ANALYSIS AND PREDICTION OF PLASMA SPRAYED ALUMINA-TITANIA COATING DEPOSITION USING NEURAL COMPUTATION

A Project Report Submitted in Partial Fulfillment of the Requirements for the Degree of

B. Tech.
(Mechanical Engineering)

By

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CERTIFICATE

This is to certify that the work in this thesis entitled **Analysis And Prediction Of Plasma Sprayed Alumina-Titania Coating Deposition Using Neural Computation** by **Lalitendu Tripathy,** has been carried out under my supervision in partial fulfillment of the requirements for the degree of **Bachelor of Technology** in ***Mechanical Engineering*** during session 2009- 2010 in the Department of Mechanical Engineering, National Institute of Technology, Rourkela.

To the best of my knowledge, this work has not been submitted to any other University/Institute for the award of any degree or diploma.

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<u>A B S T R A C T</u>

Coating deposition by plasma spraying involves a number of process variables, which contribute in a large way to the quality of the coating. During spraying, various operating parameters are determined mostly based on past experience. It therefore does not provide the optimal set of parameters for a particular objective. In order to obtain the best result with regard to any specific coating quality characteristic, accurate identification of significant control parameters is essential. Deposition efficiency of any coating is a characteristic which not only rates the effectiveness of the spraying method but also is a measure of the coatability of the material under study. This work is devoted to analyze the experimentally obtained results on the deposition efficiency of alumina-titania coatings made at different operational conditions. For this purpose, a statistical technique called Taguchi experimental design methd is used. Factors are identified according to their influence on the coating deposition. The most significant parameter is found. A prediction model using artificial neural network (ANN) is presented considering the significant factors. Inspired by the biological nervous system, an artificial neural network (ANN) approach is a fascinating computational tool, which can be used to simulate a wide variety of complex engineering problems such as deposition of ceramic coatings on metal substrates by plasma spraying. Prediction of deposition rate helps in selecting the optimum combination of process parameters and hence is essential in a plasma spray coating activity. This research shows that the use of a neural network model to simulate experiments with parametric design strategy is quite effective for prediction of wear response of materials within and beyond the experimental domain.

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CHAPTER 1

Chapter 1

INTRODUCTION

Background and Motivation

The incessant quest for higher efficiency and productivity across the entire spectrum of manufacturing and engineering industries has ensured that most modern-day components are subjected to increasingly harsh environments during routine operation. Critical industrial components are, therefore, prone to more rapid degradation as the parts fail to withstand the rigors of aggressive operating conditions and this has been taking a heavy toll of industry's economy. In an overwhelmingly large number of cases, the accelerated deterioration of parts and their eventual failure has been traced to material damage brought about by hostile environments and also by high relative motion between mating surfaces, corrosive media, extreme temperatures and cyclic stresses. Simultaneously, research efforts focused on the development of new materials for fabrication are beginning to yield diminishing returns and it appears unlikely that any significant advances in terms of component performance and durability can be made only through development of new alloys. As a result of the above, the concept of incorporating engineered surfaces capable of combating the accompanying degradation phenomena like wear, corrosion and fatigue to improve component performance, reliability and durability has gained increasing acceptance in recent years. The recognition that a vast majority of engineering components fail catastrophically in service through surface related phenomena has further fuelled this approach and led to the development of the broad interdisciplinary area of surface modifications. A protective coating deposited to act as a barrier between the surfaces of the component and the aggressive environment that it is exposed to during operation is now globally acknowledged to be an attractive means to significantly reduce/suppress damage to the actual component by acting as the first line of defense.

Surface modification is a generic term now applied to a large field of diverse technologies that can be gainfully harnessed to achieve increased reliability and enhanced performance of industrial components. The increasing utility and industrial adoption of surface engineering is a consequence of the significant recent advances in the field. Very rapid strides have been made on all fronts of science, processing, control, modeling, application developments etc. and this has made it an invaluable tool that is now being increasingly considered to be an integral part of component design. Surface modification today is best defined as "the design of substrate and surface together as a system to give a cost effective performance enhancement, of which neither is capable on its own". The development of a suitable high performance coating on a component fabricated using an appropriate high mechanical strength metal/alloy offers a promising method of meeting both the bulk and surface property requirements of virtually all imagined applications. The newer surfacing techniques, along with the traditional ones, are eminently suited to modify a wide range of engineering properties. The properties that can be modified by adopting the surface engineering approach include tribological, mechanical, thermo-mechanical, electrochemical, optical, electrical, electronic, magnetic/acoustic and biocompatible properties.

The development of surface engineering has been dynamic largely on account of the fact that it is a discipline of science and technology that is being increasingly relied upon to meet all the key modern day technological requirements: material savings, enhanced efficiencies, environmental friendliness etc. The overall utility of the surface engineering approach is further augmented by the fact that modifications to the component surface can be metallurgical, mechanical, chemical or physical. At the same time, the engineered surface can span at least five orders of magnitude in thickness and three orders of magnitude in hardness.

Driven by technological need and fuelled by exciting possibilities, novel methods for applying coatings, improvements in existing methods and new applications have proliferated in recent years. Surface modification technologies have grown rapidly, both in terms of finding better solutions and in the number of technology variants available, to offer a wide range of quality and cost. The significant increase in the availability of coating process of wide ranging complexity that are capable of

depositing a plethora of coatings and handling components of diverse geometry today, ensures that components of all imaginable shape and size can be coated economically.
Existing surface treatment processes fall under three broad categories:
(a) Overlay Coatings: This category incorporates a very wide variety of coating processes wherein a material different from the bulk is deposited on the substrate. The coating is distinct from the substrate in the as-coated condition and there exists a clear boundary at the substrate/coating interface. The adhesion of the coating to the substrate is a major issue.
(b) Diffusion Coatings: Chemical interaction of the coating-forming element(s) with the substrate by diffusion is involved in this category. New elements are diffused into the substrate surface, usually at elevated temperatures so that the composition and properties of outer layers are changed as compared to those of the bulk.
(c) Thermal or Mechanical Modifications of Surfaces: In this case, the existing metallurgy of the component surface is changed in the near-surface region either by thermal or mechanical means, usually to increase its hardness. The type of coating to be provided depends on the application. There are many techniques available, e.g. electroplating, vapour depositions, thermal spraying etc. Of all these techniques, thermal spraying is popular for its wide range of applicability, adhesion of coating with the substrate and durability. It has gradually emerged as the most industrially useful method of developing a variety of coatings, to enhance the quality of new components as well as to reclaim worn/wrongly machined parts.

The type of thermal spraying depends on the type of heat source employed and consequently flame spraying (FS), high velocity oxy-fuel spraying (HVOF), plasma spraying (PS) etc. come under the umbrella of thermal spraying. Plasma spraying utilizes the exotic properties of the plasma medium to impart new functional properties to conventional and non-conventional materials and is considered as one highly versatile and technologically sophisticated thermal spraying technique. It is a very large industry with applications in corrosion, abrasion and temperature resistant coatings and the production of monolithic and near-net shapes. The process can be applied to coat on variety of substrates of complicated shape and size using metallic, ceramic and /or polymeric consumables. The production rate of the process is very high and the coating adhesion is also adequate. Since the process is almost material independent, it has a very wide range of applicability, e.g., as thermal barrier coating, wear resistant coating etc. Zirconia (ZrO_2) is a conventional thermal barrier coating material. Wear resistant coatings are used to combat wear especially in cylinder liners, pistons, valves, spindles, textile mill rollers etc. alumina (Al_2O_3) , titania (TiO_2) and zirconia (ZrO_2) are the some of the conventional wear resistant coating materials.

Statistical methods have commonly been used for analysis, prediction and/or optimization of a number of engineering processes. Such methods enable the user to define and study the effect of every single condition possible in an experiment where numerous factors are involved. Plasma spray coating is such a complex phenomenon in which a number of control factors collectively determine the performance output i.e. the deposition rate and there is enormous scope in it for implementation of appropriate statistical techniques for process optimization. But unfortunately, such studies have not been adequately reported so far.

Against this background, the present work addresses to this aspect by adopting a systematic statistical approach called Taguchi method to optimize the process parameters and by implementing artificial neural network (ANN) as a prediction model considering the significant factors to predict deposition rate of alumina-titania coatings.

Objectives of the Present Work

The objectives of the present investigation can be outlined as:

a. Parametric appraisal of Al_2O_3-TiO_2 coating deposition by plasma spraying route using Taguchi method
b. Development of a prediction model using artificial neural network (ANN) to predict deposition rate of these coatings.

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CHAPTER 2

Chapter 2

LITERATURE REVIEW

This chapter deals with the literature survey on various aspects of plasma spraying, the coating materials and their characteristics. At the end of the chapter a summary of the literature survey and the knowledge gap in the earlier investigations are presented.

THERMAL SPRAYING

It is the generic category of surface modification technique that apply consumables in the form of a finely divided molten or semi molten droplets to produce a coating onto the substrate kept in front of the impinging jet. The melting of the consumables may be accomplished in a number of ways, and the consumable can be introduced into the heat source in wire or powder form. Thermal spray consumables can be metallic, ceramic or polymeric substances. Any material can be sprayed as long as it can be melted by the heat source employed and does not undergo degradation during heating [1, 2]. An interesting aspect of thermal spraying is that the surface temperature seldom exceeds 200^0 C. Hard metal or ceramic coating can be applied to thermosetting plastics. Stress related distortion problems are also not so significant. The spraying action is achieved by the rapid expansion of combustion gases (which transfer the momentum to the molten droplets) or by a separate supply of compressed air.

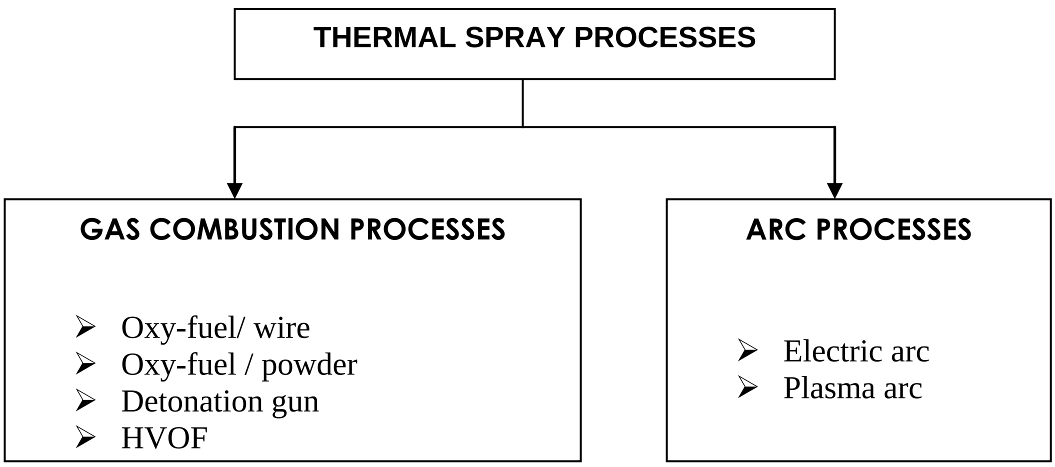

Fig. 2.1 Categorization of common thermal spray processes

Figure 2.1 shows the common thermal spray processes fitting into the above mentioned categories. The plasma spraying process is discussed briefly in the following.

Plasma Spraying

Plasma spraying is the most versatile thermal spraying process and the general arrangement is shown in Figure 2.2. An arc is created between tungsten tipped copper cathode and an annular copper anode (both water cooled). Plasma generating gas is forced to pass through the annular space between the electrodes. While passing through the arc, the gas undergoes ionization in the high temperature environment resulting plasma. The ionization is achieved by collisions of electrons of the arc with the neutral molecules of the gas. The plasma protrudes out of the electrode encasement in the form of a flame. The consumable material, in the powdered form, is poured into the flame in metered quantity. The powders melt immediately and absorb the momentum of the expanding gas and rush towards the target to form a thin deposited layer. The next layer deposits onto the first immediately after, and thus the coating builds up layer by layer. The temperature in the plasma arc can be as high as $10{,}000^{0}$C and it is capable of melting anything. Elaborate cooling arrangement is required to protect the plasmatron (i.e., the plasma generator) from excessive heating. The equipment consists of the following modules [3, 4, 5].

- <u>The plasmatron</u> : It is the device which houses the electrodes and in which the plasma reaction takes place. It has the shape of a gun and it is connected to the water cooled power supply cables, powder supply and gas supply hose.

- <u>The power supply unit</u> : Normally plasma arc works in a low voltage (40-70 volts) and high current (300-1000 Amperes), DC ambient. The available power (AC, 3-phase, 440 V) must be transformed and rectified to suit the reactor. This is taken care of by the power supply unit.

- The powder feeder : The powder is kept inside a hopper. A separate gas line directs the career gas which fluidizes the powder and carries it to the plasma arc. The flow rate of the powder can be controlled precisely.

- The coolant water supply unit : It circulates water into the plasmatron, the power supply unit, and the power cables. Units capable of supplying refrigerated water are also available.

- The control unit: Important functions (current control, gas flow rate control etc.) are performed by the control unit. It also consists of the relays and solenoid valves and other interlocking arrangements essential for safe running of the equipment. For example the arc can only be started if the coolant supply is on and water pressure and flow rate is adequate.

The Requirements for Plasma Spraying

- Roughness of the substrate surface: A rough surface provides a good coating adhesion. A rough surface provides enough room for anchorage of the splats facilitating bonding through mechanical interlocking. A rough surface is generally created by shot blasting technique. The roughness obtained is determined by shot blasting parameters, i.e., shot size, shape and material, air pressure, standoff distance between nozzle and the job, angle of impact, substrate material etc.

- Cleanliness of the substrates: The substrate to be sprayed on must be free from any dirt or grease or any other material that might prevent intimate contact of the splat and the substrate. For this purpose the substrate must be thoroughly cleaned (ultrasonically, if possible) with a solvent before spraying. Spraying must be conducted immediately after shot blasting and cleaning.

- Cooling water: For cooling purpose distilled water should be used, whenever possible. Normally a small volume of distilled water is recirculated into the gun and. it is cooled by an external water supply from a large tank. Sometime water from a large external tank is pumped directly into the gun.

Process Parameters in Plasma Spraying

In plasma spraying one has to deal with a lot of process parameters, which determine the degree of particle melting, adhesion strength and deposition efficiency of the powder. Deposition efficiency is the ratio of amount of powder deposited to the amount fed to the gun. An elaborate listing of these parameters and their effects are reported in the literature [6, 7].

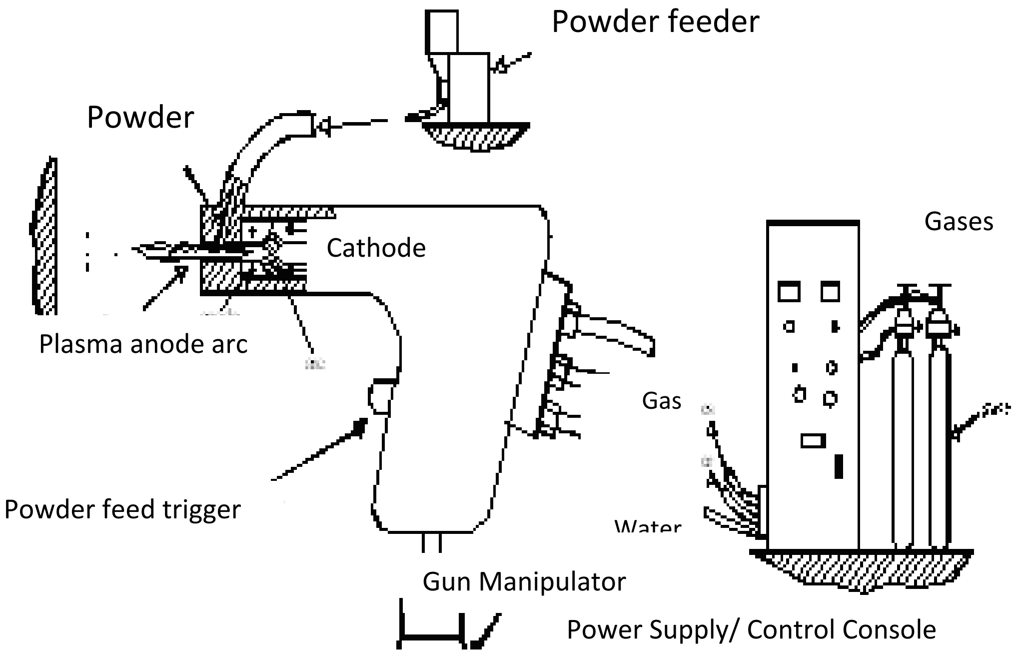

Fig. 2.2 Arrangement for the plasma spraying

Some important parameters and their roles are listed below:

<u>Arc power</u>: It is the electrical power drawn by the arc. The power is injected in to the plasma gas, which in turn heats the plasma stream. Part of the power is dissipated as radiation and also by the gun cooling water. Arc power determines the mass flow rate of a given powder that can be effectively melted by the arc. Deposition efficiency

improves to a certain extent with an increase in arc power, since it is associated with an enhanced particle melting [8]. However, increasing power beyond a certain limit may not cause a significant improvement. On the contrary, once a complete particle melting is achieved, a higher gas temperature may prove to be harmful. In the case of steel, at some point vaporization may take place lowering the deposition efficiency.

Plasma gas: Normally nitrogen or argon doped with about 10% hydrogen or helium is used as a plasma gas. The major constituent of the gas mixture is known as primary gas and the minor is known as the secondary gas. The neutral molecules are subjected to the electron bombardment resulting in their ionization. Both temperature and enthalpy of the gas increase as it absorbs energy. Since nitrogen and hydrogen are diatomic gases, they first undergo dissociation followed by ionization. Thus they need higher energy input to enter the plasma state. This extra energy increases the enthalpy of the plasma. On the other hand, the mono-atomic plasma gases, i.e. argon or helium, approach a much higher temperature in the normal enthalpy range. Good heating ability is expected from them for such high temperature. In addition, hydrogen followed by helium has a very high specific heat, and therefore is capable of acquiring very high enthalpy. When argon is doped with helium the spray cone becomes quite narrow which is especially useful for spraying on small targets.

Carrier gas: Normally the primary gas itself is used as a carrier gas. The flow rate of the carrier gas is an important factor. A very low flow rate cannot convey the powder effectively to the plasma jet, and if the flow rate is very high then the powders might escape the hottest region of the jet. There is an optimum flow rate for each powder at which the fraction of unmelted powder is minimum and hence the deposition efficiency is maximum [9,10].

Mass flow rate of powder: Ideal mass flow rate for each powder has to be determined. Spraying with a lower mass flow rate keeping all other conditions constant results in under utilization and slow coating buildup. On the other hand, a very high mass flow rate may give rise to an incomplete melting resulting in a high amount of porosity in the coating. The unmelted powders may bounce off from the substrate surface as well keeping the deposition efficiency low [11].

Torch to base distance : It is the distance between the tip of the gun and the substrate surface. A long distance may result in freezing of the melted particles before they reach the target, whereas a short standoff distance may not provide sufficient time for the particles in flight to melt [12]. The relationship between the coating properties and spray parameters in spraying alpha alumina has been studied in details [13]. It is found that the porosity increases and the thickness of the coating (hence deposition efficiency) decreases with an increase in standoff distance. The usual alpha-phase to gamma-phase transformation during plasma spraying of alumina has also been restricted by increasing this distance. A larger fraction of the unmelted particles go in the coating owing to an increase in torch to base distance.

Spraying angle: This parameter is varied to accommodate the shape of the substrate. In coating alumina on mild steel substrate, the coating porosity is found to increase as the spraying angle is increased from 30° to 60°. Beyond 60° the porosity level remains unaffected by a further increase in spraying angle. The spraying angle also affects the adhesive strength of the coating. The influence of spraying angle on the cohesive strength of chromia, zirconia 8-wt% yttria and molybdenum has been investigated, and it has been found that the spraying angle does not have much influence on the cohesive strength of the coatings.

COMMON CERAMIC COATINGS

Today a variety of materials, e.g., carbides, oxides etc., belonging to the above category are available commercially.

(i) Carbides: WC, TiC, SiC, ZrC, Cr_2C_3 etc.
(ii) Oxides: Al_2O_3, Cr_2O_3, TiO_2, ZrO2 etc.
The choice of a material depends on the application. However, the ceramic coatings are very hard and hence on an average offer more abrasion resistance than their metallic counterparts.

Carbide Coatings

Amongst carbides, WC is very popular for wear and corrosion applications [14]. The WC powders are clad with a cobalt layer. During spraying the cobalt layer undergoes melting and upon solidification form a metallic matrix in which the hard WC particles remain embedded. Spraying of WC-Co involves a close control of the process parameters such that only the cobalt phase melts without degrading the WC particles. Such degradation may occur in two ways:

- Oxidation of WC leading to the formation of $CoWO_4$ and WC_2.
- Dissolution of WC in the cobalt matrix leading to a formation of brittle phases like CoW_3C which embrittles the coating.

An increase in the spraying distance and associated increase of time in flight lead to a loss of carbon and a pickup of oxygen. As a result the hardness of the coating decreases. An increase in plasma gas flow rate reduces the dwell time and hence can control the oxidation to some extent. However, it increases the possibility of cobalt dissolution in the matrix [15]. The other option to improve the quality of such coating is to conduct the spraying procedure in vacuum.

Often carbides like TiC, TaC and NbC are provided along with WC in the cermet to improve upon the oxidation resistance, hardness, and hot strength. Similarly the binder phase is also modified by adding chromium and nickel with cobalt. The wear mechanism of plasma sprayed WC-Co coatings depends on a number of factors, e.g., mechanical properties, cobalt content, experimental conditions, mating surfaces, etc. The wear mode can be abrasive, [16] adhesive or surface fatigue. The coefficient of friction of WC-Co (in self mated condition) increases with increasing cobalt content. A WC-Co coating when tested at a temperature of 450°C exhibits signs of melting. The wear resistance of these coatings also depends on porosity. Pores can also act as source from where the cracks may grow. Thermal diffusivity of the coatings is another important factor. In narrow contact regions, an excessive heat generation may occur owing to rubbing. If the thermal diffusivity of the coating is low the heat cannot escape from a narrow region easily resulting a rise in temperature and thus failure occurs owing to thermal stress. The wear mechanism of WC-Co nano-composite coating on mild steel substrates has been studied in details [17, 18]. The wear rates of such coatings are found to be much greater than that of commercial WC-Co composite coating, presumably owing to an enhanced decomposition of nano-particles during spraying. Wear has been found to occur by subsurface cracking along the preferred crack paths provided by the binder phase or failure at the inter-splat boundary. Coatings of TiC or TiC+ TaC with a nickel cladding are alternative solutions for wear and corrosion problems. High temperature stability, low coefficient of thermal expansion, high hardness and low specific gravity of these coatings may outperform other materials, especially in steam environment. Instead of nickel, nickel chromium alloy can serve as the matrix material. The mode of wear can be adhesive, abrasive, surface fatigue or micro-fracture depending on operating conditions [19, 20].

Oxide Coatings

Metallic coatings and metal containing carbide coatings sometime are not suitable in high temperature environments in both wear and corrosion applications. Often they fail owing to oxidation or decarburization. In such case the material of choice can be an oxide ceramic coating, e.g., $Al_2O_3.Cr_2O_3$, TiO_2, ZrO_2 or their combinations. However, a high wear resistance, and chemical and thermal stability of these materials are counterbalanced by the disadvantages of low values of thermal expansion coefficient, thermal conductivity, mechanical strength, fracture toughness and somewhat weaker adhesion to substrate material. The thickness of these coatings is also limited by the residual stress that grows with thickness. Therefore, to obtain a good quality coating it is essential to exercise proper choice of bond coat, spray parameters and reinforcing additives.

Chromia (Cr_2O_3) Coatings

These coatings are applied when corrosion resistance is required in addition to abrasion resistance. It adheres well to the substrate and shows an exceptionally high hardness 2300 $HV_{0.5\ kg}$.[21,22] Chromia coatings are also useful in ship and other diesel engines, water pumps, and printing rolls. A Cr_2O_3- 40 wt% TiO_2 coating provides a very high coefficient of friction (0.8), and hence can be used as a brake liner. The wear mode of chromia coatings has been investigated under various conditions. Depending on experimental conditions, the wear mode can be abrasive, plastic deformation, micro-fracture or a conglomerate of all of these [23]. This material has also been tested under lubricated conditions, using inorganic salt solutions (NaCl, $NaNO_3$, Na_3PO_4) as lubricants and also at a high temperature. The wear rate of self-mated chromia is found to increase considerably at 450°C, and plastic deformation and surface fatigue are the predominant wear mechanisms. Under lubricated condition, the coatings exhibit tribo-chemical wear. It has also been tested for erosion resistance [24].

Zirconia (ZrO_2) Coatings

Zirconia is widely used as a thermal barrier coating. However, it is endowed with the essential qualities of a wear resistant material, i.e., hardness, chemical inertness, etc. and shows reasonably good wear behaviour. In the case of a hot pressed zirconia mated with high chromium containing iron (martensitic, austenitic, or pearlitic), it has been found that in course of rubbing the iron transfers on to the ceramic surface and the austenitic material adheres well to the ceramic as compared to their martensitic or pearlitic counterparts [25]. The thick film improves the heat transfer from the contact area keeping the contact temperature reasonably low; thus the transformation of ZrO_2 is prevented. On the other hand with the pearlitic or martensitic iron the material transfer is limited. The contact temperature is high enough to bring about a phase transformation and related volume change in ZrO_2 causing a stress induced spalling. In a similar experiment the wear behaviour of sintered, partially stabilized zirconia (PSZ) with 8 wt% yttria against PSZ and steels has been tested at 200°C. When metals are used as the mating surface, a transferred layer soon forms on the ceramic surface (coated or sintered). In ceramic-ceramic system the contact wear is abrasive in nature. However, similar worn particles remain entrapped between the contact surfaces and induce a polishing wear too. In the load range of 10 to 40 N, no transformation of ZrO_2 occurs. However, similar tests conducted at 800°C show a phase transformation from monoclinic ZrO_2 to tetragonal ZrO_2. The wear debris of ZrO_2 sometimes get compacted in repeated loading and gets attached to the worn surface forming a protective layer. During rubbing, pre-existing or newly formed cracks may grow rapidly and eventually interconnect with each other, leading to a spallation of the coating. Introduction of alumina as a dopant, has been found to improve the wear performance of the ceramic significantly. Here plastic deformation is the main wear mode. The wear performance of zirconia at 400°C and 600°C has been reported in the literature [26]. At these temperatures the adhesive mode of wear plays the major role.

Alumina (Al_2O_3) - Titania (TiO_2) Coatings

Titania coating is known for its high hardness, density, and adhesion strength. It has been used to combat abrasive, erosive and fretting wear either in essentially pure form or in association with other compounds. The mechanism of wear of TiO_2 at 450°C under both lubricated and dry contact conditions has been studied [27, 28]. It has been found to undergo a plastic smearing under lubricated contact, where as it fails owing to the surface fatigue in dry condition. TiO_2-stainless steel couples in various speed load conditions have also been investigated in details [29]. At a relatively low load, the failure is owing to the surface fatigue and adhesive wear, whereas at a high load the failure is attributed to the abrasion and delamination associated with a back and forth movement.At a high speed, Fe_3O_4 forms instead of Fe_2O_3. The TiO_2 top layer also softens and melts owing to a steep rise in temperature, which helps in reducing the temperature subsequently. The performance of the plasma sprayed pure TiO_2 has been compared with those of Al_2O_3 – 40 wt% TiO_2 and pure Al_2O_3 under both dry and lubricated contact conditions [30]. TiO_2 shows the best results. TiO_2 owing to its relatively high porosity can provide good anchorage to the transferred film and also can hold the lubricants effectively.

Alumina is obtained from a mineral called bauxite, which exists in nature as a number of hydrated phases, e.g., boehmite (γ-Al_2O_3, H_2O), hydragillate, diaspore (α-Al_2O_3. $3H_2O$). It also exists in several other metastable forms like β, δ, θ, η, κ and X [31]. α -Al_2O_3 is known to be a stable phase and it is available in nature in the form of corundum. In addition, α-Al_2O_3 can be extracted from the raw materials by fusing them. The phase transformation during freezing of the plasma sprayed alumina droplets has been studied in details [32, 33]. There are several advantages of alumina as a structural material, e.g., availability, hardness, high melting point, resistance to wear and tear etc. It bonds well with the metallic substrates when applied as a coating on them. Some of the applications of alumina are in bearings, valves, pump seals, plungers, engine components, rocket nozzles, shields for guided missiles, vacuum tube envelops, integrated circuits, etc. Plasma sprayed alumina-coated railroad components are presently being used in Japan [34].

Properties of alumina can be further complemented by the particulate (TiO_2, TiC) or whisker (SiC) reinforcement. TiC reinforcement limits the grain growth, improves strength and hardness, and also retards crack propagation through the alumina matrix. The sliding wear behaviour of both monolithic and SiC whisker reinforced alumina has been studied [35]. The whisker reinforced composite has been found to have good wear resistance. The monolithic alumina has a brittle response to sliding wear, whereas the worn surface of the composite reveals signs of plastic deformation along with fracture. The whiskers also undergo pullout or fracture.

TiO_2 is a commonly used additive in plasma sprayable alumina powder. TiO_2 , has a relatively low melting point and it effectively binds the alumina grains. However, a success of an Al_2O_3 - TiO_2 coating depends upon a judicious selection of the arc current, which can melt the powders effectively. This results in a good coating adhesion along with high wear resistance [36]. The wear performance of Al_2O_3 and Al_2O_3 -50 wt% TiO_2 has been reported in the literature [37]. In dry sand abrasion testing, alumina outperformed others presumably owing to its high hardness . In dry sliding at low velocity range, the tribocouple (ceramic and hardened stainless steel) exhibits stick-slip [38]. At relatively high speed range, the coefficient of friction drops owing to the thermal softening of the interface. The wear of alumina is found to increase appreciably beyond a critical speed and a critical load. Alumina has been found to fail by plastic deformation, shear and grain pullout. In dry and lubricated sliding as well, the mixed ceramic has been found to perform better than pure alumina.

A coating of Al_2O_3 -50 wt% TiO_2 is quite porous and hence is quite capable of holding the transferred metallic layer which protects the surface. Wear performance of such coatings can further be improved by a sealing of the pores by polymeric substances. A low thermal diffusivity of the alumina coatings results in a high localized thermal stress on the surface. The mode of wear of alumina is mainly abrasive. The pore size and pore size distribution also play a vital role in determining the wear properties. The Al_2O_3 - TiO_2 coating has a high thermal diffusivity and hence it is less prone to wear.

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CHAPTER 3

Chapter 3

ANALYSIS AND PREDICTION

Coating deposition by plasma spraying involves a number of process variables, which contribute in a large way to the quality of the coating. During spraying, various operating parameters are determined mostly based on past experience. It therefore does not provide the optimal set of parameters for a particular objective. In order to obtain the best result with regard to any specific coating quality characteristic, accurate identification of significant control parameters is essential. Deposition efficiency of any coating is a characteristic which not only rates the effectiveness of the spraying method but also is a measure of the coatability of the material under study. This chapter is devoted to analyze the experimentally obtained results on the deposition efficiency of alumina-titania coatings made at different operational conditions. For this purpose, a statistical technique called *Taguchi experimental design methd* is used. Factors are identified according to their influence on the coating deposition. The most significant parameter is found. A prediction model using *artificial neural network* (ANN) is presented considering the significant factors.

TAGUCHI EXPERIMENTAL DESIGN

Taguchi method of experimental design is a simple, efficient and systematic approach to optimize designs for performance and cost. In the present work, this method is applied to the process of plasma spraying for identifying the significant process variables/interactions influencing coating deposition efficiency. The levels of these factors are also found out so that the process variables can be optimized within the test range.

Experimental Design

Experiments are carried out to investigate the influence of the four selected control parameters (identified as significantly affecting coating deposition efficiency from the fractional factorial test) and that of the interactions among them. The code and levels of control parameters are shown in Table 3.1. This table shows that the experimental plan has three levels. A standard Taguchi experimental plan with notation $\mathbf{L_{27}\ (3^{13})}$ is chosen as outlined in table 3.1. In this method, experimental results are transformed into a signal-to-noise (S/N) ratio. It uses the S/N ratio as a measure the quality characteristics deviating from or nearing to the desired values. There are three categories of quality characteristics in the analysis of the S/N ratio, i.e. the lower-the-better, the higher-the-better, and the nominal-the-better. To obtain optimal spraying parameters, the higher-the-better quality characteristic for deposition efficiency is taken.

Parameter Code	**Levels**		
	I	**II**	**III**
Arc current intensity (amp) **A**	200	300	400
Arc voltage (volt) **B**	40	50	60
Powder feed rate (gm/min) **C**	8	12	16
Substrate surface roughness (micron) **D**	4.5	5.8	7.2
Torch to Base Distance (mm) **E**	75	100	125
TiO_2 content in feedstock (wt %) **F**	0	10	20

Table 3.1 Control factors and their selected levels

Run Number	Arc Current	Arc Voltage	Power Feed Rate	Substrate surface roughness	Torch to Base Distance	TiO_2 content in feedstock	Deposition Efficiency
1	200	40	8	4.5	75	0	17.87
2	200	40	8	4.5	100	10	13.54
3	200	40	8	4.5	125	20	21.51
4	200	50	12	5.8	75	0	16.38
5	200	50	12	5.8	100	10	18.43
6	200	50	12	5.8	125	20	23.98
7	200	60	16	7.2	75	0	18.27
8	200	60	16	7.2	100	10	21.56
9	200	60	16	7.2	125	20	25.67
10	300	40	12	7.2	75	10	27.65
11	300	40	12	7.2	100	20	31.43
12	300	40	12	7.2	125	0	23.45
13	300	50	16	4.5	75	10	27.98
14	300	50	16	4.5	100	20	32.66
15	300	50	16	4.5	125	0	29.78
16	300	60	8	5.8	75	10	32.67
17	300	60	8	5.8	100	20	35.45
18	300	60	8	5.8	125	0	27.88
19	400	40	16	5.8	75	20	37.91
20	400	40	16	5.8	100	0	29.77
21	400	40	16	5.8	125	10	32.59
22	400	50	8	7.2	75	20	36.95
23	400	50	8	7.2	100	0	30.08
24	400	50	8	7.2	125	10	33.66
25	400	60	12	4.5	75	20	37.84
26	400	60	12	4.5	100	0	32.44
27	400	60	12	4.5	125	10	34.76

Table 3.2 Experimental lay out and coating deposition efficiency values

Analysis of control factor and interaction

Table 3.2 shows the experimental lay out and Table 3.3 shows the results with calculated S/N ratios for deposition efficiency of the coatings. Analysis of the influence of each control factor on the coating efficiency is made with a signal-to-noise (S/N) response table, using MINITAB computer package. The response data of the testing process is presented in Table 3.4. The influence of control factors is also analyzed in the response table. The control factor with the strongest influence is determined by differences values. The higher the difference, the more influential is the control factor. The strongest influence on coating deposition efficiency is found out to be of plasma arc current intensity (A) followed by titania content (F) and plasma arc voltage (B) respectively.

Run Number	Deposition Efficiency	S/N Ratio
1	17.87	25.0425
2	13.54	22.6324
3	21.51	26.6528
4	16.38	24.2863
5	18.43	25.3105
6	23.98	27.5970
7	18.27	25.2348
8	21.56	26.6730
9	25.67	28.1885
10	27.65	28.8339
11	31.43	29.9469
12	23.45	27.4029
13	27.98	28.9370
14	32.66	30.2803
15	29.78	29.4785
16	32.67	30.2830
17	35.45	30.9923
18	27.88	28.9059
19	37.91	31.5751
20	29.77	29.4756
21	32.59	30.2617
22	36.95	31.3523
23	30.08	29.5656
24	33.66	30.5423
25	37.84	31.5590
26	32.44	30.2216
27	34.76	30.8216

Table 3.3 coating deposition efficiency values and the S/N ratios

Level	A	B	C	D	E	F
1	25.74	27.98	28.44	28.40	28.57	27.73
2	29.45	28.59	28.44	28.74	28.34	28.26
3	30.60	29.21	28.90	28.64	28.87	29.79
Diff.	4.86	1.23	0.46	0.34	0.53	2.06
Rank	**1**	**3**	**5**	**6**	**4**	**2**

Table 3.4 The S/N response table for coating deposition efficiency

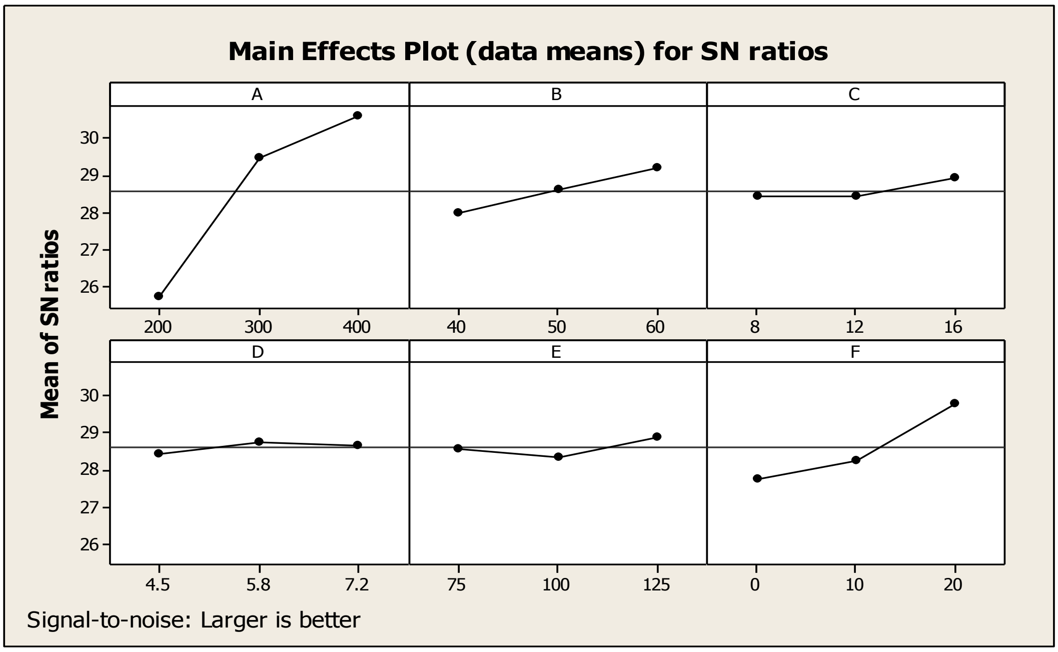

Fig 3.1 Effect of control factors

It is interesting to note that the Taguchi experimental design method identified plasma arc current as the most powerful factor influencing the deposition efficiency of the alumina-titania coatings. The weight fraction of titania (TiO_2) in the feedstock i.e. alumina-titania mixture and the plasma arc voltage emerge as the other significant factors affecting the coating deposition. The influence of interactions (among the control factors) is not included within the scope of this investigation.

NEURAL COMPUTATION

Plasma spraying is considered as a non-linear problem with respect to its variables: either materials or operating conditions. To obtain functional coatings exhibiting selected in-service properties, combinations of processing parameters have to be planned. These combinations differ by their influence on the coating properties and characteristics. In order to control the spraying process, one of the challenges nowadays is to recognize parameter interdependencies, correlations and individual effects on coating characteristics. Therefore a robust methodology is needed to study these interrelated effects. In this work, a statistical method, responding to the previous constraints, is implemented to correlate the processing parameters to the coating properties. This methodology is based on artificial neural networks (ANN), which is a technique that involves database training to predict property-parameter evolutions. This section presents the database construction, implementation protocol and a set of predicted results related to the coating deposition efficiency. The details of this methodology are described by Rajasekaran & Pai and Rao & Rao [39, 40].

ANN MODEL: DEVELOPMENT AND IMPLEMENTATION

The deposition efficiency describes the fraction of fed powder deposited on the substrate. For a given feed rate, it is mostly conditioned by the target material, coating material composition and the spraying parameters. The plasma arc current and voltage have already been identified (from the outcome of Taguchi analysis) as the parameters significantly affecting the coating deposition. Hence, in the present analysis the plasma arc current, arc voltage and the weight percentage of titania in the feedstock are taken as the three input parameters for training. Each of these parameters is characterized by one neuron and consequently the input layer in the ANN structure has three neurons.

The database is built considering experiments at the limit ranges of each parameter. Experimental result sets are used to train the ANN in order to understand the input-output correlations. The database is then divided into three categories, namely:

(i) A validation category, which is required to define the ANN architecture and adjust the number of neurons for each layer.

(ii) A training category, which is exclusively used to adjust the network weights and

(iii) A test category, which corresponds to the set that validates the results of the training protocol.

The input variables are normalized so as to lie in the same range group of 0-1. To train the neural network used for this work, about 108 data sets of different coatings applied on mild steel substrates are taken. It is ensured that these extensive data sets represent all possible input variations within the experimental domain. So a network that is trained with this data is expected to be capable of simulating the plasma spray process. Different ANN structures (I-H-O) with varying number of neurons in the hidden layer are tested at constant cycles, learning rate, error tolerance, momentum parameter and noise factor and slope parameter. Based on least error criterion, one structure, shown in Table 3.5, is selected for training of the input-output data. The learning rate is varied in the range of 0.001-0.100 during the training of the input-output data. The network optimization process (training and testing) is conducted for 1000,000 cycles for which stabilization of the error is obtained. Neuron numbers in the hidden layer is varied and in the optimized structure of the network, this number is 10. The number of cycles selected during training is high enough so that the ANN models could be rigorously trained.

Input Parameters for Training	Values
Error tolerance	0.01
Learning parameter(ß)	0.01
Momentum parameter(α)	0.02
Noise factor **(NF)**	0.002
Maximum cycles for simulations	1000,000
Slope parameter (£)	0.6
Number of hidden layer	1
Number of input layer neuron **(I)**	3
Number of output layer neuron **(O)**	1

Table 3.5 Input parameters selected for training

A software package NEURALNET for neural computing developed by Rao and Rao [40] using back propagation algorithm is used as the prediction tool for coating deposition efficiency at different operating power levels. The three-layer neural network having an input layer (I) with three input nodes, a hidden layer(H) with ten neurons and an output layer (O) with one output node employed for this work is shown in Fig. 3.2.

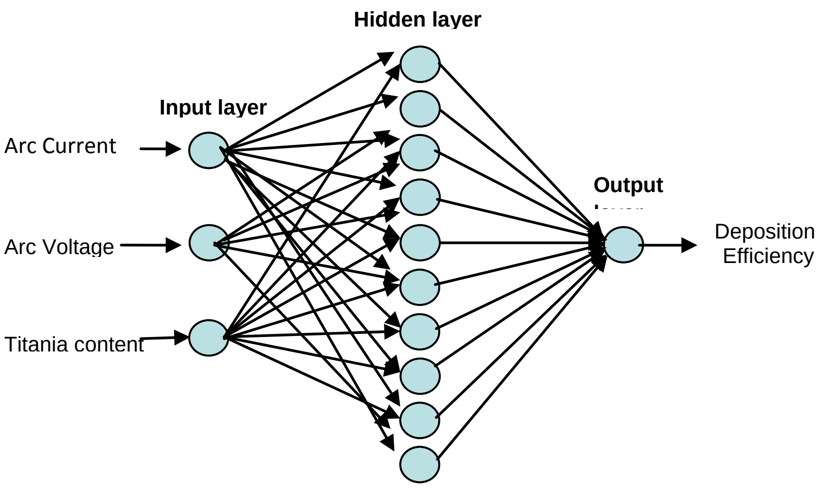

Fig. 3.2 The three layer neural network

Test run	Deposition Efficiency (%) (Experimental)	Deposition Efficiency (%) (ANN Predicted)	Error (%)
1	0.1843	0.211740	**-14.89**
2	0.2765	0.279310	**1.02**
3	0.3266	0.297240	**8.98**
4	0.3791	0.335024	**11.63**
5	0.3784	0.373018	**1.42**

Table 3.6 Comparison of experimental results and ANN results

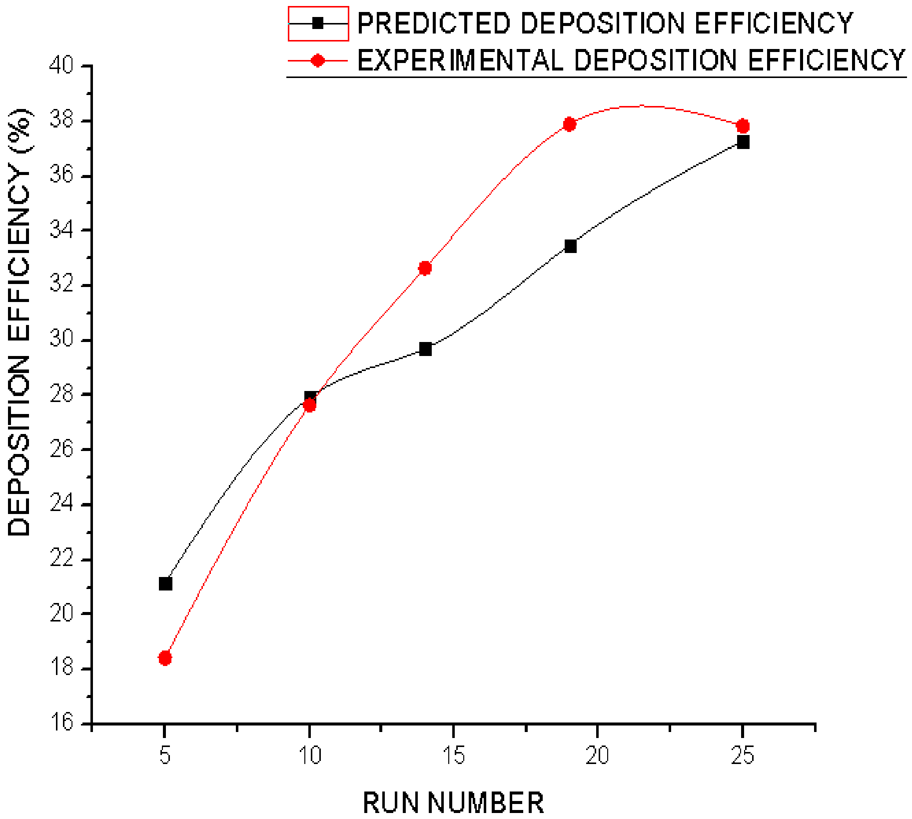

Fig. 3.3 Comparison curve showing the experimental and predicted values

ANN PREDICTION OF DEPOSITION EFFICIENCY

The prediction neural network was tested with twenty data sets from the original process data. Each data set contained inputs such as plasma arc current, arc voltage, titania content and an output value i.e. deposition efficiency was returned by the network. As further evidence of the effectiveness of the model, an arbitrary set of inputs is used in the prediction network. Results were compared to experimental sets that may or may not be considered in the training or in the test procedures. Fig. 3.3 presents the comparison of predicted output values for deposition efficiency with those obtained experimentally.

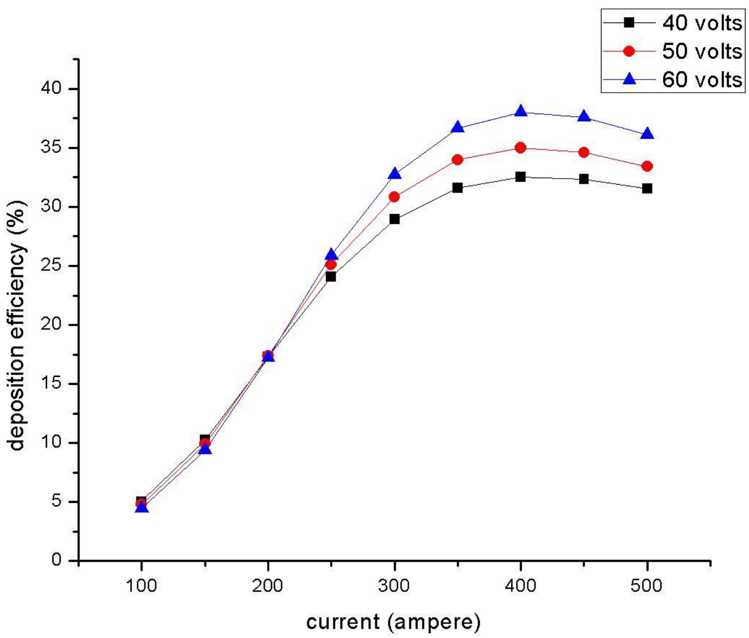

Fig. 3.4 Prediction curve showing current v/s deposition efficiency (0% TiO_2)

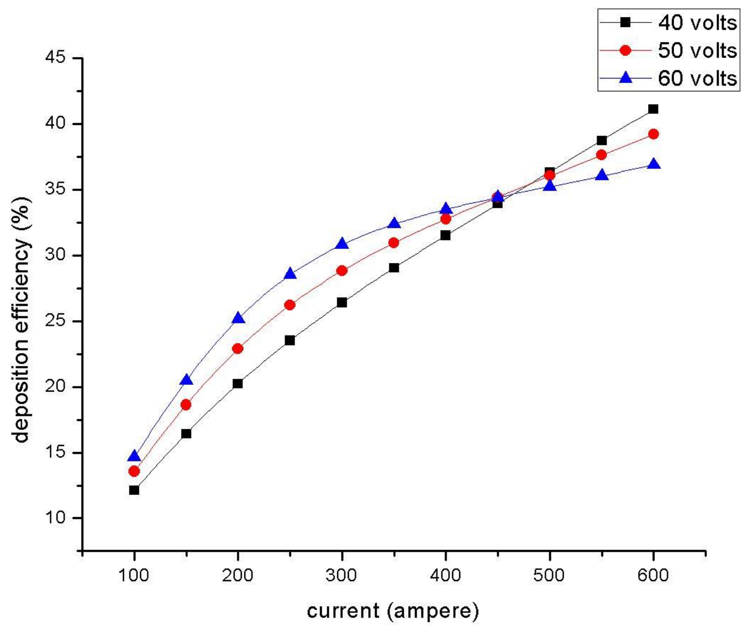

Fig. 3.5 Prediction curve showing current v/s deposition efficiency (20% TiO_2)

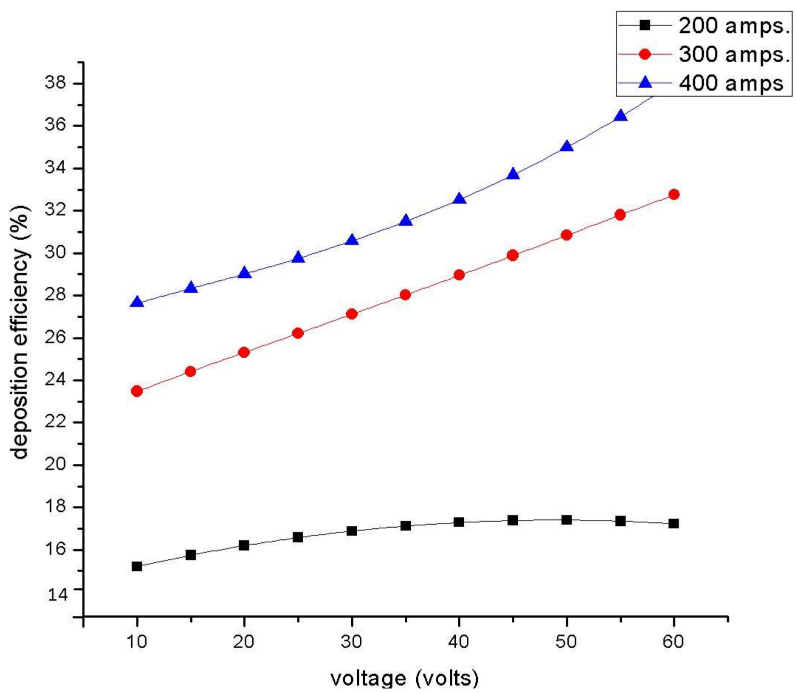

Fig. 3.6 Prediction curve showing voltage v/s deposition efficiency (0% TiO_2)

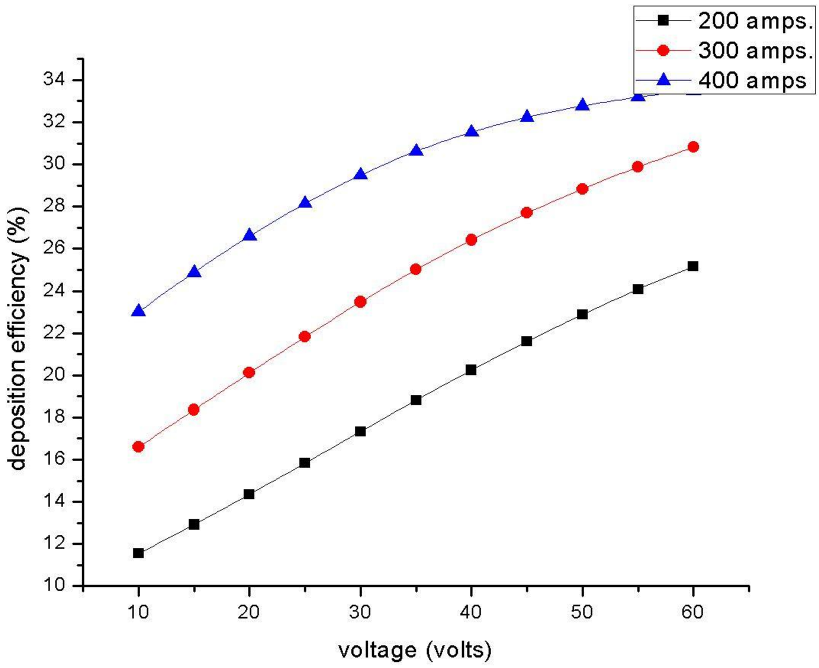

Fig. 3.7 Prediction curve showing voltage v/s deposition efficiency (20% TiO_2)

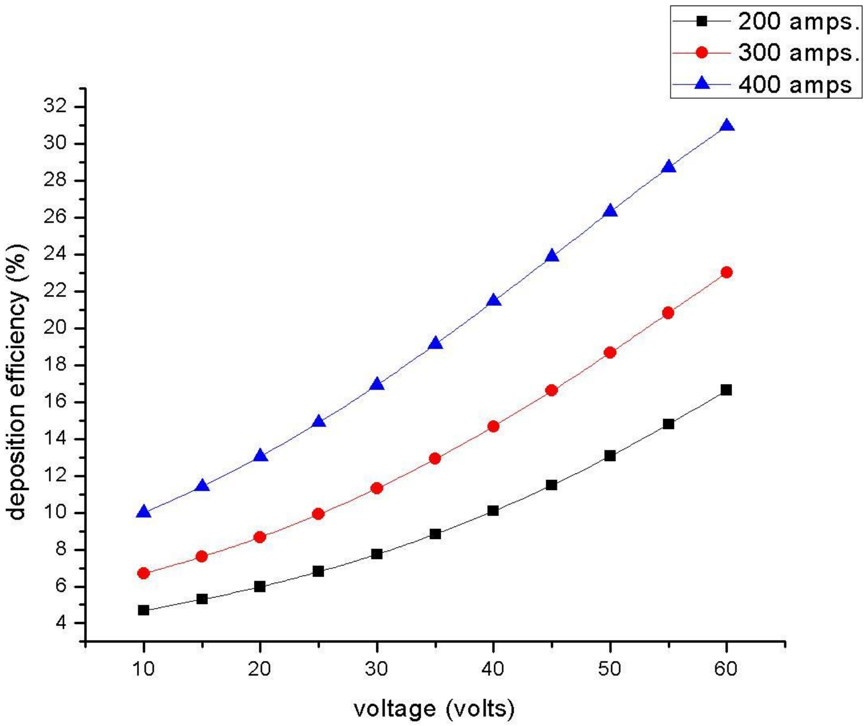

Fig. 3.8 Prediction curve showing voltage v/s deposition efficiency (40% TiO_2)

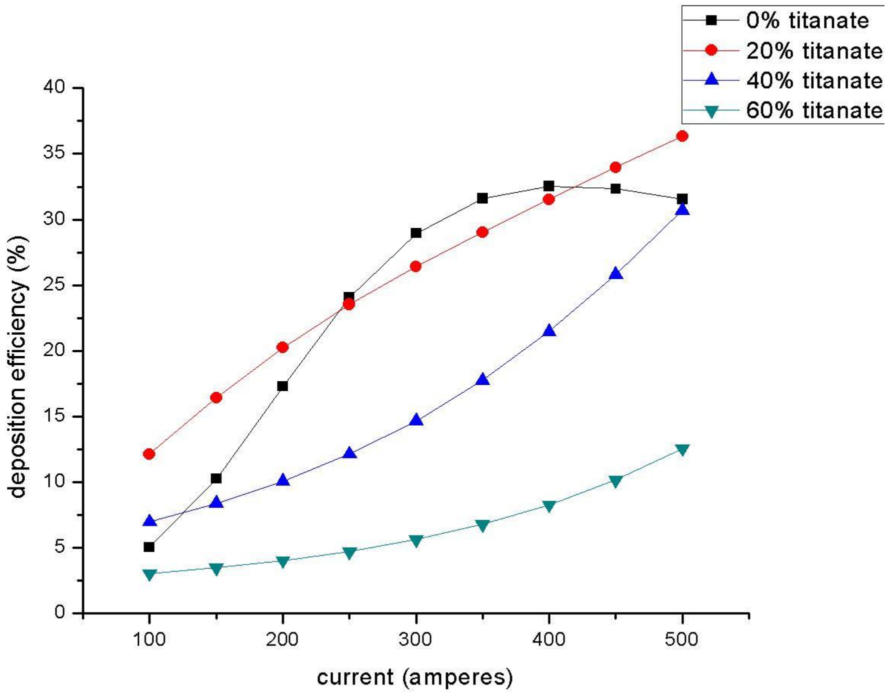

Fig. 3.9 Prediction curve showing current v/s deposition efficiency (40 volts)

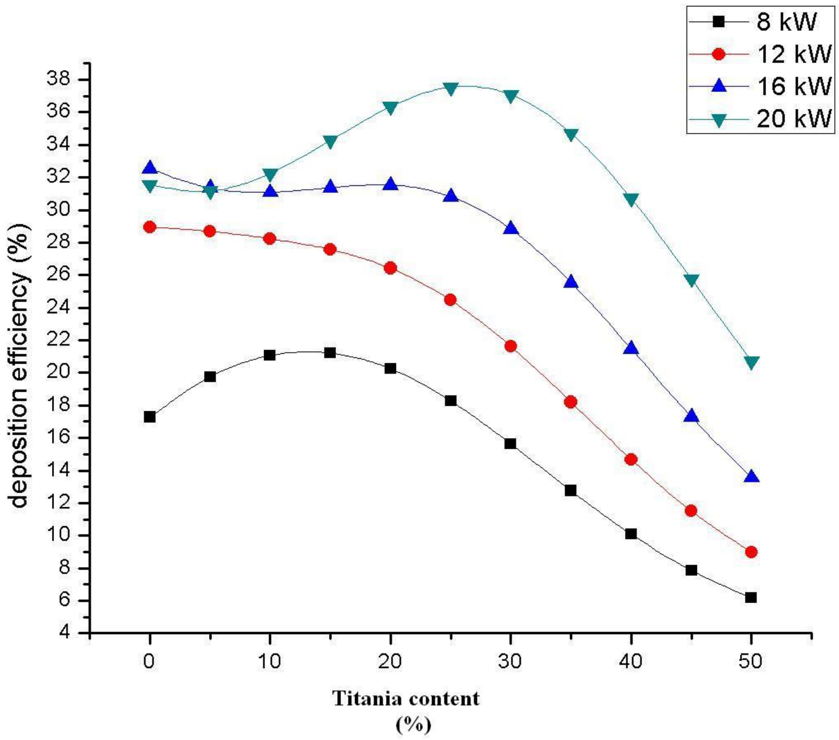

Fig. 3.10 Prediction curve showing TiO_2 content v/s deposition efficiency

It is interesting to note that the predictive results show good agreement with experimental sets realized after having generalizing the ANN structures. The optimized ANN structure further permits to study quantitatively the effect of each of the considered input parameter. The range of any chosen parameter can be larger than the actual experimental limits, thus offering the possibility to use the generalization property of ANN in a large parameter space. In the present investigation, this possibility was explored by selecting the plasma arc current in a range from 100 to 500 amps, and sets of predictions for deposition efficiency are evolved. The prediction curves are shown in Figs 3.4 and 3.5 for different titania content. Similarly Figs. 3.6 to 3.8 illustrate the predicted evolution of deposition efficiencies with respect to arc voltage in a range of 10 to 60 volts, Fig. 3.9 represents the prediction curve for deposition efficiency with the plasma arc current at a voltage of 40 volts and Fig. 3.10 represents the prediction curve for deposition efficiency with the weight fraction of titania.

The deposition efficiency presents a sigmoid-type with the plasma arc current and exponential-type evolution with the plasma arc voltage. As the power level increases, the total and the net available energies increase (the arc current intensity increases from 100A to 500A). This leads to a better in-flight particle molten state and hence to higher probability for particles to flatten. The deposition efficiency reaches a plateau for the highest current levels due to the plasma jet temperature increasing which in turn increases both the particle vaporization ratio and the plasma jet viscosity.

Factor Settings for Maximum Coating Deposition Efficiency

In this study, an attempt is made to derive optimal settings of the control factors for maximization of Coating Deposition Efficiency. The single-objective optimization requires quantitative determination of the relationship between coating deposition efficiency with combination of control factors. In order to express coating deposition efficiency in terms of the mathematical model, the following equation is suggested:

$$DE = K_0 + K_1 \times I + K_2 \times V + K_3 \times T \qquad \text{--------- (5.1)}$$

Here, *DE* is the performance output term i.e. the coating deposition efficiency and *Ki* (*i* = 0, 1, 2, 3) are the model constants. I is the plasma arc current (amp), *V* is the plasma arc voltage (volt) and *T* is the TiO_2 content in the feed stock (wt%). The constants are calculated using non-linear regression analysis with the help of SYSTAT- 7 software and the following relations are obtained:

$$DE = -5.362 + 0.072 \times I + 0.171 \times V + 0.319 \times T \qquad \text{--------- (5.2)}$$

The correctness of the calculated constants is confirmed as a high correlation coefficient (r^2) to the tune of 0.995 is obtained for Equation (5.1). Eq. 5.2 can therefore be used for predictive purpose to obtain deposition efficiency of plasma sprayed alumina-titania coatings.

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CHAPTER 4

Chapter 4

CONCLUSIONS AND SCOPE FOR FUTURE WORK

Functional coatings have to fulfil various requirements. The deposition efficiency is one the main requirements of the coatings developed by plasma spraying. It represents the effectiveness of the deposition process as well as the coatability of the powders under study. In order to achieve certain values of deposition efficiency accurately and repeatedly, the influence parameters of the process have to be controlled accordingly. Since the number of such parameters in plasma spraying is too large and the parameter-property correlations are not always known, statistical methods can be employed for precise identification of significant control parameters for optimization. Neural computation can be used as a tool to process very large data related to a spraying process and to predict any desired coating characteristic, the simulation can be extended to a parameter space larger than the domain of experimentation.

This work shows that artificial neural networks can be gainfully employed to simulate property-parameter correlations in a space larger than the experimental domain. It is evident that with an appropriate choice of processing conditions a sound and adherent alumina-titania is achievable with fairly good deposition efficiency.

This work leaves a wide scope for future investigators to explore many other aspects of plasma sprayed coatings using ANN. Some recommendations for future research include:

- Analysis and prediction of interface bond strength at different power levels
- Exploration of new coating materials and prediction of for deposition characteristics.

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