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Corrosion Investigation on Conventional and Nanocomposite (Ni-P-Al2O3-TiO2) coated Mild Steel by In-Plant Test in Digester of a Paper Mill

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Abstract

Objectives: To improve paper quality and production, the digesters are now operated at increasingly high temperature and high sulfidity, which increase different type of corrosion attack on the surface of digester. The present study aims to develop an alternative material that can enhance the service life of the digester and reduces the loss generated by corrosion. **Methods/Analysis:** The nanocomposite coating of Ni-P-Al_2O_3-TiO_2,(MS(NiPATi)) was carried out on mild steel substrate by electroless technique. To carryout coating nanosized Al_2O_3 and TiO_2 particles were dispersed in 1:1 ratio, in Ni-P electroless bath with constant stirring. The plated ($MS(NiPATi)_{plated}$) sample was heat treated at 350°C in argon atmosphere ($MS(NiPATi)_{350}$) for improving its adherence to the substrate. In-plant corrosion test was carried, to determine the corrosion attack to the samples in the actual working conditions. To carryout in-plant test, the samples of MS, SS304L, SS2205, $MS(NiPATi)_{plated}$ and $MS(NiPATi)_{350}$, were exposed for duration of six month in the 5th batch digester of SPML in the actual working conditions. After removal from the digester the exposed samples were analysed by Scanning Electron Microscopy (SEM) and metallurgical microscope. Findings: An overall corrosion view on all the samples results that MS experiences highest corrosion rate while duplex SS2205 least and samples SS304L, $MS(NiPATi)_{plated}$, $MS(NiPATi)_{350}$ is nearly the same. The lower part of digester where test rack was fitted is mainly affected due to erosion assisted corrosion. This test, therefore, suggests that in the applied conditions, it would be better to construct the bottom part of the digester either with duplex steel SS2205 or with alternative materials i.e., $MS(NiPATi)_{350}$ to minimize the risk of corrosion.

Keywords: Corrosion, Digester, Electroless Technique, Nanocomposite Coating

1. Introduction

Among the different sections of the pulp and paper industry, the most extensively affected part is digester house. A number of investigations are dealing with survey report of mill, monitoring of *in-situ* corrosion and digester inspection reports, etc[1-3]. From the literature[4-7] it has been revealed that more than 75% digesters are older than two to three decades, moreover 73% digesters had stainless steel weld. The corrosion observed in kraft digester may be due to high sulfidity, presence of high percentage of C and Si in killed steel, change in chemical concentration, etc. Huseby and Schil[8] have studied in-plant test and mentioned that corrosion rate varies linearly from 28-138 mils per year (mpy), with Si content, while heat treatment and surface preparation affect the degree of corrosion attack. It has also been reported that protective galvanic coupling on stainless steel (SS)-347 or inconel with mild steel (MS) can significantly increases the corrosion of MS. In another study[9], SS coupons and C-steel were exposed to 6 kraft digesters and it had been observed that the rate of corrosion for C-steel is too high as compared to SS without any localized corrosion. Other[10,11] have reported similar observations and proposed that increased corrosion

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rate may be due to high concentration of oxidizing chemicals i.e., thiosulfate and polysulfide, etc. That is the higher corrosive nature of liquor basically depends on its composition, e.g., on adding sulfur a reduced corrosion is observed[12]. In another in-plant test carried out by Delblac and Lundberg[13], has been observed ~0.02 mpy corrosion rate for SS 316L and SS 304L. In the present scenario, to improve paper quality and production, the process of cooking of wood chip has been changed markedly. As a result the digesters are now operated at increasingly high temperature and sulfidity, which causes corrosion attack including caustic cracking, pitting and crevice corrosion[6,14-21]. There are two ways to improve the life of the digester i) by analyzing different grade stainless steel which show improved corrosion resistance ii) by applying metallic coating, on the material used for digester.

The nanocomposite dispersion coated materials can be a good alternate option for making/fabricating digester and ancillary equipment in paper industry and provide a cost effective option too, under the actual experimental conditions. The use of nanocomposite coatings have been recommended to protect material, by improving its corrosion and wear resistance properties which in turn reduces the cost of losses generated by this[22-24]. For the present study electroless nanocomposite coatings (Ni-P-X) have been chosen owing to their uniform coating ability over the intricate part of the substrate and good corrosion resistance and tribological properties[25,26]. A number of hard and soft, second phase particles have incorporated into Ni-P matrix and studied for their corrosion resistant, wear resistant and other tribological properties[25]. Among the other second phase particles Al_2O_3 and TiO_2 nanoparticles are believed as effective second phase particles owing to their high hardness, superior wear resistance, good chemical stability and ease of availability[27,28]. To date, Ni-P-TiO_2 and Ni-P-Al_2O_3 or in combination with ZrO_2[29], PTFE[30,31], etc., have been studied so far, for their corrosion and wear resistance properties, and to ascertain the effect of the shape, size, concentration, heat treatment temperature etc. The combination of TiO_2 and Al_2O_3 into the Ni-P matrix is not known to the best of author's knowledge. Therefore, to determine the synergistic effect of the two ceramic oxides onto the Ni-P coating, a nanocomposite (Ni-P-Al_2O_3-TiO_2) coating, on adding 1:1 molar ratio of the metal oxides into the bath, has been developed on mild steel substrate by electroless coating technique. The in-plant tests are the tests which are performed in actual conditions and can give the best information in selection of materials from the corrosion point of view. The in-plant test for the coated material in the as-coated and heat treated condition has been carried out.

2. Experimental Details

2.1 Details of the In-Plant Test carried out in Digester of Paper Mill

The present in-plant test was carried out in the 5th batch digester of a Star Paper Mill limited Saharanpur (SPML) established in 1938, Uttarapradesh. It is an integrated pulp and paper mill and produces a wide range of industrial, packaging and cultural papers catering to almost all segments of consumers. For writing and printing grade paper the present paper mill uses 75% poplar, 25% eucalyptus as a raw material. For the present study the digester used is having79 m^3 capacity and is purely made up of mild steel. The digester was filled with wood chips and liquor to wood ratio was maintained as 1:2.6. Inside the digester the volume of cooking liquor was kept between 37 to 39 m^3 which consists of Na_2S (21.6 g/l as NaOH) and NaOH (90 g/l). The operating conditions are pH ~13.4, temperature maximum up to 161 C and pressure ~6.1 kg/cm^2.

2.2 Materials

For the present in-plant test, the material has been selected on the basis of their current utilization and their possible future applications for constructing a digester or allied machinery in paper and pulp industry or other process industries. MS is the basic construction material, while SS304L used for weld overlaying or cladding in the digester. Duplex stainless steel 2205 has been proposed[19,20] as a prospective material for digester construction. This is because it not only show better corrosion resistance but also prevent the localized attack and caustic stress corrosion cracking, which is an important factor in digester corrosion. Additionally and alternatively it has been observed that there is possibility with electroless Ni-P plating deposition with second phase particles for improving hardness, abrasive properties, corrosion and wear resistance[30-33]. Therefore, following conventional as well as alternate nanocomposite coated materials are considered for the present in-plant test: mild steel,

Table 1. Composition of Steel Plate Samples

Alloy	C	Si	Mn	P	S	Cr	Ni	Mo
MS	0.18	0.04	1.66	-	-	-	-	-
SS304L	0.036	0.44	1.84	0.024	0.001	18.11	8.01	0.26
SS2205	0.022	0.35	1.47	0.02	0.001	22.13	5.55	3.16

austenitic stainless steel SS304L, duplex Stainless Steel 2205 and nanocomposite-dispersion coated Ni-P-Al_2O_3-TiO_2 with base material mild steel under plated and heat treated conditions at 350°C. M/S Avesta AB, Sweden had supplied the stainless steel samples their chemical analysis and mechanical properties are listed in Table-1 and Table-2, respectively.

Table 2. Mechanical Properties of Steels

Grade	Proof Strength (N/mm²)	Tensile Strength (N/mm²)	Elongation (%)
SS 304L	276	616	62
SS 2205	636	712	40

2.3 Preparation of Nanocomposite Dispersion Coatings

The mild steel samples were coated with Ni-P-Al_2O_3-TiO_2 by electroless technique by our research group and the detail procedure is reported elsewhere[34]. For heat treatment the coated sample was kept at 350°C for 1h in tube furnace in the argon gas atmosphere and then was cooled (annealed) in the furnace to room temperature. Detail XRD, scanning electron microscopy (SEM)/energy dispersive X-ray analysis (EDAX) microstructure analysis of the as deposited and heat treated samples are given in[34]. The XRD and SEM/EDAX analysis of exposed samples are discussed here.

2.4 Exposure of Coupons

The mild steel, austenitic SS 304L, duplex Stainless Steel 2205 samples of size 20 mm x 20 mm x 4 mm, were cleaned using emery paper from coarse to fine up to 800 grit on a polishing machine for exposure in digester. All these samples were cleaned and degreased with acetone. In addition, mild steel samples of size (20 mm x 20 mm x 4 mm), were coated with Ni-P-Al_2O_3-TiO_2 by electroless technique, in as-plated and heat treated (at 350°C) form were used for exposure. The samples were weighed and their coating thickness was measured by[31]. The surface area of the samples was measured by using Screw Gauge/ Vernier calipers before putting into the test solution. To estimate crevice attack the serrated washers was used for mounting the coupons.

In digester, the samples were fixed in a rack formed of SS-316 rods and plates and then welded in bottom partof the digester (Figure 1) and was kept in the same position

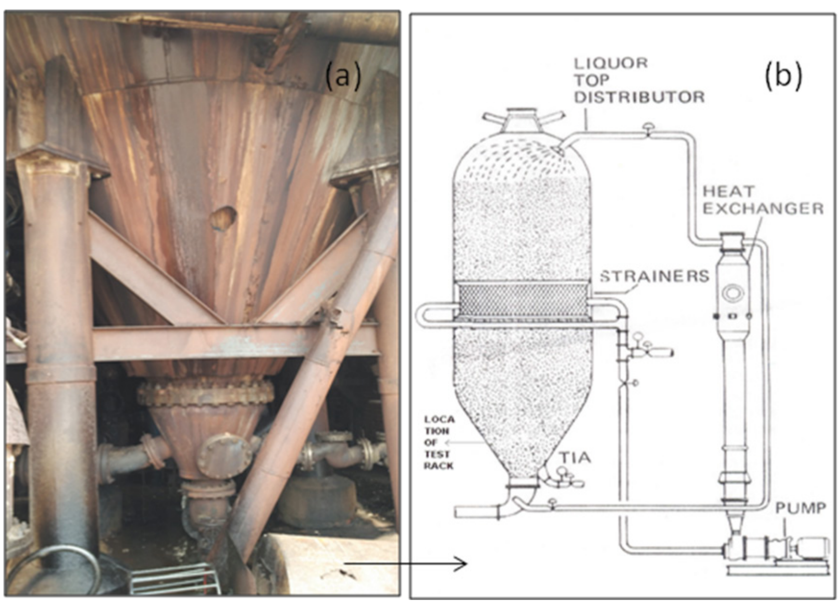

Figure 1. (a) Photograph of digester and (b) different components of digester.

for entire duration of the experiment. After six months, the samples were removed and analyzed for corrosion attack. The chemical composition and concentration of the pulping liquor was analyzed for every 20 days, in the whole duration of the experiment, by following SCAN procedure[35-37] and the chemical analysis of the pulping white liquor, is given in Table-3.

Table 3. Chemical Composition of White Pulping Liquor

Chemicals	Composition
pH	13.4
Sulfidity	~19.5%
NaOH (Sodium Hydroxide)	90.0±1.1gpl
Na_2S (Sodium Sulfide)	21.8±0.7 gpl
S_x^{2-} (Poly Sulfides)	1.2±0.17 gpl
$Na_2S_2O_3$ (Sodium Thiosulfate)	4.32±0.6 gpl
Na_2CO_3 (Sodium Carbonate)	15.6±0.8 gpl
Cl^- (Chloride)	1.87±0.43 gpl

2.5 Evaluation of Corrosion Attack

The samples without coating, as-coated and heat treated were exposed for six month in the digester. Then the samples were removed from the test rack and were cleaned mechanically and chemically by using a solution of (50 g/l $SnCl_2$ +20 g/l $SbCl_3$ in concentrated HCl), as per ASTM guidelines. The cleaned samples were weighed and their corrosion rate in mils per year (mpy) was calculated by using the following formula:

Corrosion rate (mpy)= 534W/DAT

where W is weight loss in mg, D is density of steel 7.8 gm/cm^3, A area in inch square and T is time exposure in hours.

Using metallurgical microscope (Make: Reichert Jung, USA) the samples were also analyzed for localized corrosion attack such as crevice and pitting corrosion and for weld related attacks and the results thus obtained are presented in Table-4.

3. Results and Discussion

3.1 Surface Morphology and Characterization of the Composite Coating

The mild steel samples of size (20 mm x 20 mm x 4 mm) were coated with composite Ni-P-Al_2O_3-TiO_2 coating using electroless technique, the photographs and microstructures of the coated samples are shown in Figure 2. The details of the coating procedure and their characterization were carried out by our research group and reported elsewhere[34]. The results of XRD, SEM-EDAX micrographs of as deposited and heat treated at 350^{0}C for 1h under argon atmosphere suggest that Ni-P-Al_2O_3-TiO_2 nanocomposite coated samples has smooth surface with almost uniform distribution of Al_2O_3 and TiO_2 particles over the coated surface with small porosity. It is due to the presence of the surfactants in the electroless bath. When the composite coating is heat treated at 350^{0}C for one hour, the globules of Ni and P with embedded Al_2O_3 and TiO_2 particles become more compact, which reduced porosity of the coating which is in accordance[38-44].

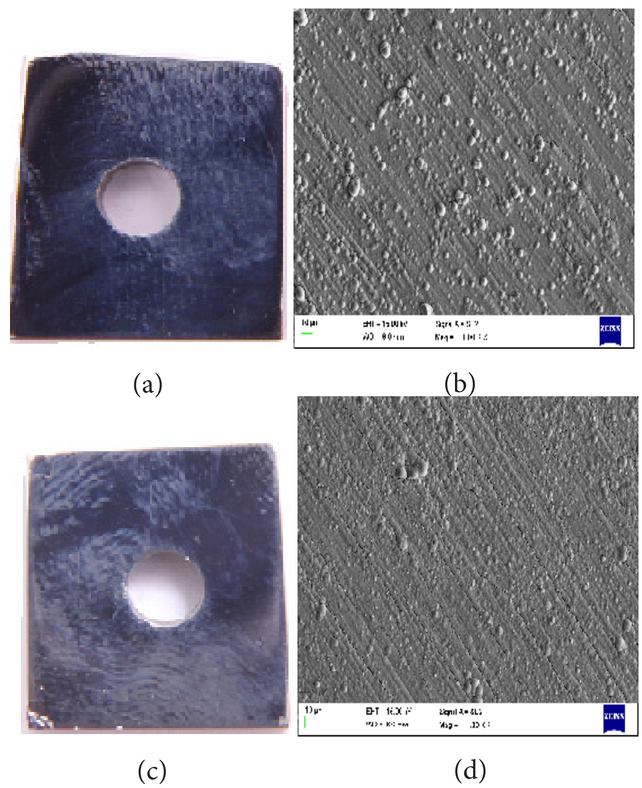

Figure 2. Mild Steel sample coated with Ni-P-Al2O3-TiO2 (a, b) photograph and microstructure of as-plated (c, d) photograph and microstructure of heat treated at 350°C.

3.2 Inplant Test Observations and their Discussion

Chemical analysis of the white pulping liquor of the mill analyzed by SCAN methods[35-37] shows that liquor has an average sulfidity of ~19.5%. The sulfidity is commonly used in all Indian paper mills, and the quantity of sulfidity used in Indian paper mills is quite less as compared to the mills of USA, Canada and Scandinavia, etc. In the present report the content of polysulfide and thiosulfate is also less. The higher concentration of the above mentioned chemicals and high operating temperature has been reported to be responsible factor of higher corrosivity of cooking liquor. A uniform corrosion has been observed for all the corroded coupons except duplex SS2205. Pitting is also observed in allcases while in duplex SS2205 no major attack has been observed. Crevice corrosion has also been observed on all the samples except MS(NiPATi)$_{Plated}$ and duplex stainless steel SS2205. No weld related attack has been observed on the coupons.

The degree of corrosion attack for the tested samples has been listed in the Table 4, and it has been observed that mild steel and duplex SS2205 samples show high (12.76 mpy) and low (0.017 mpy) degree of corrosion, respectively. The mild steel sample is experienced maximum (12.76 mpy) rate of corrosion followed by MS(NiPATi)$_{350}$(0.393mpy), MS(NiPATi)$_{Plated}$(0.310mpy), SS304L(0.28mpy) and duplex stainless steel SS2205(0.017mpy). Therefore the results suggest that mild steel is least resistance for uniform corrosion while duplex stainless steels show better corrosion resistance as compared to austenitic stainless steel SS304L, Which is just different in accordance with earlier observation by[9,35,38-39]. In digester liquor, the better resistance of duplex SS2205, may be due to the presence of higher amount of Cr and N in duplex SS 2205 in comparison to SS304L. Cr metal is capable to form astable $Cr(OH)_3$ layer which protect the metal against corrosion. At higher pH(11 to 14pH), Mo has not been considered to impart resistance against corrosion as it dissolves in the form of MoO_4^{2-}. The reason of the different result here can be dominance of $Cr(OH)_3$ layer in terms of stability over MoO_4^{2-}. However this hypothesis should also be verified bylong term immersion test, electrochemical tests and E-pH diagrams for Fe-S-H_2O system considering Cr, Ni,Mo etc as an alloying element at high temperature. MS(NiPATi)$_{plated}$ shows better performance than MS(NiPATi)$_{350}$, because former has amorphous structure and later turns into crystallized form. That may generates intergranular boundary which could act as cathodic and anodic sites and enhances corrosion rate in later case. The corrosion rate of austenitic SS304L can be challenged by MS(NiPATi)$_{Plated}$ and MS(NiPATi)$_{350}$. These results clearly indicate that coating options could be cost effective. Because, MS(NiPATi)$_{350}$ and MS(NiPATi)$_{plated}$ shows almost same uniform corrosion rate as SS304L. The photographs of the coupons after removal from digester are shown in Figure 3. In acidic chloride environment, normally the resistance against corrosion attack (including pitting), has been determined by pitting resistance equivalent number (PREN)[40]. The method is not applicable or gives misleading result in the alkaline environment.

Pitting type attack is also observed in case of all the samples. SS304L(43 μm), shows the highest pitting attack whereas the duplex stainless steels SS2205 show least (19μm) visible pitting attack. Pitting resistance of duplex SS2205 is more than that of SS304L, which is in accordance of PREN no. Pitting resistance of SS304L(43μm) can be challenged by MS(NiPATi)$_{plated}$(26 μm) and MS(NiPATi)$_{350}$(35 μm). SS 304L is observed to be lesser resistant against pitting in comparison to MS(NiPATi)$_{350}$ and MS(NiPATi)$_{plated}$. This result clearly shows the usefulness of nanocomosite dispersion coatings. In the nano composite dispersion coated materials MS(NiPATi)$_{350}$(35 μm) shows the higher side pitting attack than MS(NiPATi)$_{plated}$. The higher pitting attack of MS(NiPATi)$_{350}$ than MS(NiPATi)$_{plated}$ could be because of the changes of coated materials from amorphous structure to crystalline structure. This can also be observed by XRD structure. Crystallinity creates grain boundaries and second phases which are active sites for corrosion attack. Due to the high content of thiosulfate, chloride, polysulfide and process conditions e.g., high temperature in the cooking pulping liquor can also be responsible for the pitting attack[7,8,11].

Table 4. Corrosion Attack on the Steels Coupons

Grade	Corrosion Rate (mils per year)	Localized Corrosion Maximum Pit Depth (mm)	Crevice Corrosion Maximum Pit Depth (mm)	Weld related Attack Max Pit Depth (mm)
MS	12.760	0.027	0.051	NA
SS 304L	0.280	0.043	0.047	NVA
Duplex SS 2205	0.017	0.019	NVA	NVA
MS(NiPATi)$_{Plated}$	0.310	0.026	NVA	NA
MS(NiPATi)$_{350}$	0.393	0.035	0.025	NA

NVA (No Visible Attack) NA (Not Applicable)

(a) (b)

Figure 3. Test Coupons After removal from Digester (a) MS(NiPATi)$_{plated}$ (b) MS(NiPATi)$_{350}$.

From all the sample coupons only MS, SS304L and MS(NiPATi)$_{350}$ shows crevice type attack. In case of duplex SS2205 and MS(NiPATi)$_{plated}$ no visible crevice type attack is observed. MS has the highest crevice type attack (51 μm) followed by SS 304L (47μm), MS(NiPATi)$_{350}$(25 μm). The depth of attack under crevice is slight higher in comparison to pitting attack on open surface. The overall analysis of crevice type attack on all the samples can be explained in similar way as in case of pitting attack. The crevice attack could be due to higher velocity of the cooking liquor in the bottom part of the digester and also higher thiosulfate, poly sulfide concentration under crevice former. Weld related attack is not observed in case of SS304L and duplex SS2205 and in others case it was not applicable.

If an overall comparison against corrosion resistance is considered, it can be concluded that MS is totally rejected material for making/fabrication of digesters. Comparison of corrosion resistance between SS304L and nanocomposite dispersion coated materials, the corrosion resistance of SS304L can be challenged by these nanocomposite coated materials. Also, such nanocomposite coated materials are likely to be cost effective and could be an excellent alternative in fabrication of digesters and related allied machinery equipments. MS(NiPATi)$_{plated}$ and MS(NiPATi)$_{350}$ almost provides similar corrosion protection. Thorpe[20] has reported that duplex Stainless steel 2205 owing to its superior properties including better proof strength, better resistance against localized corrosion, comparable ductility etc., over SS 304L make it a suitable material for digesters in future. If an overall comparison against corrosion resistance in between duplex SS 2205 and MS(NiPATi)$_{plated}$, MS(NiPATi)$_{350}$, is analyzed it looks duplex SS2205 dominates over MS(NiPATi)$_{plated}$ and MS(NiPATi)$_{350}$. But considering the cost and ease of availability of material and fabrication aspects MS(NiPATi)$_{plated}$ and MS(NiPATi)$_{350}$ seems to be a good option. However this hypothesis also should be verified by electrochemical, long term immersion tests and more inplant tests.

4. Conclusion

An overall view on all the samples concludes that MS experiences highest corrosion rateand duplex SS2205 least in the tested environment. The corrosion attack on the samples SS304L and MS(NiPATi)$_{plated}$ and MS(NiPATi)$_{350}$ is nearly same in the tested environment. Increasing demand of paper will require higher sulfide and polysulfide content because of advantages associated with them. From test results it seems that under changed conditions austenitic SS304L will not be considered suitable material by considering corrosion attack and material cost. Therefore, one may look for other conventional and nonconventional materials such as duplex 2205, MS(NiPATi)$_{plated}$ and MS(NiPATi)$_{350}$. It is desirable that these aspects need to be checked in laboratory prepared solutions by long term immersion test and electrochemical test. Secondly, the lower part of digester where test rack was fitted is mainly affected due to erosion assisted corrosion. The present study suggests that in the applied conditions it would be better to construct the bottom part of the digester either with duplex steel SS2205 or with alternative materials i.e., MS(NiPATi)$_{350}$ to minimize the risk of corrosion. MS(NiPATi)$_{350}$ as an alternative materials provides a cost effective option for the construction of bottom part of the digester.

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