# INVESTIGATION OF SURFACE MODIFICATION ON ALUMINUM OXIDE USING ACID ETCHING SURFACE PRETREATMENT

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## ABSTRACT

The current work focus on surface modification of aluminum oxide using acid etching surface pre-treatment. The main objective of this thesis is to investigate the effect of surface pre-treatment using acid etching on aluminum oxide ($Al_2O_3$) cutting tool. The effect of machining performance on alumina cutting tool using this pre-treatment in term of wear rate also studied. The scope of work include alumina as cutting tool, acid hydrofluoric for etching, PVD coating process and machining by turning operation. Alumina cutting tool was subjected to acid etching surface pre-treatment for 20 minutes before it was deposited with PVD coating process. Next, the cutting tool was test by turning machine in order to determine the wear resistant and other material characterization also was performed such as microstructure study, hardness Vickers test and surface roughness test. The current work result show shows a regular rough surface with no voids and crystals emerging from the as-received surface while a non-uniform surface topography with peak and valley after pre-treatment. The surface pre-treatment with HF acid provide rough surface to alumina microstructure. Besides, the machining test performance shows the coated alumina with acid etching surface pre-treatment provided longer tool's life compared to as- received alumina and coated alumina without pre-treatment cutting tools. In addition, the micro hardness test indicated that average Vickers micro hardness of alumina as-received specimen was 1202.47 HV while coated alumina with acid etching pre-treatment specimen was 2293.43 HV. In surface roughness test, the surface roughness of alumina increase after subjected to pre-treatment but decrease after endergoes coating process. From the result, the acid etching surface pre-treatment and coating process affect the mechanical properties of alumina cutting tool such as microstructure, wear resistance, hardness and surface roughness.

# ABSTRAK

Kerja ini membentangkan kajian pengubahsuaian pada permukaan aluminium oksida dengan menggunakan asid sebagai pra-rawatan pada pemukaan. Objektif utama tesis ini adalah untuk mengkaji kesan pra-rawatan menggunakan asid pada aluminium oksida ($Al_2O_3$). Kesan prestasi alat pemotong alumina dengan menggunakan pra-rawatan ini diuji dengan mesin untuk melihat sejauh mana kadar jangka hayatnya dapat bertahan. Skop kerja termasuklah alumina sebagai alat pemoting, asid hydruoflouric untu pra-rawatan, proses salutan PVD dan proses pemesinan. Alat pemotong alumina direndam dalam pra-rawatan asid selama 20 minit. dan proses salutan PVD dilakukan. Alat pemotong alumina diuji dengan mesin untuk melihat kadar jangka hayatnya serta ujian lain seperti mikrostruktur, kekerasan dan kekasaran permukaan. Keputusan menunjukkan struktur biasa pada alumina asal manakala struktur yang kasar dengan puncak dan lembah selepas pra-rawatan dilakukan. Asid HF menghasilkan struktur yang kasar ke atas alumina. Selain itu, ujian dengan mesin menunjukkan alumina yang melalui pra-rawatan dan salutan mempunyai kadar jangka masa hayat yang tinggi berbanding alumina asal dan alumina tanpa pra-rawatan. Tambahan pula ujian kekerasan Vickers bagi alumina asal ialah 1202.47 HV mankala kekerasan alumina yang melalui pra-rawatan dan salutan ialah 2293.43 HV. Di dalam ujian kekasaran pula menunjukkan kekasaran permukaan bertambah selepas pra-rawatan asid tetapi bekurang selepas menjalani proses salutan. Daripada keputusan ini, ia menunjukkan bahawa pra-rawatan asid mempengaruhi alumina dari segi mikrostruktur, kadar jangka hayat, kekuatan dan kekasaran permukaan.

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| | |
|---|---|
| $V$ | Cutting speed |
| $Tn$ | Tool life |
| $C$ | Constant |
| $V_B$ | Flank wear |
| $A$ | Area |
| $D$ | Diameter |
| $F$ | Force |
| $\alpha$ | Alpha Vickers indenter |
| $p$ | Pressure load |

# LIST OF ABBREVIATIONS

| | |
|---|---|
| SEM | Scanning electron microscope |
| PVD | Physical vapor deposition |
| CVD | Chemical vapor deposition |
| HF | Hydrofluoric acid |
| HV | Hardness Vickers |
| TRS | Tranverse rupture strength |
| IP | Ion plating |
| PCD | Polycrystalline diamond |
| DLC | Diamond like carbon |
| ASTM | American society for testing and materials |
| ISO | International standardization organization |

# CHAPTER 1

# INTRODUCTION

## 1.1 INTRODUCTION

Nowadays, cutting tools industry was going through a big transformation development. Cutting tools are widely used in turning, shaping, grinding, polishing, drilling and other engineering applications in different industrial settings (Park, 1999). Cutting process was defined as removing material by one cutting edge. Cutting tools condition must be sharped with high hardness and high wear resistant to provided a long tool life. In order to improve hardness and wear resistant of the cutting tool, some modification has been taken for example by adding new coatings or new alloys for design changes. This step is vital to reduce the thermal heat and friction effect during cutting process in order to increase cutting tools efficiency and enhanced tool life.

According to Suzuki (1983), “Ceramics have attracted considerable attention since 1950’s because of their excellent properties such as high hardness, high resistance to chemical corrosion and good mechanical properties at high temperatures”. The most common ceramic materials are based on aluminum or also known as aluminum oxide ($Al_2O_3$). It was used almost exclusively on turning tool bits. It has several advantages such as high hardness, high thermal resistant and high chemical resistant (Gruss, 1994). In addition, it also has disadvantage such as low toughness that makes the cutting tool more brittle. Nevertheless, this problem can be solved by providing new coating with surface pr-treatment.

Campestrini (2001) stated that, “The pre-treatment can affect the composition of the alloyed surface and hence, increase the development of the conversion coatings”.

From his findings, the pre-treatment provided the best surface for adhesion and it is used to provide high surface roughness topography that plays an important role in biological interactions on tissue interface. The surface changes were suitable for coating adhesive.

While Elsentriecy (2007) stated that, “Chemical or electrochemical pre-treatment methods, such as acid activation are capable to removed the oxide layer”. Therefore, it is believed that acid etching solution roughened the etched surface. The pre-treatment methods used to remove native oxide layer that does not protect well against corrosion. It forms easily due to the high reactivity of Magnesium. Acid etching pre-treatments has several advantages such as provided highly adhering layers and deposited complex inorganic coatings.

Physical Vapor Deposition process (PVD) is the major process used to produce cutting tool coatings. According to Wertheim (1998), “The stress characteristics of the PVD coating, in combination with the usually small layer thickness from 2 to 5 pm, provided good cutting edge strength, fracture toughness and bending strength”. From his findings, coating with PVD can contribute to the uniform coating thickness. This part is important to provide sharp cutting edge on cutting tools.

## 1.2 PROBLEM STATEMENT

Ceramic cutting tool which is aluminum have attracted considerable attention since 1950’s because of their excellent properties such as high hardness, high resistance to chemical corrosion and good mechanical properties at high temperature. However, their low strength, toughness and low thermal shock resistance limit their application. This problem weakens the cutting tool for example of tool wear. Traditionally, Cutting tool improvement has been seen as a solution. In order to solve this problem, the cutting tools need to be modified using surface pre treatment and coating process. One type of the surface pre-treatment is acid etching. Acid etching is use to remove the oxide layer on cutting tool to increase surface adhesion. Uncoated cutting tools show significant wear resistance capability. Thus, their wear resistance could be improved by coating process. The application of coating technology into cutting tool can give a lot of improvement in their structure by increase high hardness, increase wear resistant, increase tool's life and provided low coefficients of friction. Therefore, the current work focused on improving the wear resistance of cutting tool which is aluminum in machining with titanium by turning operation. The main objective of this study is to increase the aluminum cutting tool's life.

## 1.3 OBJECTIVES

These are the objectives of the current work:

(i) To investigate the effect of surface pre-treatment using acid etching (hydrofluoric acid) on aluminum oxide ($Al_2O_3$) cutting tool.

(ii) To determine the effect of machining performance on aluminum oxide ($Al_2O_3$) cutting tool using different cutting speed.

(iii) To evaluate the effect of wear resistance on Titanium using coated and uncoated aluminum oxide.

## 1.4 SCOPES

The project consists of the following tasks:

(i) Aluminum oxide ($Al_2O_3$) will be used as cutting tools.

(ii) Acid Hydrofluoric acid (HF) was used to etch cutting tools surface material.

(iii) Coated the cutting tools with carbon by PVD technique.

(iv) Machining cutting tool to titanium work piece by using turning operation.

(v) Evaluate the wear rate resistant of the cutting tool, hardness and surface roughness.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 INTRODUCTION

Tool wear and breakage has been an issue with cutting tool since they were created. Tool wear decreases tool's life and increase the forced used in cutting causes a lack of consistency in material removal. There are many factors contributing to the wear of cutting tools which are cutting tool properties, workpiece properties, cutting speed, cutting feed, depth of cut and machine rigidity. Traditionally, the cutting tool's properties improvements have been seen as a solution rather than a problem in metal cutting. The example of improvement in cutting tool's life was mechanical or chemical pretreatment and additional of new coating layer. During the cutting process, the cutting tool also exposed to the change of shape due to tool surface, workpiece, the presence of particles and high temperature on contact surface. The change of shape can impair the tribological properties and increase the risk associated with tribological processes on machine tool elements. Therefore, this chapter will provide an overview of cutting tool and discussed the wear, pre-treatment, coating as well as machining process.

## 2.2 CUTTING TOOL

Nowadays, cutting tools industry is going through a transformation of material which is from hard to super hard material. Cutting tools are widely used in turning, shaping, grinding, polishing, drilling and other engineering applications in industrial manufacturing (Park, 1999). Cutting may be accomplished by single-point or multi-point tools. It can be define as removal of material by means of one cutting edge. Cutting tools must be sharped, with the correct level of hardness, accurate specifications and deliver a longer tool life. In order to improve the cutting tool's life, some research had been done for example new coatings and new material mixed together along with design changes. This step is important to reduce the heat and friction effect in order to produce efficient cutting tools with enhanced tool's life. A good cutting tools need to have the following properties:

(i) Strength at elevated temperatures
(ii) High toughness
(iii) High wear resistance
(iv) High hardness
(v) Shock resistant
(vi) Low coefficient of friction

## 2.3 CUTTING TOOL MATERIAL

Cutting tools must be made from a material which is harder than the workpiece, and the tool must be able to withstand the heat generated in the metal-cutting process. There are two types of cutting tool material which described as unstable group and stable group. The unstable group characterize as low hardness point (<HRC 70) and low heat resistant (500°C). The cutting action produces a lot of heats during machining that made the substances are inherently unstable. The stable group cutting tool material described as high hardness (>HRC 70) and high thermal resistant (1600°C).

### 2.3.1 ALUMINUM OXIDE

According to Suzuki (1983), "Ceramics have attracted considerable attention since 1950's because of their excellent properties such as high hardness, high resistance to chemical corrosion and good mechanical properties at high temperatures". Nowadays, the applications of Ti (CN) cermets tools have been extended from finish, semi-finish to rough machining, and from turning to milling machining. However, the high contents of bonding phases decrease their hardness and resistance to wear. Alumina was ceramic materials that have been developed. It was most significant used in the production of aluminum metal as abrasive because of its hardness (1600 HV) and as a refractory material by its high melting point (2072°C).

The most common ceramic materials were based on alumina. It was used almost exclusively on turning tool bits. Alumina was used as a coating material on aluminum by using anodizing or plasma electrolytic oxidation. It is widely used as a coarse or fine abrasive, including as a much less expensive substitute for industrial diamond. Cutting tools of pure alumina are usually used to process parts of grey cast iron in automobile industry. This type of cutting tool was used in processing cast iron of abrasion resistance, hardened steel and high strength steel and other difficult to machine materials (Jinbao, 2006). In addition, the alumina has many advantages to be used in high cutting speed. The following advantages were:

(i) Temperature resistant - Its higher melting point (2072°C) make it widely used in cutting tools.

(ii) High hardness - The strength and abrasive characteristics result to high hardness which value in (1400- 2000 HV).

(iii) Wear resistance and Chemical Inertness - This characteristic made alumina based ceramic cutting tools provide less ability to react with other elements or compounds.

### 2.3.2 TUNGSTEN CARBIDE

Tungsten carbide is an extremely hard material made from tungsten powder. The chemical compound is mixed between tungsten and carbon atom. Carbide tools are usually used in the form of brazed or clamped tips. High cutting speeds may be used and materials like high speed steel to machine with carbide tipped tool. Carbide tools are very abrasive’s resistant and also can withstand higher temperatures (2870°C) compared to than standard high speed steel tools (500°C). Carbide cutting surfaces are often used for machining through materials such as carbon steel or stainless steel. It maintains a sharp cutting edge better than other tools, they generally produce a better finish on parts, and their temperature resistance allows faster machining.

Based on Krupp (1927), "It was found that tungsten in carbide cuts metal more efficiently than tungsten in high-speed steel" .The cemented carbides were used for metal cutting in manufacturing industry. Selection of carbide was done due to its toughness and exceptional resistance to abrasion, catering, thermal deformation and its double torsion strength than that of steel. Today, carbide has found a number of industrial applications such as turning, milling and drilling. Carbide cutting tool are classified into three categories which are, wear grades used in machines and tool guides, impact grades used in places where high shock resistance is required and cutting tool grades used for cutting hard objects. Besides, the following are the advantages of tungsten carbide cutting tool:

(i) Strength - Carbide is a combination of carbon and tungsten atoms. The Vickers hardness is 1700 HV to 2400 HV.
(ii) Toughness – The toughness are determined by ratio of tungsten carbide to cobalt. The higher percentage of cobalt produces higher toughness.
(iii) Cost effective - Carbide maintain a sharp cutting edge that allows faster machining. The short time taken can save production cost.

## 2.4 TOOL WEAR

Tool wear is defined as the change of shape of the tool from its original shape, during cutting, resulting from the gradual loss of tool material. During machining, cutting tools removed material from the component to achieve the required shape, dimension and surface roughness. However, wear occurred during the cutting action, and it will ultimately result in the failure of the cutting tool. When the tool wear reaches at a certain extent, the tool or active edge has to be replaced to guarantee the desired cutting action. The Figure 2.1 shows the factor that can cause wear.

König (1984) stated that, "Ever since the famous Taylor started with his tool life experiments all the basic mechanisms of the tool wear process such as the wear types which can be observed on the tool". Tool wear described the gradual failure of cutting tools due to machining. In turning, catastrophic tool failure need to be avoided since it can damage the cutting tool and thus interrupt the machining process. Instead, the tool's life can be defined in terms of flank wear that occurs on the tool clearance face. The flank wear is often used as the end of effective tool life. This is physically because of the flank wear land width which when certain level is reached, it affect the dimensional accuracy and surface finish of the component as well as the stability of the machining process. One or more of the following wear modes may occur:

(i) Flank
(ii) Notch
(iii) Crater
(iv) Edge cracking
(v) Catastrophic failure

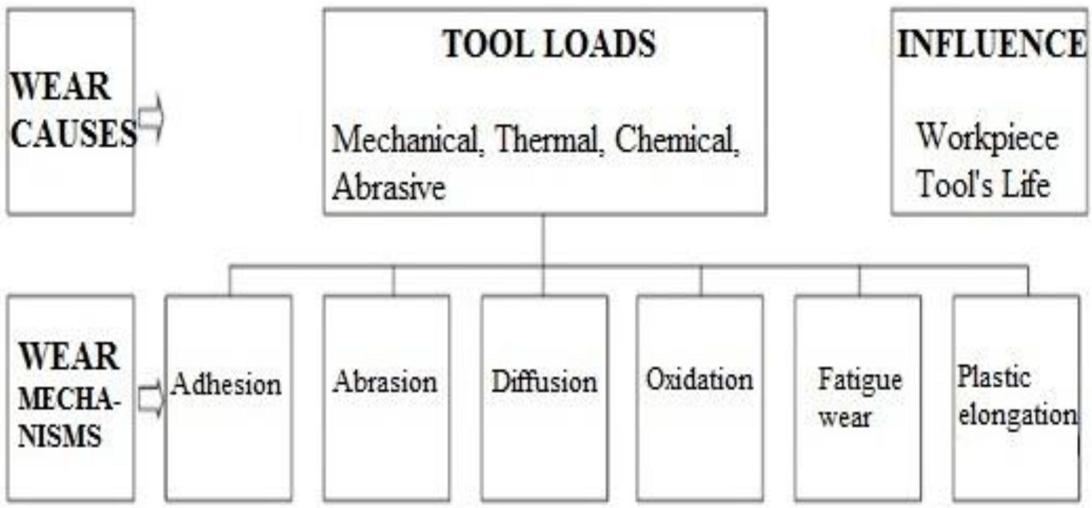


**Figure 2.1:** Factor of wear

Source: Grzesik (2001)

The cutting tools in conventional machining, particularly in continuous chip formation processes like turning, generally failed by gradual wear, abrasion, adhesion, diffusion, chemical erosion and galvanic action. It depends on the tool work materials and machining condition. Figure 2.2 show that machining parameter such as cutting speed, feed rate and depth of cut affect the tool's wear. Tool wear initially started with a relatively faster rate due to what is called a break-in wear caused by attrition and micro chipping at the sharp cutting edges. Cutting tools often failed prematurely, randomly and catastrophically by mechanical breakage and plastic deformation under adverse machining conditions caused by intensive pressure or temperature and dynamic loading at the tool tips. It is particularly if the tool material lacks strength, hot-hardness and fracture toughness. However, in the previous research show that the cutting tool, workpiece and the machining conditions result the tool failure mode which is gradual wear. The effects of tool wear on technological performance are:

(i) Increase the cutting force
(ii) Increase the surface roughness
(iii) Decrease the dimensional accuracy
(iv) Increase the temperature of surface contact
(v) Vibration
(vi) Lower the production efficiency, component quality
(vii) Increase the cost

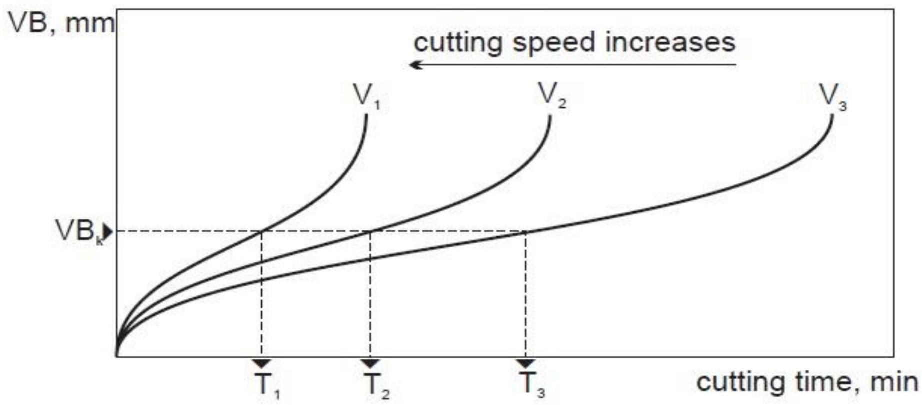


**Figure 2.2:** Effect cutting speed to tool's wear

Source: Marinov (2008)

## 2.5 TOOL LIFE

Tool life is an important characteristic that need to be considered upon tool's selection since it can affects the manufacturing costs. A short tool life will not only require additional tools to be purchased, but will also require time to change the tool each times it worn out. However, in general, the relationship between the tool's life and cutting speed is:

$$VT_n = C \qquad (2.1)$$

where; V= cutting speed in m/min, T= tool life in minutes, C= constant. For high-speed steel tools the value of C ranges from 0.14 m to 0.1 m and for ceramic tools the value would be 0.6 m.

Tool wear is a time dependent process. As cutting proceeds, the amount of tool wear increases gradually. The tool wear must not be exceeded a certain limit in order to avoid tool failure. The flank wear curve show a similar relationship occurs for other wear types. The Figure 2.3 shows general relationship of $V_B$ versus cutting time where T is tool life and $V_{Bk}$ is wear criterion.

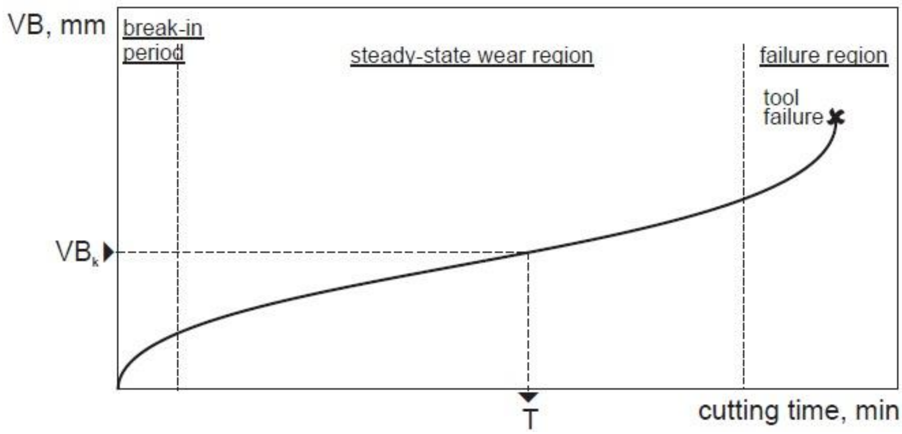


**Figure 2.3:** Tool's life curve

Source: Marinov (2008)

## 2.6 SURFACE PRE-TREATMENT

Campestrini (2001) stated that, "The pre-treatment affect the composition of the subjected surface and enhanced the development of the conversion coatings".

According to his work, the pre-treatment provided the rough topography surface which for adhesion. Surface pre-treatment used to obtain high surface roughness, because the surface topography plays an important role between coating layer and substrate interface. The surface topography was examined using scanning electron microscope (SEM) on several occasions during etching.

### 2.6.1 Acid Etching

Elsentriecy (2007) stated that, “Chemical or electrochemical pre-treatment methods, such as acid activation are capable of removing this oxide layer”. The oxide layer acted as a diffusion barrier that provides poor adhesion. The pre-treatment methods originated in the need to remove the native oxide layer that does not protect against corrosion but can easily forms due to the high reactivity of magnesium. Acid etching pre-treatments included etching the surface to form highly adhering oxides, or deposited complex inorganic coatings. The pre-treatment provides the best surface for adhesion.

### 2.6.2 Hydrofluoric Acid (HF)

According to Berishev (1995), “Studies in acid etching of materials other than silicon using HF have also reported the effect of HF concentration on the surface morphology of the etched substrate”. Acid HF is declared as a weak acid because of its lower dissociation constant compared to the strong acids. Even it was classified as weak acid, it still highly corrosive acid and capable of dissolving many materials such as glass. HF was a solution of hydrogen fluoride in water. One of the HF ability was to dissolve metal oxides is the basis of several applications. In the current work, HF had been selected as a surface pre-treatment in order to remove the oxide layer on cutting tool.

Monk and Soane (1993) stated that, “The most widespread method of HF based etching is the wet etching in a mixture of HF acid and water”. HF behavior benefits in removing the sacrificial layer in micro-machining. Apart from that, HF can etch glass through reaction with silicon dioxide and form gaseous or water-soluble silicon fluorides. In this research, HF can act as a cleaning agent of the cutting tool. Acid HF ionizes in aqueous solution in a similar fashion to other common acids but it does not fully ionize in dilute aqueous solutions. The chemical reaction is shown in Equation 2.2 and 2.3

$$HF + H_2O \leftrightarrows H_3O^+ + F^- \quad (2.2)$$

$$HF \leftrightarrows H^+ + FHF^- \quad (2.3)$$

## 2.7 COATING

An effective systematic approach was essential for the successful implementation and sophistication of modem cutting processes. One of the approaches was by coated improvement. Coating defines as a layer covering that being applied to the surface of an object or the substrate. Coated cutting tool give many advantages especially in machining such as improving the mechanical properties of the cutting tools.

Quinto, *et al.* (1999) stated that, “The use of Al-containing coating materials is reported to be advantageous, especially for machining operations which combine high cutting temperatures with high mechanical stresses on the tool material”. This can be explained by two effects. Firstly, the formation of a thin alumina layer on tool faces which come into contact with oxygen protects the coating from tribo-oxidation. This was important for interrupted cutting operations as well as for reducing notch wear at the minor cutting edge of coated carbide tools. Secondly, the good wear resistance of coatings was their comparatively high hardness at elevated temperatures. Figure 2.4 shows the substrate with the coating layer and the overview of coating thickness structure.

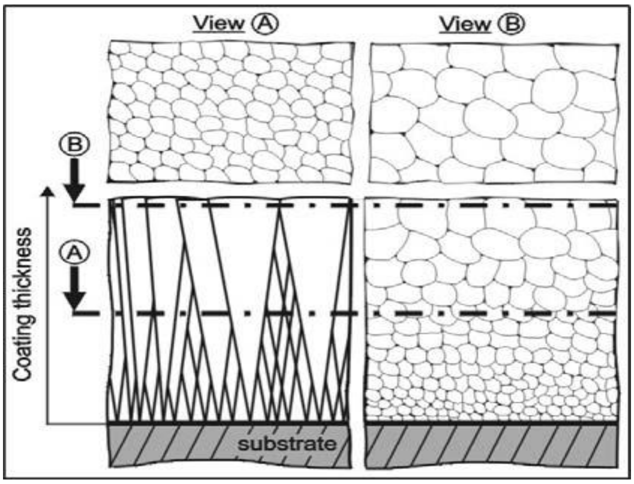


**Figure 2.4:** Coating grain sizes view a) lower thickness, b) higher thickness

Source: Bouzakis (2003)

According to Klocke *et al*. (1998), "A study including different modern tool coatings has revealed that coating materials which contain Al provide the best wear resistance to virtually all wear mechanisms, irrespective of the coating process". This was due to their comparatively great hardness, abrasion resistance, oxidation resistance and to thermal relief of the substrate in the case of volume effects like diffusion and fatigue. On the other hand, tool coatings used to vary contact conditions by altering friction, heat generation or heat flow. These are indirect means of influencing wear by decreasing wear attack. The structure of the tool coating determines both its wear resistance and the tribological conditions in the contact zones. It was essential to adapt the coating structure to the demands of a specific machining task. The main influences on the structure are:

(i) The choice of coating material
(ii) Layer growth during the coating process and
(iii) The structural design of the single layers to form a multi-layer.

Furthermore, coated cutting tool also have many advantages in machining which had been listed as follows:

(i) Improve wear resistance
(ii) Increase tool life
(iii) Improve high hardness
(iv) Improve coefficients of friction
(v) Improve result of adhesion
(vi) Improve wet ability
(vii) Improve corrosion resistance
(viii) Improve scratch resistance

## 2.8 COATING TECHNIQUE

The morphology of a coating depends mainly on the coating process applied. The relevant processes for tool's coating may be differentiated into CVD and PVD processes. CVD and PVD processes further classified with its effects on coating structures and on the tribological properties of the coated tools. The process selected depends on the tool's material composition and also on the purpose of the tool's application. The coated process used to increase mechanical properties of cutting tools such as high speed steel, cemented carbide, cermets, ceramic, and a super hard material. Other than that, many cutting tool's suppliers also combining CVD and PVD processes in an attempt to gain the advantages of each (Sokovic, 2006).

### 2.8.1 Physical Vapor Deposition Process (PVD)

This major process used to produce cutting tool's coatings. It was emerged in the 1980's as a viable process for applying hard coatings to cemented carbide tools. In PVD processes, the coating material are evaporated and subsequently condensed on the cutting tool. The coating is deposited in a vacuum chamber. Further components of the coating material can be added by using reactive gas. Nitrogen or ammonia gas was deposited onto the substrate in the chamber. The evaporation process is a main feature of a specific PVD process.