

APPLICATION OF RESPONSE SURFACE METHODOLOGY IN OPTIMIZING
ELECTROPLATING PARAMETERS OF NICKEL INTERLAYER ON
TUNGSTEN CARBIDE

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
Master of (Mechanical Engineering)

Faculty of Mechanical Engineering
Universiti Teknologi Malaysia

JULY 2013

Dedicated to my family and beloved wife

ACKNOWLEDGEMENTS

I would like to thank to Allah Almighty for blessing and strength given to accomplish this thesis. A special thank and sincere gratitude to my supervisor, Professor Dr. Noordin Bin Mohd Yusof for their great assistance, lesson, contribution and valuable guidance through this research where I never forget. To Associate Professor Dr. Izman Bin Sudin, thank for being co-supervisor with giving me opportunity and encouragement in every way I need in this research.

The acknowledgement also addressed to technician in Production Laboratory, Material Science and Metrology Laboratory for their continuous assist and cooperation in the various task. To my fellow friend, researchers and colleagues in Universiti Teknologi Malaysia, thank for your support, advice and make my daily life more colorful.

Deepest gratitude to my parents, family and my beloved wife who give me a real support in any respect together with pray and continuous encouragement for this research. I dedicate this thesis to all of them.

ABSTRACT

Tungsten carbide (WC-Co) is categorized as a high wear resistance and fracture toughness material. But, these tool materials are found to wear rapidly when machining some particular workpieces. Various techniques have been proposed to improve the WC-Co performance. One such technique is by diamond coating WC-Co. This was done due to its outstanding mechanical properties. Poor adhesion of the coating onto the substrate is the main technical barrier. This is due to the presence of cobalt in the cemented carbide which produces graphite layer. Pre-treatment step is always needed because the nucleation of specific coating is greatly affected by the initial surface conditions of the substrate. In this study, nickel was used as an interlayer and the Watt bath electroplating technique was employed to deposit a thin layer with good adhesion strength. Effect of electroplating parameters such as current density, bath temperature and surface roughness of substrate were studied. Coating thickness, hardness and surface morphology were investigated according to an experimental plan. Subsequently, full factorial experimental design followed by analysis of variance (ANOVA) were applied to analyze the statistical data. Performances of the nickel coating on the WC-Co substrate were analyzed and evaluated using Response Surface Methodology (RSM). The electrodeposition parameters have a great influence on Ni coatings properties. One can observe that lower current density produced more compact morphology and fined grain size. Increase in the thickness of Ni coatings occurred by increasing current density. Comparatively, bath temperature did not have a notable effect on coatings thickness but had great influence on surface morphology and adhesion strength of coatings. Further observation on nickel deposition found that, current density ≈ 4 to 5 A/dm^2 , bath temperature $\leq 60^\circ\text{C}$ and surface roughness $\geq 0.5\mu\text{m}$ have apparent effects on excellent quality of Ni coatings on tungsten carbide especially in producing a thin layer of coating.

ABSTRAK

Tungsten Karbida (WC-Co) dikategorikan sebagai bahan yang mempunyai rintangan kehausan dan tahap keliatan yang tinggi. Akan tetapi, bahan ini dikenalpasti akan mengalami kehausan dengan cepat apabila proses pemesinan dijalankan terhadap sesuatu benda kerja. Pelbagai kaedah telah diperkenalkan untuk meningkatkan prestasi dan sifat WC-Co itu sendiri. Salah satu kaedah yang digunakan adalah seperti saduran berlian. Saduran pada substrat yang mempunyai kelekatan yang rendah merupakan halangan teknikal utama disebabkan oleh kehadiran kobalt dalam WC-Co yang menghasilkan lapisan grafit. Rawatan awal sebelum proses saduran sentiasa diperlukan kerana memberikan kesan yang besar terhadap keadaan permukaan substrat. Dalam kajian ini, nikel bertindak sebagai lapisan antara dan teknik elektropenyaduran takungan Watt telah diaplikasikan untuk menghasilkan lapisan nipis dengan kekuatan lekatan yang baik. Kesan parameter elektropenyaduran seperti kepadatan arus, suhu larutan dan kekasaran permukaan substrat telah dikaji. Ketebalan dan kekuatan saduran serta morfologi permukaan dikaji dengan merujuk kepada pelan ujikaji. Kaedah rekabentuk ujikaji berfaktor penuh diikuti dengan analisis varians (*ANOVA*) telah digunakan untuk menilai data statistik yang diperolehi. Prestasi saduran nikel terhadap WC-Co telah dianalisis dan dinilai menggunakan kaedah tindak balas permukaan (*RSM*). Keputusan menunjukkan parameter saduran elektro mempunyai pengaruh yang besar terhadap sifat salutan Ni. Kepadatan arus yang rendah telah menghasilkan morfologi yang lebih padat dan ketebalan pada saduran Ni bertambah dengan peningkatan kepadatan arus. Suhu larutan tidak memberi kesan ketara terhadap ketebalan saduran tetapi memberikan kesan yang besar kepada kekuatan saduran dan juga morfologi permukaan. Didapati juga bahawa kepadatan arus ≈ 4 hingga 5 A/dm^2 , suhu larutan $\leq 60^\circ\text{C}$ dan kekasaran permukaan $\geq 0.5 \mu\text{m}$ memberikan kualiti yang terbaik kepada saduran Ni ke atas WC-Co untuk menghasilkan lapisan nipis.

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LIST OF SYMBOLS

SEM	-	Scanning Electron Microscopy
FESEM	-	Field Emission Scanning Electron Microscopy
ANOVA	-	Analysis of Variance
RSM	-	Response Surface Methodology
CCD	-	Central Composite Design
BWT	-	Blast Wear Tester
R^2	-	Coefficient of determination
Pred. R^2	-	Predicted R – square
Adj. R^2	-	Adjusted R- square
Adeq. precision	-	Adequate precision
CD	-	Current density
T	-	Temperature
SR	-	Surface roughness
Y	-	Response (thickness)
H	-	Response (hardness)

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CHAPTER 1

INTRODUCTION

1.1 Background

The cemented tungsten carbide cutting tools are used widely in the machining, mining and stone cutting industry. However, these tools are found to wear rapidly when machining some particular materials such as abrasive composites or high silicon-filled aluminum. Numerous research studies (Ma *et al.* 2007, Polini *et al.* 2006, Polini and Barletta, 2008) showed that efficient and energy saving cutting tools is required to reduce the tool's down-time, increase cutting productivity and improve the quality of the machined surface. Moreover, diamond coating has been proposed to improve the performance of cemented carbide tools due to its outstanding mechanical properties. Along with great wear resistance, the advantages of the diamond coating include high surface hardness, high thermal conductivity, reduced friction, better corrosion protection, and improved optical properties (Polini, 2006, Xu *et al.* 2007).

Diamond film deposition has thus made it possible to use many diamond properties in new and innovative applications. Surprisingly, simple procedures for producing low-pressure diamond films now means thin films can be obtained on wear components having complex geometries. Polini (2006) reported that the ultimate goal of using CVD diamond coatings is to extend the technical performance of components, such as tools, beyond their conventional wear life. CVD diamond films are highly resistant to chemical and abrasive wear (Kamiya *et al.* 2002). Moreover diamond-coated articles are of little use if these deposits do not remain attached to the underlying substrate during use. Consequently, in the case of CVD diamond coated articles it is mandatory to optimize both adhesion and wear resistance.

The application of diamond coatings to enhance the overall machining effectiveness of WC-Co by reducing the tools downtime; increasing the cutting productivity and improving the quality of the machined surface are becoming more attractive. Polycrystalline diamond (PCD) tools on a WC-Co substrate were generally used for this purpose. Therefore, because of their high cost, many researchers and industrial users are instead using diamond films deposited using CVD techniques which are relatively inexpensive, and could be deposited on tools of any geometry. Compared to an uncoated cemented carbide tool, the CVD diamond coated tool shows much greater abrasive wear resistance which results in up to ten times longer tool life, and less build-up edge and lower cutting forces which yield a better surface finish on the workpiece materials. Besides, one of the largest barriers to be overcome is the poor adhesion of diamond film on the cemented carbides substrate. This is due to (i) the large thermal mismatch of the diamond film with the cemented carbide tool and (ii) weak interface bonding resulting from graphite film formation during low-pressure diamond deposition due to the presence of the cobalt binder. There are many reports, which claimed to reduce the thermal mismatch and to limit the graphite formation by etching cobalt (Sarangi *et al.* 2008, Polinia *et al.* 2002, Sahoo *et al.* 2002) and/or depositing interlayer as diffusion barriers.

Due to excellent wetting ability, tungsten carbide with cobalt has made this metal the first choice as binder. However, high cost and environmental pollution impacts are basically the main issue for cobalt usage. Substitution of cobalt with other metals has always been figured out. Some other metals that have been used as binder are iron, nickel and manganese. In addition to lower cost, nickel has higher corrosion resistance than cobalt.

Among the various manufacturing methods, electroplating has received considerable attention as a feasible, inexpensive and economically viable processing technique for producing thin layer (Rashidi and Ahmadeh, 2010, Ewecharoen *et al.* 2008). Consequently, especially nickel layer, prepared by electrodeposition has high density, minimum porosity, excellent corrosion resistance and good wear resistance and as the grain size of nickel is reduced, the strength and strain hardening rate also increased (Pan *et al.* 2007, Molloy *et al.* 2011).

Performance of nickel electrodeposits is related with their microstructure (such as grain size, surface morphology and crystal orientation), which depended on electroplating conditions (such as, current density, current efficiency and time) and composition of plating bath. Xuetao *et al.* (2008) studied the influence of pulse on-time, off-time, peak current density and saccharin on the grain size, surface morphology, crystal orientation, and microhardness. Furthermore, substrate roughness is one of the electrodeposition parameter that is sometimes difficult to control as well as important in terms of quality and functionality. Vitry *et al.* (2010) indicated that mechanical preparation (such as grinding) may be difficult to implement because of the complicated geometries but also proved that surface roughness can affect the coating appearance. The effect of surface roughness on nickel characteristics was investigated by Bulasara *et al.* (2011) who found that surface roughness did not contribute much to the performance characteristics of plating efficiency and thickness. Boonyongmaneerat *et al.* (2009) noticed that current density is one of the processing parameters that controls the microstructure and hence the apparent hardness of the composite. In additional, micro-hardness and surface morphology of a nickel coating were hardly influenced by getting optimum

setup parameters for electrodeposition. Ranjan *et al.* (2011a) found that micro-hardness of the nano-composite of nickel coating increases with the increase in the stirring rate up to 450 rpm.

1.2 Problem Statement

Poor adhesion of diamond coating is the main technical barrier for commercialization of diamond-coated tools. During high temperature diamond deposition process, the presence of cobalt in cemented carbide suppresses diamond growth. Co leaching from the WC–Co substrate catalyzes the formation of non-diamond carbon (sp^2 bonded), instead of sp^3 bonded diamond. The weak graphite layer at the interface results in poor adhesion between the diamond coating and the substrate.

Removing the Co binder from the substrate using Murakami reagent and acid etching is usually conducted to grow adhered diamond coatings. However, the substrate's mechanical properties and toughness are significantly reduced after etching. Etching WC grains with Murakami solution desirably increases the substrate surface roughness substantially but also causes increase in the surface Co content. It can be realized that substantial surface roughness with low cobalt content is the most desirable surface for deposition of diamond film. A large amount of residual stress will be produced due to mismatch of the coefficient of thermal expansion (CTE). The film could easily delaminate from the substrate right after deposition. Diffusion barriers (interlayer) such as Cu, Cr, Ni, Ti-based layer and CrN have been developed to prevent the Co catalyst effect but the hardness and other interlayer properties still needs to be improved.

It has been observed that mechanical interlocking effect caused by a rough surface can contribute significantly to improve hardness and adhesion of interlayer. Enhancement of surface roughness through interlayer surface is beneficial in that the nickel can well-retained in the cavity and improve hardness and provide well anchored roots for subsequently grown of nickel layer. There is however lacking of experimental information regarding the nickel properties when being deployed as an interlayer. Also, mathematical models for various interlayer responses are also not available. The availability of such model will enable the determination of optimal process condition.

1.3 Objectives of the Research

The objectives of this research are as follows:

1. To evaluate the various nickel coating properties obtained when using different nickel electroplating parameters on tungsten carbide, WC-6%-Co substrate.
2. To develop mathematical models and provide experimental evidences for predicting various nickel coatings properties.
3. To optimize the condition of nickel electroplating parameters in terms of current density, bath temperature and surface roughness of substrate to achieve high quality of nickel coating properties based on the Design of Experiment models.

1.4 Scope of the Research

This study was limited to the following:

1. Tungsten carbide, WC-6% Co with diameter 12mm was used as substrate and electroplating technique was employed for deposition purpose.
2. Electroplating parameters were studied include current density, bath temperature and surface roughness of substrate and for the whole deposition process, pH value of nickel solution was kept constant at 4.
3. Response variables evaluated include coating hardness, thickness and surface morphology.

1.5 Significance of the Research

This study focuses on obtaining on effective nickel interlayer on tungsten carbide substrate using suitable electroplating parameters. The suitability of the interlayer was evaluated in terms of the coating hardness and thickness. Additionally, suitable empirical models were developed using the experimental data for describing the relationship between the various responses and the related factors. With the completion of this research, the viability of using the electroplating process as an alternative process for obtaining the interlayer can be produced. The mathematical models obtained will facilitate the determination of the suitable process setting as well as the prediction of the responses investigated.

REFERENCES

- Ahn, J. G., Kim, D. J., Lee, J. R., Chung, H. S., Kim, C. O., Hai, H. T. (2006). Improving the adhesion of electroless-nickel coating layer on diamond powder. *Surface and Coatings Technology*. 201: 3793-3796.
- Apachitei, I., Duszczek, J., Katgerman, L., Overkamp, P. J. B. (1998). Electroless ni–p composite coatings: The effect of heat treatment on the microhardness of substrate and coating. *Scripta Materialia*. 38(9): 1347-1353.
- Badarulzaman, N. A., Mohamad, A. A., Puwadaria, S. Ahmad, Z. A. (2010). The evaluation of nickel deposit obtained via Watts electrolyte at ambient temperature. *Journal of Coating Technology*. 7(6): 815-820.
- Bahrololoom, M. E., Sani, R. (2005). The influence of pulse plating parameters on the hardness and wear resistance of nickel–alumina composite coatings. *Surface & Coatings Technology*. 192: 154-163.
- Bai, A., Hu, C. C. (2002). Effects of electroplating variables on the composition and morphology of nickel-cobalt deposits plated through means of cyclic voltammetry. *Electrochimica Acta*. 47: 3447-3456.
- Boonyongmaneerat, Y., Saengkiattiyut, K., Saenapitak, S., Sangsuk, S. (2009). Effects of WC addition on structure and hardness of electrodeposited Ni–W. *Surface and Coatings Technology*. 203: 3590-3594.

- Borkar, T., Harimkar, S. P. (2011). Effect of electrodeposition conditions and reinforcement content on microstructure and tribological properties of nickel composite coatings. *Surface and Coatings Technology*. 205: 4124-4134.
- Bouveresse, J. D. R., Pinto, R. C., Schmidtke, L. M., Locquet, N., Rutledge, D. N. (2010). Identification of significant factors by an extension of ANOVA-PCA based on multi-block analysis. *Chemometrics and Intelligent Laboratory Systems*. xxx (2010) xxx-xxx
- Brian N. Chapman, J.C. (1974). Anderson. Science and Technology of Surface Coating.
- Bulasara, V. K., Shekar, O. C., Uppaluri, R. (2011). Effect of surface roughness and mass transfer enhancement on the performance characteristics of nickel-hypophosphite electroless plating baths formetal-ceramic composite membrane fabrication. *Chemical Engineering Research and Design*. xxx xxx-xxx
- Burzyńska, L., Rudnik, E., Koza, J., Błaż, L., Szymański, W. (2008). Electrodeposition and heat treatment of nickel/silicon carbide composites. *Surface & Coatings Technology*. 202: 2545-2556.
- Cao, J. L., Li, L. T., Wang, Y. L., Zhao, J. Q., Gui, Z. L. (2002). Mechanism of resistance degradation of lead magnesium Niobate-based ferroelectrics induced by hydrogen reduction during electroplating, *Journal of Materials Science*. 37: 3225-3228.
- Chou, C. C., Lee, J. W., Chen, Y. I. (2008). Tribological and mechanical properties of HFCVD diamond-coated WC-Co substrates with different Cr interlayers. *Surface & Coatings Technology*. 203: 704-708.
- Donev, A. N. (2004). Design of experiments in the presence of errors in factor levels. *Journal of Statistical Planning and Inference*. 126: 569-585.

- Ewecharoen, Thiravetyan, P. Nakbanpote, W. (2008). Comparison of nickel adsorption from electroplating rinse water by coir pith and modified coir pith. *Chemical Engineering Journal*. 137: 181-188.
- George Di Bari. (1994) ASM Handbook, Volume 5, Surface Engineering, *Nickel Plating*. page 201.
- Godon, A., Creus, J., Feaugas, X., Conforto, E., Pichon, L., Armand, C., Savall, C. (2011). Characterization of electrodeposited nickel coatings from sulphamate electrolyte without additive. *Materials Characterization*. 62: 164-173.
- Gu, C. D., You, Y. H., Yu, Y. L., Qu, S. X., Tu, J. P. (2011). Microstructure, nanoindentation, and electrochemical properties of the nanocrystalline nickel film electrodeposited from choline chloride–ethylene glycol. *Surface & Coatings Technology*. 205: 4928-4933.
- Gunasegaram, D. R., Farnsworth, D. J., Nguyen, T. T. (2009). Identification of critical factors affecting shrinkage porosity in permanent mold casting using numerical simulations based on design of experiments. *Journal of Materials Processing Technology*. 209: 1209-1219.
- Hamid, Z. A., Hassan, H. B., Attyia, A. M. (2010). Influence of deposition temperature and heat treatment on the performance of electroless Ni–B films. *Surface and Coatings Technology*. 205: 2348-2354.
- Haseeb, A. S. M. A., Albers, U., Badea. K. (2008). Friction and wear characteristics of electrodeposited nanocrystalline nickel–tungsten alloy films. *Wear*. 264: 106-112.
- Hua, Z., Liu, Y., Yao, G., Wang, L., Ma, J., Liang, L. (). Preparation and Characterization of Nickel-Coated Carbon Fibers by Electroplating. *Journal of Materials Engineering and Performance*.

- Jeong, D. H., Gonzalez, F., Palumbo, G., Aust, K. T., Erb, U. (2001). Effect of grain size on the wear properties of electrodeposited nanocrystalline nickel coatings. *Scripta Materials*. 44: 493-499.
- Kamiya, S., Takahashia, H., D'Antonio, P., Polinia, R., Travers, E. (2002). Quantitative comparison of adhesive toughness for various diamond films on co-cemented tungsten carbide. *Diamond and Related Materials*. 11: 716-720.
- Kang, J. X., Zhao, W. Z., Zhang, G. F. (2009). Influence of electrodeposition parameters on the deposition rate and microhardness of nanocrystalline Ni coatings. *Surface and Coatings Technology*. 203: 1815-1818.
- Khun, N. W., Liu, E., Yang, G. C. (2010). Structure, scratch resistance and corrosion performance of nickel doped diamond-like carbon thin films. *Surface and Coatings Technology*. 204: 3125-3130.
- Kim, M. S., Kim, J. Y., Kim, C. K., Kim, N. K. (2005). Study on the effect of temperature and pressure on nickel-electroplating characteristics in supercritical CO². *Chemosphere*. 58: 459-465.
- Kwon, D. H., Kang, M. C., Kim, J. S., Ok, J. T., Kim, K. H. (2005). A comparative study on cutting performance of TiN-coated tungsten carbide cutting tool with a cobalt interlayer. *Surface & Coatings Technology*. 200: 1933-1938.
- Lamovec, J., Jović, V., Randjelović, D., Aleksić, R., Radojević V. (2008). Analysis of the composite and film hardness of electrodeposited nickel coatings on different substrates. *Thin Solid Films*. 516: 8646-8654.
- Lasheras, F. S., Vilán, J. A V., Nieto, P. J. G., Díaz, J. J. C. (2010). The use of design of experiments to improve a neural network model in order to predict the thickness of the chromium layer in a hard chromium plating process. *Mathematical and Computer Modelling*. 52: 1169-1176.

- Lawrence J. Durney. *Electroplating Engineering Handbook*, 4th edition, *Van Nostrand, Reinhold Company*. (1984), p364.
- Li, C. L., Lin, Y. T., Lee, J. W. (2009). Mechanical properties evaluation of chromized tungsten carbide-cobalt hardmetals. *Surface & Coatings Technology*. 204: 1106-1111.
- Li, J. F., Liao, H. L., Ding, C. X., Coddet, C. (2005). Optimizing the plasma spray process parameters of yttria stabilized zirconia coatings using a uniform design of experiments. *Journal of Materials Processing Technology*. 160: 34-42.
- Li, Y., Jiang, H., Pang, L., Wang, B., Liang, X. (2007). Novel application of nanocrystalline nickel electrodeposit: Making good diamond tools easily, efficiently and economically. *Surface and Coatings Technology*. 201: 5925-5930.
- Lim, M., Ye, M., Panoskaltsis, N., Drakakis, E. M., Yue, X., Cass, A. E. G., Radomska, A., Mantalaris, A. (2007). Intelligent bioprocessing for haemotopoietic cell cultures using monitoring and design of experiments. *Biotechnology Advances*. 25: 353–368.
- Ma, Y. P., Sun, F. H., Xue, H. G., Zhang, Z. M., Chen, M. (2007). Deposition and characterization of nanocrystalline diamond films on Co-cemented tungsten carbide inserts, *Diamond & Related Materials*. 16: 481-485.
- Miao, J., Song, J., Xue, Y., Tong, Y., Tang, W., Lu, F. (2004). Effect of a two-step pretreatment method on adhesion of CVD diamond coatings on cemented carbide substrates. *Surface & Coatings Technology*. 187: 33- 36.
- Mirkova, L., Maurin, G., Monev, M., Tsvetkova, C. (2003). Hydrogen co evolution and permeation in nickel electroplating. *Journal of Applied Electrochemistry*. 33: 93–100.

- Molloy, D. A., Malinov, S., McNally, T., Hill, P. (2011). Thermal study of selectively plated nickel sulfamate coatings. *Organic Coatings*. 70: 330-335.
- Montgomery, D. C. (2009a). *Statistical Quality Control*. (6th.Ed). New York: Wiley and Son.
- Montgomery, D. C. (2009b). *Design and Analysis of Experiment*. (7th. Ed). New York:Wiley and Son
- Moti, E., Shariat, M. H., Bahrololoom, M. E. (2008). Electrodeposition of nanocrystalline nickel by using rotating cylindrical electrodes. *Materials Chemistry and Physics*. 111: 469-474.
- Muthukrishnan, N., Davim, J. P. (2009). Optimization of machining parameters of Al/SiC-MMC with ANOVA and ANN analysis. *Journal of Materials Processing Technology*. 209: 225-232.
- Myers, R. H., Montgomery, D. C. (2002). *Response Surface Methodology: Process and Product Optimization Using Designed Experiments*. (2nd ed). New York: John Wiley & Sons.
- Ning, Z., He, Y., Gaob, W. (2008). Mechanical attrition enhanced Ni electroplating. *Surface and Coatings Technology*. 202: 2139-2146.
- Njau, K. N., Hosseini, Y. M., Janssen, L. J. J. (1998). Electrochemical reduction of nickel ions from dilute artificial solutions in a GBC reactor. *Journal of Applied Electrochemistry*. 28: 689-696.
- Noordin, M. Y. (2003). *Performance evaluation of coated carbide and coated cemented tools when turning hardened tool steel*. Doctor Philosophy, Universiti Teknologi Malaysia, Skudai.
- Noordin, M. Y., Venkatesh, V. C., Sharif, S., Elting, S., Abdullah. (2004). Application of response surface methodology in describing the performance

of coated carbide tools when turning AISI 1045 steel. *Journal of Materials Processing Technology*. 145, 46-58

Nouraei, S., Roy, S. (2009). Design of experiments in electrochemical microfabrication. *Electrochimica Acta*. 54: 2444-2449.

Oraon, B., Majumdar, G., Ghosh, B. (2006). Application of response surface method for predicting electroless nickel plating. *Materials and Design*. 27: 1035-1045.

Oviedo, T. A. B., Batyrshin, I., Domínguez, J. M. (2009). The optimal design of experiments (ODOE) as an alternative method for catalysts libraries optimization. *Catalysis Today*. 148: 28–35.

Pan, Y., Zhou, Y. C., Zhou, Z. F., Huang, Y. L., Liao, Y. G., Sun, C. Q. (2007). Fabrication, lattice strain, corrosion resistance and mechanical strength of nanocrystalline nickel films. *Transactions of Nonferrous Metals Society of China*. 17: 1225-1229.

Parivesh. (2008). Waste Minimisation and Ecofriendly Electroplating Process.

Polini, R. (2006). Chemically vapour deposited diamond coatings on cemented tungsten carbides: Substrate pretreatments, adhesion and cutting performance. *Thin Solid Films*. 515: 4-13.

Polini, R. Barletta, M. (2008). On the use of CrN/Cr and CrN interlayers in hot filament chemical vapour deposition (HF-CVD) of diamond films onto WC-Co substrates. *Diamond and Related Materials*. 17: 325-335.

Polini, R., Delogu, M., Marcheselli, G. (2006). Adherent diamond coatings on cemented tungsten carbide substrates with new Fe/Ni/Co binder phase. *Thin Solid Films*. 494: 133-140.

- Polinia, R., Bravia, F., Casadeib, F., D'Antonio, P., Traversaa, E. (2002). Effect of substrate grain size and surface treatments on the cutting properties of diamond coated Co-cemented tungsten carbide tools. *Diamond and Related Materials*. 11: 726-730.
- Ranjan, S., Siddhartha, D., Karabi, D. (2011a). Effect of stirring rate on the microstructure and microhardness of Ni-CeO₂ nanocomposite coating and investigation of the corrosion property. *Surface & Coatings Technology*. 205: 3847-3855.
- Ranjan, S., Siddhartha, D., Karabi, D. (2011b). The effect of bath temperature on the crystallite size and microstructure of Ni-CeO₂ nanocomposite coating. *Materials characterization*. 62: 257-262.
- Rashidi, A. M. (2011). Isothermal oxidation kinetics of nanocrystalline and coarse grained nickel: Experimental results and theoretical approach. *Surface and Coatings Technology*. 205: 4117-4123.
- Rashidi, A. M., Amadeh, A. (2008). The effect of current density on the grain size of electrodeposited nanocrystalline nickel coatings. *Surface and Coatings Technology*. 202: 3772-3776.
- Rashidi, A. M., Amadeh, A. (2009a). The effect of saccharin addition and bath temperature on the grain size of nanocrystalline nickel coatings. *Surface & Coatings Technology*. 204: 353-358.
- Rashidi, A. M., Amadeh, A. (2010). Effect of Electroplating Parameters on Microstructure of Nanocrystalline Nickel Coatings. *Journal of Material Science and Technology*. 26(1): 82-86.
- Rashidi, A. M., Eivani, A. R., Amadeh, A. (2009b). Application of artificial neural networks to predict the grain size of nano-crystalline nickel coatings. *Computational Materials Science*. 45: 499-504.

- Rasmussen F. E., Ravnkilde J. T., Tang P. T., Hansen O., Bouwstra S. (2001). Electroplating and characterization of cobalt-nickel-iron and nickel-iron for magnetic microsystems application. *Sensor and Actuators. A* 92: 242-248.
- Sahoo, B., Chattopadhyay, A. K. (2002). On effectiveness of various surface treatments on adhesion of HF-CVD diamond coating to tungsten carbide inserts. *Diamond and Related Materials*. 11: 1660-1669.
- Sarangi, S. K., Chattopadhyay, A., Chattopadhyay, A. K. (2008). Effect of pretreatment methods and chamber pressure on morphology, quality and adhesion of HFCVD diamond coating on cemented carbide inserts. *Applied Surface Science*. 254: 3721-3733.
- Sarangi, S. K., Chattopadhyay, A., Chattopadhyay, A. K. (2008). Effect of pretreatment, seeding and interlayer on nucleation and growth of HFCVD diamond films on cemented carbide tools. *International Journal of Refractory Metals & Hard Materials*. 26: 220-231.
- Sein, H., Ahmed, W., Jackson, M., Polini, R., Hassan, I., Amar, M., Rego, C. (2004). Enhancing nucleation density and adhesion of polycrystalline diamond films deposited by HFCVD using surface treatments on Co cemented tungsten carbide. *Diamond and Related Materials*. 13: 610-615.
- Sun, F. H., Zhang, Z. M., Chen, M., Shen, H. S. (2003). Improvement of adhesive strength and surface roughness of diamond films on Co-cemented tungsten carbide tools. *Diamond and Related Materials*. 12: 711-718.
- Sunwang, N., Wangyao, P., Boonyongmaneerat, Y. (2011). The effects of heat treatments on hardness and wear resistance in Ni–W alloy coatings. *Surface & Coatings Technology*. xxxxxx–xxx
- Tang, W., Wang, Q., Wang, S., Lu, F. (2001). Adherent diamond coatings on cemented carbide substrates with different cobalt contents. *Diamond and Related Materials*. 10: 1700-1704.

- Tang, W., Wang, S., Lu, F. (2000). Preparation and performance of diamond coatings on cemented carbide inserts with cobalt boride interlayers. *Diamond and Related Materials*. 9: 1744-1748.
- Tian, Z. J., Wang, D. S., Wang, G. F., Shen, L. D., Liu, Z. D., Huang, Y. H. (2010). Microstructure and properties of nanocrystalline nickel coatings prepared by pulse jet electrodeposition. *Transactions of Nonferrous Metals Society of China*. 20: 1037-1042.
- Todoroki, A., Ishikawa, T. (2004). Design of experiments for stacking sequence optimizations with genetic algorithm using response surface approximation. *Composite Structures*. 64: 349-357.
- Tsay, P., Hu, C. C., Wang, C. K. (2005). Compositional effects on the physical properties of iron–nickel deposits prepared by means of pulse-reverse electroplating. *Materials Chemistry and Physics*. 89: 275-282.
- Tsuru, Y., Nomura, M., Foulkes, F. R. (2002). Effects of boric acid on hydrogen evolution and internal stress in films deposited from a nickel sulfamate bath. *Journal of Applied Electrochemistry*. 32: 629–634.
- Vitry, V., Kanta, A. F., Delaunois, F. (2010). Initiation and formation of electroless nickel–boron coatings on mild steel: Effect of substrate roughness. *Materials Science and Engineering*. B 175: 266-273.
- Vitry, V., Kanta, A. F., Delaunois, F. (2011). Mechanical and wear characterization of electroless nickel-boron coatings. *Surface & Coatings Technology*. 206: 1879-1885.
- Wahdame, B., Candusso, D., Ois, X. F., Harel, F., Kauffmann, J. M., Coquery, G. (2009). Design of experiment techniques for fuel cell characterization and development. *International journal of hydrogen energy*. 34: 967-980

- Wang, L., Gao, Y., Liu, H., Xue, Q., Xu, T. (2005). Effects of bivalent Co ion on the co-deposition of nickel and nano-diamond particles. *Surface and Coatings Technology*. 191: 1-6.
- Wu, B., Xu, B. S., Zhang, B., Dong, S. Y. (2007). The effects of parameters on the mechanical properties of Ni-based coatings prepared by automatic brush plating technology. *Surface and Coatings Technology*. 201: 5758-5765.
- Xu, Z., Lev, L., Lukitsch, M., Kumar, A. (2007). Effects of surface pretreatments on the deposition of adherent diamond coatings on cemented tungsten carbide substrates. *Diamond and Related Materials*. 16: 461-466.
- Xuetao, Y., Yu, W., Dongbai, S., Hongying, Y. (2008). Influence of pulse parameters on the microstructure and microhardness of nickel electrodeposits. *Surface and Coatings Technology*. 202: 1895-1903.
- Yao, Z., Stiglich, J. J., Sudarshan, T. S. (2003). Nano-grained Tungsten Carbide-Cobalt (WC/Co). *Material Modification*.
- Zhou, Y. B., Zhang, H. J. (2011). Effect of annealing treatment on cyclic-oxidation of electrodeposited Ni-Al nanocomposite. *Transactions of Nonferrous Metals Society of China*. 21: 322-329.