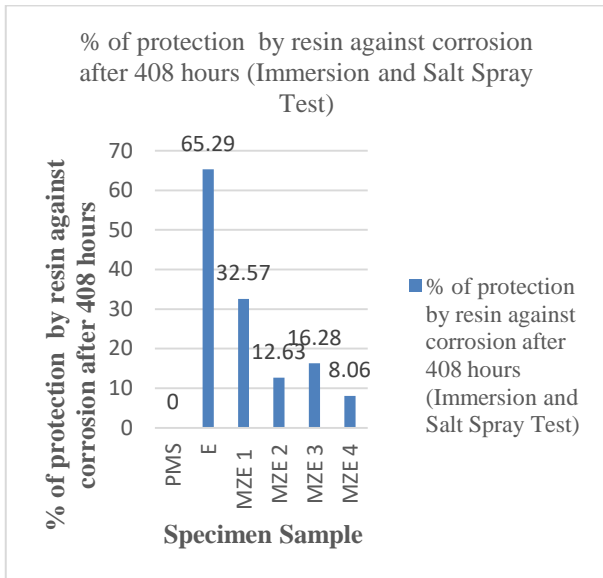


Figure. 7. Comparison of corrosion protection by MWCNT's/ZO/E in % by immersion and salt spray test.

The effect of Nano composite coated on surface of mild steel was studied by tensile test method on Universal Testing Machine. The Ultimate Tensile Strength test results and percentage increase in Ultimate Tensile Strength of plain mild steel, neat Epoxy resin, MZE-1, MZE-2, MZE-3 and MZE-4 coated samples are given in table no.7 and no.8. The standard deviation of Ultimate Tensile Strength and Percentage increase in Ultimate Tensile Strength are shown in Fig.8 and Fig.9.

The samples having MZE-1 coated samples show the maximum Ultimate Tensile Strength with 585.08 N/mm², Whereas neat Epoxy resin coated samples have 505.82 N/mm², MZE-4 have 491.11 N/mm², MZE-3 with 485.21 N/mm², MZE-2 with 479.58N/mm² and Plain mild steel have the least Ultimate Tensile Strength value of 456.23 N/mm² as shown in Figure.8.

The samples having MZE-1 shows the maximum percentage of increase in Ultimate Tensile Strength compared to mild steel samples with 22.02 %. Whereas neat Epoxy coated sample with 9.8 %, MZE-4 have 7.1 %, MZE-3 have 5.97% and the MZE-2 with least 4.86% increase in Ultimate Tensile Strength compared to mild steel samples without coating as shown in Fig.8.

1.12 Mechanical Properties

1.12.1 Tensile Strength Test (A370:2017):

Table.7.Ultimate Tensile Strength (N/mm²)

Sl. No.	Specimen Reference	Ultimate Tensile strength (KN/mm ²)
1	PMS	456.23
2	E	505.82
3	MZE 1	585.08
4	MZE 2	479.58
5	MZE 3	485.21
6	MZE 4	491.11

Table.8. % increase in Ultimate Tensile Strength

Sl. No.	Specimen Reference	% increase in Ultimate Tensile Strength
1	PMS	0
2	E	9.8
3	MZE 1	22.02
4	MZE 2	4.86
5	MZE 3	5.97
6	MZE 4	7.1

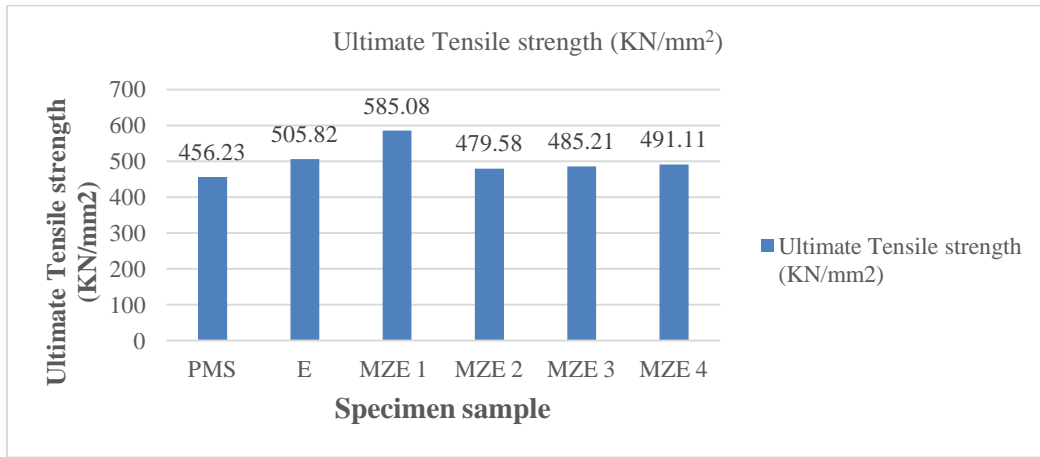


Figure.8. Ultimate Tensile Strength (N/mm²)

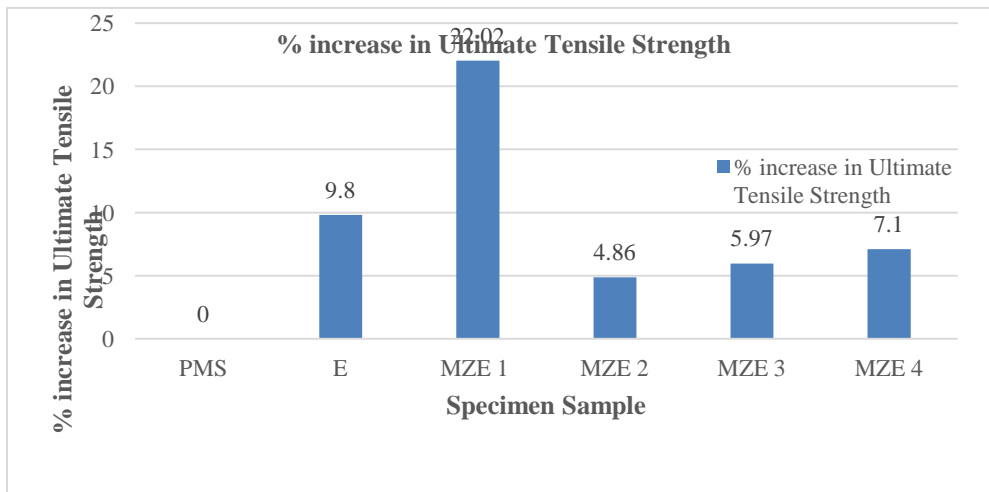


Figure.9. % increase in Ultimate Tensile Strength

1.12.2 Scratch Hardness Test:

The least load to cause failure is distinguished as the Scratch Hardness of the hybrid Nano composite coating. Scratch Hardness with standard deviation with Epoxy resin and MZE-1 coated mild steel samples are as shown in Fig.10. Neat Epoxy coated sample shows the maximum hardness with 700 mg, and MZE-1 coated sample with 500 mg. The scratch hardness values of the neat Epoxy and MZE-1 Nano composite coated surface values are as given in Table.No.9.

Table.9. Scratch Hardness (in mg)

Sl. No.	Specimen	Scratch Hardness (in mg)
1	EPOXY	700.00
2	MZE1	500.00

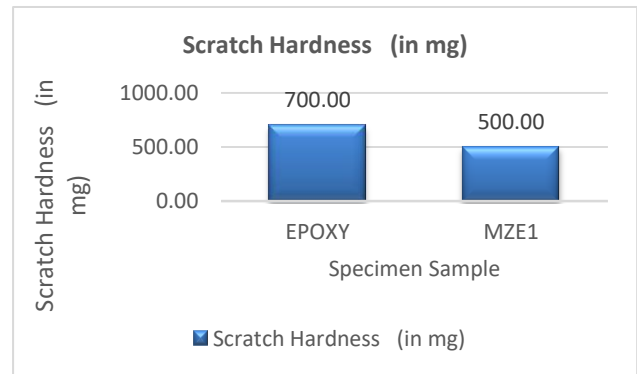


Figure.10. Scratch Hardness with standard deviation with Epoxy resin and MZE-1 coated mild steel samples.

1.13 Morphology

Figure.11 and 12 display the images of FESEM of Neat Epoxy resin coated samples before and after subjecting to corrosion. Figure.12 displays the corrosion products formed on the mild steel surface. Figure.13 and 14 are the images of MZE-1 coated samples. Figure.14 shows MZE-1 coated samples with protection of the mild steel surface with least rust with 32.57 % corrosion protection on its surface when compared to other ratios of Nano composite coating.

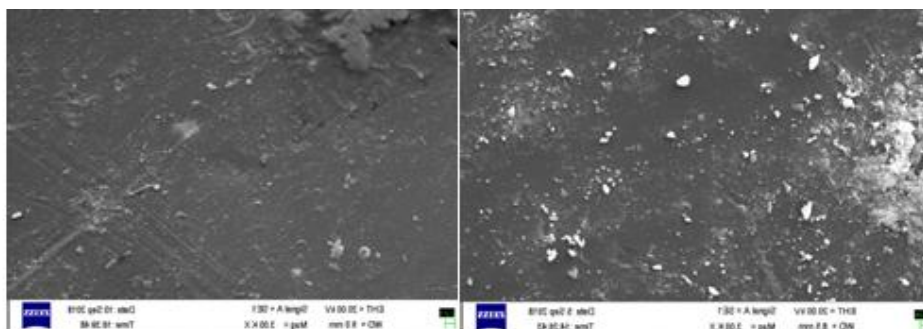


Figure.11.FESEM image of sample coated with Epoxy Resin before corrosion

Figure.12.FESEM image of sample coated with Epoxy Resin after corrosion

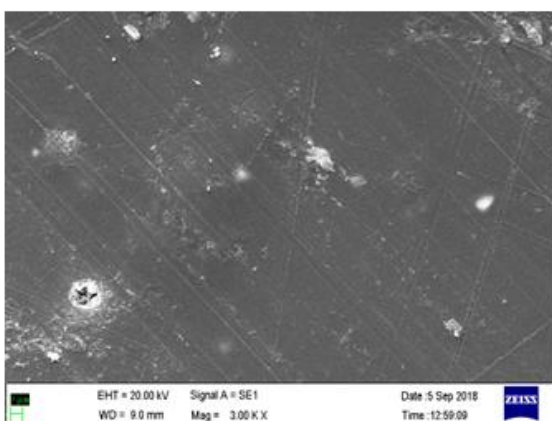


Figure.13.FESEM image of sample coated with MWCNT's/ZO/E (MZE1) Nano composite before corrosion

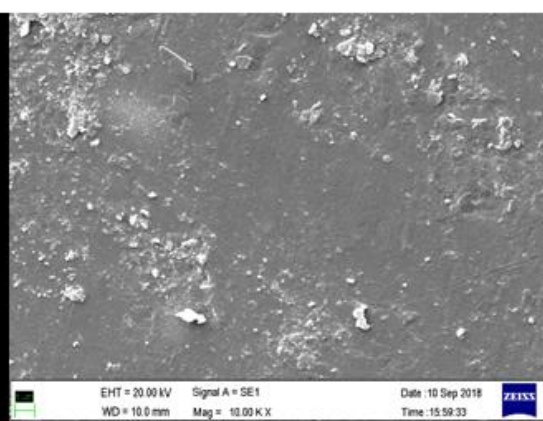


Figure.14.FESEM image of sample coated with MWCNT's/ZO/E (MZE1) Nano composite after corrosion

It is clearly seen that the mild steel samples coated with the Neat Epoxy resin (Figure.12) show the lowest corrosion products compared to MZE-1 Nano composite coated samples as per Figure.14.

IV. CONCLUSIONS:

The MWCNTs / ZO/ E (MSE1) Nano composites having 2% of ZO and 0.25% of MWCNT's indicate the upgraded properties as compared to the plain mild steel , neat Epoxy and different proportions of MWCNT's and ZO. The superior properties include anticorrosion, Ultimate Tensile Strength and scratch hardness. The results from the test of MZE-1 show 32.57% of anti-corrosiveness property, which is noted to be second maximum value. Scratch hardness property noted was 500 mg which is also the second highest value when compared to the neat Epoxy samples, the Ultimate Tensile Strength recorded was 585.08 N/mm² which is the highest value and the existence of Epoxy resin, Nano ZO and MWCNT's particles specifies that the increased mechanical properties as well as anti-corrosion properties when compared to the samples with Neat Epoxy and plain mild steel. Hence, it was concluded that the combination of 0.25% of MWCNT's, 2% of ZO with respect to weight of Epoxy resin used is an ideal combination for both anticorrosion and mechanical property. There is a wide scope for the study of mechanical and anticorrosion properties of mild steel with the combination of MWCNT's / ZO/ E with new practices of dispersion, deviation in percentages used, with upgraded properties of MWCNT's, ZO and Epoxy Resin.

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Dr. Nandini R Nadar is currently working as an assistant Professor, Department of Mechanical Engineering, B.M.S.Institute of Technology and management, Bangalore-64, Karnataka, India. She has completed her P.hD in "Development of lead free piezo-electric materials for energy harvesting applications" from Visvesvaraya Technological University, Karnataka, India. She has published various research papers in mechanical engineering, filed patents related to nano composite. He is guiding several students pursuing post graduate and under graduate students for research work in material science and mechanical engineering. She has more than 12 years of teaching experience, 1 year industrial and 1 year research experience. Ph: +91-9588922999, E-mail: nandini.nadar@bmsit.in



Kanaram Chaudhary is a graduate in Civil and infrastructure engineering from Rustomjee Academy for Global Careers, Dahanu road (E) - 401602, Maharashtra, India affiliated to University of Wolverhampton, UK. Ph: 7387165610, E-mail: kanaramchoudhary3@gmail.com



Dr. Anand. M. Hunashyal is presently working as an Associate Professor in the Department of Civil Engineering, Visvesvaraya Technological University, KLE Institute of Technology, Hubballi-580021, Karnataka, India. He has completed his P.hD from Visvesvaraya Technological University, Karnataka, India. He has published various research papers civil and mechanical engineering, filed and awarded patents related to nano composite coatings which enhance mechanical and anticorrosive properties of mild steel and many more. He is guiding several students pursuing doctorate, post graduate and under graduate students for research work in civil engineering. He has more than 20 years of teaching and research experience. Ph:9739192399, E-mail:amhunashyal@kletech.ac.in

AUTHOR'S PROFILE:



Sandeep.V.Gujjar is currently pursuing PhD from Visvesvaraya Technological University, Karnataka and working as an Assistant Professor in Department of Civil Engineering, Pillai HOC College of Engineering and Technology, Rasayani, Dist. Raigad - 410207, Maharashtra, India. He has published books related to civil engineering, research papers, filed patents related to nano composite coating which enhances mechanical and anticorrosive properties of mild steel. He has guided several students pursuing post graduate and under graduate students for research work in civil engineering. He has more than 11 years of teaching, industry and research experience in Indian and international organization. Ph: 8793169868, E-mail: sandeep.gujjar@rediffmail.com