Gage Capability Study of Thin Film Coating Thickness Measurement Method Using Scanning Electron Microscopes (SEM)

Thesis submitted in accordance with the requirements of the Universiti Teknikal Malaysia Melaka for the Degree of Bachelor of Engineering (Honors) Manufacturing (Process)

By

Saifol Azman Bin Nordin

Faculty of Manufacturing Engineering
April 2007
BORANG PENGESAHAN STATUS TESIS

JUDUL: GAUGE CAPABILITY STUDY OF THIN FILM COATING THICKNESS MEASUREMENT METHOD USING SCANNING ELECTRON MICROSCOPES (SEM)

SESJI PENGAJIAN: 2/2006-2007

Saya ____________________________

SULIT (Mengandungi maklumat yang berdarjah keselamatan atau kepentingan Malaysia yang termaktub di dalam AKTA RAHSIA RASMI 1972)

TERHAD (Mengandungi maklumat TERHAD yang telah ditentukan oleh organisasi/badan di mana penyelidikan dijalankan)

☐ TIDAK TERHAD

(TANDATANGAN PENULIS) ________________________________

Disahkan oleh: ________________________________

(TANDATANGAN PENYELIA) ________________________________

Alamat tetap:
No. 33 Kg. Pmtg Durian, 09400 Padang Serai, Kedah Darul Aman

Tarih: 15/5/07

Tarih: 15/5/07

* Tesis dimaksudkan sebagai tesis bagi ijazah Doktor Falsafah dan Sarjana secara penyelidikan, atau disertasi bagi pengajian secara kerja kursus dan penyelidikan, atau Laporan Projek Sarjana Muda (PSM).
** Jika tesis ini SULIT atau TERHAD, sila lampirkan surat daripada pihak berkuasa/organisasi berkewenangan dengan menyatakan sebab dan tempoh tesis ini perlu dikelaskan sebagai SULIT atau TERHAD.
APPROVAL

This thesis submitted to the senate of UTeM and has been accepted as partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering (Manufacturing Process). The members of the supervisory committee are as follow:

Supervisor
(Official Stamp & Date)

PROF. DR. MOHD. RAZALI BIN MUHAMAD
Dekan
Fakulti Kejuruteraan Pembuatan
Universiti Teknikal Malaysia Melaka
Karung Bekunci 1200, Ayer Keroh
75450 Melaka
DECLARATION

I hereby, declare this thesis entitled “Gage Capability Study of Thin Film Coating Thickness Measurement Method Using Scanning Electron Microscopes (SEM)” is the results of my own research except as cited in the reference.

Signature : 
Author’s Name : SAIFOL AZMAN BIN NORDIN
Date : 15 MEI 2007
ABSTRACT

Gage capability study is the procedure to determine error of measuring system represented by repeatability, reproducibility and precision to tolerance ratio (P/T). Thin film coatings on cutting tools are to improve tool properties with respect to its high hardness, low coefficient of friction, thermal stability and excellent wear resistance. These properties are influenced by the coating thickness. Due to this it is important to be able to measure coating thickness accurately. This study investigates the suitability of measuring thin film coating thickness using SEM system by conducting gage capability study. The measured artifact in this study was cutting insert with WC substrate and TiN coating. The main objectives of this study are to ascertain the suitability of Scanning Electron Microscope (SEM) thickness measurement method for thin film coating and to identify and minimize the variation of measuring tool. From the experimental data, repeatability & reproducibility and P/T ratio was calculated. Based on those data, this study found out that with proper sample preparation and measuring method SEM is a viable measuring tool to determine thin film coating thickness.
DEDICATION

Especially dedicated to my beloved family especially my Mother (Azizah Binti Saad) and my Father (Nordin Bin Daud); whose very concern, understanding, supporting and patient. Thanks for everything. To my project supervisor (Prof. Dr Mohd Razali Muhamad and Mr. Mohd Nizam Abd Rahman) and to all my Friends, I also would like to say thanks. The Work and Success will never be achieved without all of you.
ACKNOWLEDGMENTS

In the name of Allah, the most gracious, the most merciful. Alhamdulillah and grateful with the permission from Allah I have done my Projek Sarjana Muda activities. Firstly, special thanks dedicated to Universiti Teknikal Malaysia Melaka for giving the cooperation while doing this study and especially to all staff in Faculty of Manufacturing Engineering. Also a lot of thank to my family for their support because without their mental and physical supported it won’t be easy for me to complete this study. Special thanks to the important person, my supervisor whose guides me in this study Prof. Dr. Mohd Razali Muhamad and Mr. Mohd Nizam Abd.Rahman. This person that gave me a lot of advices and guidelines to make sure this study can be performed smoothly without any problem and produce the best result.

Also I would like to wish my greatest grateful especially to my family and my close friends for all their support. Lastly to all my friends and all lecturers especially my second project assessor Dr. Bagas Wardono, thank you for the support that all of you shown to me. May Allah bless all of you.
# TABLE OF CONTENTS

Abstract.........................................................................................................................i  
Dedication......................................................................................................................ii 
Acknowledgement.........................................................................................................iii  
Table of Contents..........................................................................................................iv  
List of Figures..............................................................................................................vii  
List of Tables.................................................................................................................ix  
List of abbreviation, symbols, specialized nomenclature .............................................x  
List of Appendices ........................................................................................................xi

## 1. INTRODUCTION

1.1 Background of research.........................................................................................1  
1.2 Objectives of research..........................................................................................3  
1.3 Scope project.........................................................................................................3  
1.4 Significant of study..............................................................................................3

## 2. LITERATURE REVIEW

2.1 Gauge capability study.........................................................................................7  
  2.1.1 Gauge repeatability and reproducibility (GRR)...............................................7  
  2.1.1.1 Sample calculation....................................................................................8  
2.2 Thin film coating thickness measurement method............................................10  
  2.2.1 Thickness measurement method.................................................................11  
  2.2.2 Supporting information that SEM can be used to measure thickness.........14  
  2.2.3 SEM techniques............................................................................................15  
  2.2.4 Summary.......................................................................................................17
3. METHODOLOGY

3.1 Flow chart and explanations
3.2 Gauge capability method details
3.3 Thickness measurement method
   3.3.1 Scanning Electron Microscopes
   3.3.2 SEM operating parameter
   3.3.3 Operation of SEM
3.4 Sample preparation procedure
3.5 Sample preparation technique
3.6 Measurement technique
3.7 Data analysis

4. RESULTS AND ANALYSIS

4.1 Experimental 1
   4.1.1 Data experimental 1 for Repeatability
   4.1.2 Data experimental 1 for Reproducibility
4.2 Experimental 2
   4.2.1 Data experimental 2 for Repeatability
   4.2.2 Data experimental 2 for Reproducibility
4.3 Experimental 3
   4.3.1 Data experimental 3 for Repeatability
   4.3.2 Data experimental 3 for Reproducibility

5. DISCUSSIONS

5.1 Repeatability
5.2 Reproducibility
5.3 Precision to tolerance ratio (P/T ratio)
# LIST OF FIGURES

1.1 Illustration of coating on substrate .................................................. 2

2.1 Probability density for the thickness of 2 gauge ............................... 5
2.2 Repeatability of a measuring system compared to the process spread ... 10
2.3 Schematic of the thermal electric method for coating thickness .......... 13
2.4 Microstructures of cutting tool surfaces ........................................... 16

3.1 Proposed research flow ...................................................................... 18
3.2 ZEISS EVO x Series SEM .................................................................. 22
3.3 Nomenclature of SEM ....................................................................... 23
3.4 A specimen stub and the stage ............................................................ 28
3.5 SEM setup .......................................................................................... 29
3.6 Measurement technique 1 ................................................................... 34
3.7 Measurement technique 2 ................................................................... 37

4.1 Should SEM image of thin film coating ............................................... 41
4.2 Graph range of coating thickness for experiment 1 repeatability ......... 44
4.3 Graph range of coating thickness for experiment 1 reproducibility .... 48
4.4 Graph range of coating thickness for experiment 2 repeatability ....... 52
4.5 Graph range of coating thickness for experiment 2 reproducibility .... 56
4.6 Graph range of coating thickness for experiment 3 repeatability ....... 60
4.7 Graph range of coating thickness for experiment 3 reproducibility .... 64
4.8 Results summary ............................................................................... 65

5.1 Comparison between range value for experiment 1, 2 and 3 .......... 67
5.2 Comparison a coating thickness images of a WC substrate with TiN 68
Coating between sample preparation experiment 1 and 2
5.3 Comparison of range value between experiment 1, 2 and 3 70
# LIST OF TABLES

<table>
<thead>
<tr>
<th></th>
<th>Table Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.1</td>
<td>SEM specification</td>
<td>24</td>
</tr>
<tr>
<td>3.2</td>
<td>SEM parameters</td>
<td>24</td>
</tr>
<tr>
<td>3.3</td>
<td>Specimen chamber pressure</td>
<td>27</td>
</tr>
<tr>
<td>3.4</td>
<td>Sample preparation technique 1</td>
<td>30</td>
</tr>
<tr>
<td>3.5</td>
<td>Sample preparation technique 2</td>
<td>32</td>
</tr>
<tr>
<td>3.6</td>
<td>Data form</td>
<td>36</td>
</tr>
<tr>
<td>3.7</td>
<td>Reproducibility computation</td>
<td>37</td>
</tr>
<tr>
<td>4.1</td>
<td>Data experimental 1 for repeatability</td>
<td>42</td>
</tr>
<tr>
<td>4.2</td>
<td>Data experimental 1 for reproducibility</td>
<td>45</td>
</tr>
<tr>
<td>4.3</td>
<td>Data experimental 2 for repeatability</td>
<td>50</td>
</tr>
<tr>
<td>4.4</td>
<td>Data experimental 2 for reproducibility</td>
<td>53</td>
</tr>
<tr>
<td>4.5</td>
<td>Data experimental 3 for repeatability</td>
<td>58</td>
</tr>
<tr>
<td>4.6</td>
<td>Data experimental 3 for reproducibility</td>
<td>61</td>
</tr>
</tbody>
</table>

5.1 Summarized the results of the experiment and to be referred in this discussion chapter frequently. 66
LIST OF ABBREVIATIONS, SYMBOLS, SPECIALIZED NOMENCLATURE

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>ANOVA</td>
<td>Analysis of Variance Method</td>
</tr>
<tr>
<td>BMP</td>
<td>Bitmap</td>
</tr>
<tr>
<td>CRT</td>
<td>Cathode Ray Tube</td>
</tr>
<tr>
<td>DOF</td>
<td>Depth of Field</td>
</tr>
<tr>
<td>GRR</td>
<td>Gauge Repeatability and Reproducibility</td>
</tr>
<tr>
<td>ISO</td>
<td>International Organization of Standardization</td>
</tr>
<tr>
<td>LSL</td>
<td>Lower Specification Limit</td>
</tr>
<tr>
<td>NIST</td>
<td>National Standard Institute</td>
</tr>
<tr>
<td>PVD</td>
<td>Physical Vapor Deposition</td>
</tr>
<tr>
<td>P/T</td>
<td>Precision and Tolerance</td>
</tr>
<tr>
<td>TiN</td>
<td>Titanium Nitride</td>
</tr>
<tr>
<td>TIFF</td>
<td>Tagged Image</td>
</tr>
<tr>
<td>USL</td>
<td>Upper Specification Limit</td>
</tr>
<tr>
<td>VP</td>
<td>Variable Pressure</td>
</tr>
<tr>
<td>WC</td>
<td>Tungsten Carbide</td>
</tr>
</tbody>
</table>
LIST OF APPENDICES

Appendices A  Value of $d_2$

xi
CHAPTER 1
INTRODUCTION

1.1 Background of research

Cutting tools are used in metal working for roughing, drilling, semi-finishing and finishing applications. Cutting tools are used to cut and shape metals and other materials. Additionally, wood boring tolls like auger and lathes also called cutting tools.

The aims of the coating on cutting tools are to improve tool properties with respect to its high hardness, low coefficient of friction, thermal stability and excellent wear resistance (K. Laing, 1999). All this improvement in properties decrease the diffusion coefficient and the solubility coefficient of cutting tools compared to the uncoated cutting tools (H.O. Gekonke & S.V. Subramanian, 2002).

Coating on cutting tools is invisible by common observation. It can be seen by using the optical microscope. The figure 1.1 represented the illustration of the coating on cutting tool, where the main material is called substrate and material to be coating is called Titanium Nitride (TiN).
Generally a coating thickness range of 2-15μm (80-600μin), are applied on cutting tools and inserts by the physical vapor deposition and chemical vapor deposition (Serope Kalpakjian & Steven R. Schmid, 2000). Thickness is very important to measure because its influence a coating performance. Different thickness also can give more effect in machining process and cause conductivity approached a certain value with further raise of the thickness.

Due to the importance of coating thickness in defining coating performance, capability in measuring it needs to be defined. Methods described in literatures, were done in general term, no detail step by step measuring methods were offered. Due to this, this study investigates the coating thickness measuring methods capability to ensure the selected method is capable to give meaningful data. To do this gauge capability study is conducted on the selected coating thickness measuring method.
1.2 **Objective of research**

The main objective of this study is:

- To ascertain the suitability of Scanning Electron Microscope (SEM) thickness measurement method for thin film coating.
- To identify and minimize the variation of measuring tool.

1.3 **Scope of research**

The scope of this gage capability study is limited to:

- **Equipment** - Scanning Electron Microscopes (SEM)
- **Artifact** - Cutting Tool (WC substrate with TiN Coating)

1.4 **Significant of Study**

1. Establish base line information of the equipment repeatability and reproducibility data for reference for future usage of the equipment.

2. Provide the variability data of the thickness measurement method using the equipment. This data sample size can be used to determine of future work.

3. The same gauge capability study methodology knowledge can be applied for any measurement system in the future.
CHAPTER 2
LITERATURE REVIEW

In this section, the gauge capability study methods and the thickness measurement methods are discussed in detail. At the end of this chapter, the selected gauge capability method and measurement methods are identified.

2.1 Gauge capability study

One important step before utilizing any measurement system is gauge capability study or gauge repeatability and reproducibility (GRR). GRR is a statistical tool that measures the amount of variation in the measurement system.
A measurement system is defined as the collection of operations, procedures, gauges and other equipment, software, materials, facilities and personnel used to assign a number to the characteristic being measured (Praveen Gupta, 1984). The language and techniques of GRR are oriented more to the manufacturing environment. However, measurement system analysis is a critical part of any six sigma project, regardless of the environment (e.g., transactional, service, etc.). The philosophy behind this kind of study is applicable to all project types.

Depending on the type of data, the statistical analysis will be different. For a continuous measurement, there are a variety of statistical properties that can be determined: stability, bias, precision (which can be broken down into repeatability and reproducibility), linearity, and discrimination. For a discrete measurement, estimates of the error rates can be determined for within appraiser, each appraiser versus standard, between appraisers and all appraisers versus standard (Besterfield, D.H., 2004).

Precision is the measure of the measurement system variability, and is defined as the standard deviation due to the measurement system. A traditional way to determine precision is to take a sample of representative parts from the process and to have two or
three people measure the parts two or three times. Usually, this is known as blind studies where the people aren’t aware that they are part of the measurement system analysis. Precision can be divided into repeatability and reproducibility. Repeatability is the variability of the gauge itself. Reproducibility is the variability associated with using different operators under different conditions. Variability from part-to-part is due to the process, while repeatability and reproducibility are due to the measurement system (McCarty, T. Daniels, L. Bremer& M.Gupta, 2005). The goal is to have the repeatability and reproducibility to be small.

To judge if the variability due to the measurement system is small enough, two metric are commonly used. They are %R&R and %P/T. %R&R stands for percent repeatability and reproducibility. The formula is (McCarty, T. Daniels, L. Bremer& M.Gupta, 2005).

\[
\%R&R = \frac{6\sigma_{\text{measurement system}} \times 100}{6\sigma_{\text{Total}}} \tag{5}
\]

Where the numerator contains an estimate (the U indicates an estimate) of the variability solely due to the measurement system and the denominator contains an estimate of the total variability. %P/T stands for percent precision to tolerance. The formula is:

\[
\%P/T = \frac{6\sigma_{\text{measurement system}} \times 100}{\text{USL-LSL}} \tag{4}
\]

Gauge capability study is an important aspect of controlling variation in measurement systems conforming too many quality system standards such as ISO 9000 and ISO/TS 16949 requirements.
2.1.1 Gauge Repeatability and Reproducibility (GRR)

Commonly, there are 2 methods of GRR namely short and long method. Generally, repeatability is the variation obtained from one gauge and one operator while measuring the same part in several times. Whereas reproducibility is the same measurement but made by different operators using the same gage when measuring at the same part (Hank Scutoski & Chander Sekar, 1996).

I. Gage Repeatability and Reproducibility (GRR) - Short Method

Gage Repeatability and Reproducibility (GRR) is the combined estimate of measurement system repeatability and reproducibility. The Short Term GRR does not include the effect of time in analysis.

Short method is a quick method where it can determine the acceptability of gauge variations. It is usually conducted with two operators and five parts. The two operators measure each part once, randomly. When each part is only measured once, gauge repeatability cannot be isolated from gage repeatability. The result of this study is a combination of both types of variation.

II. Gage Repeatability and Reproducibility - Long Method

Gage Repeatability and Reproducibility (GRR) is the combined estimate of measurement system repeatability and reproducibility. The Long Term GRR includes the effect of time in the analysis.

Long method study, there might be conducted with varying numbers of parts and operators. Each operator measures the same set of parts in several times. The parts of the sample should be measured in a random order each time. When each part is measured more than once by the same operator, repeatability can be isolated from reproducibility.
As the result, if reproducibility variation is large compared to repeatability, this means that the operators are getting consistently different results and if repeatability is large compared to reproducibility, the gage should be fixture more solidly or might be need maintenance.

2.1.1.1 Sample calculation

In the paper presented here, there are 3 basic method that are usually used in order to determine gage R&R which are range method, range and average method and analysis of variance method (ANOVA). Generally, the method that already used for calculate a repeatability and reproducibility is the range and average method. Although ANOVA produce good and accurate method, this study didn’t choose it because of the complex calculation involved.

The repeatability and reproducibility using the range and average method must quantify such multiple part, appraiser and trial because this 3 thing will require when using this method. From the published that are refer (Engineered Software Inc 1999), there recommended that this method is to use 10 parts, 3 appraiser and 2 trial so that the total measurement is 60. Below this is shown the example of calculation formula in repeatability system:

\[
\text{Repeatability} = \frac{5.15 \bar{R}}{d_2} \tag{1}
\]

Where, \( \bar{R} \) is the average of the ranges for all appraisers and parts. \\
\( d_2 \) is found in Appendix A with \( Z = \) the number of part times the number of appraisers and \( W \) is found as the number of trials.
For reproducibility,

\[
\text{Reproducibility} = \left( \frac{5.15 \bar{X}_{\text{range}}}{d_2} \right)^2 - \frac{\text{Repeatability}^2}{n r}
\]

(2)

Where

\(\bar{X}_{\text{range}}\) is the average of the difference in the average measurement between the appraiser with the highest average measurement, and the appraiser with the lowest average measurement for all appraisers and parts.

\(d_2\) is found in Appendix A with \(Z = 1\) and \(W\) is found as the number of appraisers.

\(n\) is the number of part

\(r\) is the number of trials

The measurement system repeatability and reproducibility:

\[
R & R = \sqrt{\text{Repeatability}^2 + \text{Reproducibility}^2}
\]

(3)

To define \(\%P/T\) stands for percent precision to tolerance. The formula is:

\[
\%P/T = 6\sigma_{\text{measurement system}} \times 100 \quad \text{USL - LSL}
\]

(4)