



Experimental and Analytical Investigation of Electrodeposited Ni-Al₂O₃ Composite Coatings

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Abstract: Electrodeposited composites are gaining importance for their advantages including low cost, ease and simplicity of operation. Composite electroplating is a method of co-depositing fine particles of metallic, nonmetallic compounds or polymers in the plated layer to improve material properties such as wear resistance, hardness, lubrication, or corrosion resistance. Electrodeposition has been identified to be a technologically feasible and for many applications, economically superior technique for the production of nanocrystalline materials. With increasing availability of micron-sized particles of Al₂O₃, there is growing interest in the electrolytic and electroless co-deposition of these particles. In this research work an attempt is made to produce electrodeposited Ni – Al₂O₃ coating on a mild steel substrate by electroplating process. By adjusting the plating parameters such as current density, pH value of the electrolyte bath, amount of Al₂O₃ particles in bath, agitation speed and temperature of the bath, electroplating is carried out for the investigation. The mass of deposit for various Ni- Al₂O₃ composite coated specimens at different plating parameters are measured experimentally. The plating parameters and their mass of the effects on the deposited coatings were investigated by using neural networks technique.

1.0 Introduction

Electrodeposited composite coatings consist of a metal matrix with either a ceramic or cermets particle addition which represents the new development in the field of coating processes. Electro-composite coating is a co-deposition of a homogeneously dispersed second phase material on the surface of the bulk material in the form of a particulate material, whisker, and fibre in a metal matrix with improved or new engineering properties. Inert particles such as diamond, powdered ceramics (for example aluminum oxide and silicon carbide) or polytetrafluoroethylene (PTFE or Teflon) can be deposited on the nickel matrix, forming a composite coating with enhanced wear resistance. Electrodeposition has been identified to be a technologically feasible and for many applications, economically superior technique for the production of nanocrystalline materials. The objective of giving a coating to a substrate is to enhance Engineering and physical properties of the substrate material or to obtain coating technology has been used extensively in many manufacturing areas such as coatings for engine cylinders, Textile mill rollers, Small size cams and gears.

In this work an attempt is made for preparation of Ni-Al₂O₃ composite coatings by adjusting the plating parameters such as current density, pH value of the bath, Amount of Al₂O₃ particles in the bath, Agitation speed and temperature of the bath. The mass of deposit for various Ni- Al₂O₃ composite coated specimens at different plating parameters are measured experimentally. A neural network model is also developed for the prediction of mass of deposit in the coated specimens for various plating parameters.

2.0 literature survey

Begona Ferrari et al [1] reported that ceramic coatings are often used to improve the surface behavior of metals, thus allowing to be used in high temperature applications, such as thermal barrier coatings etc., One of the most widely studied metal – ceramic systems is the Ni – Al₂O₃, due to the high refractoriness and stability of nickel. Chan et al [2] studied, the discontinuously reinforced metal-matrix composites are attractive for their high elastic modulus and strength, good wear resistance, and good dimensional stability. Clark et al [3] have found that most techniques to produce nanostructure powder, thin films or bulk materials operate at conditions which are conducive to the formation of non- equilibrium structures.. Clark have suggested that the initial capital investment to produce nanostructure materials by electrode position is low and often requiring only minor modifications to existing conventional plating lines. Scale-up is relatively easy, and high production rates can be achieved. The costs for nanostructure plating are comparable to those of conventional electrode position processes as the bath constituents are essentially the same. Clint and Michae [4] reported that surface coatings have gained tremendous popularity over the past several decades and are prevalent in a large number of industries which include aerospace, automobile, computer, machining and precision manufacturing industries. Grosjean et al [5] concluded from the experimental results that the hardness of nickel increases with increasing the rate of incorporation of articles until a maximal value. In addition, it has been found that hardness increases with increasing SiC particle size. Hagedorn and Weinert [6] mentioned that metal matrix composites (MMCs) are very often used to combine low structural weight with high wear resistances in the component development of automobiles... Hongzhi et al [7] suggested that the composite electroplating technique is one of the most advanced production routes of functionally graded materials (FGM) The major advantages of this method over other techniques are the simplicity to control, possibility to process complicated parts, and the low initial capital investment. The Ni/SiC gradient deposit is prepared by the composite electro deposition method. Hou et al[8] determined from the experimental results that the contents of submicron-size SiC particles in the deposition layer increased with the increasing concentration of both the SiC and the surfactant Cetyl trimethyl ammonium bromide (CTAB) in the electrolyte. Huang [9] reported that electro less nickel (EN) coatings are well known for their wear and corrosion resistance. Co deposition of hard or lubricant particles into the EN to form a composite coating can further improve the tribological properties of the coating and also add on additional functions. Junhong jia et al[10] reported that Ni-based composites can be designed with the capability of having self-lubricating property in a wide temperature range, for example, the turbine engines used in aviation and electric industries, as well as the radiator sealing systems of the automobile engines. Kezheng and Zhihao Jin [11] reported that oxide formed on nickel alloy surface could provide a lubricating function. Another research for SiC TiC/SiC–TiC and SiC–TiB₂/SiC–TiB₂ showed that oxide of titanium has been formed on friction surface, but friction coefficient was still high except that of SiC–TiC/SiC–TiC at room temperature. Kılıc kap et al [12] reported that aluminium, titanium and magnesium alloy can be used as a metal matrix element and the popular reinforcements are silicon carbide (SiC) and alumina (Al₂O₃). Aluminium-based SiC particle reinforced MMC materials have become useful engineering materials due to their properties such as low weight, heat-resistant, wear-resistant and low cost. Medeliene [13] found that non-metallic inclusions in the metal can change its electron structure and break up the crystal lattice, resulting in changes in the physical and mechanical properties of the electrodeposits. Ming-derger[14] found that, however, the reduction of particle size will decrease the co deposition content of the particles. SiC particles, the amount incorporated during rotating disc experiments in a nickel sulfate solution never exceeded 0.7 weight percentage. Similar results reported by Ming-Der Ger that the highest volume fraction of co deposited SiC have been achieved with 5 µm particles, rather than with 0.7 or 0.3 µm particles. In addition, for a Ni–Al₂O₃ composite, the amount of co deposited Al₂O₃ of 10 µm particle size has been found as four times larger than that of Al₂O₃ of 0.3 µm in the composite coating. Xu Jianga et al [15] investigated that corrosion resistance of brush plated alloy Ni–Fe–W–S coating is superior to that of electrodeposited chromium and a better wear resistance than that of electrodeposited chromium at high speed and heavy load under normal conditions where lubricant was applied between the contact surfaces.

3.0 Al₂O₃ as Composite Particles

The different composite particles normally used are Silicon carbide, Silicon nitride, Boron carbide Diamond particles & aluminium oxide for improved wear resistance. The reasons for chosen the aluminium oxide particles as composite material in the above electro deposition is due to the following favorable reasons.

- a) Excellent Heat and Wear resistance.
- b) Hard Ceramic Material.

- c) C.Provides good adherent with metal matrix.
- d) Cheap and ease of availability.
- e) Good chemical stability.

4.0 Experimental Setup

The Electro deposition as a method of Composite coating is chosen in this work due to the following significant features of the Electroplating process.

The experimental set up consists of a glass beaker containing the Watt's bath electrolyte solution along with two electrodes namely, a Nickel plate as anode and a mild steel plate as cathode (Substrate material) with the size of 75mm X 25.4mm X 1.8mm. The Nickel anode and mild steel plate cathode are connected to a 5 Volts 10 Amps stabilized D.C Power supply. A small amount of surfactant is also assorted in the bath to avoid coherence of Al₂O₃ particles. The Watt's Nickel Bath contains the following:

Nickel Sulphate = 250 gpl
Nickel Chloride = 30 gpl
Boric Acid = 40 gpl
Surfactant = 0.02 m



Fig .1. Electroplating Experimental Setup

The Al₂O₃ composite particles of 5 microns size is also assorted in the above Watt's Bath electrolyte and continuously agitated to maintain the composite particles always in suspension in the electrolyte solution by mechanical stirrer.

The plating parameters such as pH value of the bath, Current density, rotational agitation speed and Al₂O₃ amount in the bath and temperature of the electrolyte are continuously monitored with appropriate instruments. The mass of the deposition of composite material in the substrate is measured by electronic balance weighing unit.

6.0 Electroplating Parameters

The experimental trails are taken for different set of readings between the minimum and maximum values given in the table no.1. Rotational speed of the stirrer is kept at constant Rotational speed of 300 rpm and unit size of the particles Al₂O₃ is taken as 5micron size for the entire experiment.

The minimum and maximum ranges of plating parameters under current investigation are follows:

Table.1 Bath parameters.

Bath parameters	Symbol	Range	Units
Current density	X ₁	1- 4	A /dm ²
pH Value of bath	X ₂	2.5 -4.5	---
Temperature of bath	X ₃	30-60	°C
Powder (Al ₂ O ₃) concentration in the bath	X ₄	20	Grams /Lit. (Gpl)
Rotational speed of Mechanical stirrer	X ₅	300	RPM

Table.2 Experimental trials

Sl. No.	X1	X2	X3	X4	X5	Mass of deposit in mg
1	1	3	30	20	300	88.5
2	2	3	30	20	300	112.9
3	3	3	30	20	300	180.6
4	1	3	45	20	300	91.6
5	2	3	45	20	300	116.6
6	3	3	45	20	300	184.6
7	1	3	60	20	300	98.6
8	2	3	60	20	300	124.5
9	3	3	60	20	300	193.5
10	1	3.5	30	20	300	91.5
11	2	3.5	30	20	300	126.5
12	3	3.5	30	20	300	190.3
13	1	3.5	45	20	300	94.6
14	2	3.5	45	20	300	129.4
15	3	3.5	45	20	300	188.8
16	1	3.5	60	20	300	103.8
17	2	3.5	60	20	300	135.2
18	3	3.5	60	20	300	199.3
19	1	4	30	20	300	89.1
20	2	4	30	20	300	120.5
21	3	4	30	20	300	184.2
22	1	4	45	20	300	92.7
23	2	4	45	20	300	125.8
24	3	4	45	20	300	186.6
25	1	4	60	20	300	99.5
26	2	4	60	20	300	130.8
27	3	4	60	20	300	196.7

8.2 Effect of Current Density

The mass of the deposit is studied at three different current densities i.e. at 1, 2 and 3. The variation of mass of the deposit with current densities at different pH values are given below.

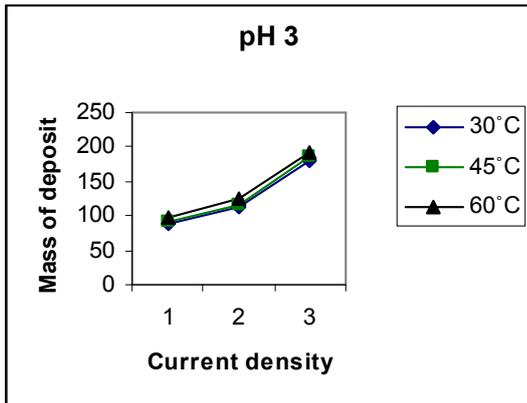


Fig.2.Current density in A/dm² Vs Mass of deposit at pH 3

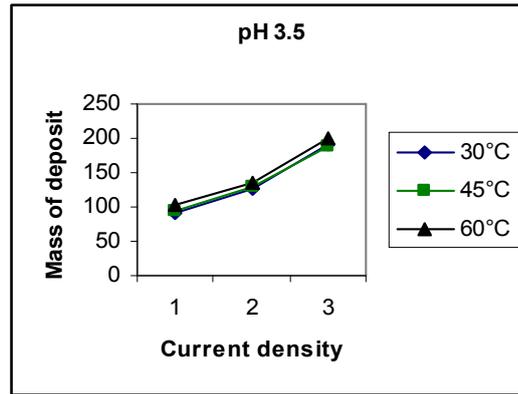


Fig.3.Current density in A/dm² Vs Mass of deposit at pH 3.5

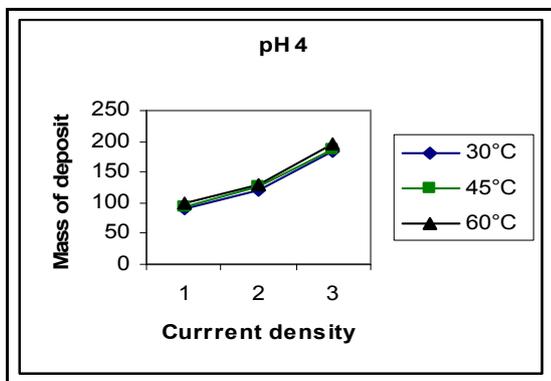


Fig.4.Current density in A/dm² Vs Mass of deposit at pH 4

It is evident from the graph that the mass of the deposit increases with current density. The maximum deposit is obtained when the current density value is maintained at three. In all the above graphs the variation of mass of deposit with current density is plotted at three different temperature values i.e. at 30°C , 45°C and 60°C .The deposition is maximum at 60°C. It is known from figure3, that the deposition is maximum when the pH value is 3.5.Based on the variation of mass of the deposit with current density, as shown in figure, the optimal value occurs when

Current Density = 3 A/dm²
 Temperature = 60°C
 pH = 3.5.

The corresponding maximum value of mass of the deposit is 199.3.

8.3 Effect of Ph

The effect of coating is studied at three different pH values i.e. at 3, 3.5, and 4. The variation of mass of the deposit at different pH values are shown in the graph.

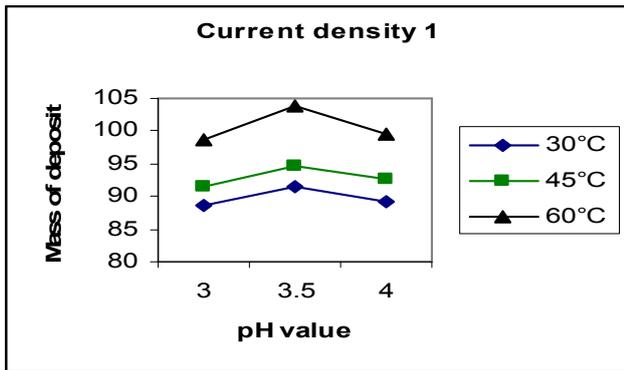


Fig.5.pH Vs Mass of deposit in milligram at Current density 1in A/dm²

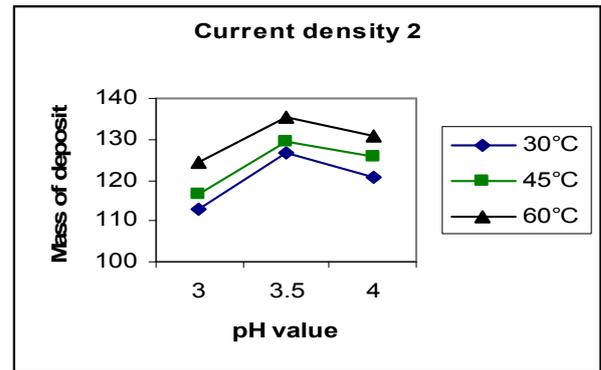


Fig.6.pH Vs Mass of deposit in milligrams at Current density 2 A/dm²

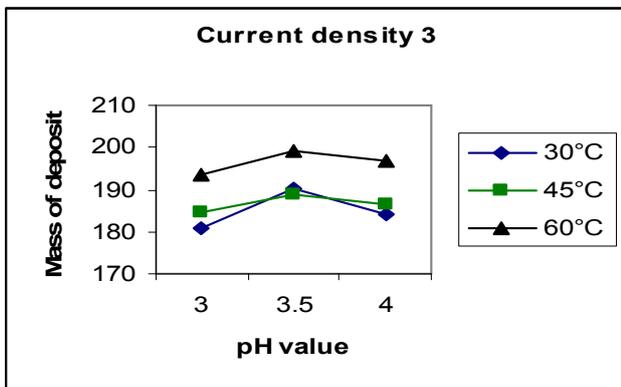


Fig.7.pH Vs Mass of deposit in milligrams at Current density 3 A/dm²

The maximum deposition is obtained when the pH is maintained at 3.5 for a given value of current density and temperature.

8.4 Effect of Temperature

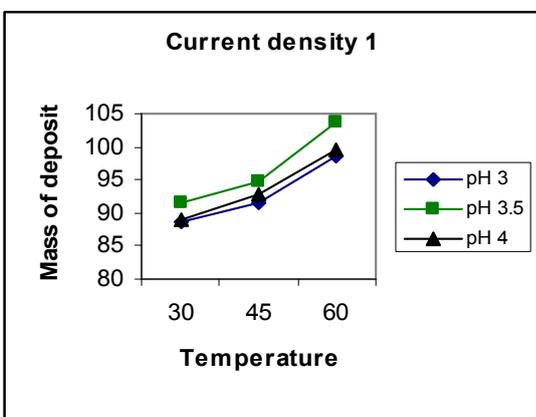


Fig.8.Temperature in °C Vs Mass of deposit

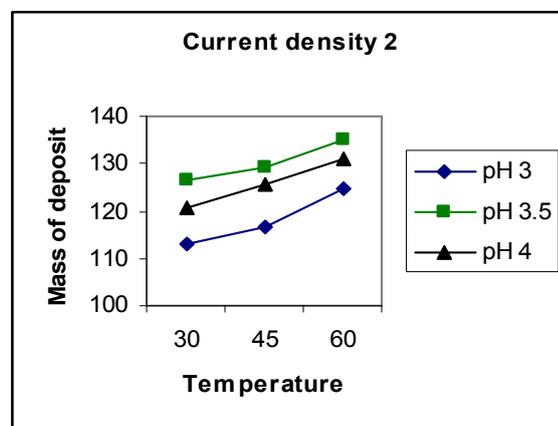


Fig.9.Temperature in °C Vs Mass of deposit at Current density 2 A/dm²

at Current density 1 A/dm²

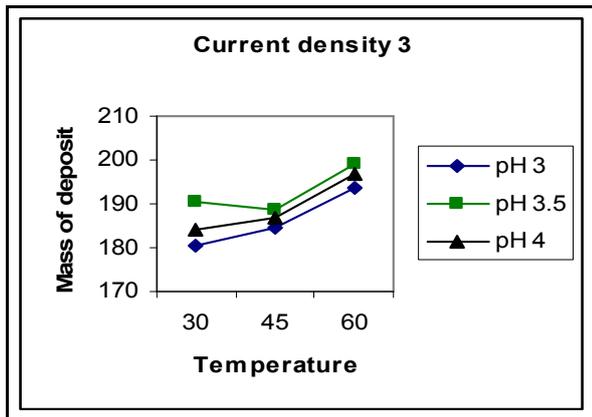


Fig.10. Temperature °C Vs Mass of deposit at Current density 3 A/dm²

8.5 Micro Structure Of Ni-Al₂O₃ Composite Coatings Using SEM

Figure 11 to Figure 12 shows the surface morphology of coating with various magnifications. It can be seen that incorporation of Al₂O₃ particle in Nickel matrix with the mentioned plating parameters

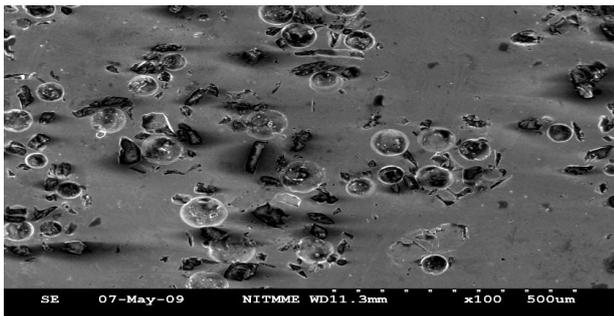


Figure. 11. SEM photo-100X Current-2 A/dm², pH 3.5, Temperature- 30°C Powder size-5 micron at 10 gpl of Al₂O₃(Magnification-100X)

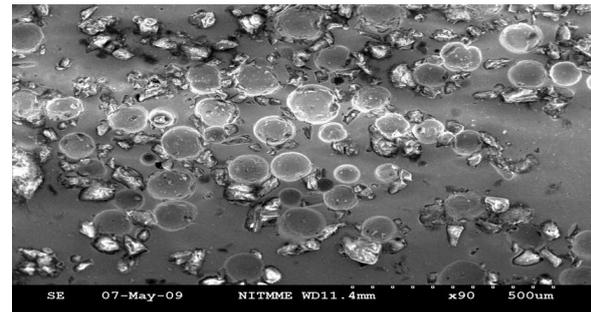


Figure .12. SEM Photo-90X Current-2 A/dm², pH- 3.5, Temperature- 45°C Powder size-5 micron at 10 gpl of Al₂O₃ (Magnification-90X)

8.6 Artificial neural network

ANN is an information processing system and consists of a large number of very simple in a highly interconnected processor called neurodes or neurons. The neurodes perform in a manner that it is analogous to the most elementary function of the biological neuron (of brain). The neurodes are connected to each other by a large number of weighted links. Other which signal can pass each neurodes may receive one or more input signal. ANN exhibits surprising number of brains characteristic. They learn from experience, generalized from previous examples to new ones and abstract essential characteristics from inputs containing irrelevant data. ANN's can modify their behavior in response to their environment. Shown as a set of inputs (and desired output if available). They self adjust to produce consistent response, based on the training they received. A wide variety of training algorithms have been developed. .

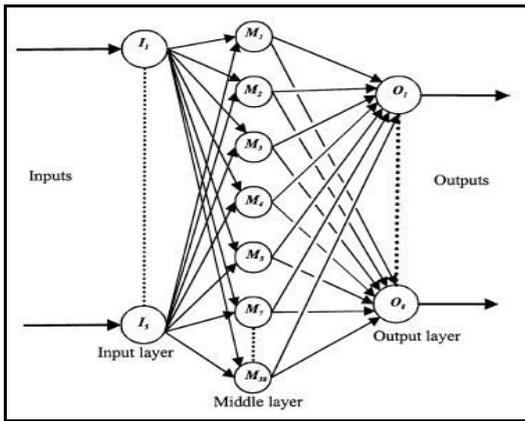


Fig.13.Model Architecture of Neural Network

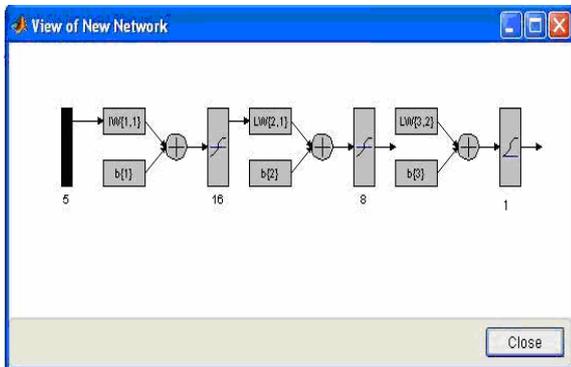


Fig.14..Feed Forward Back Propagation Network Architecture

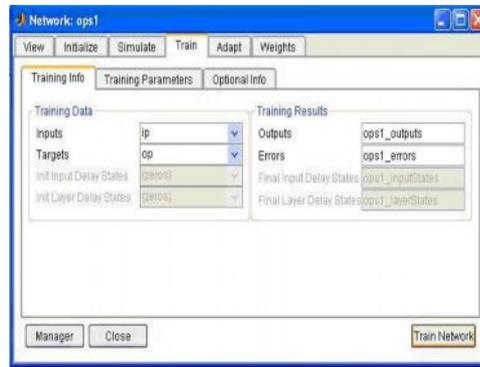


Fig.15.Selections of Parameters for Training

8.7 Normalized input parameters

Table.3 Normalized input parameters

Specime n No.	Current Density	pH	Temper ature	Bath Conc.	Speed	Mass of Deposit
1	0.1	0.1	0.1	1	1	0.10000
2	0.14	0.1	0.1	1	1	0.11762
3	0.18	0.1	0.1	1	1	0.16650
4	0.1	0.1	0.14	1	1	0.10224
5	0.14	0.1	0.14	1	1	0.12029
6	0.18	0.1	0.14	1	1	0.16939
7	0.1	0.1	0.18	1	1	0.10729
8	0.14	0.1	0.18	1	1	0.12599
9	0.18	0.1	0.18	1	1	0.17581
10	0.1	0.14	0.1	1	1	0.10217
11	0.14	0.14	0.1	1	1	0.12744
12	0.18	0.14	0.1	1	1	0.17350
13	0.1	0.14	0.14	1	1	0.10440

14	0.14	0.14	0.14	1	1	0.12953
15	0.18	0.14	0.14	1	1	0.17242
16	0.1	0.14	0.18	1	1	0.11105
17	0.14	0.14	0.18	1	1	0.13372
18	0.18	0.14	0.18	1	1	0.18000
19	0.1	0.18	0.1	1	1	0.10043
20	0.14	0.18	0.1	1	1	0.12310
21	0.18	0.18	0.1	1	1	0.16910
22	0.1	0.18	0.14	1	1	0.10303
23	0.14	0.18	0.14	1	1	0.12693
24	0.18	0.18	0.14	1	1	0.17083
25	0.1	0.18	0.18	1	1	0.10794
26	0.14	0.18	0.18	1	1	0.13054
27	0.18	0.18	0.18	1	1	0.17812

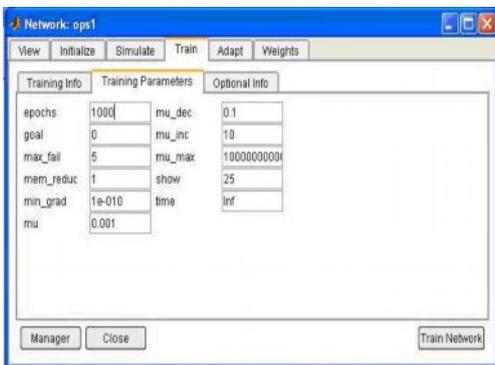


Fig.16.Parameter Setting Tool

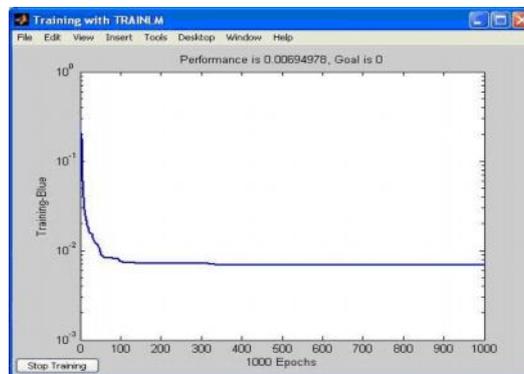


Fig.17.Training Response Chart

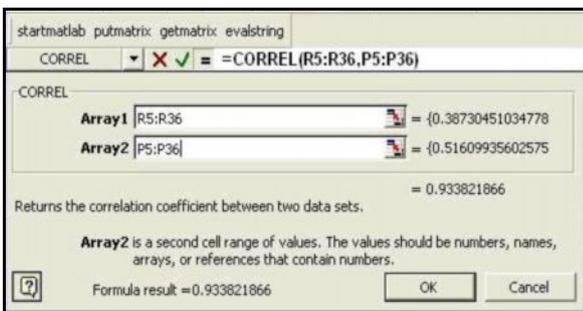


Fig.18.ANN Training Results

8.8 Output results of experimental method and ANN model

The following table shows the Comparison results of Volume fraction of Al₂O₃ in deposit and hardness analyzed by experimental method and neural network model

Table.4.Comparisons of Results

Specimen No.	Experimental Out put	ANN out put
	Mass of Deposit	Mass of Deposit
1.	0.100000	0.093382
2.	0.11761733	0.109834
3.	0.16649819	0.15548
4.	0.10223827	0.095472
5.	0.12028881	0.112328
6.	0.16938628	0.158177
7.	0.10729242	0.100192
8.	0.12599278	0.117655
9.	0.17581227	0.164177
10.	0.10216606	0.095405
11.	0.12743682	0.119003
12.	0.17350181	0.16202
13.	0.10440433	0.097495
14.	0.12953069	0.120959
15.	0.17241877	0.161008
16.	0.11104693	0.103698
17.	0.13371841	0.124869
18.	0.180000	0.168088
19.	0.10043321	0.093787
20.	0.12310469	0.114958
21.	0.16909747	0.157907
22.	0.10303249	0.096214
23.	0.12693141	0.118531
24.	0.17083032	0.159525
25.	0.10794224	0.100799
26.	0.13054152	0.121903
27.	0.17812274	0.166335

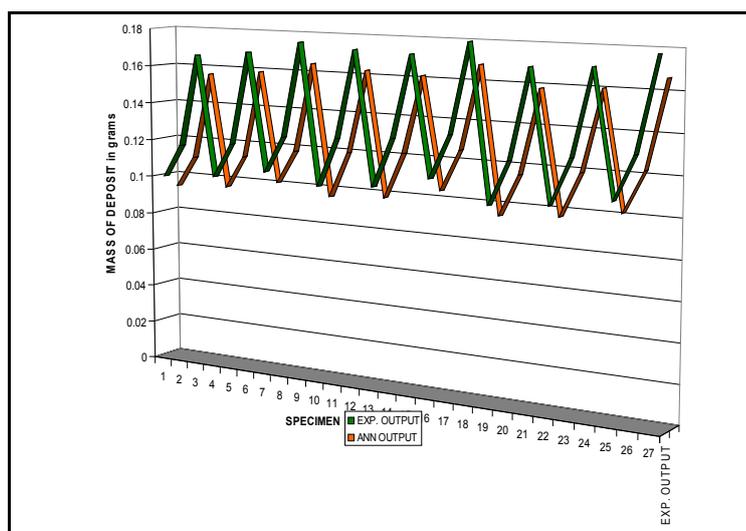


Fig.19. Comparison chart for Experimental and ANN results

Conclusions

The above neural network model is developed based on 5 plating parameters such as pH value of the bath, current density, rotational agitation speed, temperature of the bath and amount of aluminium oxide particles in the bath only. It is also noted that the experimental and neural network model developed is only for prediction of mass of deposit.

Based on those results, the following conclusions have been made. The optimum conditions for plating are found to be,

Current Density = 3 A/dm²
 Temperature = 60°C
 pH = 3.5

The mass of the deposit is increased with the increase in current density. Also the mass of the deposit is increased with increase in pH up to the value of 3.5 and then decreases above the level. The corresponding optimal value of mass of the deposit is 199.3 milligrams.

The mass of deposit of Ni - Al₂O₃ composite coatings is predicted by ANN is found to be of which is closer to that obtained by experimental output results and its correlation coefficient is 0.933821 .

In future, it is also possible to include few more variables of electrodeposition such as time of deposition, volume of the bath, size of the Al₂O₃ particles etc, can also be included and models can also be developed for prediction of wear index, roughness, micro hardness etc., By conducting more and more experiments, it is possible to enhance the accuracy of the predicted value by training the neural network in a more systematic way.

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