

Electrodeposition of Transition Metal Composites on Mild Steel: Structural and Wear Behaviour

P. Sathyabalan, M. D. Puhazhendhi, K. R. Aranganayagam, R. Kannan



ABSTRACT--- Wear characteristics of the transition metal composite (TMC) coated mild steel are investigated. TMC coatings were performed using electrodeposition technique on mild steel. Different concentrations of transition metals were subjected to prepare the TMC's and studied. The structural and the micro structural studies of the composites coatings were studied through X-ray diffraction (XRD) and scanning electron microscopy (SEM), respectively. The elemental compositions of coated composites were evaluated using Energy dispersive X-ray diffraction (EDS) studies. Both the structural and micro structural characterizations confirmed the formation of composite coatings. Further, it is evident from the EDS analyses that TMC's are coated with the desired concentrations. In order to understand the wear resistance of coated mild steel, the specimen were subjected to load on pin-on-disc type wear tester. The effects of concentration of composite and thickness of the coating on wear resistance are discussed. The coating results in improving the wear resistance and hardness of the specimen.

Keywords - Composite coating, Electrodeposition, Wear, XRD, EDX

I. INTRODUCTION

Life of a product is directly controlled not only by material aspects but also, by technical boundaries enforced by environmental conditions. These boundaries impose limiting criteria to access the functional feasibility of the product. For centuries people have been working on to improve the life of a product. Components are deemed to be unfit once their surfaces have degraded. This limit may vary from the appearance of minor pitting or scoring marks in bearing surfaces to the removal of several millimeters of material from the bucket of an excavating tool. In moving a car the frictional losses account for 21.3% of the total loss [1]. Reducing the frictional losses will decrease the fuel consumption. A number of approaches have been carried out to improve the products life. Chief among them are adding alloying elements, using composite materials [2], graded

materials [3] and employing surface treatments [4]. Structural modification of service properties of material parts area is increased by depositing adherent tiny coatings of small and/or nano dimension on the surface of the material [5]. The deposition might either be to enhance aesthetics or to improve engineering properties [6][7]. The structural modification imparts new service properties to the materials [8][9]. Rejuvenating techniques also are capable of increasing worn parts to their original tolerances, therefore reducing waste and replacement costs.

Electrodeposition is one of the important process of plating a material over another. It is used for various applications such as wear resistance, corrosion resistance [10] and decorative appearance [11]. In addition to those classical plating applications, recent development of composites to new areas, like electro-catalysis, photoactive materials or energy storage, have also been determined. Due to simplicity, low cost and less time consuming, the electrodeposition technique is selected for this composite coating work. Most composite coatings contain micron-sized particles, though there is growing interest within the codeposition of nano particles as a result of their increasing handiness [12]. Mild steel is widely used material in structural applications due to good performance and balanced properties of strength, plasticity and weldability. It is widely used in the fabrication of mechanical components and abrasive tools in the less strict situation. However, the steel is susceptible to pitting corrosion in the natural environment because of its low abrasive resistance and chemical stability. Compared to the steel, nickel has higher chemical stability, drawability and plasticity, which makes it much harder and more resistant to abrasion and corrosion as coating material [13]. There are many techniques available for coating on MS materials, with varying thickness from several microns to several millimeters [14][15].

The prime objective of the present investigation is on the effect of transition metals composites on the mild steel towards wear resistance and hardness.

II. EXPERIMENT

A. Plating Sample Preparation

Experimental mild steel samples were sheared into required size of 10 mm diameter and 32 mm length from a lengthy mild steel rod. The samples were cleaned from heavy oils and dirt using dilute sulphuric acid and acetone.

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Then samples were washed in distilled water and dried [16]. After the cleaning process, emery sheets and grinding/polishing machine were used to polish the specimen. Transition metal composites were prepared with five different stoichiometric ratio of plating substance were taken and prepared for electrodeposition.

B. Plating Procedure

The weight of the samples was taken and noted before the start-up of the plating process. The deposition parameters considered in the present investigation, varied at different levels to study their effects on the weight of composite deposited, were voltage and plating time. The parameters were considered in the following order: plating temperature is from 30° to 45°C and current density is 60-75 mA. A magnetic stirrer was used in the Watts bath at 550 rpm. The optimum plating time is 30 minutes.

C. Preparation and Process of Electrodeposition

All the metal precursors (Merck) are in Analar grade and used as such. Solvents and reagents were purified under standard procedures. The electroplating solutions were prepared with desired concentration in the 200 ml beaker with doubly distilled water. The concentrations of electrolyte solution are listed in the Table 1 to 5 for the sample 1 to 5. The schematic representation and electrode assembly of the electro deposition technique are shown in Fig. 1. Attempts were made to optimize the input conditions such as pH, temperature, current density etc. After the successful completion of optimization, the input conditions (discussed in Section II B), were fixed for the composite coatings process [11].

TABLE I. Basic bath Compositions for Ni coating (Sample 1)

Concentration of NiSO ₄ .6H ₂ O	100 g/L
Boric acid	20 g/L

TABLE II. Basic bath Compositions for Ni + Zn coating (Sample 2)

Concentration of NiSO ₄ .6H ₂ O	52 g/L
Zinc acetate dehydrate	44 g/L
Boric acid	10 g/L

TABLE III. Basic bath Compositions for Ni + Mn coating (Sample 3)

Concentration of NiSO ₄ · 6H ₂ O	52 g/L
Concentration of MnSO ₄ · 6H ₂ O	34 g/L
Boric acid	10 g/L

TABLE IV. Basic bath Compositions for Zn + Mn coating (Sample 4)

Zinc acetate dehydrate	44 g/L
Concentration of MnSO ₄ .6H ₂ O	34 g/L

After completion of the plating process, the weight of the substrate was noted. Difference between the initial and final weight of the coating in the plating process denotes the amount of composite coating on mild steel substrate.

TABLE V. Basic bath Compositions for Ni + Zn + Mn coating (Sample 5)

Concentration of NiSO ₄ .6H ₂ O	52 g/L
Zinc acetate dehydrate	44 g/L
Concentration of MnSO ₄ .6H ₂ O	34 g/L
Boric acid	10 g/L

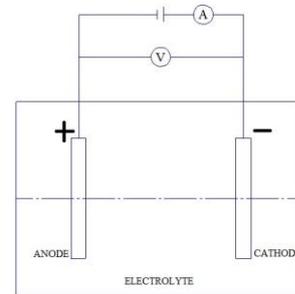


Figure 1. Electro deposition apparatus setup model

III. RESULTS AND DISCUSSION

A. Surface Morphology studies

The coating uniformity and micro structural behaviour of the uncoated and TMC coated specimens were studied using Scanning Electron Microscope (SEM) under a 20 kV accelerating voltage. SEM images of uncoated base material, Ni+Zn, Ni+Mn, Zn+Mn and Ni+Zn+Mn composites electrodeposited samples are presented in Fig. 2 to 6.

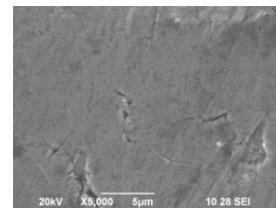


Figure 2. SEM Micrograph of uncoated at magnification of X5000, 5 µm

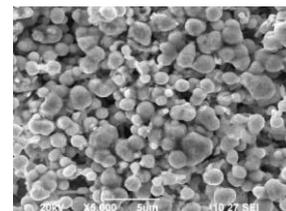


Figure 3. SEM Micrograph of the Ni+Zn coated surface at magnification of X5000, 5 µm

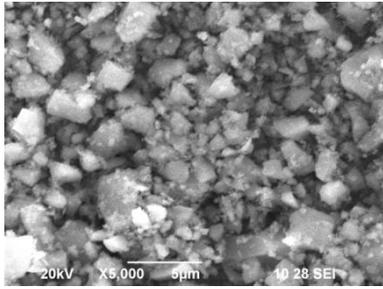


Figure 4. SEM Micrograph of the Ni+Mn coated surface at magnification of X5000, 5 μm

Fig. 3 SEM micrograph explains the distribution of Ni and Zn particles on the surface of base material. Ni and Zn particles are spread over the base surface giving the impression of another layer like structure. Comparing to the uncoated surface of the base substrate, the Ni and Zn particles, found evenly distributed on the surface, improve the micro hardness and wear resistivity. Fig. 4 SEM micrograph explains the distribution of Ni and Mn particles on the surface of base material. Ni and Mn particles are evenly spread over the base surface giving the impression of another layer like structure. This distribution on the surface, improve the micro hardness and wear resistivity.

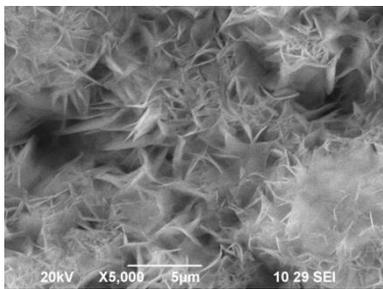


Figure 5. SEM Micrograph of the Zn+Mn coated surface at magnification of X5000, 5 μm

Fig. 5 SEM micrograph explains the distribution of Zn and Mn particles on the surface of base material. Comparing to the uncoated surface of the base substrate, the Zn and Mn particles are evenly distributed on the surface.

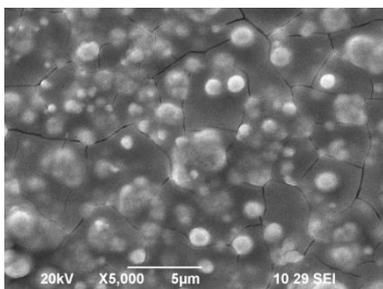


Figure 6. SEM Micrograph of the Ni+Zn+Mn coated surface at magnification of X5000, 5 μm

Fig. 6 SEM micrograph explains the distribution of Ni, Zn and Mn particles on the surface of base material. Ni, Zn and Mn particles are spread over the base surface producing a frame work of a number of cotton balls spread densely on the surface. Comparing to the uncoated surface of the base substrate, the Ni, Zn and Mn particles are evenly distributed on the surface and less wear resistivity than the Ni and Mn

coating which having very less wear of 19 μm was recorded in the experiment.

B. Elemental compositions of coated composites study using energy dispersive X-ray diffraction (EDS)

The elemental composition of the specimens was studied using EDS analyses and the results show that the corresponding peaks to confirm the presence of elements like Ni, Mn and Zn in the specimen. In all the samples, theoretical values are correlated with the experimental results.

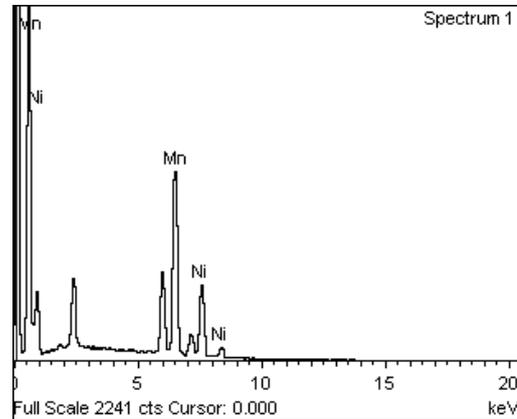


Figure 7. EDX result for Ni+Mn coated substrate

TABLE VI. Ni and Mn elemental composition

Element	App. Conc.	Intensity Corr.	Weight %	Weight % Sigma	Atomic %
Mn K	15.87	1.0452	41.90	1.21	43.53
Ni K	20.21	0.9599	58.10	1.21	56.47
Totals			100.00		

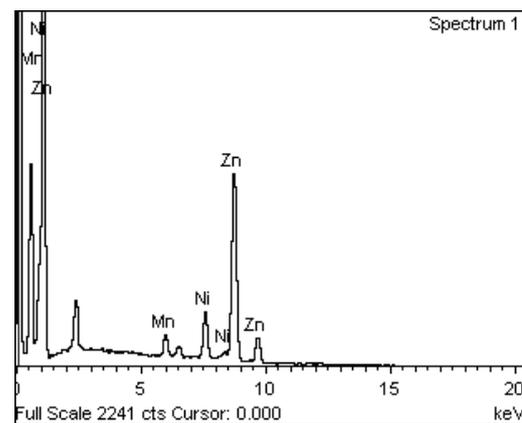


Figure 8. EDX result for Ni+Zn+Mn coated substrate

TABLE VII. Ni, Zn and Mn elemental composition

Element	App. Conc.	Intensity Corr.	Weight %	Weight % Sigma	Atomic %
Mn K	3.52	1.0688	5.51	0.45	5.99
Ni K	9.29	1.2589	11.55	0.70	12.57
Zn K	51.93	0.9752	83.31	0.81	81.44
Total			100.00		

C. X-ray diffraction Characterization

In order to understand the structural properties of the specimens XRD were performed with $\text{CuK}\alpha$ wavelength and scanning in the ranges from 10 to 90 degree. The sharp and well defined Bragg peaks indicate that the coated materials are in crystalline in nature. All the observed diffraction peaks are corresponds to the formation of metal coating on the surface of the specimen.

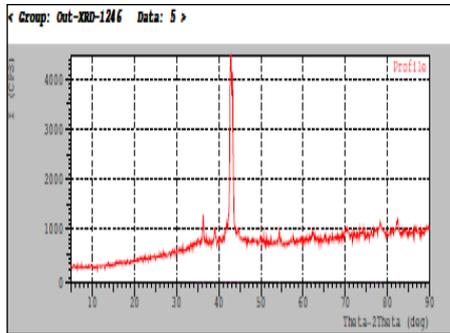


Figure 9. Indication of coated materials are in crystalline in nature

D. Hardness test result

Table 8 shows that hardness value of Ni+Mn coated sample have high hardness value of 77.25 HRB than the uncoated sample, and Ni+Zn+Mn composition.

TABLE VIII. Hardness value

S.No	Material	Hardness value (HRB)
1.	Uncoated sample	65
2.	Ni	75
3.	Ni+Zn	71.75
4.	Ni+Mn	77.25
5.	Zn+Mn	72.5
6.	Ni+Zn+Mn	75.3

E. Wear results

Pin-on-disc test was utilized to observe the tribological properties of the substrate. The experiment was as per ASTM G99-95 standards at the speed of 300 rpm for 6 minutes under a load of 10 N. Using acetone the disc was cleaned before conducting the test. The substrates wear, coefficient of friction and frictional force are given in fig. 10 to 15. In this regard, the aim of this work is to find the important combination of factors influencing the process to achieve the minimum wear and co-efficient of friction.

The wear test, the graph represents the amount of wear (y axis) with respect to increasing time (x axis). From the Fig. 10 to 15, the wear rate is directly proportional to the time i.e., when the time is increase, the wear rate of the uncoated and coated substrates also increase. For the composite coated substrate, the result gave the less wear compared to the uncoated substrate. From the table 9 weight variation of before and after the wear test of Ni, Ni+Zn, Ni+Mn, Zn+Mn, Ni+Zn+Mn coated substrates to find the wear loss as 0.01g, 0.02g, 0.01g, 0.007g, 0.01g with respectively.

TABLE IX. Data of wear loss of composites coated samples

S.No	Samples	Weight (g)	
		Before test	After test
1.	Ni	19.450	19.440
2.	Ni+Zn	19.620	19.600
3.	Ni+Mn	19.290	19.280
4.	Zn+Mn	20.120	20.113
5.	Ni+Zn+Mn	19.390	19.380

Uncoated sample's wear, coefficient of friction and frictional force for 6 minutes are shown in Fig 10. It is inferred that, when the time was increasing, the amount wear also increased up to 49 μm . Nickel coated sample's wear, coefficient of friction and frictional force for 6 minutes are shown in Fig. 11. The graph represents, the amounts wear increasing up to 22 μm at first 260 seconds. Then afterwards with time increasing, wear hikes to 34 μm . The coefficient of friction gradually decreased from 0.8 to 0.55 and frictional force maintained at 5.8 N.

Nickel and zinc coated sample's wear, coefficient of friction and frictional force with respect to 6 minutes are presented in Fig 12. The graph represents, the amounts wear increasing up to 40 μm at first 180 seconds. Then afterwards on increasing time, wear hikes into 45 μm . The coefficient of friction gradually decreased from 0.75 to 0.5 and frictional force maintained at 5.5 N.

Nickel and manganese coated sample's wear, coefficient of friction and frictional force for 6 minutes are shown in Fig. 13. The graph represents, the amount of wear increasing up to 12 μm at first 180 seconds. Then after the time increasing, wear hikes into 19 μm . Coefficient of friction gradually decreased from 0.55 to 0.5 and frictional force remained at 5.5 N. It is having more wear resistance than other coatings.

Zinc and manganese coated sample's wear, coefficient of friction and frictional force with respect to 6 minutes as shown in Fig. 14. The graph represents, the amounts wear increasing up to 30 μm at first 180 seconds. Then after the time increasing, wear hikes into 38 μm . Coefficient of friction gradually decreased from 0.8 to 0.6 and frictional force also decreased from 8 N to 6 N.

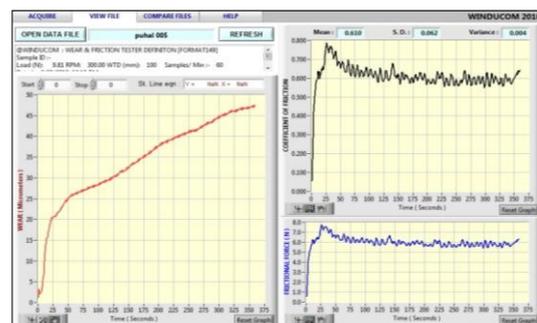


Figure 10. Wear, coefficient of friction and frictional force for the uncoated sample

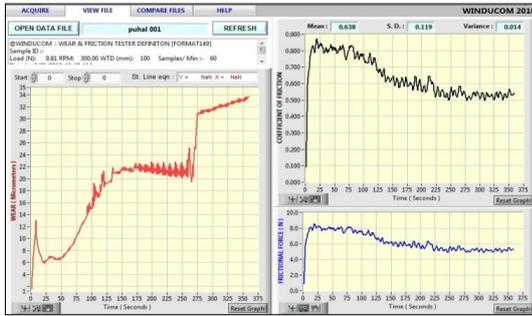


Figure 11. Wear, friction coefficient and frictional force for pure Ni coated sample

Nickel, zinc and manganese coated sample's wear, coefficient of friction and frictional force are shown in Fig. 15. The graph represents, the amounts wear increasing up to 35 μm at first 180 seconds. Then after the time increasing, due to the resistance layer formation, wear was maintained at 36 μm . Coefficient of friction gradually decreased from 0.8 to 0.5 and frictional force also decreased from 8 N to 5 N.

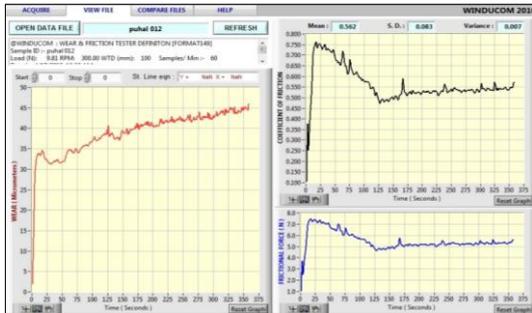


Figure 12. Wear, coefficient of friction and frictional force for Ni+Zn coated sample

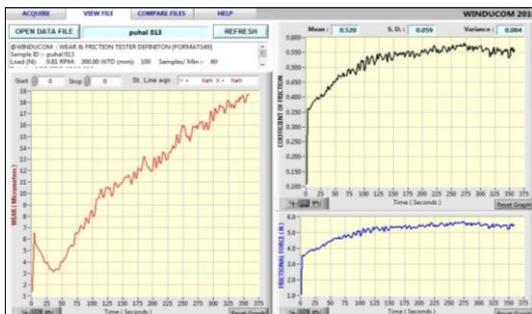


Figure 13. Wear, friction coefficient and frictional force for Ni+Mn coated samples

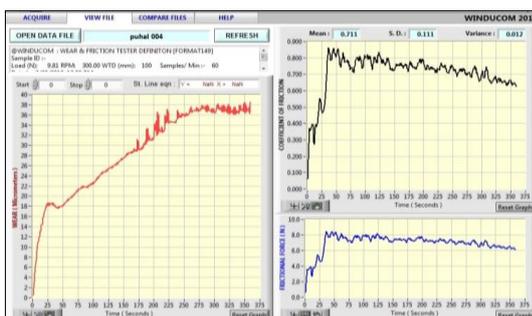


Figure 14. Wear, friction coefficient and frictional force for Zn+Mn coated samples

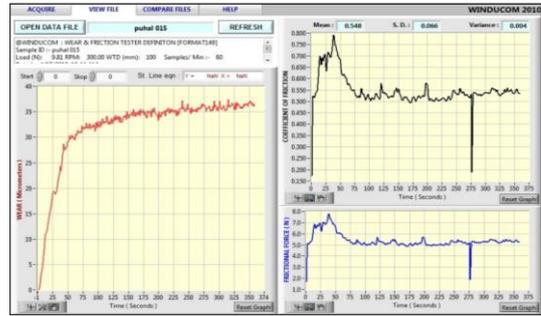


Figure 15. Wear, friction coefficient and frictional force for Ni+Zn+Mn coated samples

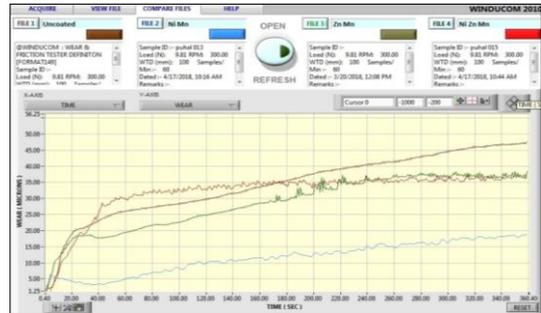


Figure 16. Comparison of frictional wear among uncoated, Ni+Mn, Zn+Mn and Ni+Zn+Mn coated samples



Figure 17. Comparison of coefficient of friction among uncoated, Ni+Mn, Zn+Mn and Ni+Zn+Mn coated samples

Fig. 16 represents the comparison of wear among uncoated, Ni+Mn, Zn+Mn and Ni+Zn+Mn coated samples. From that, Ni+Mn and Ni+Zn+Mn coated samples having high wear resistance than the remaining uncoated, Zn+Mn composite coated samples.

Fig. 17 represents the comparison of coefficient friction among uncoated, Ni+Mn, Zn+Mn and Ni+Zn+Mn coated samples. From that, Ni+Mn and Ni+Zn+Mn coated samples having less coefficient of friction by comparing with remaining uncoated and Zn+Mn composite coated samples, which having a high coefficient of friction.

IV. CONCLUSION

Composite coatings were carried out on mild steel by using electrodeposition technique. SEM micrograph shows even distribution of Ni, Zn and Mn particles on the surface of the base material. EDX analysis shows that the corresponding peaks to confirm the presence of elements like Ni, Mn and Zn in the specimens. XRD studies reveal that, the sharp and well defined Bragg peaks indicate that the coated materials are in crystalline in nature. From the hardness test, compared with uncoated sample, Ni+Mn and Ni+Zn+Mn coated samples having the high hardness value. Thus, the composite coated sample's hardness was improved.

Wear characteristics of the composite coated mild steel are investigated. All the coated samples had better wear resistance. On both coated and uncoated substrates, tribological test was conducted. From the wear test results, comparing with uncoated sample, Ni, Zn and Mn coating had less wear resistivity than the Ni and Mn coating, which having very less amount wear was recorded in the experiment. Thus the coated sample proved that, it has improved wear resistance. This enhancement is due to the Ni+Mn and Ni+Zn+Mn composite coatings on the mild steel and the evidenced better wear resistance shown in the wear test result.

V. ACKNOWLEDGEMENT

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