

Copyright Undertaking

This thesis is protected by copyright, with all rights reserved.

By reading and using the thesis, the reader understands and agrees to the following terms:

1. The reader will abide by the rules and legal ordinances governing copyright regarding the use of the thesis.
2. The reader will use the thesis for the purpose of research or private study only and not for distribution or further reproduction or any other purpose.
3. The reader agrees to indemnify and hold the University harmless from and against any loss, damage, cost, liability or expenses arising from copyright infringement or unauthorized usage.

If you have reasons to believe that any materials in this thesis are deemed not suitable to be distributed in this form, or a copyright owner having difficulty with the material being included in our database, please contact lbsys@polyu.edu.hk providing details. The Library will look into your claim and consider taking remedial action upon receipt of the written requests.

THE HONG KONG POLYTECHNIC UNIVERSITY

Department of Mechanical Engineering

Hard Chrome Plating
for
Ring Grooves of Piston Crown

By

Andrew Ning

Supervisor: Dr. T.T. Wong

Co-Supervisor: Prof. C.W. Leung

A Thesis submitted in partial fulfillment of the
requirements for the Degree of Master Philosophy at the
Hong Kong Polytechnic University

November, 2003



Pao Yue-kong Library
PolyU • Hong Kong

CERTIFICATE OF ORIGINALITY

I hereby declare that this thesis is my own work and that, to the best of my knowledge and belief, it reproduces no material previously published or written nor material which has been accepted for the award of any other degree or diploma, except where due acknowledgement has been made in the text.

Andrew Ning

ACKNOWLEDGEMENTS

The author wishes to thank **Dr. T.T. Wong, Prof. C.W. Leung** and **Mr. Harvey Wong**. These are the people who encourage my continual growth in all facets of my life. The author also wishes to thank the Innovation and Technology Commission of the Government of the Hong Kong Special Administrative Region and Brigantine Services (Hong Kong) Limited for the financial support of the project. (ITF Project Reference. UIT/010)

TABLE OF CONTENTS

Table of Contents i
List of Figures ii
List of Tables iii
Abstract iv
1. Introduction 1
1.1 Background	
1.2 Objectives of study	
1.3 Layout of thesis	
2. Literature review 9
2.1 Process variables of hard chrome plating	
2.1.1 Bath composition	
2.1.2 Current density	
2.1.3 Throwing power	
2.2 Process modeling and prediction - statistical approach	
2.3 Process modeling and prediction - artificial intelligence approach	
2.4 Proposed approach	
3. Design of pilot plant for rotary plating system 27
3.1 The original hard chrome plating system in the teaching company	
3.2 Practical constraints of hard chrome plating system in the teaching company	
3.3 Design of the pilot plant	
4. Construction of experimental setup 39
5. Process modeling and prediction by Full Factorial Design (FFD) approach 42
5.1 The influential factors for the hard chrome plating process	
5.1.1 Bath composition	
5.1.2 Temperature setting	
5.1.3 Current density	
5.1.4 Plating time	
5.1.5 Rotary speed	
5.2 Experimental design	
5.3 Experimental procedure	
5.4 Analysis of experimental data	
5.5 Multiple regression analysis	

5.6	Multiple coefficients of correlation	
5.7	Model adequacy diagnosis	
5.7.1	Normal probability plot of residuals	
5.7.2	Interaction analysis	
5.7.3	Outlier analysis	
5.7.4	Influential cases	
5.7.5	Leverage analysis	
5.8	Verification of FFD results	
6.	Process modeling and prediction by Neural Network (NN) approach	75
6.1	NN approach for process modeling and prediction	
6.1.1	Types of NN models	
6.1.2	Back-propagation NN model architecture	
6.1.3	The learning ability of NN model	
6.1.4	The advantage of using NN model for process modeling and prediction	
6.2	The development of NN model for process modeling and prediction	
6.2.1	Training of NN model	
6.2.2	Testing and validation of NN model	
7.	Discussion of results	93
7.1.	Comparison of FFD and NN methods	
7.2.	Discussion on research methods	
7.3.	Effect of impurities	
7.4.	Effect of solution conductivity	
7.5.	Effect of chromic/sulphuric acid ratio	
7.6.	Limitations of study	
7.6.1.	Limitation of time	
7.6.2.	Limitation of NN	
8.	Conclusion	103
9.	Suggestions for future work	106
	References	108
	Appendices	
Appendix A	Details of the assembly process of the rotary plating machine parts	113
Appendix B	Details of the commissioning of the rotary plating machine	117
Appendix C	Determination of Chromium(VI) ion in hard chrome plating solution	122

Appendix D	Determination of Sulphate ion in hard chrome plating solution 123
Appendix E	The procedure for the training of a NN model using Qnet software 125
Appendix F	The data sets for the training, testing and validation of a NN model 130

LIST OF FIGURES

Figure 1:	A photo shows a sample piston crown of a diameter 900mm 5
Figure 2:	A photo shows the appearance and dimension of the piston crown ring grooves 6
Figure 3:	A photo shows a sample of the original static hard chrome plating system in the teaching company 6
Figure 4:	Flow chart for the proposed approach in this study 26
Figure 5:	Flow chart for the hard chromium plating process in the teaching company 29
Figure 6:	Dimensions of the ring grooves 31
Figure 7:	A sample CAD drawing of piston crown specification 31
Figure 8:	Construction of the piston crown and the electric anodes 32
Figure 9:	Construction of the piston crown and the supporting shaft 32
Figure 10:	The plating machine is about to be put into the plating tank 33
Figure 11:	The rinsing process 33
Figure 12:	A photo shows the appearance of ring grooves of the refurbished piston crown 34
Figure 13:	The primary current distribution on a rotary cylinder electrode with concentric counter electrode 37
Figure 14:	A machine drawing of the rotary plating machine 38
Figure 15:	The rotary plating machine is ready for action 41
Figure 16:	The effect of chromic acid concentration on current efficiency 43
Figure 17:	Flow chart for the experimental procedure 54

Figure 18:	The plot of plating thickness vs. actual run order 56
Figure 19:	A normal plot of residuals 65
Figure 20:	The interaction graph between chromic acid concentration and electric current 67
Figure 21:	The interaction graph between chromic acid concentration and sulphuric acid concentration 68
Figure 22:	The interaction graph between chromic acid concentration and plating time 69
Figure 23:	A plot between the Run numbers and the Outlier T 70
Figure 24:	A plot between the Run numbers and the Cook's distance 71
Figure 25:	A plot between the Run numbers and the Leverage values 72
Figure 26:	Back-propagation NN models with multi-layered architecture 79
Figure 27:	A schematic diagram for the training of NN model 90
Figure 28:	The transfer function used in the training of NN model.....	90
Figure 29:	The plot of RMS error after 300,000 iterations of the training of NN model 90
Figure 30:	A plot of the difference between the predicted (FFD) values vs. the actual values 95
Figure 31:	A plot of the difference between the predicted (NN) values vs. the actual values 95
Figure 32:	The side view of the rotary plating machine 113
Figure 33:	The back view of the rotary plating machine 113
Figure 34:	A closer view of the internal section of the rotary plating machine 114
Figure 35:	The bearing section and the carbon brush section of the rotary plating machine 114
Figure 36:	The piston holding section of the rotary plating machine 115

Figure 37:	The commissioning of the rotary plating machine 115
Figure 38:	The new design of the electric anode 116
Figure 39:	The appearance of the rotary plating machine 117
Figure 40:	Damage of the anode upon the completion of plating process 118
Figure 41:	The modified anode 119
Figure 42:	The final design of anode 121

LIST OF TABLES

Table 1:	Basic chromium plating baths 12
Table 2:	The influential factors and their ranges in this study 46
Table 3:	The number of runs for 2^k FFD method 47
Table 4:	The High (+1) and Low (-1) Setting for the experiment 48
Table 5:	A 2^4 two-level, FFD table showing runs in preset run order 50
Table 6:	A design table for a two-level FFD with four factors, replicated twice 52
Table 7:	A design table for a two-level FFD with four factors, replicated twice, with random run order indicated 53
Table 8:	A design table with the plating thickness of the hard chrome deposition as outcome 57
Table 9:	The design summary of the hard chrome plating process for a piston crown of diameter 900mm 59
Table 10:	Analysis Of Variance (ANOVA) for the initial FFD model 60
Table 11:	The calculation of R-squared coefficient 61
Table 12:	A comparison between the predicted (FFD) values vs. the actual values 63
Table 13:	A comparison between the values obtained from predicted (FFD) model vs. the ones obtained from the confirmation run 73
Table 14:	The configuration for the training of a NN model 91
Table 15:	Typical sample data for hard chrome plating process with the use of rotary plating machine 91
Table 16:	A comparison between the predicted (NN) values vs. the actual values 91
Table 17:	The average and maximum errors in the predictions by FFD and NN 95

Abstract

Today hard chromium plating is generally accepted in engineering as an invaluable means of prolonging the life of all types of metal parts subjected to wear by friction or abrasion. Such parts can be protected from new or they can be reconditioned to as good as new state when they are worn and would otherwise be scrapped.

At the commencement of this Innovation Technology Fund project, the chromium plating bath employed in the Teaching Company was a static system, which involved fixing the anode close to the surface of the work-piece while the work-piece was kept at the cathode. Electric current of appropriate strength was then applied and passed from the anode through the plating solution to the cathode. Chromium was then gradually deposited onto the unmasked surface of the work-piece. Thickness and quality of the chromium deposits depend on several operating parameters, viz. current density, temperature and concentration of the plating solution. Best electroplating result depends on the optimization of these operating parameters, which involves a very complicated analytic process. This project was aimed to improve the hard chrome plating process through (i) the development of a more efficient plating system and (ii) to predict plating thickness through the development of an empirical model between plating thicknesses and the input variables.

On completion of this study, a rotary electroplating system was constructed and predictions of the plating thickness were achieved by using the Full Factorial Design (FFD) and the Neural Network (NN) methods. The values obtained by both

methods were compared with the experimental values of the plating thickness to evaluate about the accuracy of the predictions.

Within the range of input variables for the present study, the results showed that NN came ahead of the FFD in nearness of the predictions to the experimental values of plating thickness. The average errors in the plating thickness in the case of NN were less than that obtained using FFD (average error is 1.5% for NN as compared to 4.4% in case of FFD predictions; maximum deviation is 4.2% for NN as compared to 9.8% in case of FFD predictions).

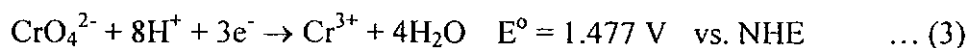
The information gained as well as the proposed methodology in this study contributes reliable and reproducible data on the absorption of chromium metal. A significant contribution has been made to provide in-depth understanding of the industrial electroplating process. Another significant contribution made to the present investigation is the development of a new approach in the hard chrome plating of ring grooves of piston crowns.

1. Introduction

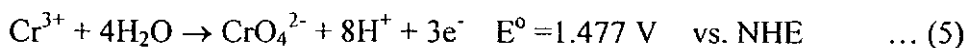
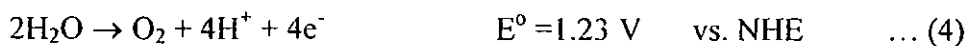
1.1 Background

Electroplated chromium is widely used in military and industry. The extreme hardness of the metal and its low coefficient of friction, combined with its corrosion resistance, make it particularly valuable as a coating where resistance to the various types of wear is important [Dubpernell, 1963; Shreir, 1976]. Thick functional deposits applied for this purpose are referred to as 'hard chrome' to distinguish them from thin decorative deposits [Dennis & Such, 1993]. Chromium plating is infamous for its low current efficiency (10-25%) and low throwing power.

The vast majority of decorative, and almost all hard, chromium plating is carried out using CrO_3 as the electrolyte. The electrochemical reactions of chromium are complicated, but the main reactions on cathode can be summarized [Griffin, 1971] as:



And those reactions occurring on the anode are:



where E° = Electromotive Force and the superscript refers the
standard state pressure 1 atmosphere

NHE = Normal Hydrogen Electrode at standard state pressure
1 atmosphere

More than 50% of electric charges are consumed by the unwanted hydrogen and oxygen evolving on the cathode and anode, respectively. The fact that chromium can be deposited from hexavalent (Cr^{6+}) solutions but not from simple aqueous solutions of salts is a disadvantage for the following reasons [Edwards, 1997; Greenwood, 1981]:

- Because the electrochemical equivalent of Cr in a CrO_3 solution is 0.3234 g/h and cathode current efficiency is typically 10% to 20%, the passage of current of one-hour yields only 0.032 to 0.64 gram of metal. This is 15 to 30 times less than nickel, and 18 to 36 times less than for copper from acid solution. The only way to offset this is to increase the working current density via increase in mass transport and temperature and/or plating time.
- The minimum current density at which electro-deposition takes place is two to three orders of magnitude larger than that in the case of other metals (Zn, Ni, Ag, etc.).

- ♦ The electro-deposition of chromium is more sensitive to operating conditions (temperature and current density) than any other deposition process.
- ♦ In contrast to other processes, the cathodic current efficiency varies inversely with temperature but is proportional to current density (which causes low throwing power).
- ♦ Chromium will plate only in the presence of a catalyst (e.g. H_2SO_4), whose concentration influences the plating rate.
- ♦ On the positive side, hexavalent chromium electrolytes are relatively less sensitive to the presence of impurities, and the anode material is usually lead or lead alloy, which can easily be made to conform to any shape.

Despite its paramount technological and with all the advances of modern science and instrumentation, the exact mechanisms of chrome plating are still open to considerable assumption. The chrome-plating bath, used for decorative and hard chrome baths, is still mostly of the type originally investigated by Sargent [1997]. It is the simplest plating bath to make up, and it consists of two essential ingredients:

- ♦ A water-soluble salt of chromium

- A small but critical amount of an anion, which acts as a catalyst. Such catalyst is supplied in the form of sulphuric acid alone or in combination with another acid radical, usually fluoride, fluoroborate or a mixture of them.

In 1986, an organic acid radical in the form of alkene-sulphonic acid, has been successfully included in the high-energy efficiency formulations (HEEF) introduced by Atotech Company (USA) [Chessin & Newby, 1986].

Because chromium metal will not serve satisfactory as an anode, owing to its close to 100% anodic dissolution efficiency, insoluble anodes are used generally as a lead alloy. The source for the chromium trioxide, CrO_3 (chromic anhydride), is commonly referred to as chromic acid. It is a deep red to reddish-brown crystal that volatilizes at 110°C . It is highly soluble in water (165g/100g at 0°C and 206g/100g at 100°C), producing a solution containing a mixture of $\text{H}_2\text{Cr}_2\text{O}_7$ and polychromic acids.

Despite the established understanding of the principle, such hard chrome plating process usually has to be tailored in order to produce a specific hard chrome deposition. This is due to the cost and inefficiency of the empirical approach currently used to determine the process parameters to yield a specific hard chrome composition, and as a result such electroplating process has been utilized only in a limited manner.

Furthermore, at the commencement of this Innovation Technology Fund project, the chromium plating bath employed in the Teaching Company was a static system, which involved fixing the anode close to the surface of the work-piece while the work-piece was kept at the cathode. The deposition material was hard chrome and the electroplating area was the ring grooves of a piston crown. The diameter of the piston crown varies from 350mm to 900mm, and it weighs up to 1000 kilograms. Electric current of appropriate strength was then applied and passed from the anode through the plating solution to the cathode. Chromium was then gradually deposited onto the unmasked surface of the work-piece. Figure 1 shows a sample piston crown of diameter 900mm and Figure 2 shows the appearance as well as the dimension of the ring grooves. Figure 3 shows a typical sample of the original static hard chrome plating system.

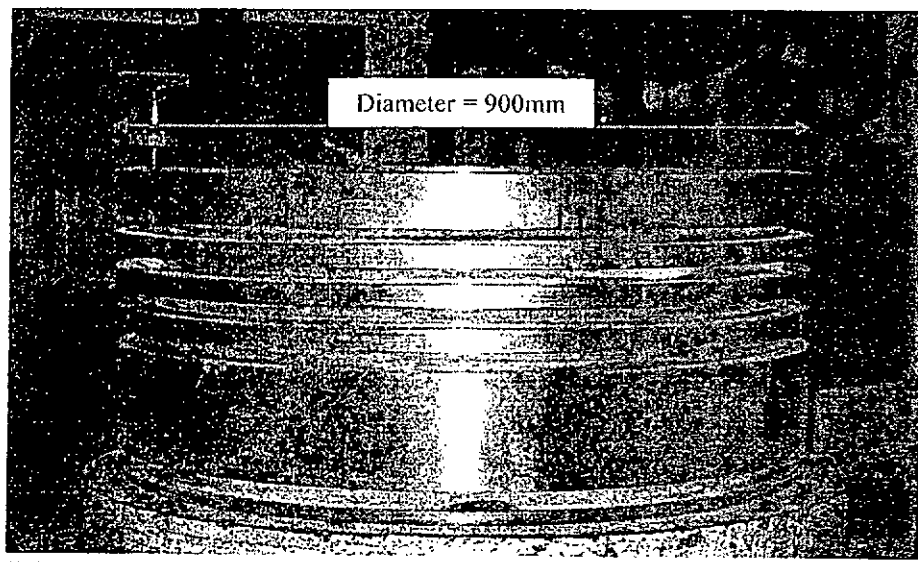


Figure 1: A photo shows a sample piston crown of a diameter 900mm

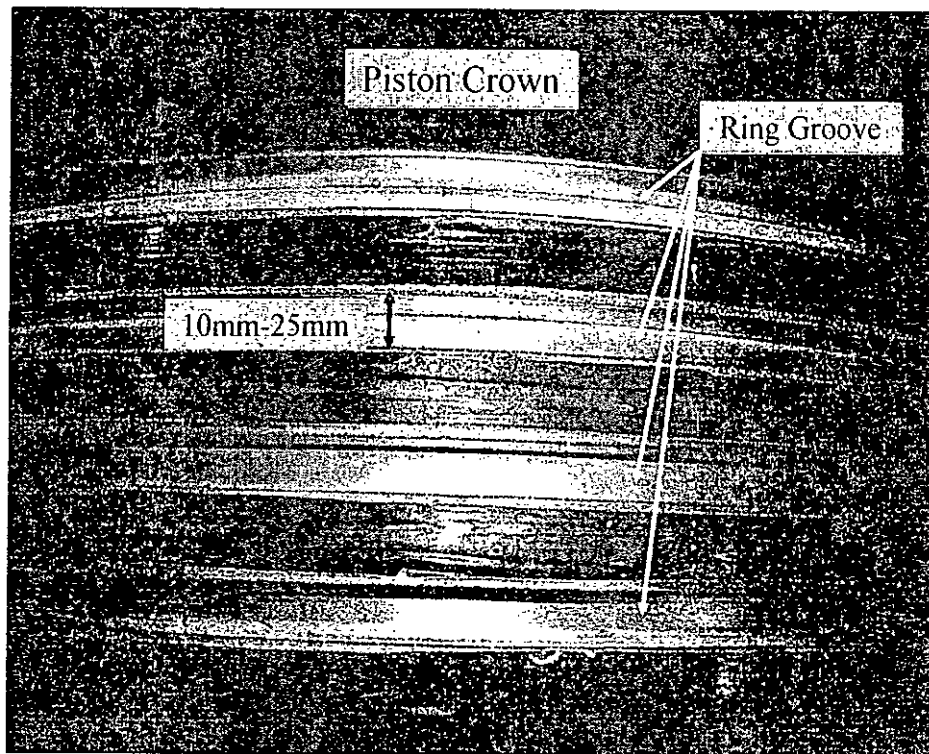


Figure 2: A photo shows the appearance and dimension of the piston crown ring grooves

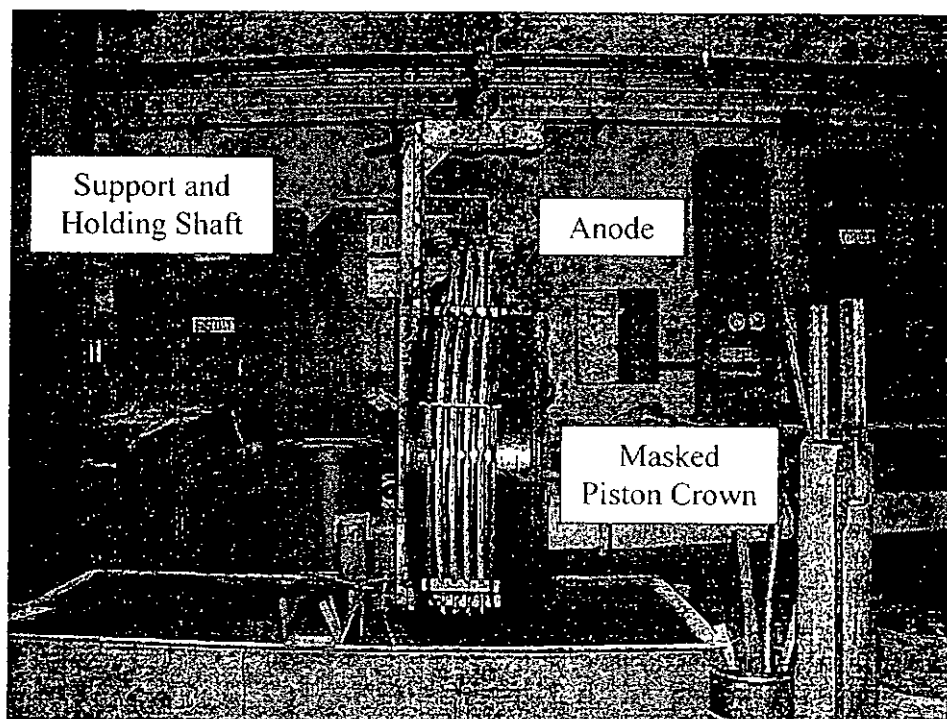


Figure 3: A photo shows a sample of the original static hard chrome playing system in the teaching company

The greatest limitations of the original static hard chrome plating system include the very large size of the substrate (piston crown) and the very long process time (the set-up time plus the plating time for 1 plating process can take up to 120 man hours). In addition, there is a lack of understanding in the combined effects of process variables on plating thickness. As a result, the workers have to use their knowledge (accumulated through many trial-and-error operations) in order to estimate the process variables to achieve a particular hard chrome deposition. Moreover, it can be quite difficult to produce a uniform chromium distribution onto product parts. Best electroplating result depends on the process modelling of these operating parameters, which involves a very complicated analytic process.

1.2 Objectives

The objectives of this research study comprise the development of a faster speed plating system and the determination of the optimal operating conditions for such system. Using the experimental results established in the early stages of this study, the individual and combined effects of various process variables on the hard chrome deposition would be evaluated, i.e. the influences of apparent current density, anode diameter, plating temperature, etc. would be assessed. Then a process prediction with respect to the thickness of the chrome-plating outcome would be carried out through statistical modeling technique. It is anticipated that with an increase in the understanding of relationships between process parameters in hard chrome plating, a more effective maintenance and process control will be achieved.

1.3 Layout of thesis

A literature review in the areas of hard chrome plating, process modeling and prediction techniques is presented in the next chapter. In chapter three, the design of pilot plant for rotary plating system is presented. The construction of the experimental setup is depicted in chapter four. The process modeling and prediction by full factorial design approach is described in chapter five. The neural network approach is presented in chapter six. A discussion of results is provided in chapter seven. The conclusion is given in chapter eight, and the suggestion for future work is given in chapter nine.

2. Literature review

2.1 Process variables of hard chrome plating

Electroplated chromium can be classified among the most important plated metals and is used almost exclusively as the final deposit on parts. Without the physical properties offered by such deposits, the service life of most parts would be much shorter due to wear, corrosion, etc [Edwards, 1997]. Product parts would have to be replaced or repaired more frequently, or they would have to be made from more expensive materials, thus wasting valuable resources.

In general there are two categories of electroplated chromium, namely decorative and functional. The thickness of decorative chromium deposit is usually about several micrometers. Decorative chromium deposits offer a pleasing, reflective appearance while also providing corrosion resistance, and durability. They are typically plated over nickel but are occasionally plated directly over the substrate of the part.

On the other hand, the thickness of functional chromium (hard chrome) deposits is usually from several micrometers to several millimetres. Hard chrome deposits are usually plated directly on the substrate, which is the other way round when comparing to decorative chromium deposits. Industrial coatings take advantage of the special properties of chromium, including resistance to heat, hardness, wear, corrosion, and erosion, and a low coefficient of friction. In

addition, hard chrome deposits are also used on parts such as cutting tools and strip steel [Greenwood, 1981]. Moreover, they have been used to protect against abrasion, oxidation and hot corrosion; or to provide lubrication [Mevrel, 1989; Sofer, Yarnitzky & Dirnfeld, 1990]. The hard chrome plating process can be used to make dispersion-strengthened alloy components and high surface area cathodes that are used electro catalysts for hydrogen electrodes in industrial water electrolysis [Pushpavanam & Co., 1993].

The most common and oldest commercial type of chromium process utilizes hexavalent chromium (Cr^{6+}) in an aqueous solution containing one or more catalysts. The commercial process of hexavalent chromium plating resulted principally from the work in 1923 and 1924 of Fink and Eldridge [Fink, 1931; Dubpernell, 1963]. Liebreich [1927] made similar discoveries more or less simultaneously, but his work was masked by an overemphasis of the importance of the trivalent chromium ion. Then early in the 1970s aqueous trivalent chromium (Cr^{3+}) solutions started to attain commercial success for decorative applications. Even though much work has been done on functional trivalent chromium deposits, there are only very restricted number of applications due to limitations in the physical properties of thick deposits.

Significant improvement in hexavalent chromium plating came with the introduction of double and organic catalysed systems. Double catalysed (or mixed catalyst) systems introduced in 1949 generally contain sulphate and silico-fluoride in forms, which are either self-regulating [Stareck, Passal, & Mahlstedt, 1950] or operator regulated. In comparison to the initial commercial processes

(which are only sulphate catalysed), the double catalysed processes offer higher plating speeds and help activate the part prior to plating by mildly etching the substrate. Another noteworthy improvement in hexavalent chromium plating came with the introduction of organic catalysed systems. Such systems offer an increase in plating speeds, an improvement of physical properties in chromium deposit, and they do not etch iron substrate [Morisset, Oswald, Draper, & Pinner, 1954; Dubpernell, 1963].

In the early 1990s, several review papers regarding electroplating have been published over the years. Papers by Buelens & Co. [1992] provided good reviews in the early 1990s, of proposed electroplating mechanisms and models of the process. Hovestad & Janssen [1995] published an overview in 1995 which presented more details of the experimental trends, possible mechanisms and process models. More recently, Helle & Walsh [1996] presented a review addressing the shortcomings of the theories to predict the composition of the deposits.

According to the researches on hard chrome plating, temperature, current density and bath composition affect the film characteristics and current efficiency [Dennis and Such, 1993; Edwards, 1997]. These parameters are therefore carefully controlled in order to obtain specific deposit properties and plating rates. The influence that a particular variable has on the process is typically assessed by the change in the amount of particle incorporation obtained when that variable is adjusted.

2.1.1 Bath composition

The conversion of a pure chromic acid solution to a chrome-plating bath solution constitutes a sulfate catalyst. With a given set of conditions of bath temperature, current density, and chromic acid concentration, too low amounts of catalyst will result in either no current flow, at first, or no plate or in an iridescent to brown oxide stains. Too high a catalyst content will result in an adverse effect: either partial plating with poor throwing power or, with great excess, no plate at all. The later effect is due to depolarization action or easy formation of trivalent chromium (Cr^{3+}) at the cathode. A typical formula for chromium plating using a sulfate as the catalyst acid radical is presented in Table 1.

Table 1: Basic chromium plating baths

	Dilute Bath		Standard Bath		Concentrated Bath	
	Gram/Liter	Molarity	Gram/Liter	Molarity	Gram/Liter	Molarity
Chromic Acid, CrO_3	100	1.000	250.0	2.500	400	4.00
Sulfate, SO_4^{2-}	1	0.001	2.5	0.025	4	0.04
Ratio CrO_3/SO_4	100		100		100	

Although concentrations of chromic acid from about 50 gram/liter up to saturation (about 900 gram/liter) can be used, most commercial baths are operated between 150 and 400 gram/liter. Still higher concentration gives very low current efficiencies. The important requirement is the proper ratio already mentioned.

Bath containing 200 gram/liter chromic acid has a slightly higher current efficiency than more concentrated solutions. They also have a lower conductivity

and therefore a higher voltage for a given current density. The more dilute baths are also more sensitive to the changes of catalyst from drag-in and drag-out. Hence they require more frequent and more careful adjustment for maintenance. Usually the more concentrated solutions are favored for decorative applications and more dilute baths for heavy hard chrome plating.

Silicofluoride has had wide use as a catalyst in chromic acid baths since Fink and McLeese first proposed it in 1932 [Fink & McLeese 1932]. Such solutions were difficult to analyze and maintain. Yet those baths have definite advantages compared to sulfate only catalyzed baths. They have inherently higher current efficiency, can be operated at higher deposition rates, and produce somewhat harder and brighter deposits. Fluorides give better throwing power and covering power. On the other hand, there are some important disadvantages. These metals are sensitive toward changes in composition and toward impurities such as iron and aluminum, and consequently more careful attention to bath purification, frequent analytical control is required. Also analytical control of simple or complex fluorides is relatively more complicated, and finally, those solutions will attack or etch the base metal at low current density as well as unmasked areas such as blind holes. If masking is less than optimum, which sometimes cannot be avoided, proper attention must be paid to the possible etching effect. The solutions are aggressive toward plating equipment such as tank liners and heating/cooling coils.

The extra efficiency available from fluoride containing baths still resulted in chromium deposition rates which, were much lower than for most other

plating baths (in relation to the high current densities employed). However, in 1986, proprietary plating solutions were introduced that had higher cathodic efficiencies than obtainable from the fluoride containing bath and these baths were established as viable industrial processes. They are based on chromic acid solutions that do not contain any fluorides or other halogens. Their chromic acid content is between 250 and 300 gram/liter. The only other component of these solutions that is known is the primary catalyst (presumably a sulfate ion), within the ratio of 100:1, and 1% to 3% of alkene sulfonic acid as secondary catalyst. These proprietary solutions provide extra cathodic efficiencies of up to 25%. These components catalysts have either been patented [Chessin & Newby, 1986; Korbach, 1989; Korbach & McMullen, 1989; Martyak, 1989] or kept secret. The properties of the deposits have been documented [Newby, 1999], together with optimum operating parameters of these plating processes. The solutions are usually operated at temperatures between 55°C and 60°C and typical cathodic current densities are 30 to 50 A/dm². Even at these high current densities, deposit distribution is superior to that obtained from conventional baths, with less edge buildup. The deposits have good hardness (about 1000HV Vickers), and retain such hardness better than conventional chromium when heated. The chromium plate always shows plenty of micro-cracks, e.g. 200 to 400 cracks per centimeter.

One of the greatest benefits of these fluoride-free plating solutions is that they do not attack steel on those portions of the cathodes where the current density is too low for chromium to be deposited. This low current density etching is especially detrimental when complex shaped steel objects are hard chrome plated for a long period of time in fluoride containing baths. The fluoride ion

dissolves the protective oxide film off those portions of the substrate steel exposed to low current densities and thus the acid solution can then dissolve it with consequent iron buildup. This etching attack has been a limiting factor in the use of those baths for hard chrome deposit.

In practice, relatively high concentrations of chromic acid are used, e.g. from 250 to 400 gram/liter of CrO_3 . This increase in concentrations increases the conductivity up to a maximum but decreases the cathode efficiency. It should be noted that in some cases these two factors, concentration and conductivity, may offset each other at higher current density obtainable at a given voltage in a more concentrated chromic acid bath and may not yield a faster rate of chromium deposition.

2.1.2 Current density

The amount of chrome particles deposition in Cr- Al_2O_3 system has been found to increase when the current density is increased. In addition, a minimum of chrome particle deposition as a function of current density was also found when the loading in suspension exceeded 100 gram/liter [Narayan & Chattopadhyay, 1982]. The relationship between the current density and applied voltage can be revealed using polarization scans. The effect of particles in suspension on the polarization scans have been reported for some electrolytes used in electroplating systems. A higher current density has been observed for a given cathodic potential in the presence of particles at high voltage where the transport of metal ions to the cathode becomes an important factor. Moreover, it

has been discovered that by adding particles to the electrolyte, mass transport could be enhanced. At low voltage, the particles near cathode suppress metal ion reduction, which effectively lowers the current density at a given voltage [Hovestad & Janssen, 1995; Edwards, 1997].

2.1.3 Throwing power

Besides keeping the current density as nearly uniform as possible, the throwing power is another measure towards a good chrome-plating outcome. By definition, the throwing power of a plating bath means its ability to uniformly deposit chromium on the cathode surface [Greenwood, 1981]. The major factors influencing the throwing power in a chrome-plating bath are the primary current distribution, polarization, secondary current distribution, and cathode efficiency. The primary current distribution is a function of the geometrical properties of the plating system, meaning the shape and the distance between the anode and the cathode. Increasing the anode to cathode gap is helpful, but this usually requires higher operating voltage, and also an initial high strike current may be necessary in order to obtain adequate coverage of chromium deposit.

Increasing the plating bath temperature and plating current density generally improve the throwing power [Edwards, 1997]. However, in the case of irregular cathodes, the current density varies widely, being highest on corners, edges and areas closest to the anode, whereas it is lowest in recesses, re-entrant angles, and areas farthest from the anode. It is then evident that cathode efficiencies being highest at the high current density areas result in heavier

chromium deposition, whereas the low current density areas have thinner chromium deposition.

As shown in the above review, hard chrome plating process can be very application-specific, thus the operating conditions for a particular hard chrome plating process to obtain a uniform chromium distribution can be significantly different from those necessary to obtain a gradient chrome deposition. Although the effect of each of the process variables on such plating process has been reported in the literature, the results are usually contradictory. In addition, while considerable researches have been conducted on hard chrome plating, they usually focus on improving a specific characteristic of the hard chrome deposition rather than evaluating the interdependence between process variables. In spite of this, there seems to be a common thread which unites the bulk of the electroplating literature and that is the lack of discussion regarding the possibility of interaction relationship between process variables.

It is the aim of this research work to establish a thorough experimental foundation on hard chrome plating. With a further understanding of interdependent relationships between process variables, a more effective maintenance and process control could be achieved.

2.2 Process modeling and prediction - statistical approach

It is very often that the end-product of a research study is a statistical model or set of models, whether it is aimed for prediction, explanation, or quality control. A statistical model is an abstract description of the real world. A model is a simple representation of the complex forms, processes, and functional relationships of a “real world” counterpart. Because many of the processes underlying scientific research are complex and a function of many inputs (independent variables), the statistical models representing them by necessity are much simpler [Cichocki & Amari, 2002]. In fact, a primary objective of statistical modeling is to represent complex phenomenon with as simple a model as possible.

Although it is usually possible to identify the primary objective of a research project, models estimated for predictive or explanatory purposes are related. When the analyst can identify functional or cause-effect relationships between objects or events, then superior models result, and often are useful for both explanation and prediction. In many cases, however, the suspected ‘causal’ independent variables are not directly measurable, are too expensive or impractical to measure, and so the analyst seeks surrogate variables that are correlated with the causal ones.

Two fundamental features characterize all models: form (relationships) and content (variables.) The choice of form establishes the ease of manipulating

and interpreting the content; detecting errors of omission and commission; and refining and improving the model to better serve its purpose.

Process optimization involves the minimization (or maximization) of an objective function, which can be established from a technical and/or economic viewpoint. In general, the decision variables are subject to constraints such as valid ranges (maximum and minimum limits) as well as constraints related to safety considerations and those that arise from the process model equations. Another most challenging aspects of multivariate analysis is finding the optimal variable settings that maximize (or minimize) a response [Shaffer, 1996]. On the other hand, the algorithms for process modeling, prediction and optimization can also be regarded as a general name for techniques that are designed to solve such problems. The process modeling, prediction and optimization can be defined mathematically as the search for the settings of the n variables of a function, such as $f(x_1, x_2, \dots, x_n)$, that optimize $f(x)$.

Broadly speaking, there are two classes of these kinds of problems. One class includes the problems that can be solved analytically through direct means. These kinds of problems require the knowledge of the mathematical form of the function being optimized. A simple example is finding the root(s) of a polynomial function. However, process modeling and prediction applications in electroplating are usually not simple. The relationships between the process variables and $f(x)$ may not be well understood and indirect methods need to be employed.

If indirect methods are employed in trying to model and/or predict a function $f(x)$, “Guesses” for the settings for the n variables are carefully calculated. Hence the employment of indirect methods is sometimes called iterative trial and error approaches. Decisions about what “Guesses” to make are called search heuristics. When indirect methods are being used, the setting of process variables is modified in some logical way after each iteration and then presented to an objective function to determine whether this particular combination of process variables is an improvement. Such objective function would include numerically the performance of a particular combination of process variable.

The principles of the most common indirect process modeling and prediction method, single-factor-at-a-time, can be illustrated by way of an example in hard chrome plating process. In this case, the objective function is the thickness of hard chrome deposition and the variables to be modeled and predicted are the chromic acid concentration and sulphuric acid concentration. In the first step of this approach, the settings for chromic acid concentration are modified, while the setting for sulphuric acid concentration is held constant. The variable setting for the chromic acid concentration that caused the thickness of hard chrome deposition to be the largest is considered the setting for that variable. Next, the sulphuric acid concentration is modeled and predicted, while the chromic acid concentration is held constant at its preset setting. The single-factor-at-a-time optimization procedure is inherently flawed, however, because it does not take into account any interdependencies that may be present among the variables. Interdependencies are caused by variables that affect the objective

function differently based on the settings for the other variables. In analytical applications, interdependencies among the variables are the rule rather than the exception. If, in the hard chrome plating example above, the impact that chromic acid concentration had in the thickness of hard chrome deposition changed as a function of the sulphuric acid concentration, a univariate approach such as the single-factor-at-a-time approach would not be able to predict the thickness of hard chrome deposition. However, a multivariate approach in which both variables are modified at each step would overcome this drawback. Multivariate methods have been employed to solve challenging problems in many areas of science and engineering [Cooper, 1974; Law & Kelton, 1982; Press & Co, 1986; Arora, 1989; Hajela, & Berke, 1991; Peace, 1993; Hinkelman, 1994; Wang & Grandhi, 1994; Montgomery, 1997].

According to the algorithms for process modeling and prediction most frequently seen in the science and engineering literature, they can be divided into three general groups: experimental design methods, methods that require derivatives of the function being optimized, and methods that do not require derivatives to be computed [Cooper, 1974; Press & Co, 1986; Montgomery, 2001]. Experimental design methods offer valuable insights into the improvement and understanding of the response from a system and are considered very general and versatile [Daniel, 1976]. As it is very often a scientific experimental outcome may depend on many factors, the factors chosen for examination would be tested at a number of levels reflecting likely operating levels for such factors. Experiments of this type come under the domain of Factorial (Experimental) Designs where both effects and interaction effects can

be investigated to determine the level of influence, if any, such factors have and how it may be occurring in the observed response measurements. (One advantage of this kind of design is that it allows one to examine how different process variables operate together (or "interact") to produce a combined effect.)

In general, there are two types of Factorial Design, namely Full Factorial Design and Fractional Factorial Design. In simple terms, going through the Full Factorial Design approach would exhaustively try every possible combination of all levels of all factors, while only a fraction of possible combination of all levels of all factors would be performed in the Fractional Factorial Design approach. In this research study, Full Factorial Design approach has been adopted because the initial guess for the number of process variables is not going to be many and the Teaching Company Associate would like to find the individual and combined effects of the process variables.

2.3 Process modeling and prediction - artificial intelligence approach

In many applications, the calculation of the derivative is not possible analytically or it is very time consuming or numerically inaccurate. An alternative to the statistical approach for the development of controlled systems is the artificial intelligence approach. There are many techniques available in the artificial intelligence approach and one of the popular choices is Neural Network (NN).

Generally speaking, NN is an information processing paradigm that is inspired by the way biological nervous systems, such as the brain, process information. According to the DARPA Neural Network Study (1988), the definition of NN can be regarded as a system composed of many simple processing elements operating in parallel whose function is determined by network structure, connection strengths, and the processing performed at computing elements or nodes. In addition, according to Haykin (1994), a NN is a massively parallel distributed processor that has a natural propensity for storing experiential knowledge and making it available for use. It resembles the brain in two respects: (i) Knowledge is acquired by the network through a learning process and (ii) interneuron connection strengths known as synaptic weights are used to store the knowledge.

The key element of the NN paradigm is the novel structure of the information processing system. It is composed of a large number of highly interconnected processing elements (neurons) working in unison to solve specific problems. NN possess the ability to 'learn' what happens in the process without actually modeling the physical and chemical laws that govern the system. Furthermore, NN can be configured for a specific application, such as pattern recognition or data classification, through a learning process. Learning in biological systems involves adjustments to the synaptic connections that exist between the neurons and this is true for NN as well.

Recently, NN has been applied to an increasing number of real-world problems of considerable complexity. Their most important advantage is in solving problems that are too complex for conventional technologies -- problems that do not have an algorithmic solution or for which an algorithmic solution is too complex to be found. In general, because of their abstraction from the biological brain, NN are well suited to problems that people are good at solving, but for which computers are not. Moreover, the distinct features of the NN make the model itself very useful in situations where a functional dependence between the inputs and outputs is not clear. In this context, a NN model can handle multiple independent and dependent process variables simultaneously in one model (e.g., back propagation model). The functional relationship between the independent and dependent process variables need not be known since the NN model can learn the latent relationships between the causal factors and response. [Vanderplaats, 1984; Carpenter & Hoffman, 1995].

On the other hand, the NN model has modeling, prediction and formulation capabilities, and can be updated with new data. It can also be used to predict the response for new experimental conditions after the models are trained [Haykin, 1994; Takahara, Takayama & Nagai, 1997; Basheer & Hajmeer, 2000].

In this research study, the NN approach has been adopted because of its remarkable ability to derive meaning from complicated or imprecise data, not to mention the possible usage to extract patterns and predict trends that are too complex to be noticed by either humans or other computer techniques. A trained NN can be thought of as an "expert" in the category of information it has been

given to analyze. This expert can then be used to provide projections given new situations of interest and answer "what if" questions.

Despite the availability of various algorithms for process modeling and prediction, not much work have been done in trying to adopt these techniques in the area of electroplating process in order to model and predict chrome plating conditions for selected piston crowns. It is the aim of this research study to compare the prediction results generated from the full factorial design approach, the trained NN and the actual experiments.

2.4 Proposed approach

At the commencement of this Innovation and Technology Fund (ITF) project, the Teaching Company Associate (TCA) was expected to interview the relevant staff in the Teaching Company in order to understand the details of the company operation. For the data collection process, the TCA needed to work with company staff in order to obtain the necessary information regarding the current practice of the hard chrome plating operation. With the Teaching Company's expectation to shorten the process time of the current plating system, the development of a new plating machine was the main target.

On the other hand, the TCA needs to evaluate the process variables of the hard chrome plating system for the purpose of developing a model for the hard chrome plating system. In particular, a statistical approach (such as Full Factorial Design) is suggested because it is essential to find the individual and combined

effects of every process variables. Alternatively, an artificial intelligence approach (such as Neural Network) can be adopted because of its processing paradigm that is inspired by the way biological nervous systems. Next, a comparison of results obtained from the actual experiments, the statistical approach and the computational approach would be presented. Figure 4 provides a flow chart for the proposed approach in this research study.

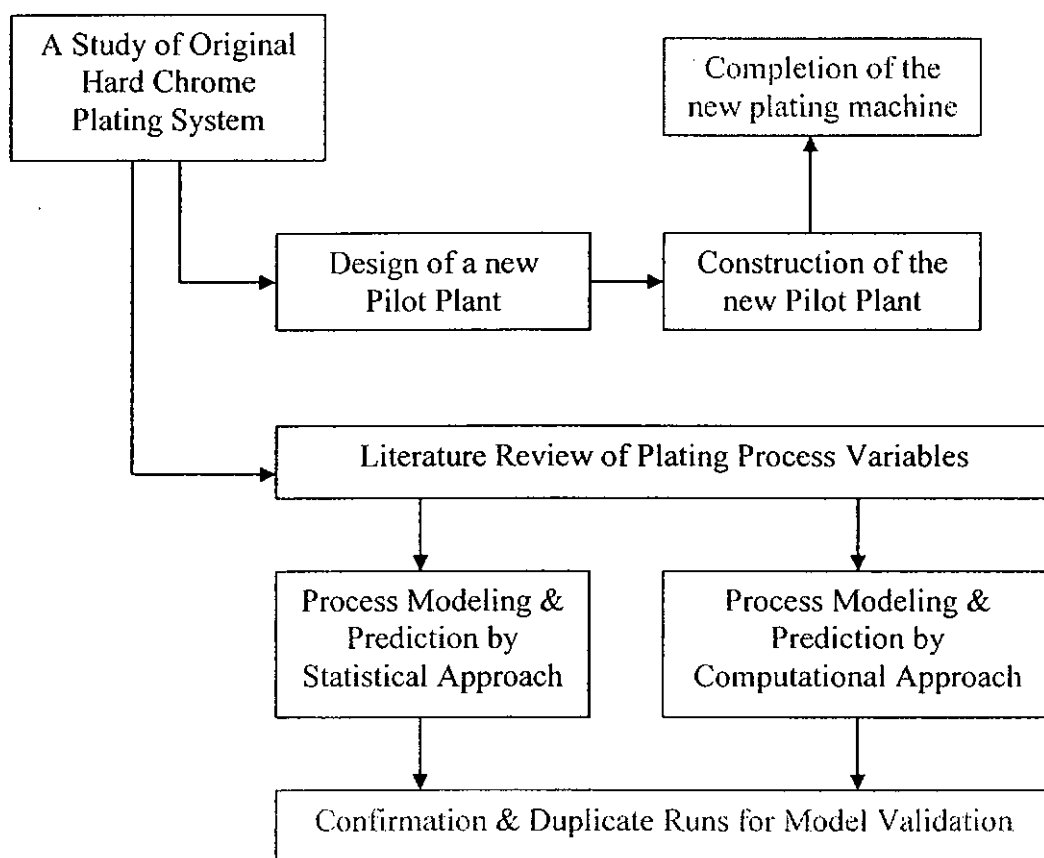


Figure 4: Flow chart for the proposed approach in this study

3. Design of pilot plant for rotary plating system

3.1 The original hard chrome plating system in the teaching company

Electroplating is the process of applying a metallic coating to an object by passing an electric current through an electrolyte in contact with the object, thereby forming a surface having properties or dimensions different from those of the article. Essentially any electrically conductive surface can be electroplated. Special techniques, such as coating with metallic-loaded paints or silver-reduced spray, can be used to make nonconductive surfaces, such as plastic, electrically conductive for electroplating. Electroplated materials are generally used for a specific property or function, although there may be some overlap, e.g., a material may be electroplated for decorative use as well as for corrosion resistance [Metal Finishing Guidebook and Directory, 1993; Horner, 1994].

The essential components of an electroplating process are an electrode to be plated (the cathode or substrate), a second electrode to complete the circuit (the anode), an electrolyte containing the metal ions to be deposited, and a direct current power source. The electrodes are immersed in the electrolyte with the anode connected to the positive leg of the power supply and the cathode to the negative leg. As the current is increased from zero, a point is reached where metal plating begins to occur on the cathode. The plating tank is either made of or lined with totally inert materials to protect the tank. Anodes can be either

soluble or insoluble, with most electroplating baths using one or the other type. The majority of power supplies are solid-state silicon rectifiers, which may have a variety of modifications, such as constant current and constant voltage.

At the commencement of this Innovation Technology Fund project, the hard chrome plating system employed in the Teaching Company was a static system, which involved fixing the anode (lead alloy) close to the surface of the work-piece while the work-piece was kept at the cathode, as shown in Figure 1, 2 & 3. Figure 5 presents a process flow diagram for hard chrome electroplating. The process consists of pretreatment, alkaline cleaning, acid dipping, chromic acid anodizing, and chromium electroplating. The pretreatment step would include polishing, grinding, and degreasing. Degreasing consists of either dipping the part in organic solvents, such as trichloroethylene or perchloroethylene, or using the vapors from organic solvents to remove surface grease. Chromic acid anodic treatment, which was optional, cleaned the metal surface and enhances the adhesion of chromium in the electroplating step. The final step in the process is the electroplating operation itself.

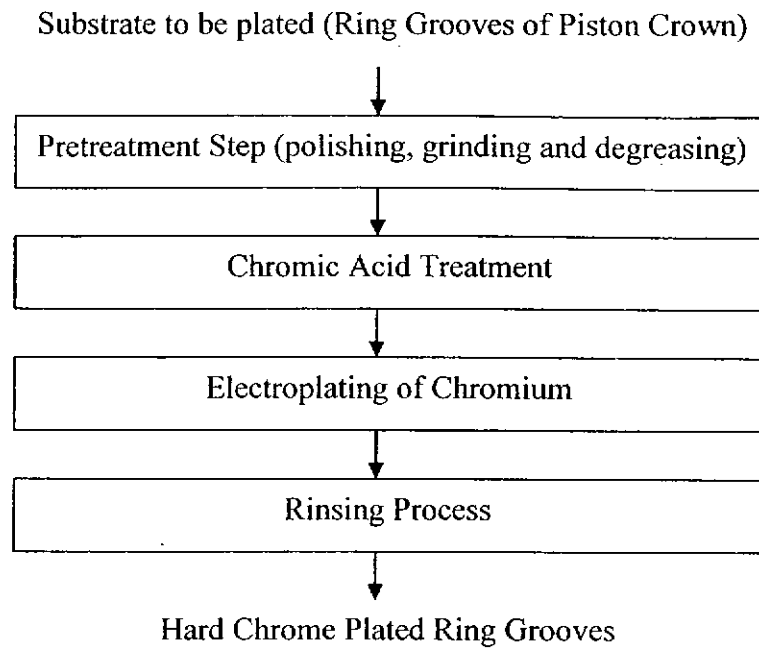


Figure 5: Flow chart for the hard chromium plating process in the teaching company

Ideally the plating tanks typically are equipped with some types of heat exchangers. And preferably, mechanical agitators or compressed air supplied through pipes on the tank bottom would provide uniformity of bath temperature and composition. Unfortunately, no such device was installed in the Teaching Company.

Chromium electroplating requires constant control of the plating bath temperature, current density, plating time, and bath composition. Hard chrome plating baths are the most widely used baths to deposit chromium on metal. Such hard chrome baths are composed of chromic acid, sulfuric acid, and water. The chromic acid is the source of the hard chrome that reacts and deposits on the metal and is emitted to the atmosphere. The sulfuric acid in the bath catalyzes the

chromium deposition reactions. The evolution of hydrogen gas from chemical reactions at the cathode consumes 80 to 90 percent of the power supplied to the plating bath, leaving the remaining 10 to 20 percent for the deposition reaction. When the hydrogen gas evolves, it causes misting at the surface of the plating bath, which results in the loss of chromic acid to the atmosphere.

The original hard chrome plating system in the Teaching Company aims to refurbish the ring grooves of piston crown. The depth of a ring groove is usually within the range 15mm ~ 30mm. The width is usually within the range 8mm ~ 28mm. Usually it is required that 0.7mm thickness of chromium is to be deposited only onto the top and bottom part of the ring grooves. Figure 6 describes the range of dimensions of the ring grooves. The piston crown is in cylindrical shape. Its diameter is usually within the range 450mm ~ 900mm and its height is usually within the range 210mm ~ 550mm. A sample CAD drawing of piston crown specification has been shown in Figure 7.

Once the surface of the ring grooves, electric anodes and supporting shaft have been cleaned, masked, those items would be constructed as shown in Figure 8 & 9. When the preparation work has been finished, the whole plating machine could be put into the plating tank, as shown in Figure 10. The plating time for each operation ranges between 50 hours to 80 hours depending on the dimension of the piston crown. When the time is due, the plating machine would be taken out of the plating tank and be gone through the rinsing process, as shown in Figure 11. After dismantling the plating machine, the refurbished piston crown is

ready for precise surface machining and eventually to be delivered to the customer (Figure 12).

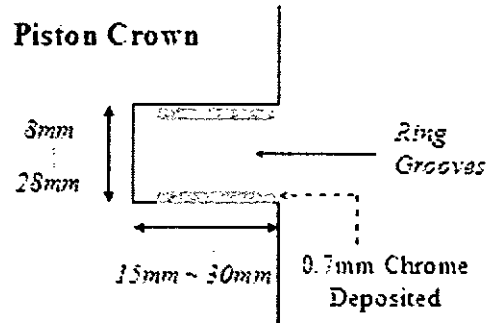


Figure 6: Dimensions of the ring grooves

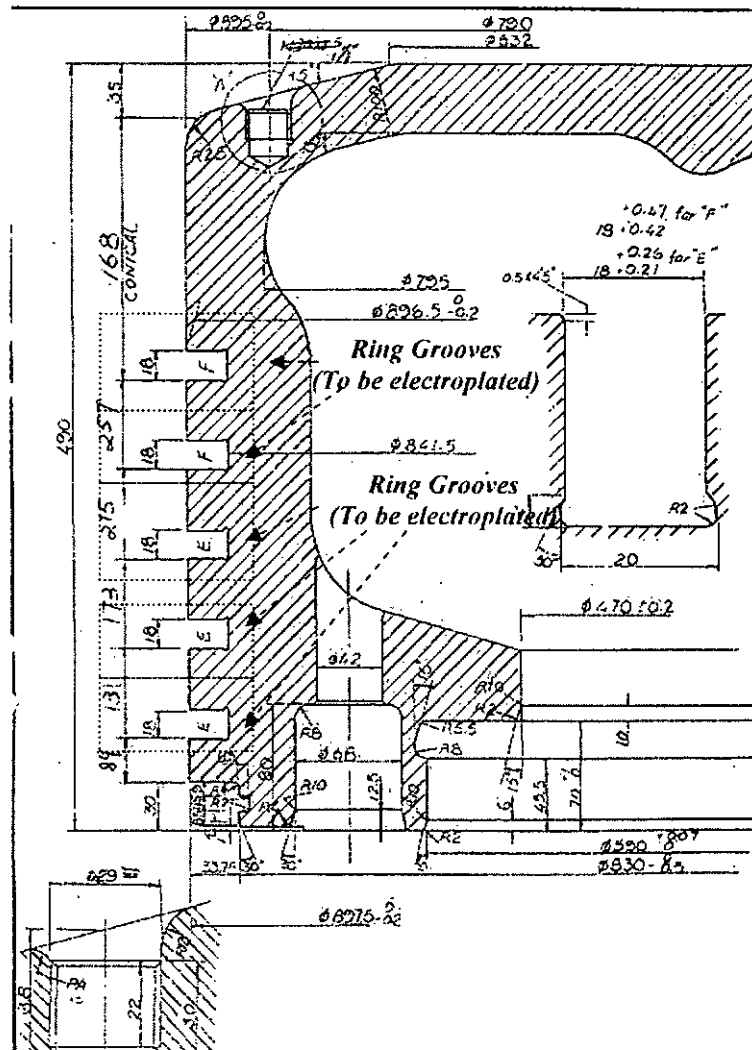


Figure 7: A sample CAD drawing of piston crown specification

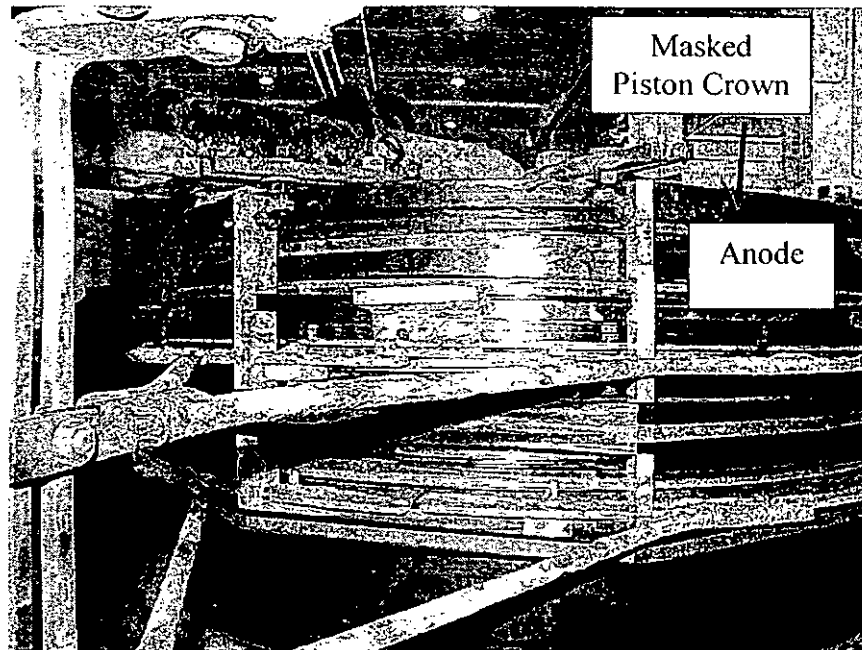


Figure 8: Construction of the piston crown and the electric anodes

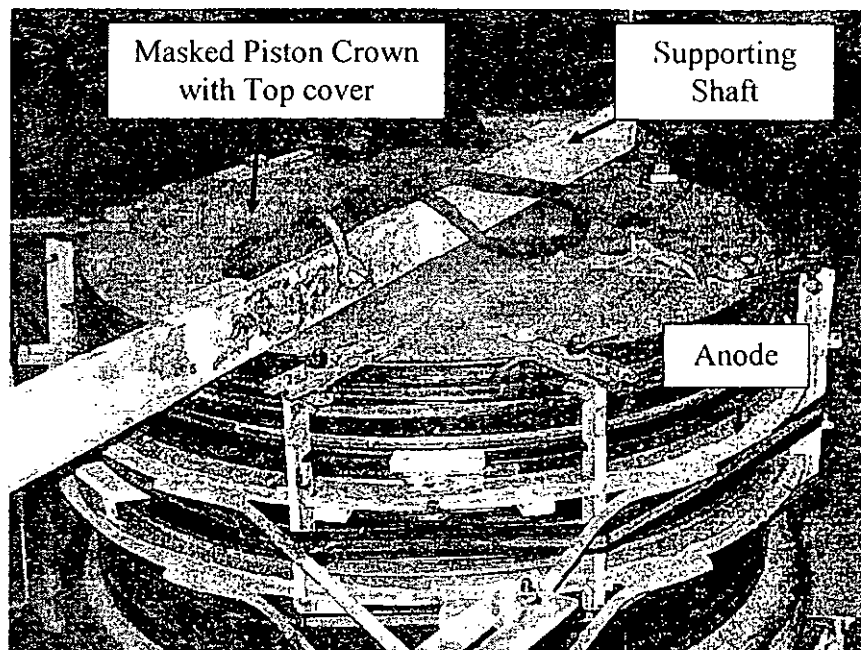


Figure 9: Construction of the piston crown and the supporting shaft

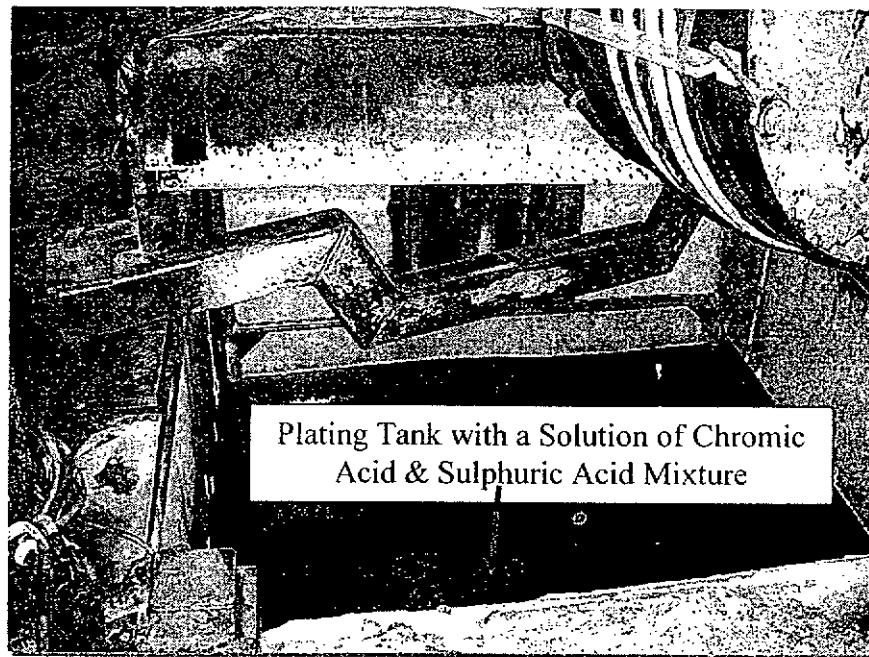


Figure 10: The plating machine is about to be put into the plating tank

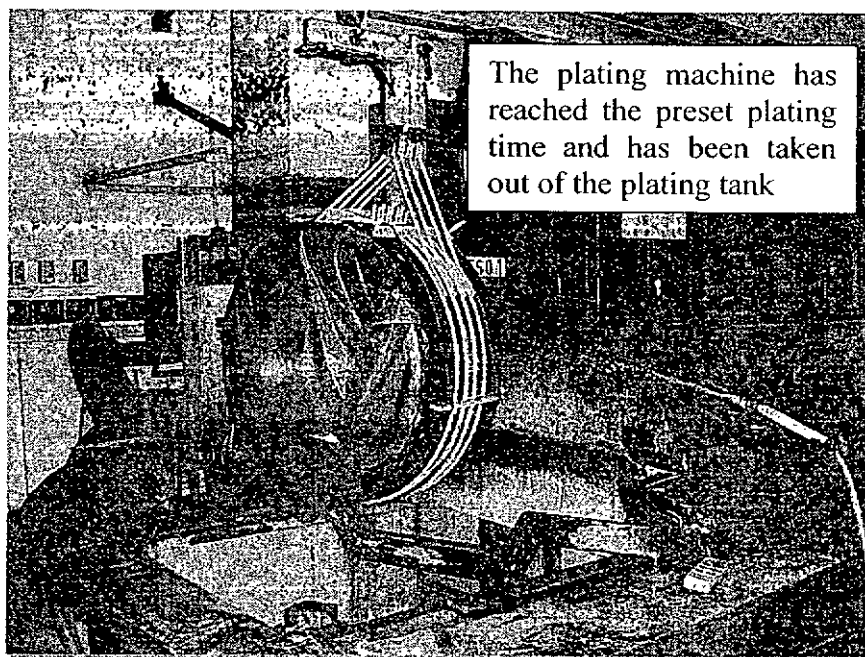


Figure 11: The rinsing process

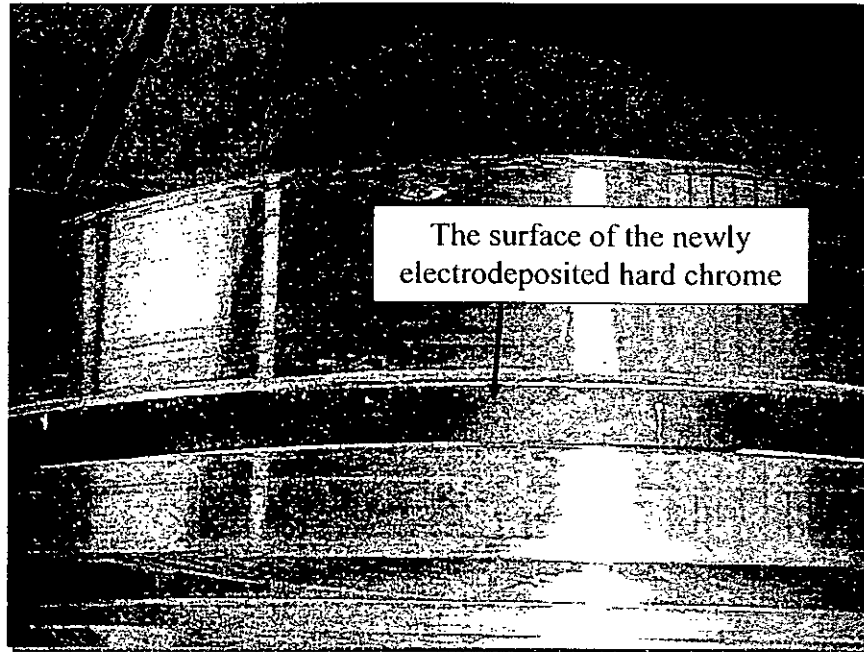


Figure 12: A photo shows the appearance of ring grooves of the refurbished piston crown

3.2 Practical constraints of hard chrome plating system in the teaching company

There are a number of constraints for the original static hard chrome plating system and these include:

- The very large size of the substrate (piston crown), thus a thorough cleaning operation (a necessary pre-treatment prior to the start of the actual plating operation) consumes a lot of time;
- The relatively small area to be hard chrome plated (ring groove), and
- The long process time (the set-up time plus the plating time for 1 plating process can take up to 120 man hours)

In addition, there seems to be a lack of understanding in the interaction of process variables. As a result, the workers have to make use of their experience (accumulated through many trial-and-error operations) in order to estimate the process variables to achieve a particular hard chrome deposition. Moreover, it can be quite difficult to produce a uniform chromium distribution onto product parts. Best electroplating result depends on the optimization of these operating parameters, which involves a very complicated analytic process. Hence, this research project has been aimed to improve the hard chrome plating process through (i) the development of a more efficient plating system and (ii) to predict plating thickness through an estimation of the statistical relationship between different thicknesses and the input variables. Such objectives have been agreed by the Teaching Company and the Supervisors of this research study.

3.3 Design of the pilot plant

Design of an appropriate electroplating machine is crucial for successful investigation of the relationships among parameters in electroplating operation. Such machine would allow accurate determination and control over as many process variables as possible, thereby providing reproducible experiments. Also accurate control and measurement of process values is important to obtaining meaningful data [Gileadi, 1993].

The process variables affected by the machine design include the total current and potential difference (between the working electrode and the plating

solution), and the hydrodynamics. The current-potential relationship is affected by the hydrodynamics of the electroplating system. In particular, if the plating system is not mixed sufficiently, the electrode reactions will cause a depletion of reactants. This in turn limits the amount of current that can be passed through the plating system [Brimi & Luck, 1965].

In a plating system without forced convection, natural convection would eventually develop due to the concentration gradients resulting from the electrochemical reactions. In addition, there would be vapors, mist and gases that are generated during the plating process. The main source of vapors, mist and gases is the small gas bubbles which form at the electrodes when the electric current is on. The bubbles rise to the surface and burst, creating a fine mist above the bath. However, the bubbles form resistive gas 'blankets' that would result a possible reduction in the deposition current. In this case, by adopting an agitation approach such as cathode motion, the forced convection would remove the resistive gas 'blankets' away from the plating surface, thus ensuring good quality in the plating outcome.

Furthermore, because of the greater reproducibility and higher allowable rates of reaction, forced convection is generally preferred to natural convection. This argument explains the reason behind the long plating time (up to 90 hours) in order to achieve the necessary thickness of hard chrome deposition. As a result, the plating time is expected to be less if a rotary approach can be employed in the design of the new plating system.

With the need to adopt a rotary approach as a research direction, the rotary cylinder electrode approach [Pickett, 1979] seems to have an advantage over the rotary disk electrode approach [Gabe, 1974]. First, the rotary cylinder approach has the hydrodynamic advantage over the rotary disk electrode approach in that the average shear force on the rotary cylinder electrode is uniform over the entire surface. Next, the hydrodynamic diffusion layer is uniform regardless of whether the flow is laminar or turbulent. Figure 13 schematically illustrates the uniformity in potential, hence current density, for the concentric cylinder configuration. The need of uniformity in current density distribution is essential because it will result a more uniform hard chrome deposition, thus reducing the process time for the final precise surface machining.

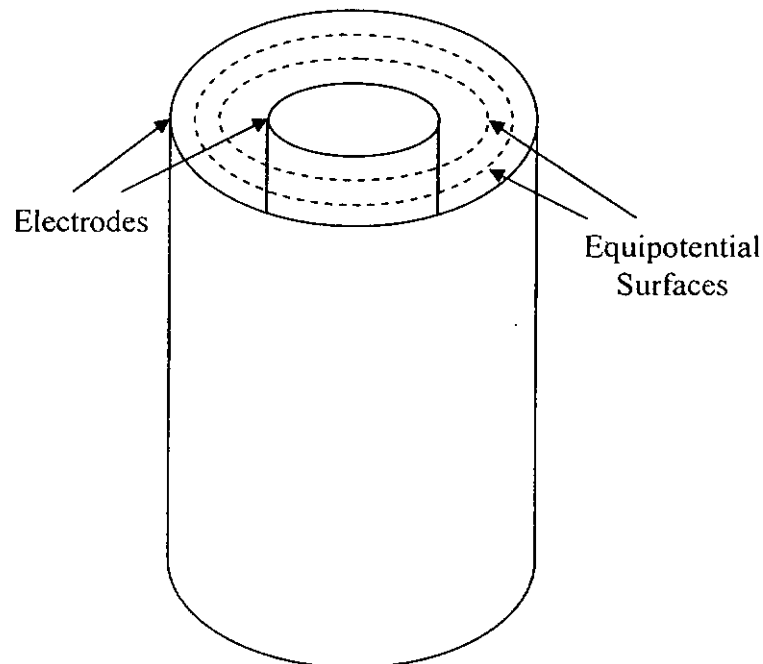
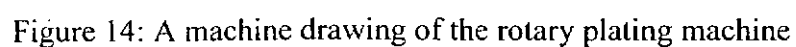


Figure 13: The primary current distribution on a rotary cylinder electrode with concentric counter electrode

In addition, the rotary cylinder electrode approach allows the best control over the process variables that are believed to influence the hard chrome plating

Eventually, after taking account of the characteristics of piston crowns, anodes and the choice of construction materials, the pilot plant has been designed, as shown in Figure 14.



4. Construction of experimental setup

The construction of the rotary plating machine was started in early April 2001 and finished in late October 2001. The course of the construction and commission of the new rotary plating machine was not a smooth process. The details of the assembly of the rotary plating machine parts have been listed in Appendix A. The details of the commissioning of the rotary plating machine have been listed in Appendix B.

The first successful trial was completed in late October 2001. The feedback showed that the surface of the ring grooves were shinny and clean, just like the plating outcomes normally observed from the static plating process. However, some observations were:

- ✎ The minimum rotary speed of the new plating machine was 10 revolutions per minute (rpm) and should be no higher than 15 rpm. If the rotary speed was set below 10 rpm, then the new plating machine would not start rotating. The problem could be due to the piston crown, which could weigh up to 1000 kilograms. Because of its weight, it would need a very large force to initialize the rotary action. On the other hand, if the rotary plating speed was set to higher than 15 rpm, smoke fume would start coming out from carbon brush section of the plating machine. Therefore, the rotary speed for the subsequent trials was set between 10~15 rpm.

- ✎ With the temperature of the plating bath fixed at 55 degree C (the optimum plating temperature), plus the adoption of the rotary plating approach, it is believed that enough turbulence would be created by such forced convection for mixing the plating solution and transferring heat from the solution to the air by evaporative cooling.
- ✎ It appeared that it was quite difficult to hold the electric anode at the centerline of the ring groove. This was a necessary requirement to be met, otherwise short-circuit would happen if the electric anode got in contact with the ring groove surface (the electric cathode). Later, with a change of design of the electric anode (as shown in Figure 40 & 41 in Appendix A), this major obstacle was cleared.

After the specified hardness (>750 HV) for the newly hard chrome plated surface had been achieved with several runs, the Teaching Company Associate was asked to optimize the rotary plating process. Figure 15 shows the eventual design and appearance of the new rotary plating machine.

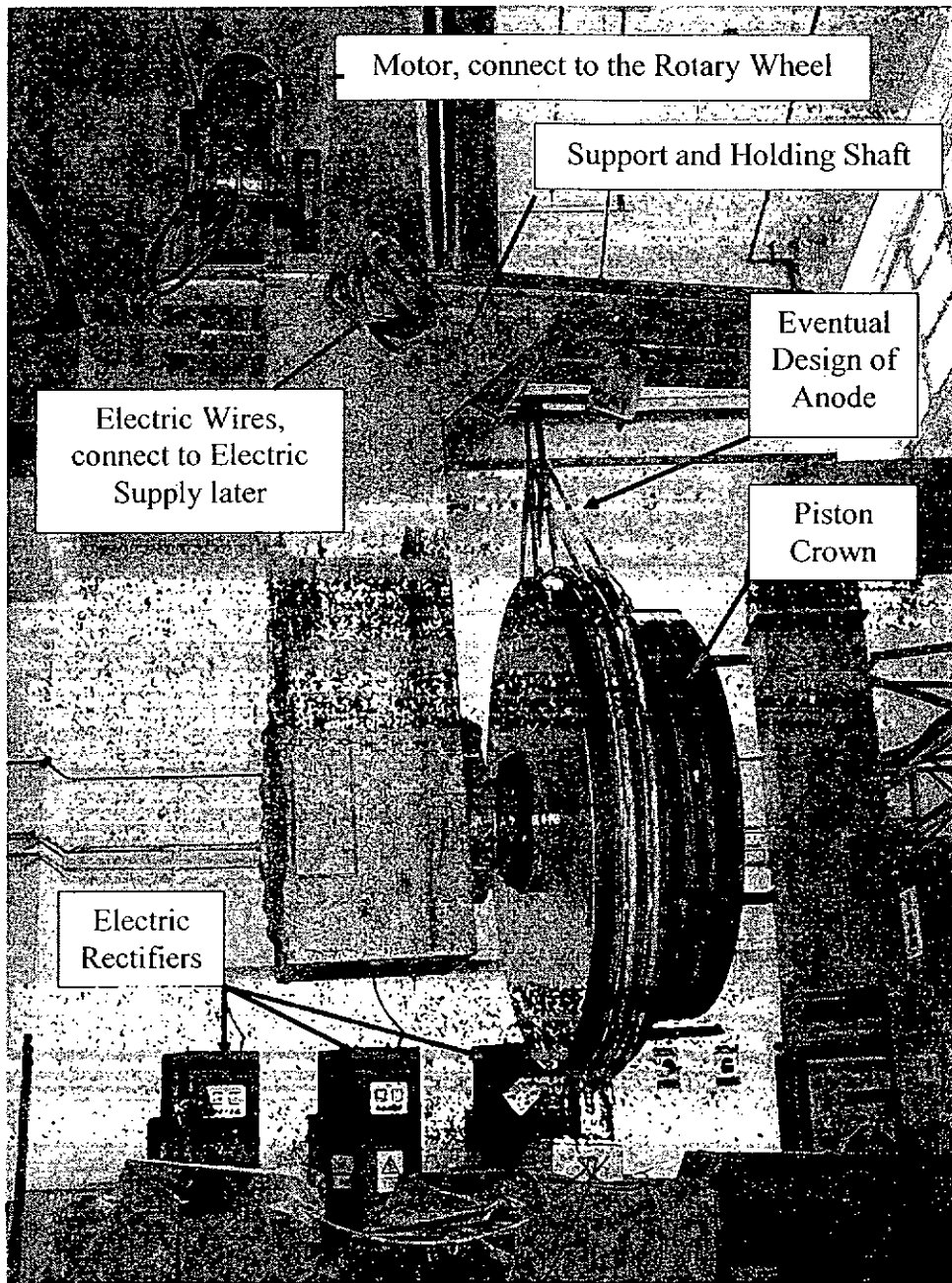


Figure 15: The rotary plating machine is ready for action

5. Process modeling and prediction by Full Factorial Design (FFD) approach

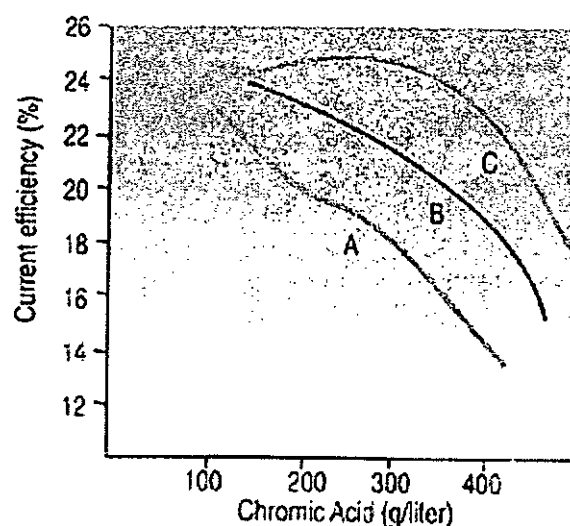
5.1 The influential factors for the hard chrome plating process

The factors which contribute to the enhancement of hard chrome plating process by the application of rotary approach are listed below:

- Bath Composition
- Temperature
- Current Density
- Plating Time
- Rotary Speed

5.1.1 Bath composition

Chromic acid and Sulphate (generated from Sulphuric Acid) are the necessary ingredients. Chromic-to-sulfate ratios range from 75:1 to 250:1. The composition depends primarily on the application. In single-catalyst baths, cathode efficiency decreases proportionally with chromic acid concentration (see Figure 16). Solutions with higher concentrations of chromic acid tolerate a higher level of trivalent chromium and iron oxide contaminants. Addition of a secondary catalyst improves cathode efficiency at high concentrations of chromic acid, up to 300 g/liter.



- A. Single catalyst (sulfate)
- B. Co-catalyzed bath
- C. High-speed self-regulated bath

Figure 16: The effect of chromic acid concentration on current efficiency [Zaki, 2000] (A single catalyst bath has been employed in this study)

In the Teaching Company, the density of each plating bath sample is measured using accurate Baume hydrometers. In addition, the Teaching Company also quantifies, by titration, the amount of chromic acid present. Such procedure [Brigantine Services Ltd., 2003] is listed in Appendix C. Furthermore, the Teaching Company specifies the density of a brand new bath, and can relate this density to the amount of chromium trioxide that has been dissolved in water. In other words, there exists a strong relationship between the density of the bath, as expressed in degrees of Baume, and the chromic acid concentration. This relationship is often shown in tabular form in Baume charts from various sources.

On the other hand, the Teaching Company uses a chemical analysis method for the measurement of concentration of Sulphuric Acid. Such procedure [Brigantine Services Ltd., 2003] has been listed in Appendix D.

5.1.2 Temperature setting

Generally, the higher the current density, the higher the temperature requirement. For hard chrome plating, the range is 49-65.5 degree C.

However, according to the pilot study that had been done by the Teaching Company Associate, there was not much difference ($\pm 3\%$) in terms of the thickness of the hard chrome deposition when different temperature settings (50-65 degree C) within the operating range were used. Therefore for the core experiments, the Teaching Company Associate has decided that process temperature would be maintained at 55 degree C for the subsequent trials.

The new plating machine had been preheated to the expected bath temperature (55 degree C) before it was introduced to the plating tank to ensure uniformity of deposit. In addition, both heating and cooling coils have been used in the same tank in order to maintain a precise temperature.

5.1.3 Current density

At given solution composition and temperature, the current density affects cathode efficiency. Generally, the optimum current density is recommended by the manufacturer of the plating chemicals used. At too high a current density, burning or roughness of hard chrome deposition occurs. At too low a current density, lack of chromium coverage can be expected.

Current Density is calculated by the amount of applied electric current divided by the surface area to be hard chrome plated. Since in this research study, only one type of piston crown (with a diameter = 900mm) has been used, so the expected area to be hard chrome plated is fixed, leaving the amount of applied electric current as the process variable.

The Teaching Company has used three-phase rectifiers with a maximum of 5% AC ripple, which would supply an uninterrupted flow of current throughout the plating cycle. Standard current densities are in the range of 23.25-100 amp/dm² for hard chromium plating.

5.1.4 Plating time

Generally speaking, it is believed that the longer the plating time, more hard chrome deposition would be found on the surface of the ring grooves. However, one should note that in general, if the plating time is higher than 100 hours, the uniformity of hard chrome deposition would generally be disturbed.

5.1.5 Rotary speed

Since the chromic acid solutions used are fairly concentrated and viscous, stratification may occur. This results in uneven temperature distribution within the solution. Agitation is therefore required to equalize the bath temperature, produce uniform brightness and, in the case of hard chromium, improve deposit hardness.

Unfortunately, according to the report described in Chapter 4, the rotary speed for the new plating machine has to be set within the range 10~15 rpm. Therefore, the Teaching Company has decided that the rotary speed for the subsequent trials would be set at 10 rpm.

In summary, the new hard chrome plating practice is expected to have 4 influential factors. Their operating ranges are listed in Table 2.

Table 2: The influential factors and their ranges in this study

Factors	Minimum	Maximum	Unit
Chromic Acid Concentration	160.0	240.0	Gram/Liter
Sulphuric Acid Concentration	2.4	3.6	Gram/Liter
Electric Current	800.0	950.0	Amps
Plating Time	39.0	52.0	Hours

The Temperature would be maintained at 55 degree C and the Rotary Speed would be set at 10rpm. It was assumed that the errors were independent and identically distributed with zero mean and common variance.

5.2 Experimental design

As discussed in Chapter 2, Full Factorial Design (FFD) method is selected in the expectation to obtain valuable insights in terms of the improvement and understanding of the response from the hard chrome plating system. A common FFD method is one with all input factors set at two levels each. These levels are called 'high' and 'Low' or '+1' and '-1' respectively. A design with all possible high/low combinations of all the input factors is called a full factorial design in two levels. If there are 'k' factors, each at 2 levels, a FFD method has 2^k runs. Table 3 shows the number of runs for 2^k FFD and Table 4 shows the High (+1) and Low (-1) setting for the experiment.

Table 3: The number of Runs for 2^k FFD method

Number of Factors	Number of Runs
2	4
3	8
4	16
5	32
6	64
7	128
8	256

Table 4: The High (+1) and Low (-1) setting for the experiment

Factors	Low (-1)	High (+1)	Unit
Chromic Acid Concentration (X_1)	160.0	240.0	Gram/Liter
Electric Current (X_2)	800.0	950.0	Amps
Sulphuric Acid Concentration (X_3)	2.4	3.6	Gram/Liter
Plating Time (X_4)	39.0	52.0	Hours

As shown by the above table, when the number of factors is 5 or greater, a FFD requires a large number of runs and is not very efficient. For 5 or more factors, a “Fractional Factorial Design” or a “Plackett-Burman Design” would be a better choice.

In this research study, one of the objectives is that, given a set of process variables, the thickness of the hard chrome deposition on the ring groove surface can be confidently predicted. The four inputs (factors) that are considered important to the Teaching Company’s new hard chrome plating process are the Chromic Acid Concentration (X_1), Electric Current (X_2), Sulphuric Acid Concentration (X_3) and Plating Time (X_4). It is the aim to establish the individual and combined effect of each of these factors on the Thickness of the hard chrome deposition (Y).

The process variables (Chromic Acid Concentration, Electric Current, Sulphuric Acid Concentration and Plating Time) can all be varied continuously along their respective scales, from a low to a high setting. The Thickness of hard

chrome deposition is observed to vary smoothly when progressive changes are made to the inputs.

Note that if there are k factors present, each run at two levels, there will be 2^k different combinations of the levels. In this research study, $k = 4$ and $2^4 = 16$.

Running the full complement of all possible factor combinations means that all the main and interaction effects can be estimated. There are a mean effect, four main effects, six two-factor interactions, four three-factor interactions and one four-factor interaction, all of which appear in the full model as follows:

$$\begin{aligned}
 Y = & \lambda_0 + \lambda_1(X_1) + \lambda_2(X_2) + \lambda_3(X_3) + \lambda_4(X_4) \\
 & + \lambda_{12}(X_1X_2) + \lambda_{13}(X_1X_3) + \lambda_{14}(X_1X_4) \\
 & + \lambda_{23}(X_2X_3) + \lambda_{24}(X_2X_4) + \lambda_{34}(X_3X_4) \\
 & + \lambda_{123}(X_1X_2X_3) + \lambda_{134}(X_1X_3X_4) + \lambda_{124}(X_1X_2X_4) + \lambda_{234}(X_2X_3X_4) \\
 & + \lambda_{1234}(X_1X_2X_3X_4) \quad \dots (6)
 \end{aligned}$$

where (X_1) = Chromic Acid Concentration

(X_2) = Electric Current

(X_3) = Sulphuric Acid Concentration

(X_4) = Plating Time

(Y) = Plating Thickness of Hard Chrome Deposition

A full factorial design allows us to estimate all fifteen ' λ ' coefficients ($\lambda_0 \dots \lambda_{1234}$).

Next, the factor settings need to be coded, replacing the low setting by -1 and the high setting by +1, as shown in Table 5.

Table 5: A 2^4 two-level, FFD table showing runs in preset run order

Preset Run Order	X_1	X_2	X_3	X_4
1	-1	-1	-1	-1
2	+1	+1	-1	-1
3	+1	+1	+1	+1
4	-1	+1	+1	+1
5	+1	-1	-1	+1
6	-1	+1	-1	+1
7	-1	-1	+1	-1
8	-1	-1	+1	+1
9	-1	-1	-1	+1
10	+1	+1	+1	-1
11	-1	+1	+1	-1
12	+1	-1	+1	+1
13	+1	-1	-1	-1
14	-1	+1	-1	-1
15	+1	-1	+1	-1
16	+1	+1	-1	+1

Running the entire design more than once makes for easier data analysis. The benefit is that for each run, an average value of the response as well as some

idea about the dispersion (variability, consistency) of the response at that setting. One of the usual analysis assumptions is that the response dispersion is uniform across the experimental space.

Replication permits check this assumption and possibly find the setting combinations that give inconsistent yields, thus avoiding that area of the factor space. Table 6 shows the constructed design table for a two-level full factorial in four factors, replicated twice.

If the design is going to be run, as shown in Table 6, one key deficiency would appear, i.e. no randomization. Using randomization is the most reliable method of creating homogeneous treatment groups, without involving any potential biases or judgments. The more freely one can randomize experimental runs, the more insurance one has against extraneous factors possibly affecting the results.

Table 7 shows the design table with the rows randomized. The preset run order column is also shown for comparison.

Table 6: A constructed design table for a two-level FFD with four factors, replicated twice

Preset Run Order	X_1	X_2	X_3	X_4
1	-1	-1	-1	-1
2	+1	+1	-1	-1
3	+1	+1	+1	+1
4	-1	+1	+1	+1
5	+1	-1	-1	+1
6	-1	+1	-1	+1
7	-1	-1	+1	-1
8	-1	-1	+1	+1
9	-1	-1	-1	+1
10	+1	+1	+1	-1
11	-1	+1	+1	-1
12	+1	-1	+1	+1
13	+1	-1	-1	-1
14	-1	+1	-1	-1
15	+1	-1	+1	-1
16	+1	+1	-1	+1
17	-1	+1	+1	+1
18	-1	-1	-1	+1
19	-1	-1	+1	-1
20	-1	-1	-1	-1
21	-1	+1	+1	-1
22	+1	+1	-1	-1
23	+1	+1	-1	+1
24	+1	-1	+1	+1
25	+1	+1	+1	-1
26	-1	-1	+1	+1
27	+1	-1	-1	-1
28	-1	+1	-1	-1
29	+1	+1	+1	+1
30	+1	-1	+1	-1
31	-1	+1	-1	+1
32	+1	-1	-1	+1

Table 7: A constructed design table for a two-level FFD with four factors, replicated twice, with random run order indicated

Random Run Order	Preset Run Order	X ₁	X ₂	X ₃	X ₄
2	1	-1	-1	-1	-1
7	2	+1	+1	-1	-1
31	3	+1	+1	+1	+1
6	4	-1	+1	+1	+1
19	5	+1	-1	-1	+1
22	6	-1	+1	-1	+1
10	7	-1	-1	+1	-1
20	8	-1	-1	+1	+1
17	9	-1	-1	-1	+1
16	10	+1	+1	+1	-1
21	11	-1	+1	+1	-1
28	12	+1	-1	+1	+1
4	13	+1	-1	-1	-1
5	14	-1	+1	-1	-1
12	15	+1	-1	+1	-1
9	16	+1	+1	-1	+1
29	17	-1	+1	+1	+1
18	18	-1	-1	-1	+1
30	19	-1	-1	+1	-1
1	20	-1	-1	-1	-1
14	21	-1	+1	+1	-1
8	22	+1	+1	-1	-1
23	23	+1	+1	-1	+1
27	24	+1	-1	+1	+1
15	25	+1	+1	+1	-1
25	26	-1	-1	+1	+1
3	27	+1	-1	-1	-1
13	28	-1	+1	-1	-1
32	29	+1	+1	+1	+1
11	30	+1	-1	+1	-1
26	31	-1	+1	-1	+1
24	32	+1	-1	-1	+1

5.3 Experimental procedure

The flow chart of the experimental procedure for the new hard chrome plating system is shown in Figure 17.

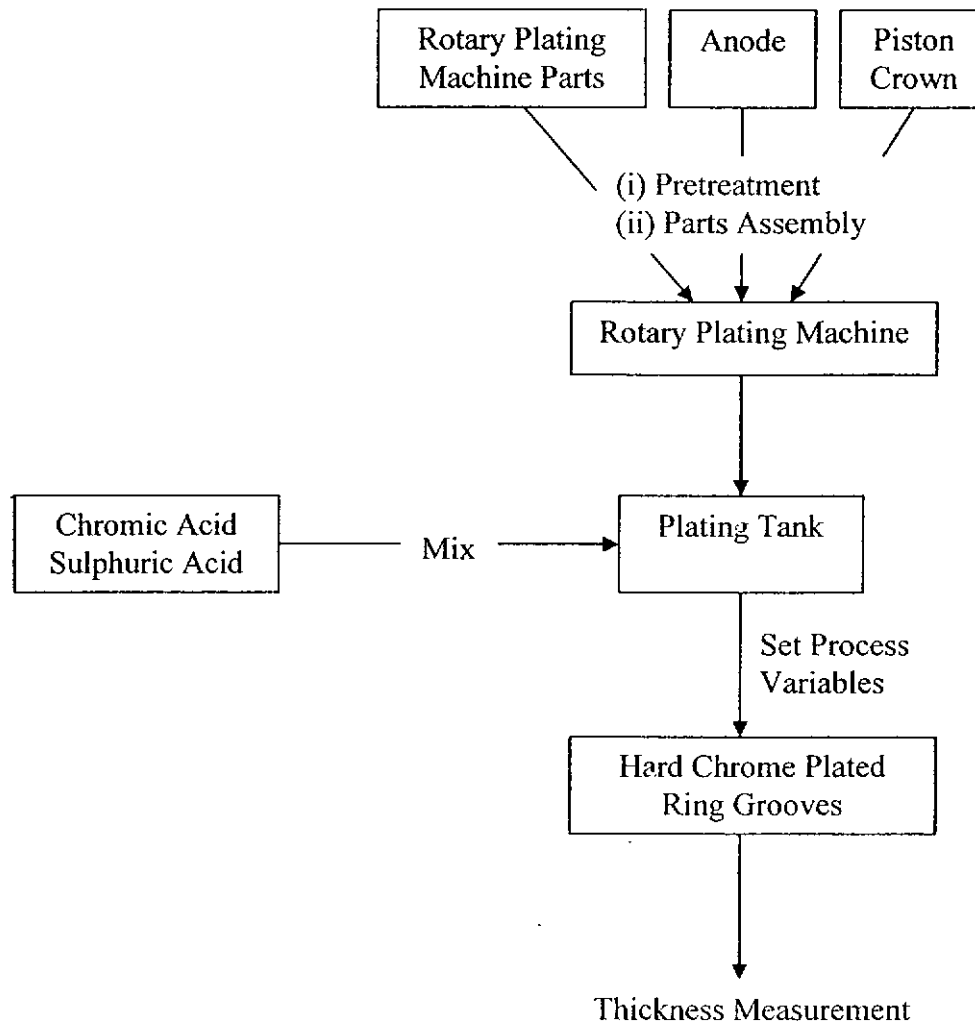


Figure 17: Flow chart for the experimental procedure

5.4 Analysis of experimental data

Recalling the description of this research experiment:

Purpose: To determine the effect of process factors on plating thickness

Response variable: Plating Thickness of Hard Chrome Deposition (10^{-2} mm)

Number of observations = 16 (a complete 2^4 factorial design)

Response Variable Y = Plating Thickness of Hard Chrome Deposition

Factor 1 = Chromic Acid Concentration

[2 levels: Low (160 g/l) and High (240 g/l)]

Factor 2 = Electric Current

[2 levels: Low (800 amps) and High (950 amps)]

Factor 3 = Sulphuric Acid Concentration

[2 levels: Low (2.4 g/l) and high (3.6 g/l)]

Factor 4 = Plating Time

[2 levels: Low (39 hrs) and High (52 hrs)]

The design matrix, with the plating thickness of the hard chrome deposition responses, appears in Table 8. The actual randomized run order is given in the first column.

In the context of the FFD method, the investigation procedure would include the following steps:

- To identify any constraints on the selected key factors in order to specify the experimental region.
- To identify the key response variable to be measured.
- To propose an appropriate model for modeling the response data as function of the factors selected.
- To select an experimental design that is sufficient not only to fit the proposed model, but which allows a test of model adequacy as well.

Through the study, it is assumed that the errors are independent and identically distributed with zero mean and common variance.

Figure 18 shows the Plot of Responses versus Run Order to check whether there might be a time sequence component affecting the response levels. As expected, this plot does not indicate that time order has much to do with the response levels.

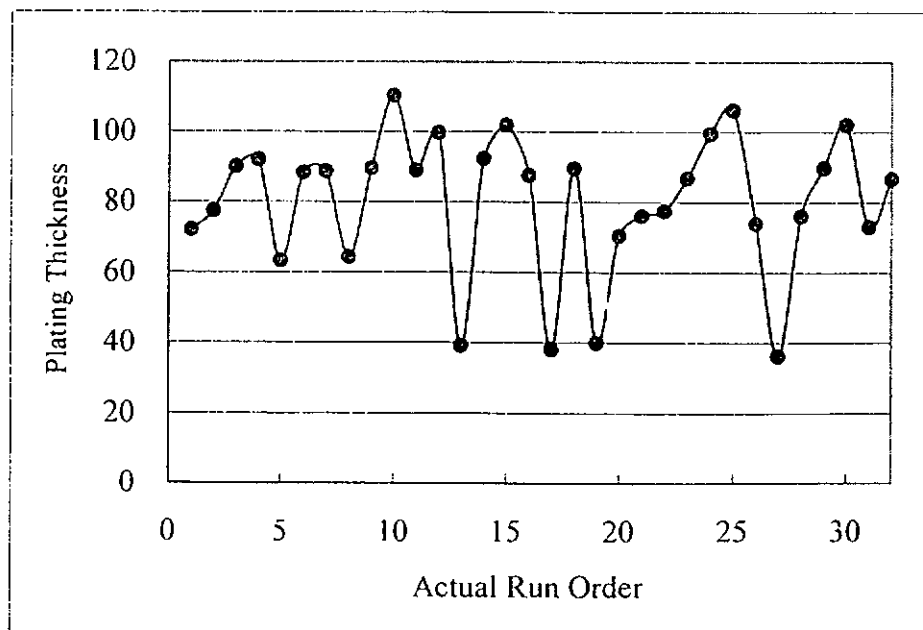


Figure 18: The plot of plating thickness vs. actual run order

Table 8: A design table with the plating thickness of the hard chrome deposition as outcome

Random Order	Run	Chromic Acid Conc.	Electric Current	Sulphuric Acid Conc.	Plating time	Actual Result
2	1	160	800	2.4	39	72
7	2	214	950	2.4	39	77
31	3	214	950	3.6	52	90
6	4	160	950	2.4	39	92
19	5	214	800	2.4	52	63
22	6	160	950	2.4	52	88
10	7	160	800	3.6	39	89
20	8	160	800	3.6	52	64
17	9	160	800	2.4	52	90
16	10	214	950	3.6	39	110
21	11	160	950	3.6	39	89
28	12	214	800	3.6	52	100
4	13	214	800	2.4	39	39
5	14	160	950	2.4	39	92
12	15	214	800	3.6	39	102
9	16	214	950	2.4	52	88
29	17	160	950	3.6	52	38
18	18	160	800	2.4	52	90
30	19	160	800	3.6	39	40
1	20	160	800	2.4	39	70
14	21	160	950	3.6	39	76
8	22	214	950	2.4	39	77
23	23	214	950	2.4	52	87
27	24	214	800	3.6	52	99
15	25	214	950	3.6	39	106
25	26	160	800	3.6	52	74
3	27	214	800	2.4	39	36
13	28	160	950	2.4	39	76
32	29	214	950	3.6	52	90
11	30	214	800	3.6	39	102
26	31	160	950	2.4	52	73
24	32	214	800	2.4	52	87

5.5 Multiple regression analysis

The most powerful application in using FFD method is for understanding how the changes in process variables would affect the response of the system. This is performed by determining the individual and combined effects of the process variables. For instance, if three process variables jointly influence the response, this would be a three-way combined effect. Such stimulus is judged statistically significant if the variation in the response caused by changing the combination of variable settings is larger than the experimental error in the measurement of the response. Several statistical methods can be employed for the determination of the main and interaction effects including Yate's algorithm and Analysis Of Variance (ANOVA). Discussion of Yate's algorithm is beyond the scope of this review, but is fully described in several textbooks on experimental design methodology [Daniel, 1976; Law & Kelton, 1982; Arora, 1989; Hinkelman, 1994].

With the assistance of JMP[®] software [SAS Institute, Inc., 1995], the experimental design matrix for the hard chrome plating process for a piston crown of diameter 900mm is constructed as shown in Table 9. Multiple regression technique was used to model the main effects and interactions between selected key factors.

Table 9: The design summary of the hard chrome plating process for a piston crown of diameter 900mm

Study Type	Factorial
Experiments	32
Initial Design	2 Level Factorial
Center Points	0
Design Model	2FI

Response	Name	Units	Observations	Min	Max
Y1	Thickness	0.01mm	32	36.3	110.0

Factor	Name	Units	Low Actual	High Actual	Low Coded	High Coded
X ₁	Chromic Acid	Gram/Liter	160.00	214.00	-1	1
X ₂	Electric Current	Amps	800.00	950.00	-1	1
X ₃	Sulphuric Acid	Gram/Liter	2.40	3.60	-1	1
X ₄	Plating Time	Hours	39.00	52.00	-1	1

The analyze procedure for this research study comprises the following:

- Form an initial model
- Perform statistical testing
- Refine model
- Analyze residuals
- Interpret results

With a 2^4 full factorial experiment, one can fit a model containing a mean term, four main effect terms, six two-factor interaction terms, four three-factor interaction terms and one four-factor interaction term (16 parameters). However, it can be assumed that all three factor and higher interaction terms are non-existent, since it is infrequently that for such high-order interactions to be significant. That allows us to accumulate the sums of squares for these terms and

use them to estimate an error term. As a result, a theoretical model can be started out with 11 unknown constants, hoping the data will clarify which of these are the significant main effects and interactions for the development of the final model. Table 10 shows the ANOVA for the initial FFD model.

Table 10: Analysis Of Variance (ANOVA) for the initial FFD model

Response: Plating Thickness

ANOVA for Selected Factorial Model

Analysis of variance table

Source	Sum of Squares	D	Mean Square	F Value	Probability > F
Model	12688.95	10	1268.90	47.15	< 0.0001
X ₁	658.85	1	658.85	24.48	< 0.0001
X ₂	588.25	1	588.25	21.86	< 0.0001
X ₃	159.31	1	159.31	5.92	< 0.0024
X ₄	413.28	1	413.28	15.36	< 0.0008
X ₁ X ₂	1800.00	1	1800.00	66.89	< 0.0001
X ₁ X ₃	3473.61	1	3473.61	129.08	< 0.0001
X ₁ X ₄	42.78	1	42.78	1.59	0.2212
X ₂ X ₃	3030.31	1	3030.31	112.61	< 0.0001
X ₂ X ₄	1267.56	1	1267.56	47.10	< 0.0001
X ₃ X ₄	1255.01	1	1255.01	46.64	< 0.0001
Residual	565.10	21	26.91		
Pure Error	54.24	16	3.39		

According to Table 11, this fit has high R^2 and adjusted R^2 coefficients, but has one high (>0.10) p-values (in the "Prob>F" column), makes it clear that the source (X_1X_4) can be regarded as an unnecessary term. In other words, X_1 , X_2 , X_3 , X_4 , (X_1X_2), (X_1X_3), (X_2X_3), (X_2X_4), (X_3X_4) are significant model terms. The Model F-value of 47.15 implies the model is significant. Also, there is only a 0.01% chance that a "Model F-Value" of this magnitude could occur due to noise. Furthermore, the values of "Probability > F" less than 0.0500 indicate model terms are significant.

5.6 Multiple coefficients of correlation

In this section, the objective is to find out how accurate is the model, and it is done via the examination of R-squared coefficient. The R-squared coefficient is the square of the correlation coefficient between Y (the observed value of dependent variable) and the predicted value of Y from the fitted line. If all the observations fall on the regression line, R^2 is 1. If there is no linear relationship between the dependent and independent variables, R^2 is 0.

The statistically adjusted R^2 attempts to correct R^2 to more closely reflect the goodness of fit of the model in the population. Adjusted R^2 is given by the following formula:

$$(\text{Adjusted } R)^2 = R^2 - [p(1 - R^2)/(N - p - 1)]$$

where p is the number of independent variables (4 in the present case).

Table 11: The calculation of R-squared coefficient

<u>Std. Dev.</u>	5.19		<u>R-Squared</u>	0.9574
<u>Mean</u>	81.69		<u>Adj R-Squared</u>	0.9371
<u>C.V.</u>	6.35		<u>Pred R-Squared</u>	0.9010
<u>PRESS</u>	1312.17		<u>Adeq Precision</u>	25.3870

According to Table 11, the “Predicted R-Squared” (measures the amount of variation in new data explained by the model) of 0.9010 is in reasonable agreement with the “Adj R-Squared” of 0.9371. The term “Adeq Precision” measures the range in predicted response relative to its associated error, in other

words a signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 25.387 indicates an adequate signal. This model can then be used to navigate the design space.

The next step is to do a result comparison between the FFD and the actual experiment. From the above examination, one can classify the following equation as a valid equation:

Plating Thickness (Y)

$$\begin{aligned}
 = & -263.72037 \\
 & -5.30185 (X_1) + 0.60074 (X_2) + 145.73418 (X_3) + 14.32888 (X_4) \\
 & +3.70370E-003 (X_1X_2) + 0.64313 (X_1X_3) - 0.21625 (X_2X_3) \\
 & - 0.012910 (X_2X_4) - 1.60577 (X_3X_4)
 \end{aligned}$$

where (X_1) = Chromic Acid Concentration

(X_2) = Electric Current

(X_3) = Sulphuric Acid Concentration

(X_4) = Plating Time

(Y) = Plating Thickness of Hard Chrome Deposition

A comparison between the values obtained from the FFD vs. the actual values is shown in Table 12. According to the table, the maximum deviation between the predicted (FFD) value and the actual value is 9.8%. As the maximum deviation is less than $\pm 10\%$ of the actual value, the equation obtained

from the FFD method can be regarded as a good tool to predict the plating thickness of hard chrome deposition.

Table 12: A comparison between the predicted (FFD) values vs. the actual values

Random Order	Run	Chromic Acid Conc.	Electric Current	Sulphuric Acid Conc.	Plating Time	FFD Result	Actual Result	Diff. (%)
2	1	160	800	2.4	39	71	72	-1.4
7	2	214	950	2.4	39	85	77	9.8
31	3	214	950	3.6	52	86	90	-4.6
6	4	160	950	2.4	39	97	92	5.4
19	5	214	800	2.4	52	62	63	-1.9
22	6	160	950	2.4	52	87	88	-1.6
10	7	160	800	3.6	39	87	89	-1.8
20	8	214	800	2.4	52	62	64	-3.6
17	9	160	800	2.4	52	87	90	-2.9
16	10	214	950	3.6	39	116	110	5.2
21	11	160	950	2.4	52	87	89	-2.1
28	12	214	800	3.6	52	94	100	-5.7
4	13	214	800	2.4	39	42	39	7.7
5	14	160	950	2.4	39	97	92	5.1
12	15	214	800	3.6	39	99	102	-2.8
9	16	160	800	3.6	39	87	88	-0.7
29	17	160	950	3.6	52	39	38	2.6
18	18	160	800	2.4	52	87	90	-2.9
30	19	160	950	3.6	52	39	40	-2.0
1	20	160	800	2.4	39	71	70	1.0
14	21	160	950	3.6	39	73	76	-3.9
8	22	214	950	2.4	39	84	77	8.5
23	23	214	950	2.4	52	93	87	7.1
27	24	214	800	3.6	52	94	99	-5.4
15	25	214	950	3.6	39	116	106	9.3
25	26	160	800	3.6	52	77	74	4.2
3	27	214	800	2.4	39	39	36	8.0
13	28	160	950	3.6	39	73	76	-3.9
32	29	214	950	3.6	52	86	90	-4.2
11	30	214	800	3.6	39	99	102	-2.9
26	31	160	800	3.6	52	77	73	5.5
24	32	214	950	2.4	52	93	87	7.3

With the completion of the comparison between the predicted vs. the actual values, the focus is now switched to model adequacy checking, including the checking of normality of residuals, outliers, and influential cases.

5.7 Model adequacy diagnosis

5.7.1 Normal probability plot of residuals

Non-significant effects would effectively follow an approximately normal distribution with the same location and scale. Significant effects will vary from this normal distribution. Therefore, another method of determining significant effects is to generate a normal plot of all 32 effects. Those effects that are substantially away from the straight line fitted to the normal plot are considered significant. Although this is a somewhat subjective criterion, it tends to work well in practice. It is helpful to use both the numerical output from the fit and graphical techniques such as the normal plot in deciding which terms to keep in the model.

Linearity shown on a normal probability plot of the studentized residuals will indicate the normality of residuals. The studentized residual is the number of standard deviations that separate the actual and predicted response values. It is the residual divided by the estimated standard deviation of the residual. As shown in Figure 19, almost all of the studentized residuals fall into a straight gradient line. Therefore, it indicates no abnormalities, meaning the plot looks OK in this case.

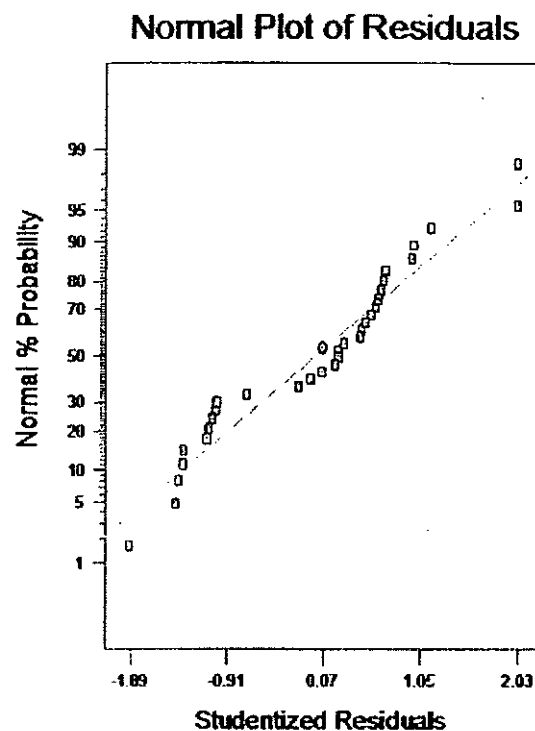


Figure 19: A normal plot of residuals

5.7.2 Interaction analysis

An interaction effect usually means a combined and simultaneous influence of two or more independent variables on the dependent variable. Alternatively, interaction effects are sometimes called moderator effects because the interacting third variable which changes the relation between two original variables is a moderator variable which moderates the original relationship. For instance, the relation between income and conservatism may be moderated depending on the level of education. The significance of an interaction effect is the same as for any other variable, except in the case of a set of dummy variables representing a single ordinal variable. When an ordinal variable has been entered as a set of dummy variables, the interaction of another variable with the ordinal variable will involve multiple interaction terms.

Figure 20 shows an interaction relationship exists between the Chromic Acid Concentration and the applied Electric Current, with the concentration of the Sulphuric Acid is set at 3 grams/liter and the Plating Time is set at 45.5 hours. According to Figure 20, it shows that when the amount of Electric Current being applied is 800 amps, an increase in the concentration of Chromic Acid would have a decreasing effect in the plating thickness of the hard chrome deposition. On the other hand, when the amount of Electric Current being applied is 950 amps, an increase in the concentration of Chromic Acid would have an increasing effect in the plating thickness of the hard chrome deposition. Those points that have non-overlapping intervals are significantly different. In this case the spread of the points on the right side of the graph (where concentration of Chromic Acid is high) is much larger than the spread between the points at the left side of the graph (where concentration of Chromic Acid is low.) In other words, the effect of the Electric Current is much more significant at the high level of the concentration of Chromic Acid. Therefore, the experimenters usually need to maintain a high concentration of Chromic Acid and supply a high amount of Electric Current in order to maintain the required rate of hard chrome deposition.

AB interaction

X = A: Chromic Acid Conc
Y = B: Current

■ B- 800.000
▲ B+ 950.000

Actual Factors

C: Sulphuric Acid Conc = 3.00

D: Plating time = 45.50

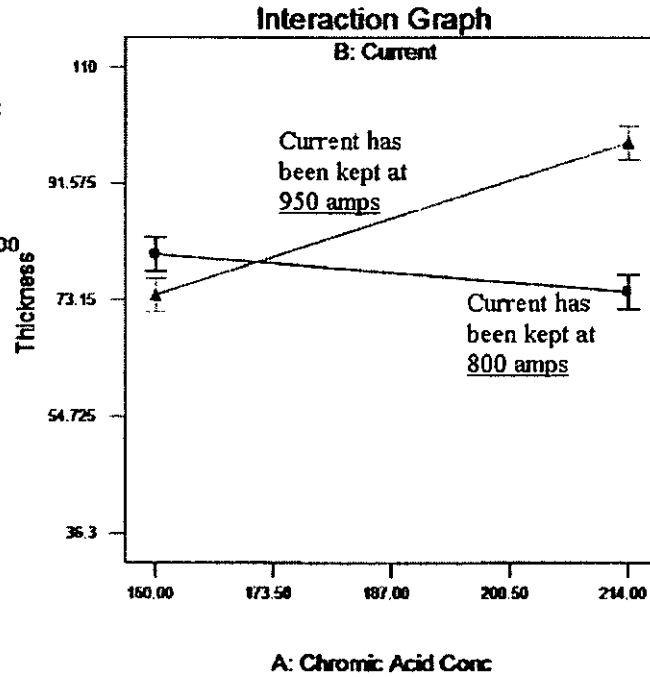


Figure 20: The interaction graph between chromic acid concentration and electric current

And in Figure 21, an interaction relationship also exists between the Chromic Acid Concentration and the Sulphuric Acid Concentration, with the Electric Current being set at 875 amps and the Plating Time at 45.5 hours. According to Figure 21, it shows that when the concentration of Sulphuric Acid is set at 2.4 gram/liter, an increase in the concentration of Chromic Acid would have a decreasing effect in the plating thickness of the hard chrome deposition. On the other hand, when the concentration of Sulphuric Acid is set at 3.6 gram/liter, an increase in the concentration of chromic acid would have an increasing effect in the plating thickness of the hard chrome deposition. Those points that have non-overlapping intervals are different. In this case the spread of the points on the right side of the graph (where concentration of Chromic Acid is high) is slightly larger than the spread between the points at the left side of the graph (where concentration of Chromic Acid is low.) In other words, the effect of

the concentration of Sulphuric Acid is slightly more significant at the high level of the concentration of Chromic Acid. Therefore, if the experimenters want to increase the concentration of Chromic Acid, they need to increase concentration of Sulphuric Acid as well in order to maintain the required rate of hard chrome deposition.

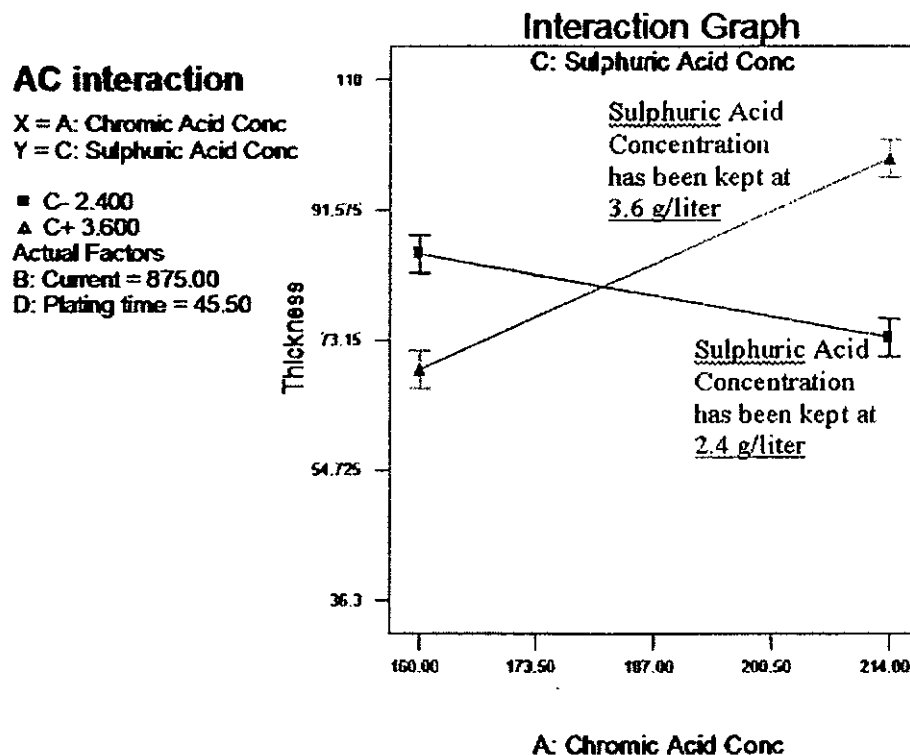


Figure 21: The interaction graph between chromic acid concentration and sulphuric acid concentration

Figure 22 shows that, with the Electric Current being set at 875 amps and the concentration of Sulphuric Acid being set at 3.6 gram/liter, there is no interaction relationship between the Chromic Acid Concentration and the Plating Time. In fact, this is the only case that shows no interaction relationship.

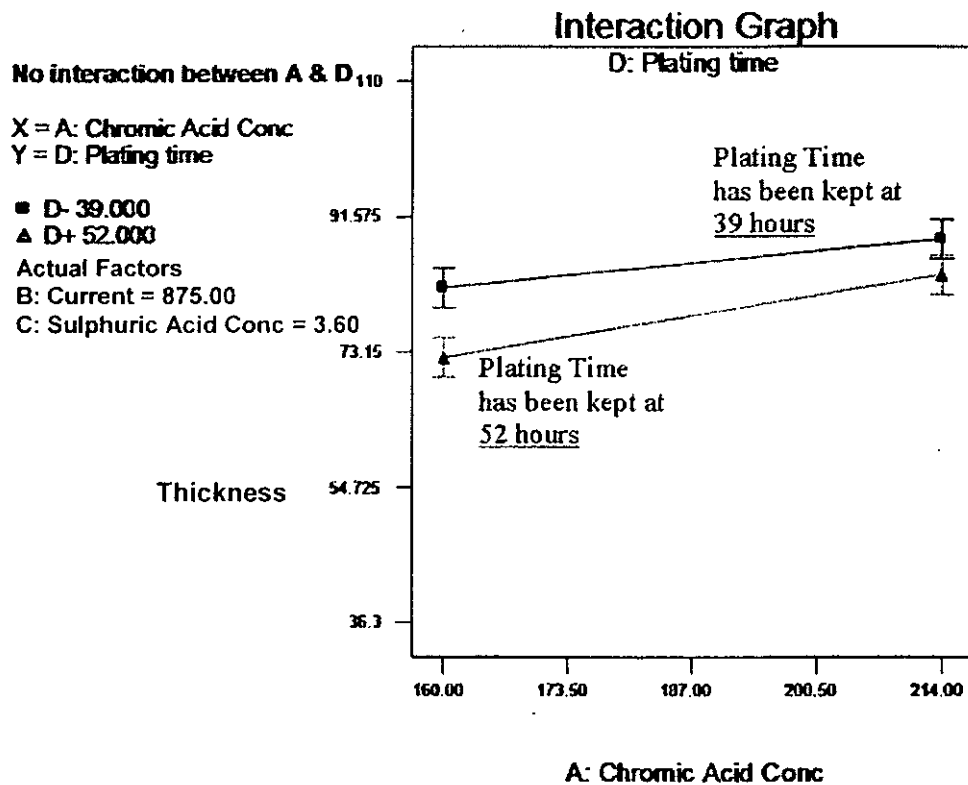


Figure 22: The interaction graph between chromic acid concentration and plating time

5.7.3 Outlier analysis

Outliers are data points which lie outside the general linear pattern of which the midline is the regression line. A rule of thumb is that outliers are points whose standardized residual is greater than 3.5. The removal of outliers from the data set under analysis can at times dramatically affect the performance of a regression model. Outliers should be removed if there is reason to believe that other variables not in the model explain why the outlier cases are unusual -- that is, these cases need a separate model. Alternatively, outliers may suggest that additional explanatory variables need to be brought into the model. Another alternative is to use robust regression, whose algorithm gives less weight to outliers but does not discard them.

In the case of the possibility in locating any outliers, one can spot outliers readily on residual plots, since they are cases with very large positive or negative residuals. In general standardized residual values greater than an absolute value of 3 are considered outliers. From Figure 23, there seems nothing out of the ordinary here - all the points fall well within the red lines set at plus-or-minus 3.5.

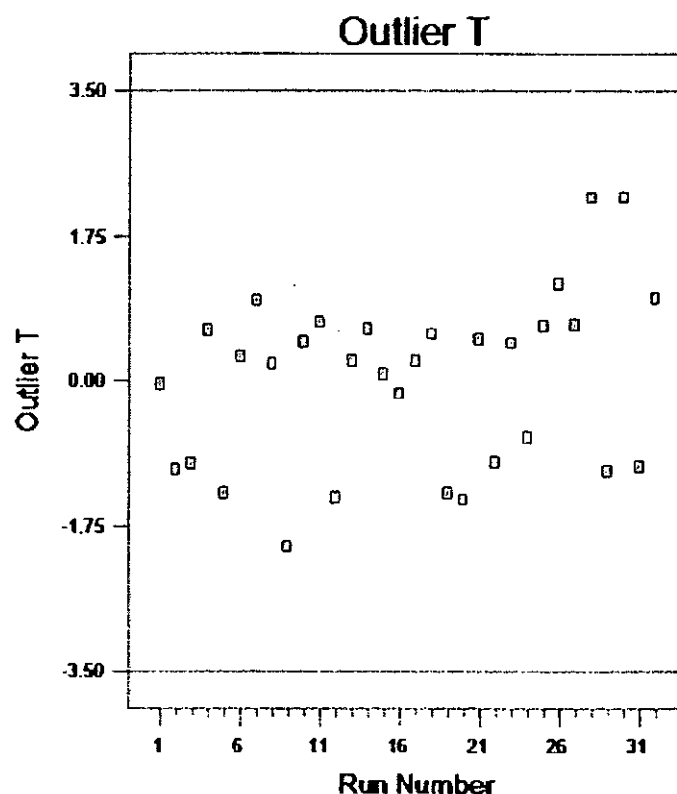


Figure 23: A plot between Run numbers and the Outlier T

5.7.4 Influential cases

Certain observations in a set of data can have a large influence on estimates of the dependent variable. One way to identify an influential case is to consider changes in all residuals when a certain case is omitted – Cook's distance. Cook's distance is one of the measures of the influence of a case. Cases with larger Cook's distance values than the rest of the data are those which have

unusual leverage. Fox [1991] suggests as a cut-off for detecting influential cases, values of the Cook's distance greater than $4/(n - k - 1)$, where n is the number of cases and k is the number of independents. A large Cook's distance identifies the case as an influential point. The plot of Cook's Distance can review the influence of each point on the model fit to help you decide whether to remove an outlier. In this case, according to Figure 24, the probability to have any influential cases seems to be quite small.

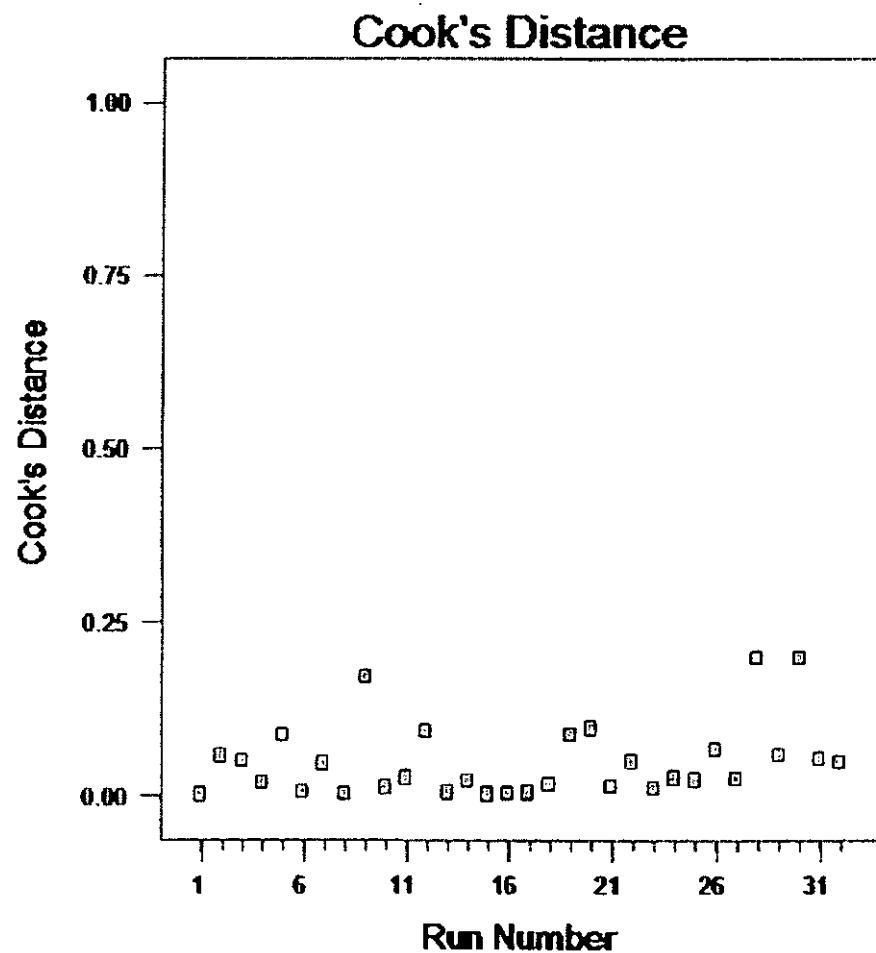


Figure 24: A plot between the Run numbers and the Cook's distance

5.7.5 Leverage analysis

The leverage analysis is another method to identify cases which influence the regression model more than others. The leverage statistic varies from 0 (no influence on the model) to 1 (completely determines the model). A rule of thumb is that cases with leverage under 0.2 are not a problem, but if a case has leverage over 0.5, the case has undue leverage and should be examined for the possibility of measurement error or the need to model such cases separately. In this case, according to Figure 25, there seems to be little chance for a design point to influence the fit of the model coefficients. However, it should be noted that Leverage near unity should be avoided. Generally, one can replicate the point or add more design points to reduce leverage.

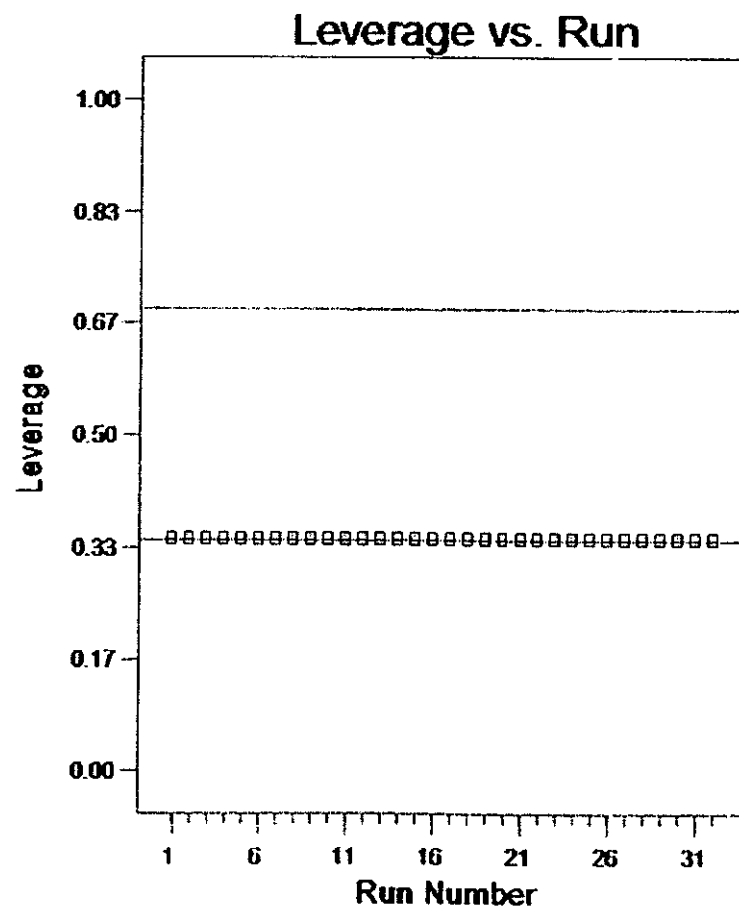


Figure 25: A plot between the Run numbers and the Leverage values

5.8 Verification of FFD results

When the analysis of the experiment is completed, one must verify that the predictions are good. These are called confirmation runs. The interpretation and conclusions from an experiment may include a "best" setting to use to meet the goals of the experiment. Even if this "best" setting were included in the design, such experimental design would be run again as part of the confirmation runs to make sure nothing has changed and that the response values are close to their predicted values would get. In an industrial setting, it is very desirable to have a stable process.

Therefore, in this research study, 6 runs have been conducted to allow an estimate of variability at that setting. Apart from the two extreme cases (the case with coded variables -1,-1,-1,-1; the case with coded variables +1,+1,+1,+1), the rest of confirmation runs had changes in either Electric Current or Plating Time. The results obtained from the confirmation run were shown in Table 13. It was verified that the predictions are good since the maximum difference was still within the range obtained from the FFD experiment.

Table 13: A comparison between the values obtained from the predicted (FFD) model vs. the ones obtained from the confirmation run

Run	Chromic Acid Conc.	Electric Current	Sulphuric Acid Conc.	Plating time	FFD Result	Confirmation Run Result	Diff (%)
1	160, (-1)	800, (-1)	2.4, (-1)	39, (-1)	71	78	-9.0
3	214 (+1)	950 (+1)	3.6 (+1)	52 (+1)	86	88	-2.3
4	160, (-1)	950, (+1)	2.4, (-1)	39, (-1)	97	98	-1.0
6	160, (-1)	950, (+1)	2.4, (-1)	52, (+1)	87	87	0.0
9	160, (-1)	800, (-1)	2.4, (-1)	52, (+1)	87	92	-5.4
10	214, (+1)	950, (+1)	3.6, (+1)	39, (-1)	116	110	5.5

Despite the difficulties, such as the handling of large size of the piston crown and the working environment, these confirmation runs were conducted in an environment as similar as possible to the original experiment. For example, if the experiments were initialized in the afternoon and the rotary plating machine needed to be pre-heated before actual operation, the confirmation runs would then be initialized in the afternoon after the rotary plating machine had been preheated. Other extraneous factors that might change or affect the results of the confirmation runs include the possible break down of the motor, the person/operator on the equipment, the sudden collapse of the rectifier/heat exchanger, etc.

6. Process modeling and prediction by Neural Network (NN) Approach

In engineering applications, process variable-based control systems are normally employed in situations where variables such as heating temperature and component concentration need to be adjusted to achieve the required outcome of the overall condition. Traditionally, Proportional-Integral-Derivative (PID) control algorithms are adopted to deal with these parameter-based control situations albeit complex mathematical equations need to be used to analyze the operating conditions [Buchanan & Shortliffe, 1989; Lau & Wong, 2000]. However, the mathematical analysis based on relevant algorithms may become more complex when dealing with multiple input/output control situations where more than one input is used with more than one output. The mathematical equations involved in the PID control algorithms of such situation are rather complex. Computational methods of non-linear programming with constraints usually have to cope with problems such as numerical evaluation of derivatives and feasibility issues. On the other hand, direct search methods, are usually less efficient and more time-consuming, as they usually require a higher number of iterations. The lower efficiency of direct search methods results from the necessity of solving the non-linear model equations in each iteration process.

This chapter intends to introduce a process modeling and prediction approach incorporating NN capabilities. Such approach can be more reliable,

readily deals with constraints, avoids several typical numerical problems of conventional optimization tools, and is not computationally time-consuming.

6.1 NN approach for process modeling and prediction

A Neural Network (NN) comprises computer programs that are designed to simulate some functions of the human brain using different learning algorithms, which can learn from experience. NN has the remarkable information processing features of human brain such as nonlinearity, high parallelism, robustness, fault and failure tolerance, learning, ability to handle imprecise and fuzzy information and their capability to generalize [Basheer & Hajmeer, 2000]. Thus, NN can be used to solve complicated real life problems such as pattern classification, clustering, function approximation, process modeling and prediction.

There are many kinds of NN models that have been developed for numerous different applications. Based on the learning (training) algorithm, training of the NN model could be supervised or unsupervised. For supervised training, the NN model is presented with input/output data sets; for unsupervised training, the NN model is presented with input data alone, and the model learns to recognize patterns in the data. Based on the topology, the connection of NN could be feedforward and feedback. In a feedforward NN model, the connections between the nodes do not form cycles. In a feedback or recurrent NN model, there are cycles in the connections. In some feedback NN models, each time an input is presented, the NN model must iterate for a potentially long time before it

produces a response. Feedback NN models are usually more difficult to train than feedforward NN models [Sarle, 2002].

6.1.1 Types of NN models

NN models can be classified into three categories based on their functions [Bourquin et. al., 1997]:

- Associating networks
- Feature extracting networks
- Nonadaptive networks

Associating networks, which are employed for data classification and prediction, need input (independent variable) and correlated output (dependent variable) values to perform supervised learning. Feature-extracting networks, which are used for data dimension reduction, need only input values to perform unsupervised or competitive learning. Nonadaptive networks need input values to learn the pattern of the inputs and reconstruct them when presented with incomplete data set. Among these three types of NN models, associating networks can be employed to develop controlled release formulations, because the relationship in hard chrome plating between formulation and process variables may not be linear, and is not well understood.

Associating NN models can map the relationship between the formulation and process variables through learning or training processes. These networks can then be used to predict the plating thickness of hard chrome deposition with

different piston crown dimensions and process variables. These networks can also be used for optimization, in which the optimal formulation and process parameters can be selected by a trained NN model. Rumelhart et al. [1986] first introduced the associating NN based on delta rule back-propagation of errors. Since then, many learning algorithms based on the back-propagation have been developed. These algorithms are the most commonly employed in NN for process modeling and prediction.

6.1.2 Back-propagation NN model architecture

Back-propagation NN models have multi-layered architecture as shown in Figure 26 [Wu et al., 2000]. The first layer is called the input layer, which does not have computing activity. It is simply used to input independent variables such as various significant formulations and process factors (inputs), to the first hidden layer. The last layer is called the output layer, which is used to process outcome for the dependent variables such as in vitro drug release profiles (outputs). Hidden layers stay in between the input and output layers, and provide the interconnection between the input and output layers. The connection could be fully connected or partially connected. For a fully connected NN model, each node on the first layer is connected to every node on the second layer. For partially connected NN models, a node of the first layer does not have to be connected to all the nodes on the second layer. The direction of the connection can be feed forward and bi-directional. For feed forward connection, the nodes on the first layer send their output to the nodes on the second layer, but they do not receive any input back from the nodes on the second layer. For the bi-

directional connection, there is another set of connections carrying the output of the nodes of the second layer into the nodes of the first layer.

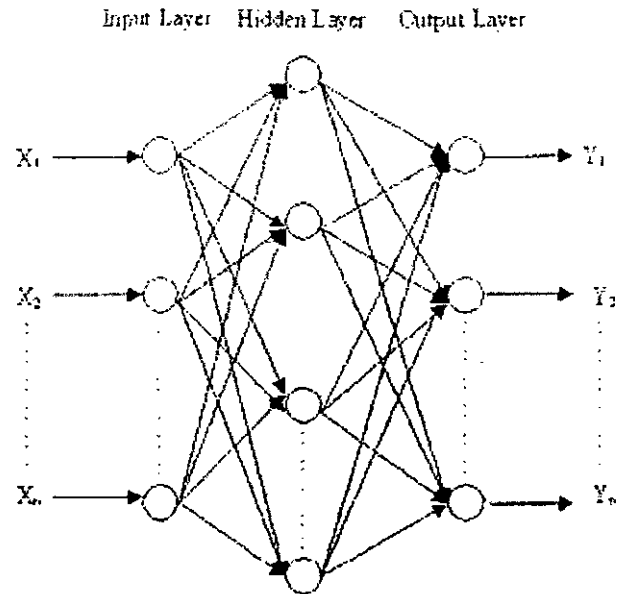


Figure 26: Back-propagation NN model with multi-layered architecture [Wu et al., 2000]

The number of hidden layers is determined by the complexity of the problem. Many NN models consist of only one hidden layer, since one hidden layer is normally adequate to provide an accurate prediction. More than one hidden layer can be used for modeling complex problems.

The building components in NN are processing elements, which are called artificial neurons or nodes. These artificial nodes process information based on weighted inputs using their transfer function and send out outputs. The nodes in adjacent layers are fully or partially interconnected with weighted links. The net input into the j th layer node ($i[j]$) equals the sum of weighted outputs from the prior i th layer ($o[i]$) [Erb, 1993]:

$$\text{Net input to a node} = i[j] = \sum_i \{w[ji] o[i]\} \quad \dots (7)$$

where, $w[ji]$ is the weight factor.

The weight factors of the links between the processing nodes play a critical role during the learning process by the NN model. They are part of the memory capacity of the NN model, since the numerical values of the weight factors change according to the training data sets, in order to minimize the difference between the actual outputs and model predicted outputs. Thus, the relationship between causal factors and response is mapped during the learning process.

The transfer function of processing nodes is used to determine the output value of the node based on the total net input from nodes in prior layer. The most widely used transfer function is a sigmoid function, which is shown in the following equation [Erb, 1993]:

$$\text{Output from a node} = o[j] = 1/\{1+\exp(-i[j])\} \quad \dots (8)$$

where, $o[j]$ is the output from each j th hidden layer node and; $i[j]$ is the sum of the net inputs from nodes in the prior layer.

The number of nodes in the input layer is determined by the number of independent variables that need to be investigated. The number of nodes in the output layer is determined by the number of dependent variables. The number of hidden layers and number of hidden nodes in each layer is strongly dependent on the complexity of the problems such as number of input and output variables,

number of training data set and required prediction accuracy, which need to be studied. The optimal number of hidden nodes depends on the following number of factors:

- numbers of input and output nodes
- number of training data sets
- amount of noise in the targets
- complexity of the function or classification to be learned
- architecture
- type of hidden node activation function
- training algorithm

Too few hidden layers and number of hidden nodes would hamper the learning capability of the NN model, while too many of them can cause over fitting or memorization of the training data set. Several rules of thumb to select the number of hidden nodes in an NN model have been proposed by various investigators. Kolomogorov's theorem states that twice the number of input variables plus one is enough hidden nodes to compute any arbitrary continuous function [Hecht-Nielsen, 1987]. Jadid and Fairbairn [1996] proposed an upper limit of number of hidden nodes using the following equation:

$$N_{\text{hidden}} = N_{\text{trn}} / [R + (N_{\text{inp}} + N_{\text{out}})] \quad \dots (9)$$

where N_{hidden} is the number of hidden nodes;

N_{trn} is the number of training sample;

R is a constant with values ranging from 5 to 10;

N_{inp} is the number of inputs;

N_{out} is the number of outputs.

The number of nodes in the hidden layer is normally defined on the basis of heuristic guidelines. A very common approach to select the optimal number of hidden nodes is by trial and error method using the aforementioned rules as guidance. Some other optimizing techniques such as growing and pruning methods have also been used to select the optimal number of hidden nodes [Sietsma & Dow, 1988].

6.1.3 The learning ability of NN model

NN models learn from experience, which is acquired through a training process. The training process involves fitting of the data to a neural network model. Training is supervised for an associating NN model in which the model is presented with input/output data sets. Training or learning by the NN model is the process of adjusting the weighting factors of links among the processing nodes when the training data sets (known input/output data sets) or data are presented to the NN model. Training data set can be presented to the network model example-by-example or as the whole batch [Wythoff, 1993; Zupan & Gasteiger, 1993]. The weights are updated after processing each sample for the incremental training process or after processing the entire training set for the batch training process. The method used to adjust the weight factor is called training algorithm. For back-propagation NN models, delta rule back-propagation of errors is used as the training algorithm. After an NN model has

been designed and the initial weight factors assigned random small values, the NN model can be trained through an iterative process of presenting training data sets to the model and adjusting weight factors. Each iteration includes two steps, a feedforward step and a back-propagation step.

During the feedforward step, the training data set is presented to the model. The processing nodes in the hidden layer sum the inputs based on the randomly assigned weight values as shown in Equation (7), and then apply the sigmoid transfer function to compute the output, as shown in Equation (8). The predicted output(s) for this input can be obtained at the output layer. During the back-propagation step, the error for the output is calculated first. This is accomplished by comparing the actual output values to the predicted output values. Errors for all the processing nodes are calculated and weight adjustment are then computed for all interconnections. The weight adjustment is then sent back to the model for slight weight correction. This iterative training process keeps on going until the error has reached the criteria set by the model designer.

The back-propagation learning rate and the momentum coefficient are two parameters that need to be defined for the back-propagation NN model training. The learning rate is an adjustable factor that controls the speed of the learning process. With a faster learning rate, the NN model will learn faster. However, if the learning rate is too high, the oscillation of weight change can impede the convergence of the error surface, and may lead to overshooting of a near-optimal weight factor w . In contrast, if the learning rate is too slow, the NN model may get caught in a local error minimum instead of the global minimum.

The learning process can be facilitated by starting with a high learning rate initially, followed by a gradual reduction in the learning rate. A constant learning rate of 0.1–10 throughout the training process has also been proposed by Wythoff [1993] and 0.3–0.6 has been proposed by Zupan & Gasteiger [1993].

Momentum coefficient is used in weight updating for back-propagation NN to avoid local minima and to reduce oscillation of weight change. To obtain faster learning without oscillation, the weight change is related to the previous weight change to provide a smooth effect. The momentum coefficient determines the proportion of the last weight change that is added into the new weight change.

Convergence is the process of searching a set of weight factors for the NN model so that the prediction errors can be reduced to a minimum. The most common criterion of convergence is based on the sum of squared errors. Supervised NN networks measure the difference (error) between the predicted output value and the actual output value during the training process. The sum of squared errors (SSE) for the training and test subsets can be calculated using the following equation [Basheer & Hajmeer, 2000].

$$SSE = \frac{1}{N} \sum_{p=1}^N \sum_{i=1}^M (t_{pi} - o_{pi})^2 \quad \dots (10)$$

where, o_{pi} is the actual output of i th output node from p th sample; t_{pi} is the target output of i th output node from p th sample; N is the number of training samples; and M is the number of output nodes.

Training error such as SSE for the training data set decreases indefinitely with increasing number of hidden nodes or training iterations. The initial quick drop of SSE is due to learning. However, the subsequent slow reduction of SSE could be attributed to memorization or over-fitting because of the excessively large number of training cycles or excessive number of hidden nodes. On the other hand, the test error decreases initially, but subsequently increases due to memorization and over-fitting of the NN model. Thus, the training would be stopped when the test error starts to increase, and the optimal number of hidden nodes would be picked when the test error is the minimum.

6.1.4 The advantage of using NN model for process modeling and prediction

Process modeling and prediction of a particular process can be achieved through a number of approaches. The initial attempt to develop a mathematical model is probably through the derivative from first principal models. However, in most of the engineering problems, the first principle models are non-linear and computationally time-consuming. The replacement of the first-principle model by an equivalent NN for the process modeling and prediction takes the advantage of high speed processing, since simulation with a NN involves only a few non-iterative algebraic calculations [Haykin, 1994; Aleksander & Morton, 1995]. In these works, the NN model was obtained from data simulated by a previously available first-principle model. In the modeling step, the first-principle model is used to generate a large set of simulated data under different operation conditions and these simulated data are used to train the NN model. In this sense, it is

possible to obtain a NN model that represents the first principle model in the range of interest.

The use of a NN takes advantage of the comparative rapidity of the NN simulation. In this way, even a detailed grid search can be achieved in reasonable time, as long as there were not too many variables been optimized. This approach is more reliable, readily deals with constraints, avoids several typical numerical problems of conventional optimization tools, and is not computationally time-consuming. As an additional benefit, the full mapping of the objective function allows one to identify multiple optima easily, an important feature not presented by conventional optimization methods. Moreover, the constraints are easily treated afterwards since the points with violated constraints can be recognized and classified.

Once the map is obtained, it is easy to choose the optimum point, to identify whether multiple optima are present, to check if constraints were violated, and so on. This approach definitely provides more comprehensive information for the engineer's analysis than the conventional non-linear programming procedure. This algorithm can be straightforwardly extended to treat multi-objective optimization problems as well.

6.2 The development of NN model for process modeling and prediction

The main objective of employing NN model is to model the hard chrome

plating process as well as predict the plating thickness of hard chrome deposition (with no more than $\pm 10\%$ deviation). To build an NN model, the architecture of the NN model, which includes number of nodes in both the input and output layers, number of hidden layer, and number of hidden nodes, need to be defined first. The number of inputs and outputs are defined based on the hard chrome plating operation. The number of inputs is expected to be four (the Chromic Acid Concentration, Electric Current, Sulphuric Acid Concentration and Plating Time). The most important decision in the NN model building is to decide how many hidden nodes to be used. As mentioned in the above NN basic architecture section, there is no magic formula to select the number of hidden nodes. In this research study, by using Kolomogorov's theorem [Hecht-Nielsen, 1987]:

$$\begin{aligned} \text{Number of inputs} = 4 &>>>> \text{Number of hidden nodes} &&= (4*2) + 1 \\ &&&&&&= 9 \end{aligned}$$

6.2.1 Training of NN model

To train an NN model, the data collected from experiments are normally divided into three sets, namely, training set, test set and validation set. The training set is used to train the NN model by adjusting the link weights of network model, which would include the data covering the entire experimental space. This means that the training data set has to be fairly large to contain all the required information and must include a wide variety of data from different experimental conditions.

The test data set is an independent data set, which is reserved, and not

actually used for training, in the back propagation algorithm. It is presented to the NN model periodically, and is used to check the training progress of the NN model. In other words, it is used to keep an independent check on the progress of the algorithm. As mentioned earlier, both the training and the test errors initially drop. However, when the test error stops decreasing, or alternatively starts to rise, it indicates that the NN model is starting to over-fit the data, and at this point, the training must be stopped. When over-fitting or over-learning occurs during the training process, it is usually advisable to decrease the number of hidden units and/ or hidden layers. In contrast, if the network is not sufficiently powerful to model the underlying function, over-learning is not likely to occur, and neither the training nor test errors will drop to a satisfactory level. Therefore, the test data can be used to check the architecture and training progress of a NN model.

The validation data set is used to ensure that the relationship between inputs and outputs, based on the training and test sets are real, and not artifacts of the training process. The validation data set should include data, which are not included in the other data sets, but lie in the data boundaries of the training data set. The final model is tested with the validation data set to confirm its accuracy before it becomes trained for implementation.

In this research study, for the training of the NN model, the process variables had been set and the resulting schematic diagram for such NN model was shown in Figure 27. The configuration for the training of the NN model was shown in Table 14. The transfer function that had been employed was a sigmoid transfer function, as shown in Figure 28. Such transfer function took the input,

which might have any value between plus and minus infinity, and squashed the output into the range 0 to 1. The reason of choosing this transfer function was that it was differentiable.

With the assistance of Qnet software [Qnet, 2003], a trained NN model has been developed. The procedure of using Qnet software to train a NN model has been listed in Appendix E. Figure 29 show the value of RMS error has reached the desirable value after 300,000 iterations of the training of such NN. Two historical data that have been obtained from industrial plant are shown in Table 15 and Table 16 shows a comparison between the predicted (NN) vs. the actual values.

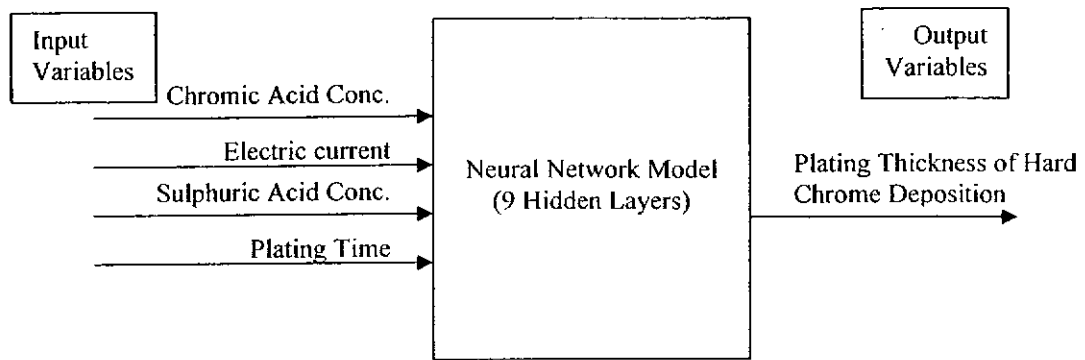
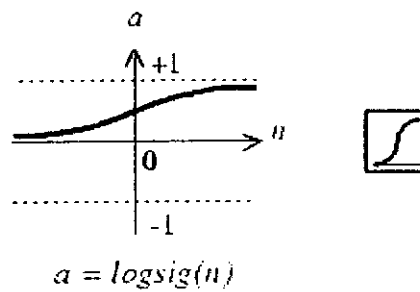


Figure 27: A schematic diagram for the training of NN model



Log-Sigmoid Transfer Function

Figure 28: The transfer function used in the training of NN model

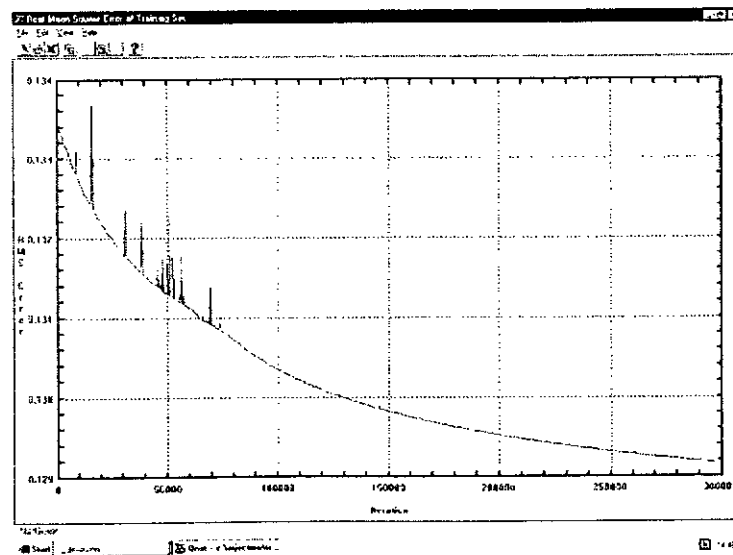


Figure 29: The plot of RMS error after 300,000 iterations for the training of NN model

Table 14: The configuration for the training of a NN model

Desire Network Name	HCP NN
Number of layers	3
Number of input nodes	4
Number of hidden layers	1
Number of nodes in hidden layer	9
Number of output nodes	1

Table 15: Typical sample data for hard chrome plating process with the use of rotary plating machine

Piston Model No.	Run	Chromic Acid Concentration (gram/liter)	Sulphuric Acid Concentration (gram/liter)	Electric Current (amps)	Plating Time (hours)	Thickness [mm]
MC90	1	160	2.4	800	39	0.71
MC90	3	214	3.6	950	52	0.86

Table 16: A comparison between the predicted (NN) values vs. the actual values

Standard	Run	Chromic Acid Conc.	Electric Current	Sulphuric Acid Conc.	Plating time	NN Result	Actual Result	Diff (%)
2	1	160	800	2.4	39	72	72	-0.6
7	2	214	950	2.4	39	78	77	1.2
31	3	214	950	3.6	52	90	90	-0.2
6	4	160	950	2.4	39	93	92	1.5
19	5	214	800	2.4	52	65	63	3.0
22	6	160	950	2.4	52	89	88	0.8
10	7	160	800	3.6	39	89	89	0.0
20	8	214	800	2.4	52	64	64	-0.6
17	9	160	800	2.4	52	90	90	0.8
16	10	214	950	3.6	39	112	110	1.2
21	11	160	950	2.4	52	90	89	1.1
28	12	214	800	3.6	52	98	100	-1.6
4	13	214	800	2.4	39	41	39	3.8
5	14	160	950	2.4	39	91	92	-1.2
12	15	214	800	3.6	39	100	102	-1.5
9	16	160	800	3.6	39	89	88	1.0
29	17	160	950	3.6	52	39	38	2.1
18	18	160	800	2.4	52	90	90	0.6
30	19	160	950	3.6	52	41	40	1.8
1	20	160	800	2.4	39	70	70	0.0
14	21	160	950	3.6	39	75	76	-1.1
8	22	214	950	2.4	39	79	77	1.9
23	23	214	950	2.4	52	89	87	2.6
27	24	214	800	3.6	52	99	99	-0.5
15	25	214	950	3.6	39	110	106	3.4
25	26	160	800	3.6	52	76	74	2.6
3	27	214	800	2.4	39	38	36	4.2
13	28	160	950	3.6	39	76	76	-0.4
32	29	214	950	3.6	52	89	90	-1.0
11	30	214	800	3.6	39	102	102	0.4
26	31	160	800	3.6	52	74	73	1.6
24	32	214	950	2.4	52	89	87	2.2

A comparison between the values obtained from the NN method vs. the actual values is shown in Table 16. According to such table, the maximum deviation between the predicted (NN) value and the actual value is 4.2%. As the maximum deviation is less than $\pm 10\%$ of the actual value, the developed NN model can be regarded as a good tool to predict the plating thickness of hard chrome deposition.

6.2.2 Testing and validation of NN model

For the testing and validating of NN model, there are no mathematical rules to determine the required number of data in each set for training the NN models. [Basheer and Hajmeer, 2000] There are only some rules of thumb that can be used as guidance to divide the collected data into training set, test set and validation set. Baum and Haussler [1989] proposed that the training subset should be at least equal to the product of the number of weights in the model multiplied by the inverse of the minimum target error. Dowla and Rogers [1995] suggested that the ratio of sample in training subset to number of weight factors should be larger than 10. Looney (1996) recommended that 65% of data should be used for training, 25% of data for testing and 10% for validation.

In this study, the Looney (1996) approach has been adopted. There are a total number of 140 data sets (including the original 32 data sets) for the NN model development. Those 140 data sets have been divided into 3 groups, namely the training data set, the testing data set and the validation data set.

7. Discussion of results

7.1 Comparison of FFD and NN methods

In this research study, FFD method and regression modeling techniques were shown to be efficient tools for exploring little known processes, as was the case with hard chrome plating process. A limited experimental effort, consisting of only 32 trials with the test conditions structured according to a central composite statistical design, was sufficient to provide a relatively detailed overview of the process behavior. The statistical models revealed that this process behavior was quite complex and depends on a number of synergistic and antagonistic interactions between the process variables. These interactions had not only been identified but their relative impact on the plating thickness of hard chrome deposition had been quantified. The predictive models developed here using regression analysis had served to simulate the process at various operating regimes, thereby making it possible to identify certain key reaction mechanisms. A further advantage offered by the statistical modeling performed in this study resided in the fact that it provided a comprehensive picture of the process which links the isolated elements of the basic electrochemistry. In turn, these electrochemical analyses supported the behavior predicted by the models and helped shed some light on the underlying fundamental mechanisms responsible.

The empirical model presented in Table 10 indicated that there exists a number of interactions between process variables which significantly affected the plating thickness of hard chrome deposition. The effect of the Sulphuric Acid Concentration was coupled with that of several other process variables. While the

linear effect (or first-order effect) of Sulphuric Acid Concentration alone was of minor significance, the interactions of Chromic Acid Concentration with Sulphuric Acid Concentration as well as with Electric Current were important contributors to accelerating both the principal reaction mechanisms in place as well as that of the side reactions.

After determining the regression equations of all the process variables and also the training of NN model, the predictions by both the techniques were found out. The predicted values of the plating thickness, obtained through FFD method and NN method were compared with the actual values for the validation set of experiments. These comparisons had been described in terms of percentage error in Figure 30 & 31 for validation set of experiments.

From Table 17 it is evident that for the set of data that had been used for this research study, the NN model predicted thicknesses are nearer to the actual experimental values than the FFD model.

The use of FFD and NN methods to approximate the limit state function could reduce the total effort on the hard chrome plating evaluation. For parametric evaluations significant reduction of the total computational effort with a relative good precision might be attained. More studies would need to be performed to compare other possible alternatives, although the FFD and NN methods seem to be very promising techniques in the case of hard chrome plating operation.

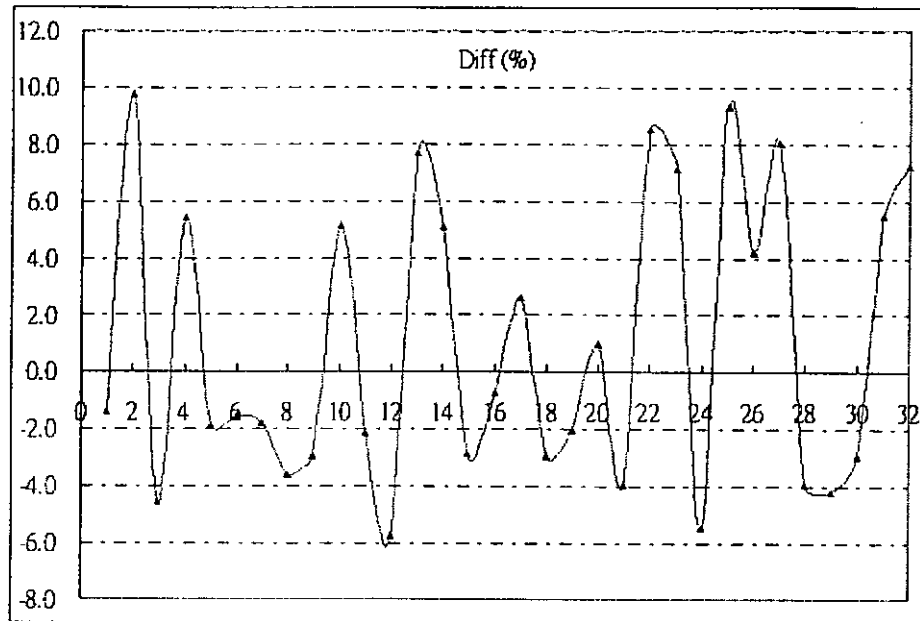


Figure 29: A plot of the difference between the predicted (FFD) values vs. the actual values

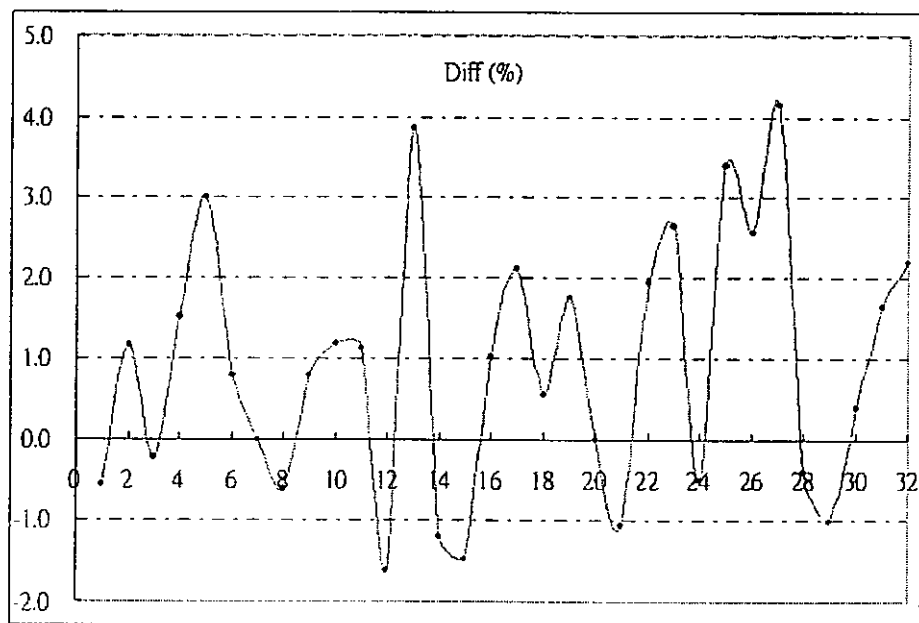


Figure 30: A plot of the difference between the predicted (NN) values vs. the actual values

Table 17: The average and maximum errors in the predictions by FFD and NN

	Percentage Error (FFD)	Percentage Error (NN)
Average	4.4	1.5
Maximum	9.8	4.2

7.2 Discussion on research methods

In this research study, FFD method had been chosen due to its important features. First, it shows greater flexibility for exploring or enhancing the “signal” (treatment) in this research study. Whenever it is interested in examining treatment variations, FFD method would be strong candidates as the designs of choice. Second, FFD method was efficient. Instead of conducting a series of independent studies one would effectively be able to combine these studies into one.

On the other hand, FFD method is the only effective way to examine interaction effects. There are two ways one could determine whether there is an interaction. First, when the statistical analysis was initialized, the statistical table would report on all main effects and interactions. Second, one could always spot an interaction in the graphs of group means -- whenever there were lines that were not parallel there was an interaction present. If one checks out the main effect graphs above, then all of the lines within a graph were parallel. On the contrary, for all of the interaction graphs, one would see that the lines are not parallel.

Secondly, the FFD method and regression modeling techniques have been shown to be efficient tools for exploring little known processes, as is the case with hard chrome plating process. A limited experimental effort, consisting of only 32 trials with the test conditions structured according to a central composite statistical design, was sufficient to provide a relatively detailed overview of the process behavior. The statistical models revealed that this process behavior was

quite complex and depended on a number of synergistic and antagonistic interactions between the process variables. These interactions had not only been identified but their relative impact on the plating thickness of hard chrome deposition was also quantified. The predictive models developed here using regression analysis has served to simulate the process at various operating regimes, thereby making it possible to identify certain key reaction mechanisms. A further advantage offered by the statistical modeling performed in this research study resided in the fact that it provides a comprehensive picture of the process which links the isolated elements of the basic electrochemistry. In turn, these electrochemical analyses supported the behavior predicted by the models and helped shed some light on the underlying fundamental mechanisms responsible.

Conversely, while the developed FFD-based model is very useful to the understanding of the physical mechanisms involved in hard chrome plating process, it could also possess application difficulties, essentially for the following reasons: (1) they required a large number of parameters for modeling the complexity of hard chrome plating process dynamics; and (2) extension of such model to even slightly different situations could be very difficult. The black-box models, on the other hand, though might not necessarily lead to a better understanding of the hard chrome plating process (in a physically realistic manner), have an advantage in that they are easier to apply under different conditions since the modeling and forecasting procedure is usually analogous. Furthermore, the analysis of the characteristic parameters of the black-box models could furnish useful information on the dynamics of the phenomenon.

In the absence of accurate information about the physical mechanisms underlying or the 'exact' equations involved in the dynamics of hard chrome plating process, the use of a black-box model seems to have an edge over the use of a FFD-based model, since the former was capable of representing arbitrarily the complex non-linear hard chrome plating process by relating the inputs and the outputs of the underlying system. In view of this, this research study has incorporated the investigation of using a non-linear black-box approach for predicting the hard chrome plating process dynamics. The NN method, which has been employed in this research study, is a global approximation approach, in the sense that it uses all the values that were generated in the past as input for the prediction. In other words, the NN method has the capability of relating the input and output parameters, learning from examples through iteration, without requiring a prior knowledge of the relationships of the process parameters. Its structure is relatively simple, with connections in parallel and sequence between neurons. This means a much shorter computing time and a high potential of robustness and adaptive performance.

7.3 Effect of impurities

One process variable was not taken into consideration for this study, i.e. the effect of impurities/contaminations within chrome plating solution. There are two general classes of impurities in a chrome-plating bath: (1) inorganic impurities such as chlorides, excess of sulfates or fluorides, and (2) metallic impurities. The former is most common in the form of detrimental impurities such as iron, copper and zinc. These metals enter the plating solution from parts accidentally dropped into the plating bath and not recovered, from the attack of

the solution on supporting racks, and from attack on anode and cathode. The maximum acceptable concentrations of these metals would depend on the overall bath composition, and the approximate limits for iron and copper are 10 gram/liter and 0.2 gram/liter respectively. At or near these concentrations, copper and iron would reduce the brightness of the plating outcome, especially in the low current density area. However, it should be noted that the harm caused by metallic impurities was very much synergetic. What it means is that while one impurity alone even at high concentration is not necessary highly damaging, a combination of impurities (even with less concentration) would certainly cause significant damage. Nevertheless, if the level of impurities/contaminations has exceeded the general industrial standard, the efficiency of the hard chrome plating process could be significantly reduced or even stopped. As a result, further development of this research model would best incorporate the effect of impurities/contaminations.

7.4 Effect of solution conductivity

Solution conductivity is often associated with power consumption (electric current). Usually, the effect of conductivity loss can be primary compensated by increasing the electric current. In this study, the power input is quite high (> 800 amps), therefore the effect of conductivity loss is not significant.

Although there are bubbles forming resistive gas 'blankets' during the plating process, it is believed that the amount of (hydrogen) gas generated is

relatively small and would not contribute to a significant lowering of the solution conductivity.

7.5 Effect of chromic/sulphuric acid ratio

According to Dubpernell [1963], Dennis & Such [1993] and Edwards [1997], the normal range for chromic/sulphuric acid ratio is between 80 and 120. In the case of a high chromic/sulphuric acid ratio, milky or hazy hard chrome deposit will result. On the contrary, rough deposit is usually caused by low chromic/sulphuric acid ratio. In this study, it is the Teaching Company's policy to maintain a steady deposition rate; hence some of the chromic/sulphuric acid ratios used are below 80. Consequently, the surfaces of some of the plated ring grooves require additional surface machining. It is suggested that this ratio will be incorporated in future study.

7.6 Limitations of study

7.6.1 Limitation of time

A major objective of the study is to improve productivity of the chrome plating process for marine pistons. However, it should be noted that the necessary time required for completing a hard chrome plating process can be enormous (up to 120 man-hours). This is due to the rather long setup time for the preparation of a piston crown, which includes the processes of machining, pretreatment, etc. To achieve this aim, the idea of a rotary plating system instead

of expanding the original static system was discussed and finally it was agreed to put into action.

In view of this deviation from the original static design, considerable time and effort was spent on the design and construction of a prototype rotary plating machine. Additional trials have been performed. Throughout the commissioning process, quite a number of mechanical and process design modifications were made and new machine parts have been constructed. For example, the replacement of a damaged bright shaft with a new elongated one, the introduction of an extra bearing at the end of the shaft, etc. This kind of modifications has taken more time than expected, and because of this, the commissioning process has been extended considerably.

Although subsequent trial results showed less and less deviations in terms of deposited chromium thickness, the successful rate (<60%) did not meet the company expectation. It took quite a while to identify the causes of the problem. Eventually it was found that the main problem lied in the positioning of the anode. However, this problem was not an easy one to solve, because of the very limited space in the ring groove and also the rotary movement of the piston crown during the electroplating process.

Later, with the development of a new anode design, which repositioned the anode to a central position, the above-mentioned problem was solved. Providing that right procedures are followed at the pre-treatment stage, there would be little or almost no rotary movement of the piston crown throughout the plating process.

Because of this accomplishment, much less deviations in deposited chromium thickness were resulted and the successful rate of the chrome plating process has been greatly enhanced.

As depicted in the above paragraphs, design and commissioning of the new rotary plating machine has taken up more time than expected in this research study. In addition, this study has to follow the operating schedule in the Teaching Company. As a result, the number of completed trial runs could be obtained is restricted by the availability of company resources, not to mention the sudden occurrence of machine failure.

7.6.2 Limitation of NN

It should be noted that NN models could not be used to elucidate the mechanistic nature of the correlation established between the process variables. To obtain a reliable and trained NN model, a formulator might need a lot of training data and computer time to do the training. Because of the rather long process time of the original hard chrome plating system (even with the development of a new rotary plating machine), the data set for the NN training could not be regarded as large. As time goes on, more process information could be accumulated, thus a more reliable NN model could be developed.

8. Conclusion

At the commencement of this Innovation Technology Fund project, the chromium plating bath employed in the Teaching Company was a static system, which involved fixing the anode close to the surface of the work-piece while the work-piece was kept at the cathode. Due to the fact that the original plating system had become a bottleneck when the whole piston crown recondition process was considered, the demand from the Teaching Company was to improve the hard chrome plating process through (i) the development of a more efficient plating system and (ii) to predict plating thickness through an empirical model between different thicknesses and the input variables.

Towards the latter half of the project, a rotary electroplating system was constructed and predictions of the response variables were made using the Full Factorial Design (FFD) and the Neural Network (NN) methods. The models and the predicted values obtained by both of the methods were compared with the experimental values of the response variables to evaluate the accuracy of the predictions.

On completion of project, quite a number of the staff in the Teaching Company acquired a better knowledge about its electroplating process because of this research study. Furthermore, the electroplating solution has been systematically monitored ever since. As a result, the probability in getting a poor electroplating outcome is greatly reduced. For a particular piston crown model, a cost saving of 50% in plating parts (e.g. anodes: 8 pairs \rightarrow 4 pairs) and 40%

saving in process lead time (e.g. total time for the whole piston crown recondition process: 96 hours → 58 hours) could be achieved if the recommended process parameters were used. More importantly, the pilot plant of the rotary plating machine was accepted by the Teaching Company and would be mass produced in the near future.

Within the range of input variables for the present case, the results showed that the NN method came ahead of the FFD method in nearness of the predictions to the experimental values of plating thickness as the average errors in the plating thickness in case of NN are less than that obtained using FFD (average error is 1.5% for NN as compared to 4.4% in the case of FFD predictions; maximum deviation is 4.2% for NN as compared to 9.8% in the case of FFD predictions). Both the FFD and NN models found could be used to predict the plating thickness given process parameters.

In conclusion, the benefits of the adoption of the proposed approach are obvious. The suggested models (either FFD or NN) with an easy-to-follow roadmap illustrated here offered an alternative approach for those who are interested to investigate the various options for solving complex process control problems. In particular, the suggested approaches, which have not been adequately discussed in the area of electroplating, provide a novel technique to deal with the process variables in hard chrome plating system, thereby minimizing the tedious analytical process associated with traditional plating control algorithm. This would pave the way for future research.

Finally, two technical publications based on the results obtained in this study, have been accepted for publication:

- A. Ning, T.T. Wong & and C.W. Leung (2002), Optimization of Chrome Plating Process Design: A Neural Network Approach, The 8th ISSAT International Conference on Reliability and Quality Design, Anaheim, California: USA, pp. 331-335.
- A. Ning, T.T. Wong & and C.W. Leung (accepted for publication), Hard Chrome Plating of Marine Engine Pistons, The 4th Conference for New Ship and Marine Technology, Oct 26-29, 2004, Shanghai, China.

9. Suggestions for future work

The results of this research study further confirmed that a statistical approach and an artificial intelligence approach is a highly efficient and reliable way of providing credible models of the process. The next step in this research study will be to apply these models to identify economical conditions for optimizing the plating thickness of hard chrome deposition at commercial production rates. The complexities of the interactions which exist between process variables suggest that a numerical optimization will be required. Once a promising set of conditions is identified, commercial tests can be conducted to validate model predictions.

Another area which can be suggested for future work is to incorporate the status of the surface roughness of the refurbished ring groove as another objective function on top of the thickness of the hard chrome deposition. This is because any changes in the originally quoted process variables (chromic acid concentration, sulphuric acid concentration, electric current and plating time), may sometime produce different surface roughness. A knowledge of the plating surface roughness will facilitate the planning of surface machining process.

Subsequently, the Response Surface Method (RSM) can be used to optimize both the plating thickness and surface roughness simultaneously. RSM is a blend of statistical design techniques, empirical model-building and optimization methods. In other words, it allows the calculation and prediction of process variables. RSM has been applied extensively in the industrial world for process and product development and optimization. It is possible to integrate

RSM into the suggested FFD and NN models to enhance the accuracy of developed models.

References

- Aleksander, I. & Morton, H. (1995), *An Introduction to Neural Computing*, Thomson.
- Arora, J.S. (1989), *Introduction to optimum design*. New York: McGraw-Hill.
- Basheer, I.A. and Hajmeer, M. (2000), Artificial neural networks: fundamentals, computing, design, and application, *J. Micro. Methods*, 43, pp. 3–31.
- Bourquin, J., Schmidli, H., van Hoogevest, P. and Leuenberger, H. (1997), Basic concepts of artificial neural networks modeling in the application to pharmaceutical development. *Pharm. Dev. Technol.* 2, pp. 95–109.
- Brimi, M.A. and Luck, J.R. (1965), *Electrofinishing*, American Elsevier Publishing Company, Inc., New York.
- Buchanan B, Shortliffe E.H. (1989), *Rule-based expert systems: the MYCIN experiments of the Stanford Heuristic Programming Project*. Addison-Wesley series in artificial intelligence. Reading, Mass.: Addison-Wesley.
- Buelens, C., Fransaer, J., Celis, J.P. and Roos, J.R. (1992), The mechanism of electrolytic codeposition of particles with metals: a review, *Bulletin of Electrochemistry*, 8, (8), pp. 371-375.
- Basheer, I.A. and Hajmeer, M. (2000), Artificial neural networks: fundamentals, computing, design, and application, *Journal of Microbiology Methods*, 43, pp. 3–31.
- Carpenter, J.C. and Hoffman, M.E. (1995), Understanding neural network approximations and polynomial approximations helps neural network performance, *AI Expert*, 10, pp. 31–33.
- Chan, W. M., & Nascimento, C.A.O. (1994), Use of neural networks for modeling of olefin polymerisation in high-pressure tubular reactors. *Journal of Applied Polymer Science*, Vol. 53, pp. 1277–1289.
- Chessin, H. and Newby, K. (1986), U.S. Patent 4,588,481.
- Cichocki, A. and Amari, S. (2002), *Adaptive blind signal and image processing: Learning Algorithms and Applications*, Published by John Wiley & Sons, Chichester UK.
- Cooper, L. (1974), *Applied nonlinear programming for engineers and scientists*; Aloray: Englewood, NJ.

Daniel, C. (1976) Applications of statistics to industrial experimentation; Wiley: New York.

Dennis, I.K. and Such, T.E. (1993), Nickel and chromium plating, 3rd edition, Woodhead, Publishing, Cambridge, UK.

DARPA Neural Network Study (1989). A connectionist/neural network bibliography, MIT Lincoln Lab.

Dubpernell, G. (1963), Modern electroplating, 2nd Edition, Wiley, New York, pp. 80-140.

Edwards, J. (1997), Coating and surface treatment systems for metals: a comprehensive guide to selection, Materials Park, Ohio: ASM International; Stevenage, England: Finishing Publications.

Elektro-Chrom-G.m.b.H. (assignee of Leibreich, E.), (1927) German Patent 448,526; (1925) British Patent 237,288; (1926) French Patent 601,059; (1927) Swiss Patent 118,632.

Erb, R.J. (1993), Introduction to backpropagation neural network computation. Pharm. Res. 10, pp. 165-170.

Fink, C.G., (1926) U.S. Patent 1,581,188; (1931) 1,802,463.

Fink, C.G. and McLeese (to United Chromium, Inc.), (1932) U.S. Patent 1,844,751.

Fox, J. (1991), Regression diagnostics. Thousand Oaks, CA: Sage Publications. Quantitative Applications in the Social Sciences Series No. 79.

Gabe, D.R. (1974), The rotating cylinder electrode: its continued development and application, Journal of Applied Electrochemistry, 4, pp. 91.

Gileadi, E. (1993), Electrode kinetics for chemists, chemical engineers and material scientists, VCH Publishers, Inc., New York.

Greenwood, J.D. (1981), Hard chromium plating: a handbook of modern practice, 3rd edition, Redhill: Portcullis.

Griffin, J.L. (1971), nya-Technical methods of analysis, Plating, 61, pp. 129.

Hajela, P. and Berke, L. (1991), Neural network based decomposition in optimal structural synthesis, Comput. Systems Engrg. 2, pp. 473-481.

Haykin S. (1994), Neural networks: a comprehensive foundation. New York: Macmillan Ed.

Hecht-Nielsen, R. (1987), Kolmogorov's mapping neural network existence theorem. In:, pp. 11-14.

Helle, K. and Walsh, F. (1996), The theories for the composition of the deposits, Proceedings of the 26th International Congress of Metal Finishing (Interfinish'96), Birmingham, England, 10-12 September, pp. 291.

Hinkelman, K. (1994), Design and analysis of experiments. New York: John Wiley & Sons.

Horner, J. (1994), "Electroplating", Kirk-Othmer Encyclopedia Of Chemical Technology, 4th Ed., Volume No. 9, John Wiley and Sons, Inc., New York, NY.

Hovestad, A. and Janssen, L.J. (1995), Reviews in electroplating, Journal of Applied Electrochemistry, 40, pp. 519.

Jadid, M.N. and Fairbairn, D.R. (1996), Predicting moment-curvature parameters from experimental data. Eng. Applied Artificial Intelligence 9, pp. 303–319.

Korbach, W. (1989), U.S. Patent 4,790,674.

Korbach, W. and McMullen, W. (1989), U.S. Patent 4,828,650.

Lau, H.C.W. & Wong, T.N. (2000), An expert system for complex closed-loop control: a non-mathematical approach". International Journal of Expert Systems, Vol. 15, No. 2, pp. 98-109.

Law, M.A. and Kelton, W.D. (1982), Simulation modeling and analysis. New York: McGraw-Hill.

Martyak, N. (1989), U.S. Patent 4,810,336.

Metal Finishing Guidebook And Directory (1993), Issue '93k, Volume 91, Issue 1A, Elsevier Science Publishing Company, Inc., New York, NY.

Mevrel, R. (1989), Use of chromium plating, Material Science and Engineering, A120, pp. 13.

Montgomery, D. (1997), Design and analysis of experiments, Wiley, Chichester.

Montgomery, D .C. (2001), Response surface methods and other approaches to process optimization in Design and Analysis of Experiments, Wiley, New York, pp. 427–500.

Morisset, P., Oswald, J.W., Draper, C.R. and Pinner, R. (1954), Chromium plating, Teddington, Middlesex, England.

Narayan, R. and Chattopadhyay, S. (1982), Chrome plating characteristics, Surface Technology, 16, pp. 227.

Nascimento, C. A. O., Giudici, R., & Scherbakoff, N. (1996), Use of neural networks for modelling of nylon-6,6 polymerisation in a twin-screw extruder

reactor. In: Proceedings of the 5th world congress of chemical engineering, San Diego, CA, USA, Vol. IV, July 14–18, pp. 515–520.

Newby, K. (1999), New techniques in hard chrome plating, Proc. AESF Surface Finishing.

Peace, G. (1993), Taguchi Method: A Hands-on Approach, Addison-Wesley, Reading, MA.

Pickett, D.J. (1979), Electrochemical Reactor Design, Elsevier Scientific Publishing Company, New York.

Press, W.H., Flannery, B.P., Teukolsky, S.A. and Vetterling, W.T. (1986), Numerical recipes; Cambridge University Press: Cambridge.

Pushpavanam, S., Pushpavanam, M., Natarajan, S.R., Narasimham, K.C. and Chinnasamy, S. (1993), The electrolysis of industrial water, International Journal of Hydrogen Energy, 18 (4), pp. 277.

Qnet, (2003), Vesta Services, Inc., <http://www.qnetv2k.com/> .

Rumelhart, D.E., Hinton, G.E. and Williams, R.J. (1986), Learning internal representations by error propagation. In: D.E. Rumelhart and J.L. McClelland, Editors, Parallel Distributed Processing: Explorations in the Microstructure of Cognition Vol. 1, MIT Press, Cambridge, MA, pp. 318–362.

Shaffer, R.E. (1996), Ph.D. Dissertation "Optimization Methods for the Multivariate Analysis of Infrared Spectral and Interferogram Data", Ohio University, Athens, OH.

Sarle, W.S. (2002), Neural networks model, <ftp.sas.com/pub/neural/FAQ.html> .

SAS Institute, Inc. (1995), JMP statistics and graphics guide, Version 3.1, SAS institute, Inc.: Cary, NC.

Shreir, L.L. (1976), Corrosion, Butterworths, London, pp. 14–86.

Sietsma, J. and Dow, R.J.F. (1988), Neural Net Pruning - Why and How. IEEE International Conference in Neural Networks, 1, pp. 325–333.

Sofer, Y., Yarnitzky, Y. and Dirnfeld, S.F. (1990), Hard chrome plating, Surface Coating Technology, 42, pp. 227.

Stareck, J., Passal, F. and Mahlstedt, H. (1950), Self-regulated hard chrome plating, Process of American Electroplating Society, 37, pp. 31.

Takahara, J., Takayama, K. and Nagai, T. (1997), Multi-objective simultaneous optimization technique based on an artificial neural network in sustained release formulations, J. Control. Release, 49, pp. 11–20.

Wang, L. and Grandhi, R. (1994), Optimal design of frame structures using multivariate spline approximation, AIAA J. 32, pp. 2090–2098.

Wu, T., Pan, W., Chen, J. and Zhang, R. (2000), Formulation optimization technique based on artificial neural network in salbutamol sulfate osmotic pump tablets. Drug Development of Industrial Pharmacy. 26, pp. 211–215.

Vanderplaats, G.N. (1984), Numerical Optimization Techniques for Engineering Design: With Applications, McGraw-Hill, New York, NY.

Wythoff, B.J. (1993), Backpropagation neural networks: a tutorial. Chemometr. Intell. Lab. Syst. 18, pp. 115–155.

Zaki, N. (2000), Chromium plating, PF Online, www.pfonline.com .

Zupan, J. and Gasteiger, J. (1993), In: Neural Networks For Chemists: An Introduction, VCH, New York.

Appendices

Appendix A: Details of the assembly process of the rotary plating machine parts

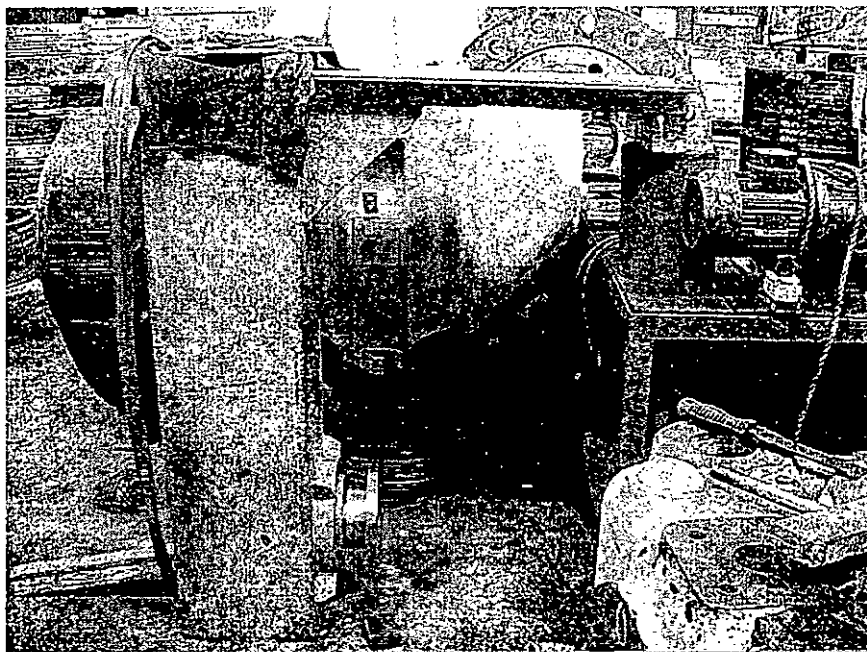


Figure 32: The side view of the rotary plating machine

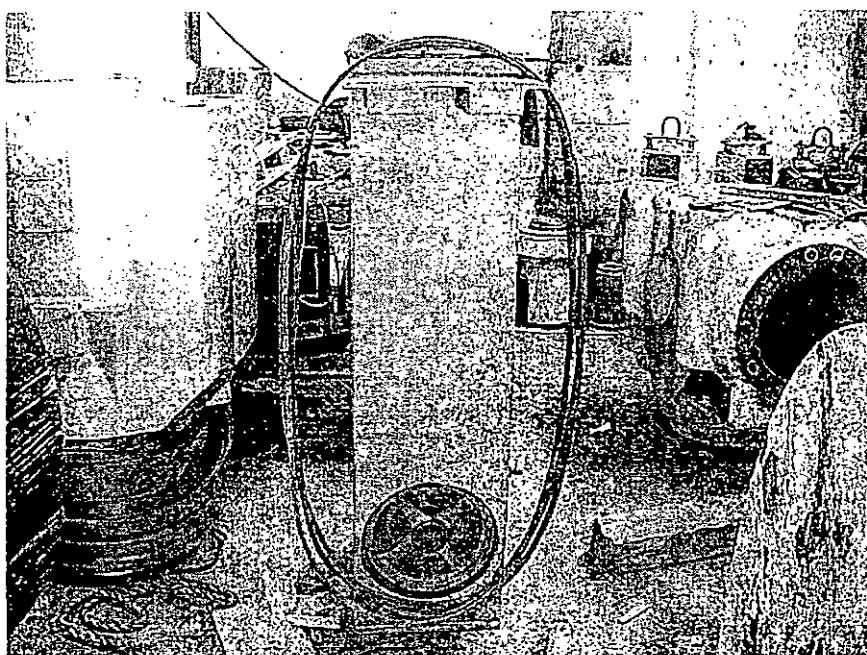


Figure 33: The back view of the rotary plating machine

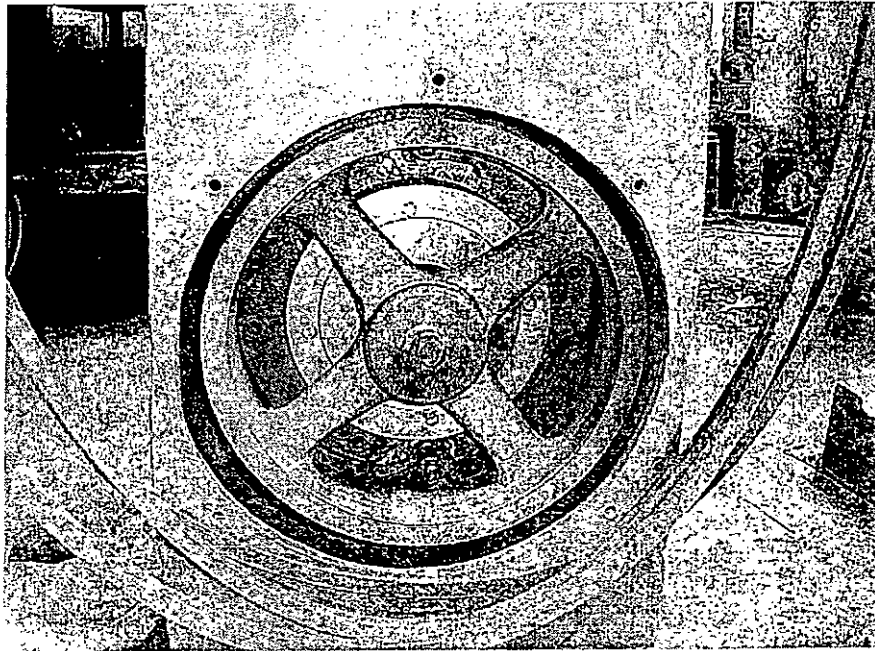


Figure 34: A closer view of the internal section of the rotary plating machine

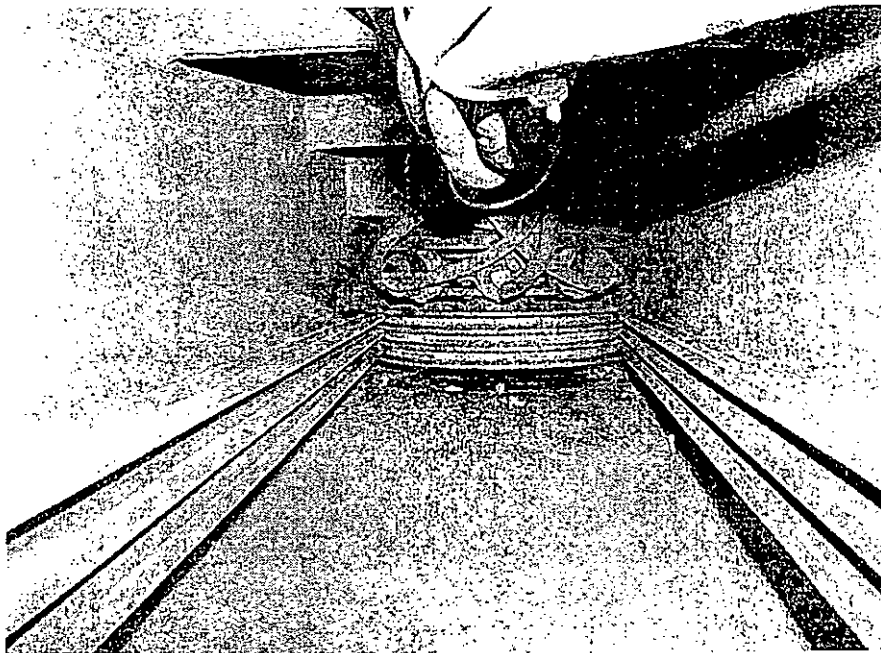


Figure 35: The bearing section and the carbon brush section of the rotary plating machine

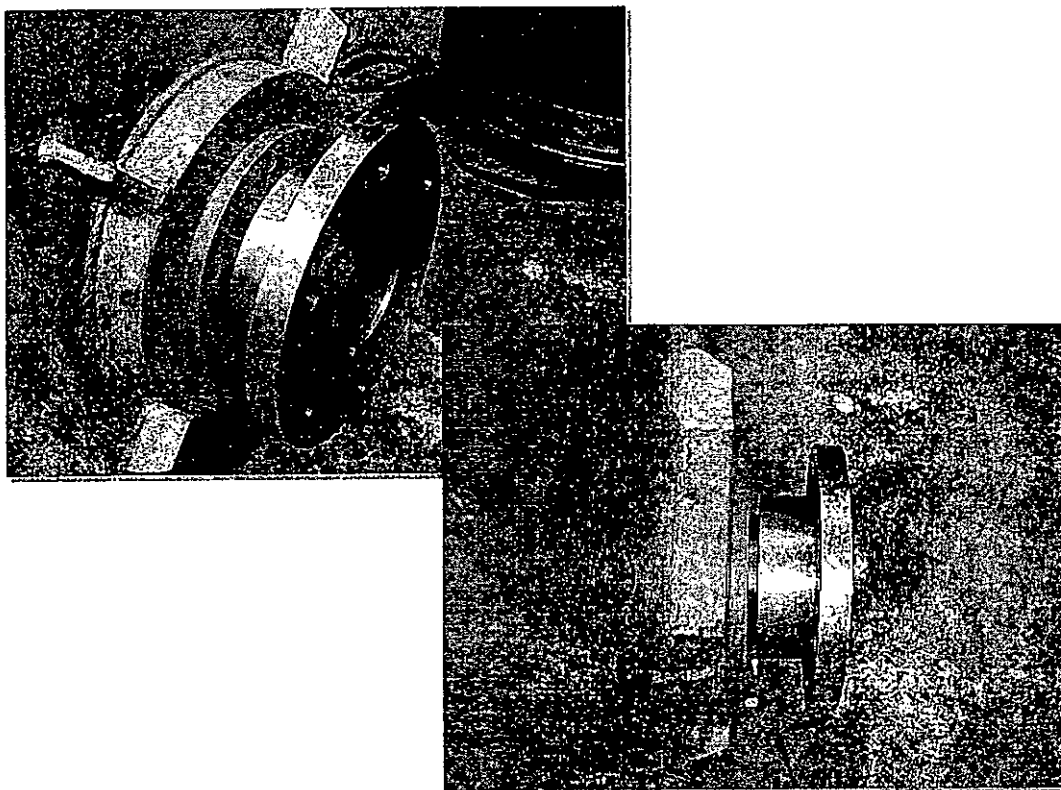


Figure 36: The piston holding section of the rotary plating machine

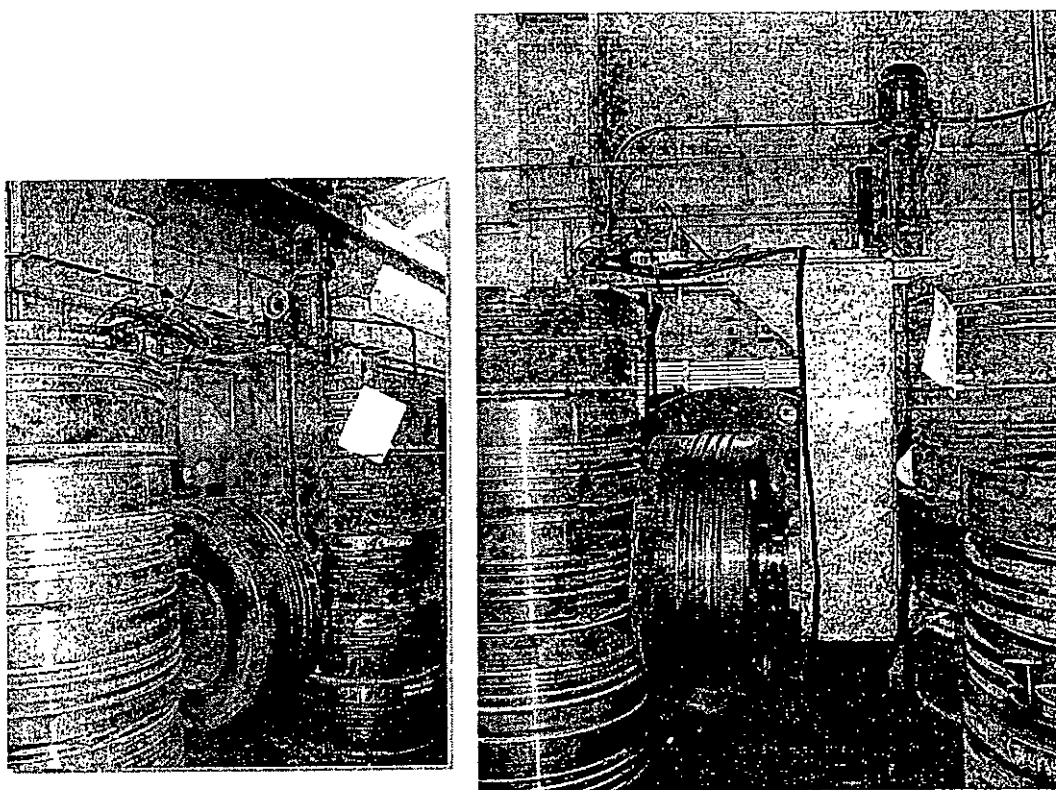


Figure 37: The testing of the rotary plating machine

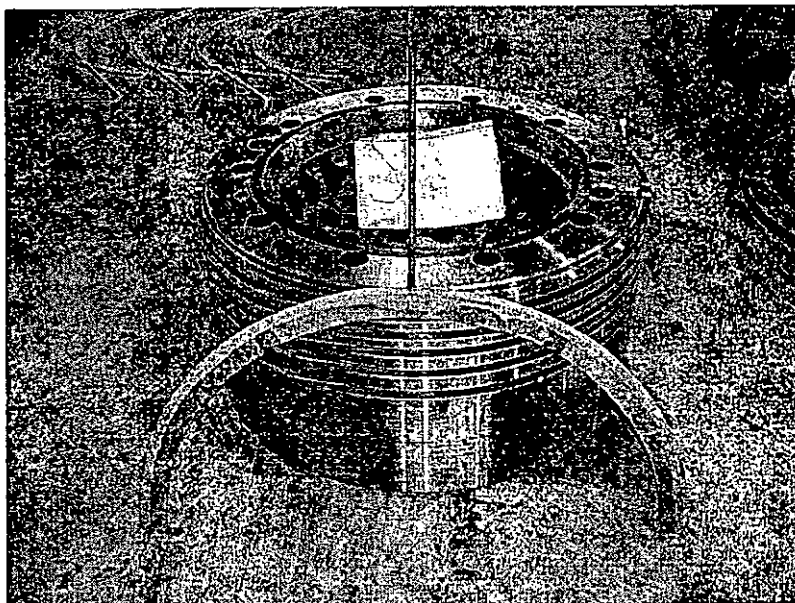


Figure 38: The new design of the electric anode

Appendix B: Details of the commissioning of the rotary plating machine

- The construction of the rotary plating machine was finished in October 2001.
- The first successful trial was completed on 23rd October 2001, using Full-circle anodes (2 semi-circle ones). For the subsequent trials a detachable piston crown was used in order to test the hardness after rotary plating process. Figure 39 shows the appearance of the rotary plating machine

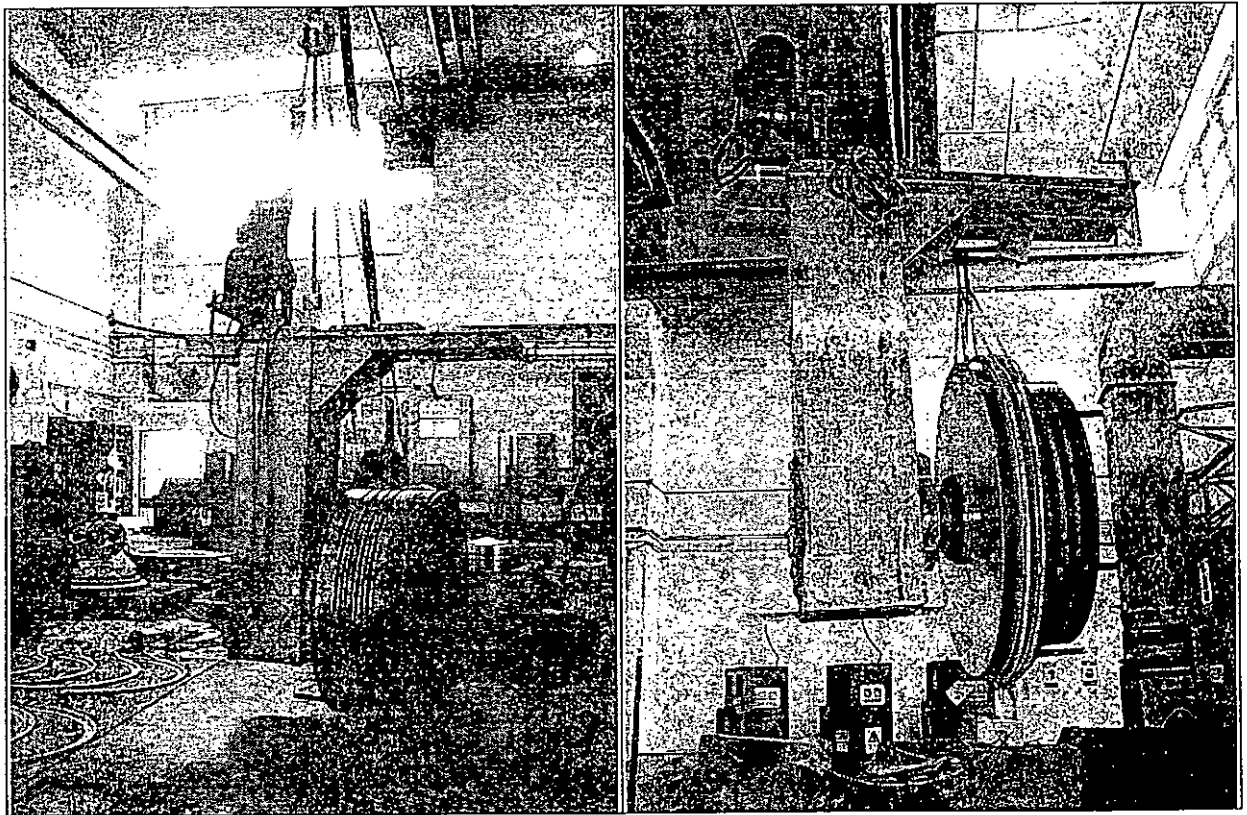


Figure 39: The appearance of the rotary plating machine

- General plating outcome observations from the trials between October 2001 and February 2002:

- | | |
|-------------------|--|
| Positive Feedback | <ul style="list-style-type: none">– Development of “needle” has been kept at minimum– Appearance is shinny, like the plating outcomes normally observed from the static plating process |
| Negative Feedback | <ul style="list-style-type: none">– It is very difficult to hold anodes at the centerline of ring groove– Sometimes the anode restrictions have been thrown out– The anodes are usually not straight in shape– Teflon buttons are stuck within ring grooves after the plating process (Figure 40 shows the damage of anode upon the completion of plating process)– The hardness measurements are not totally satisfactory |

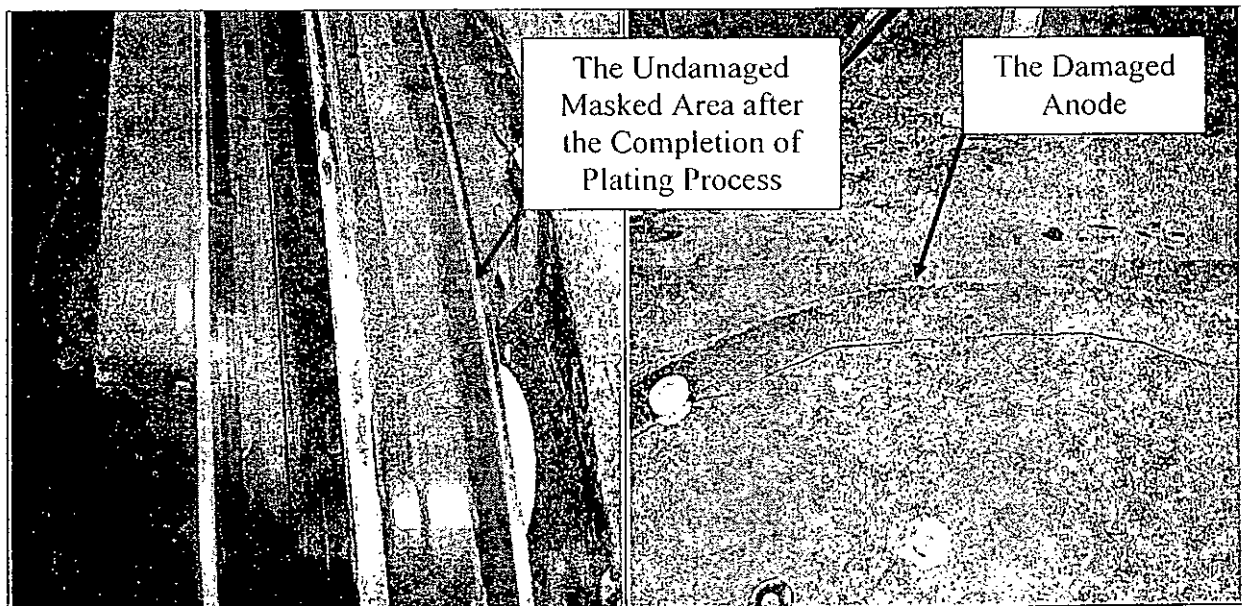


Figure 40: Damage of the anode upon the completion of plating process

- In order to address the positioning of anodes, it is decided that only 1 semi-circle anode (covering half of the ring groove) would be used, instead of 2 semi-circle ones, as shown in Figure 41. Such modification was applied to trials after 7th March 2002.

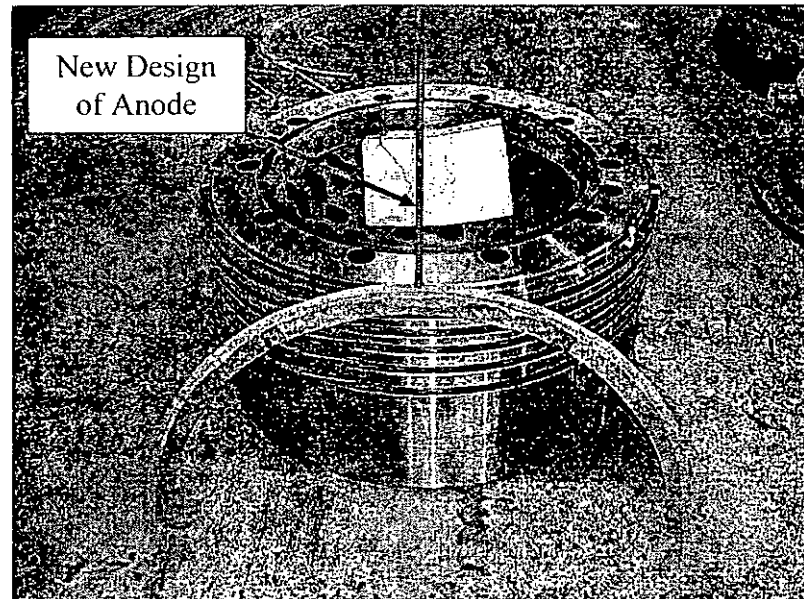


Figure 41: The modified anode

Plating outcome observations from the trial on 7th March 2002:

- | | |
|-------------------|--|
| Positive Feedback | <ul style="list-style-type: none"> – It is easier to keep the anodes at the centerline – The appearance and the deposit rate are very much the same as the ones observed from the static plating process – Only half number of anodes are needed, meaning saving in resources |
| Negative Feedback | <ul style="list-style-type: none"> – The anodes have touched the surface of ring grooves – The anodes are usually not straight in shape – The hardness measurements are not totally satisfactory still |

- The rotary wheels have swapped position in order to increase the rotating speed. The original maximum rotary speed was 8.5 rpm, but now 15rpm could be achieved easily. The results show that less deviation in terms of chrome thickness on the same ring groove surface is observed.
- In order to reduce/eliminate the development of “needle”, plastic strip was used on the edge of the ring groove.
- Using the detachable piston crown as a testing medium, the results showed that if the thickness of the newly deposited chromium reached 0.5mm or above, its hardness value would be not less than 760HV Vickers.
- Because of the collected experience and confidence, it had been decided that scrap piston crown would be used for subsequent trials from May 2002.
- In July 2002, some major modifications in machine design were carried out. For example the shaft was elongated and an extra bearing was added. The trial showed that the appearance of the chrome-plated surface was the same as the one using traditional method, which was shinny and flat. On the other hand, it was found that no more than 1000 amps of electric current should be applied to the anodes.
- In September 2002, the design of anodes has been modified. PVC Through Thread Rods were used to replace the Steel Through Thread Rods. Also two

extra restriction Rods have been used, as shown in Figure 42. The trial confirmed that such modifications seemed to have solved the anode-positioning problem.

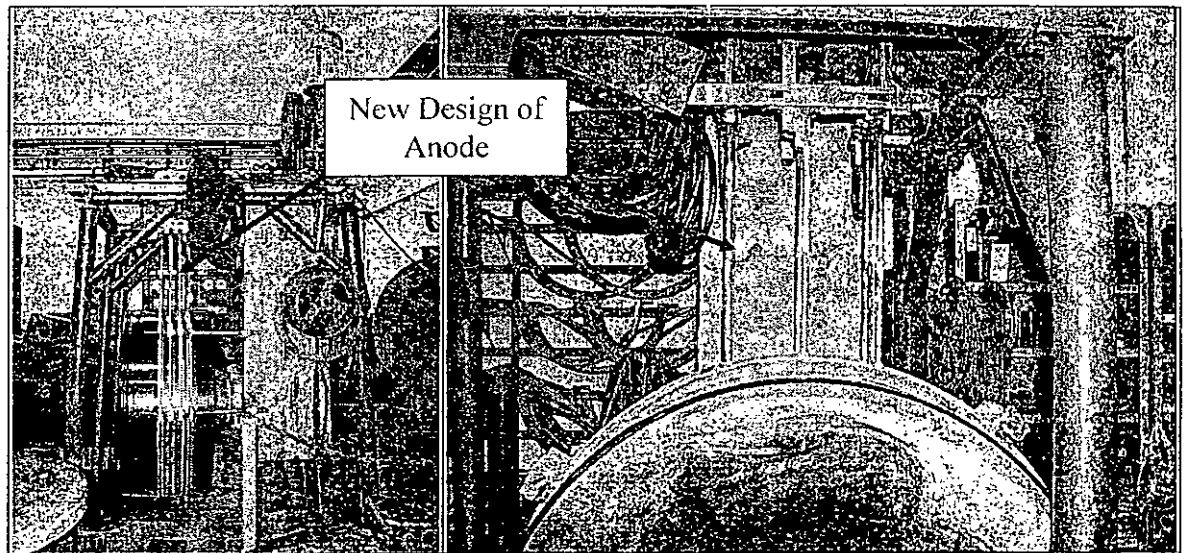


Figure 42: The final design of anode

- In October 2002, new copper-graphite brushes replaced the copper brushes. The use of graphite element is to provide lubricating effect during shaft rotation.
- In November 2002, Zinc strip was used instead of hard plastic strip in order to further reduce/eliminate the development of “needle”. However, the result reviewed that although the shininess and flatness was better, the chromium deposition rate was about 20% slower. As a result, it was decided not to switch from hard plastic strip to Zinc strip.
- The probability in obtaining a satisfactory result by using the rotary chrome plating result could now be regarded as over 90%.

Appendix C: Determination of Chromium(VI) ion in hard chrome plating solution

Reagents

50% v/v sulphuric acid

0.1N ferrous ammonium sulphate

0.1N potassium permanganate (standard volumetric solution)

Method

1. Pipette 40ml of 0.1N ferrous ammonium sulphate into a 250ml conical flask.
2. Add 50ml DI water and 10ml 50% sulphuric acid.
3. Titrate with 0.1N potassium permanganate to pink end point.
4. Record titre = A mls.
5. Pipette a 10ml aliquot of the plating solution into a 500ml volumetric flask and make up to the mark with DI water.
6. Mix thoroughly.
7. Pipette 10ml of this dilution into a 250ml conical flask.
8. Add 50ml DI water and 10ml 50% sulphuric acid.
9. Pipette 40ml 0.1N ferrous ammonium sulphate into the flask.
10. Titrate with 0.1N potassium permanganate to a pink end point.
11. Record titre = B mls.

Calculation

$$(A - B) \times 16.64 = \text{g/L chromic acid}$$

Appendix D: Determination of Sulphate ion in hard chrome plating solution

Reagents

Conc. hydrochloric acid

Hydrogen peroxide solution

30% barium chloride solution

5% v/v hydrochloric acid

Method

1. Filter a sample of the working solution.
2. Pipette a 10ml aliquot of the filtered solution into a 500ml beaker.
3. Add 20ml DI water and 20ml conc. hydrochloric acid.
4. Add hydrogen peroxide dropwise until no further effervescence is seen and the solution is a blue-green colour.
5. Heat to boiling and add 2ml of 30% barium chloride solution.
6. Continue boiling for 2 mins.
7. Add 200mls boiling DI water and continue boiling for 2 hours.
8. Allow to stand overnight.
9. Filter into a Whatman 542 filter paper.
10. Bobby out the beaker and rinse into filter with hot DI water.
11. Rinse beaker into filter paper with hot 5% hydrochloric acid.

12. Rinse filter with hot DI water until paper is clean.
13. Weigh a dried crucible and record weight as 'A' grams.
14. Transfer paper to the crucible and burn off in furnace for 1 hour at 800 degree C.
15. Ensure no carbon residues remain in the crucible.
16. Transfer crucible to a dessicator and allow cooling.
17. Weigh the crucible and record weight as 'B' grams.

Calculation

$(B-A) \times 42.02 = \text{g/L sulphuric acid (sulphate)}.$

Appendix E: The procedure for the training of a NN model using Qnet software

Step	Target	Procedure
1	Prepare a set of data	<ul style="list-style-type: none">▪ Sample data file has been prepared, named as HCP NN data.xls
2	Define the input and output of the Neural Network	<ul style="list-style-type: none">▪ Input: Chromic Acid Conc. / Electric Current / Sulphuric Acid Conc. / Plating Time▪ Output: Plating Thickness of Hard Chrome Deposition▪ Rearrange the sets of data that fit the setup▪ Put # in the 1st data record, as shown in the highlight box▪ Save file as File02.xls.
3	Convert Excel table into a Text-format file.	<ul style="list-style-type: none">▪ From the menu bar, choose File → Save As▪ From Save as type (bottom of the dialogue), choose Text (Tab delimited) (*.txt)▪ Save file as <u>File02.txt</u>▪ Choose OK▪ <u>File02.txt</u> looks like the picture as shown here.
4	Start to use the Qnet software program	<ul style="list-style-type: none">▪ Open Qnet software program▪ A main dialogue would be displayed

- | | | |
|---|--|---|
| 5 | To train a Neural Network | <ul style="list-style-type: none"> ▪ Click New, and a dialogue appears |
| 6 | Setup the Design of the Neural Network | <ul style="list-style-type: none"> ▪ Click Network Design ▪ Input the following information: <ul style="list-style-type: none"> – Desire Network Name (HCP NN) – Number of layers (3) – Number of input nodes (4) – Number of hidden layer (1) – Number of nodes in hidden layer (9) – Number of output nodes (1) ▪ Click OK |
| 7 | Define the Training Data for the training process | <ul style="list-style-type: none"> ▪ Click Training Data ▪ Click Input Node Data File, and select <u>File02.txt</u> ▪ Next to Data Start Column, input 1 (∵ Chromic Acid Conc. is in the 1st column) ▪ Click Target Node Data File, and select <u>File02.txt</u> ▪ Next to Data Start Column, input 3 (∵ Plating Thickness is in the 5th column) ▪ Next to Number of Test Cases, input 500 ▪ Choose <u>None</u> inclusion method ▪ Click OK |
| 8 | Input the Training Parameters for the training process | <ul style="list-style-type: none"> ▪ Click Training Parameters ▪ Next to Max Iteration, input 100,000 ▪ Next to Learn Rate Control Start Iteration, input 100001 ▪ Click OK |
| 9 | Save the definitions | <ul style="list-style-type: none"> ▪ Click Save Network Setup |

- | | | |
|----|--|--|
| | of the Training process | <ul style="list-style-type: none"> ▪ Input File.net ▪ Click OK ▪ Then click OK to start the Neural Network training process |
| 10 | Observing the training process | <ul style="list-style-type: none"> ▪ During the training process, notice the changes in the RMS Error value and Correlation value ▪ Wait until the training process has been finished (Max Iteration reached!) |
| 11 | Refine the training of the Neural Network | <ul style="list-style-type: none"> ▪ Click the icon <u>RMS Error History vs Iteration</u> to get a rough picture about the performance of the training. ▪ User may like to change the previously set training parameters in order to see the difference of several training results. |
| 12 | Save the trained Neural Network | <ul style="list-style-type: none"> ▪ If desire result has been achieved, the trained Neural Network has to be saved to form a data repository for the development of future application. ▪ From the menu bar, choose File → Save Network As ▪ Input <u>File Trained.net</u> ▪ Click Save <p>Then restart Qnet.</p> |
| 13 | Preparing a set of recall data to generate a prediction feedback | <ul style="list-style-type: none"> ▪ With the available of a trained Neural Network, it is time to prepare a set of data, which is then being input into the Qnet program, in order to get a prediction feedback. ▪ A sample file has been created, named as <u>File02 Recall.xls</u> |

- Remember to put # in the 1st data record, as shown in the highlight box.

- 14 Convert the Recall data set into a Text-format file.
 - Save the Excel file into a Text file (File02_Recall.txt)

- 15 Prepare the prediction process
 - Restarted the Qnet program:
 - From the Main dialogue, select Recall mode

- 16 Recall the Trained Neural Network and input the prepared data set
 - Click the Open icon
 - Choose File_Trained.net
 - Click Input Node Data File, and select File02_Recall.txt
 - Next to Data Start Column, input 1 (∵ Chromic Acid Conc. is in the 1st column)
 - Click OK

- 17 Completing the prediction process
 - A screen that shows the network definitions has been displayed.

- 18 Gather the prediction result
 - To see the predicted results that are created from the trained Neural Network:
 - From the menu bar, choose Info
 - From the drop down menu, choose Network Outputs/Targets
 - A result that is similar to the shown figure has been displayed.

- 19 Saving the predicted result
 - Close the displayed result.
 - From the reappeared dialogue:

- From the menu bar, choose File
 - From the drop down menu, choose Save Outputs/Targets
 - Save the output data into a file called File02_Output.txt (Note: A Text-format file is needed for the next step).
- 20 Importing the predicted result
- Open the Excel program
 - From the menu bar, choose File
 - From the drop down menu, choose Open
 - Select File02_Output.txt, a Text Import Wizard would appeared
 - Press Next 2 times, then press Finish to complete the import process.
 - As shown from the figure, the File02_Output.txt file has been converted into an Excel table.
- 21 Constructing final Excel prediction result table
- Finally, by combining the File02_Recall.txt and the File02_Output.txt, a predicted result Excel table can be gathered.

Appendix F: The data sets for the training, testing and validation of a NN model (total number = 140)

Training data sets = 91 (65% of 140)

<u>Run</u>	<u>Chromic Acid Conc.</u>	<u>Electric Current</u>	<u>Sulphuric Acid Conc.</u>	<u>Plating Time</u>	<u>NN Result</u>	<u>Actual Result</u>	<u>Diff (%)</u>
1	160	800	2.4	39	72	72	-0.6
2	214	950	2.4	39	78	77	1.2
3	214	950	3.6	52	90	90	-0.2
4	160	950	2.4	39	93	92	1.5
5	214	800	2.4	52	65	63	3.0
6	160	950	2.4	52	89	88	0.8
7	160	800	3.6	39	89	89	0.0
8	214	800	2.4	52	64	64	-0.6
9	160	800	2.4	52	90	90	0.8
10	214	950	3.6	39	112	110	1.2
11	160	950	2.4	52	90	89	1.1
12	214	800	3.6	52	98	100	-1.6
13	214	800	2.4	39	41	39	3.8
14	160	950	2.4	39	91	92	-1.2
15	214	800	3.6	39	100	102	-1.5
16	160	800	3.6	39	89	88	1.0
17	160	950	3.6	52	39	38	2.1
18	160	800	2.4	52	90	90	0.6
19	160	950	3.6	52	41	40	1.8
20	160	800	2.4	39	70	70	0.0
21	160	950	3.6	39	75	76	-1.1
22	214	950	2.4	39	79	77	1.9
23	214	950	2.4	52	89	87	2.6
24	214	800	3.6	52	99	99	-0.5
25	214	950	3.6	39	110	106	3.4
26	160	800	3.6	52	76	74	2.6
27	214	800	2.4	39	38	36	4.2
28	160	950	3.6	39	76	76	-0.4
29	214	950	3.6	52	89	90	-1.0
30	214	800	3.6	39	102	102	0.4
31	160	800	3.6	52	74	73	1.6
32	214	950	2.4	52	89	87	2.2
33	160	800	2.4	39	72	73	-1.4
34	214	950	2.4	39	78	78	0.4
35	214	950	3.6	52	90	89	1.0
36	160	950	2.4	39	93	92	1.5
37	214	800	2.4	52	65	64	1.7
38	160	950	2.4	52	89	87	2.4
39	160	800	3.6	39	89	88	0.7

40	214	800	2.4	52	64	65	-1.7
41	160	800	2.4	52	90	88	2.6
42	214	950	3.6	39	112	109	2.4
43	160	950	2.4	52	90	89	1.0
44	214	800	3.6	52	98	98	0.1
45	214	800	2.4	39	41	40	1.3
46	160	950	2.4	39	91	91	0.2
47	214	800	3.6	39	100	104	-3.5
48	160	800	3.6	39	89	89	-0.6
49	160	950	3.6	52	39	40	-3.0
50	160	800	2.4	52	90	91	-1.0
51	160	950	3.6	52	41	40	1.3
52	160	800	2.4	39	70	72	-2.4
53	160	950	3.6	39	75	76	-1.1
54	214	950	2.4	39	79	79	-0.1
55	214	950	2.4	52	89	88	1.2
56	214	800	3.6	52	99	100	-1.1
57	214	950	3.6	39	110	106	3.5
58	160	800	3.6	52	76	73	3.8
59	214	800	2.4	39	38	36	4.2
60	160	950	3.6	39	76	74	2.3
61	214	950	3.6	52	89	88	1.0
62	214	800	3.6	39	102	103	-0.6
63	160	800	3.6	52	74	75	-1.1
64	214	950	2.4	52	89	87	1.8
65	160	800	2.4	39	72	70	2.9
66	214	950	2.4	39	78	80	-2.1
67	214	950	3.6	52	90	91	-1.2
68	160	950	2.4	39	93	92	1.5
69	214	800	2.4	52	65	65	0.2
70	160	950	2.4	52	89	91	-2.1
71	160	800	3.6	39	89	91	-2.6
72	214	800	2.4	52	64	62	3.1
73	160	800	2.4	52	90	92	-1.8
74	214	950	3.6	39	112	108	3.3
75	160	950	2.4	52	90	91	-1.2
76	214	800	3.6	52	98	98	0.1
77	214	800	2.4	39	41	42	-3.6
78	160	950	2.4	39	91	93	-1.9
79	214	800	3.6	39	100	99	1.4
80	160	800	3.6	39	89	90	-1.7
81	160	950	3.6	52	39	39	-0.5
82	160	800	2.4	52	90	88	2.4
83	160	950	3.6	52	41	40	1.8
84	160	800	2.4	39	70	72	-2.4
85	160	950	3.6	39	75	77	-2.3
86	214	950	2.4	39	79	81	-2.6
87	214	950	2.4	52	89	86	3.6
88	214	800	3.6	52	99	101	-2.1
89	214	950	3.6	39	110	111	-1.2
90	160	800	3.6	52	76	76	-0.3
91	214	800	2.4	39	38	39	-3.6

Testing data sets = 35 (25% of 140)

<u>Run</u>	<u>Chromic Acid Conc.</u>	<u>Electric Current</u>	<u>Sulphuric Acid Conc.</u>	<u>Plating Time</u>	<u>NN Result</u>	<u>Actual Result</u>	<u>Diff (%)</u>
92	160	950	3.6	39	76	78	-2.9
93	214	950	3.6	52	89	92	-3.4
94	214	800	3.6	39	102	100	2.4
95	160	800	3.6	52	74	72	3.1
96	214	950	2.4	52	89	85	4.2
97	214	950	2.4	52	89	86	3.0
98	160	950	3.6	39	76	78	-2.9
99	160	950	3.6	52	41	40	1.3
100	214	950	3.6	39	112	113	-1.2
101	160	800	2.4	39	72	72	-0.6
102	214	950	2.4	39	78	79	-0.9
103	214	950	3.6	52	90	91	-1.2
104	160	950	2.4	39	93	92	1.5
105	214	800	2.4	52	65	64	1.7
106	160	950	2.4	52	89	90	-1.0
107	160	800	3.6	39	89	88	0.7
108	214	800	2.4	52	64	65	-1.7
109	160	800	2.4	52	90	91	-0.8
110	214	950	3.6	39	112	111	0.5
111	160	950	2.4	52	90	90	-0.1
112	214	800	3.6	52	98	99	-0.9
113	214	800	2.4	39	41	40	1.3
114	160	950	2.4	39	91	92	-1.2
115	214	800	3.6	39	100	101	-0.6
116	160	800	3.6	39	89	90	-1.7
117	160	950	3.6	52	39	40	-3.0
118	160	800	2.4	52	90	92	-2.1
119	160	950	3.6	52	41	42	-3.6
120	160	800	2.4	39	70	71	-1.0
121	160	950	3.6	39	75	77	-2.3
122	214	950	2.4	39	79	78	1.2
123	214	950	2.4	52	89	91	-2.1
124	214	800	3.6	52	99	98	0.9
125	214	950	3.6	39	110	108	1.6
126	160	800	3.6	52	76	78	-2.8

Validation data sets = 14 (10% of 140)

<u>Run</u>	<u>Chromic Acid Conc.</u>	<u>Electric Current</u>	<u>Sulphuric Acid Conc.</u>	<u>Plating Time</u>	<u>NN Result</u>	<u>Actual Result</u>	<u>Diff (%)</u>
127	214	800	2.4	39	38	36	4.2
128	160	950	3.6	39	76	77	-1.7
129	214	950	3.6	52	89	86	3.4
130	214	800	3.6	39	102	100	2.4
131	160	800	3.6	52	74	75	-1.1
132	214	950	2.4	52	89	88	0.7
133	214	950	2.4	39	78	77	1.7
134	160	950	2.4	52	89	90	-1.0
135	160	950	2.4	52	90	90	-0.1
136	160	800	2.4	52	90	92	-2.1
137	214	950	2.4	52	89	86	3.6
138	214	800	2.4	39	38	39	-3.6
139	214	800	3.6	39	102	106	-3.4
140	214	950	2.4	52	89	88	0.7