SURFACE ROUGHNESS AND WETTABILITY INVESTIGATION OF VARIED PLASMA PARAMETER EFFECT ON PVD DEPOSITED ALUMINIUM

by

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A thesis submitted in fulfillment of the requirements for the degree of Master of Science (Microelectronic Engineering)

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2013

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Academic Session	:	2011 – 2013

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ACKNOWLEDGEMENTS

Postgraduate lifestyle has taught me a very important lesson of my life. This has many important persons that need to be thanked for being such a support and guidance throughout this important part of my life. First and foremost I would like to thank my mentors throughout the journey which will be my supervisor, Major Assoc Prof Dr Zaliman Sauli and my co-supervisor, Captain Dr. Vithyacharan Retnasamy. This journey will not been started or completed without both of them.

Besides that I would like to thank my dearest family members for their support in my decision to pursue my studies. They would be my respected father Mr. Palianysamy Nallasami, my lovely mother Mrs Sarojini Divi Muthusamy and my dear sister Miss Kalaashangery. Another important companion who was with me throughout my studies was Miss Hema Rani where she guided me with full support.

A good guidance is a must for a success which was given to me by few good mentors which are Mr Steven Taniselass, Mr Ruhaizi, Mr Wan Mokhdzani, Mr Hafiz and Mrs Hafizah. This guidance was well supported by the university administration led by the ever young Vice Chancellor, R&D Dean, Mrs Rozila and Mrs Suhana, Mrs Intan ,Mrs Zehan, Mr Muzamir and other staffs.

This important part of my life would not be completed if not for my close friends who were my pillars of strength. They are the SHARKZ, specially my fellow master mate Mr. Rajendaran Vairavan and others who are Mr Ong Tee Say, Mrs Shakirina, Miss They Yee Chin, Mr. Aaron, Mr Khor Kang Nan, Mr Lim Wei Jer and others. I would like to thank all the others who were my strength throughout this whole process.

TABLE OF CONTENTS

CHAPTER 3: RESEARCH METHODOLOGY

CHAPTER 4: RESULTS AND DISCUSSION

CHAPTER 5: CONCLUSION AND FUTURE WORK

LIST OF TABLES

LIST OF FIGURES

FIGURE **PAGE**

LIST OF ABBREVIATIONS

RIE	=	Reactive Ion Etching
O_2	=	Oxygen
Al	=	Aluminum
CF_4	=	Carbon Tetrafluoride
DOE	=	Design of Experiment
FFED	=	Full Factorial Experiment Design
PVD	=	Physical Vapor Deposition
Al_2O_3	=	Aluminium Oxide
AFM	=	Atomic Force Microscopy
Ar	=	Argon
DIW	=	De-ionized Water
XPS	=	X-ray Photoelectron Spectroscopy
FTIR	=	Fourier Transform Infra-Red Spectroscopy
SEM	=	Scanning Electron Microscopy
FTS	=	Facing Target Sputtering
CGDS	=	Cold Gas Dynamic Spray
UHV STM	=	Ultra High Vacuum Scanning Tunneling Microscope
EDS	=	Energy Dispersive Spectroscopy
CVD	=	Chemical Vapor Deposition
TEM	=	Transmission Electron microscopy
LSM	=	Laser Scanning Microscopy
HPT	=	High Pressure Torsion

Penyiasatan Kekasaran Permukaan Dan Kebolehbasahan Mengunakan Parameter Plasma Pada Permukaan Pad Ikatan Aluminium Yang Didepositkan Melalui Kaedah Pemendapan Wap Fizikal

ABSTRAK

Metrologi permukaan telah menjadi bidang penyelidikan fokus dan disiasat dengan teliti sejak kemunculan fabrikasi peranti. Peralihan teknologi semasa dalam MEMS dan nano telah sekali lagi menjadikan metrologi permukaan sebagai bidang tumpuan. Lekatan lapisan dan ikatan mempunyai kepentingan metrologi permukaan tersendiri. Dalam kajian ini, permukaan pad ikatan aluminium dicirikan menggunakan ujian titisan air dan analisis AFM. Rekabentuk Eksperimen Faktorial Penuh telah digunakan untuk menjalankan kajian eksperimen ini. Aluminium telah didepositkan menggunakan teknik Pemendapan Wap Fizikal (PVD). Ujian titisan air telah dilakukan dengan menggunakan ruang ujian yang disediakan. Permukaan dirawat menggunakan parameter plasma, di mana parameter plasma dan kekasaran dikaitkan. Parameter plasma dimanipulasikan dengan dua pendekatan yang berbeza. Eksperimen pertama telah dijalankan mengunakan gabungan parameter aliran gas oksigen, aliran gas argon, kuasa ICP dan kuasa BIAS. Eksperimen kedua terdiri daripada parameter aliran gas Karbon Tetrafluorida(CF_4), aliran gas Argon, kuasa ICP dan kuasa BIAS. Keputusan pencirian permukaan dianalisis dengan menggunakan analisis statistik yang terdiri daripada analisis kesan utama dan analisis interaksi antara faktor. Ini akan memberikan maklumat berkenaan kesan setiap faktor dan gabungan faktor-faktor yang memberi kesan kepada hasil eksperimen. Korelasi parameter plasma untuk kekasaran permukaan dan ujian titisan air akan membolehkan kita memahami manipulasi permukaan aluminium untuk ikatan dan tujuan melekat lain. Keputusan akhir menunjukkan bahawa eksperimen yang mengunakan aliran gas Karbon Tetrafluorida(CF_4), aliran gas Argon, kuasa ICP dan kuasa BIAS menghasilkan permukaan dengan ciri-ciri hidrofilik yang lebih tinggi terbukti dengan sudut sentuh yang telah diukur di mana purata sudut sentuh adalah 56°. Sudut sentuh ini dihasilkan oleh kekasaran permukaan yang mempunyai nilai dari 2.46nm hingga 19.53nm. Manakala, eksperimen yang terdiri daripada aliran gas oksigen, aliran gasargon, kuasa ICP dan kuasa BIAS menghasilkan permukaan hidrofilik yang lebih rendah yang menghasilkan sudut sentuh dengan purata 70°. Sudut sentuh ini dihasilkan oleh kekasaran permukaan yang mempunyai nilai dari 4.09nm hingga 20.09nm.

Surface Roughness And Wettability Investigation Of Varied Plasma Parameter Effect On PVD Deposited Aluminium

ABSTRACT

Surface metrology has been a focused and thoroughly investigated research niche since the advent of device fabrication. The current technology shift into MEMS and nano has again made surface metrology a focused area. Layer adhesion and bonding have surface metrology importance here. In this work, surface of aluminium bond pad is characterized using water droplet test and Atomic Force Microscopy(AFM) analysis. Full Factorial Experiment Design was used to carry out this experimental study. Aluminium was deposited using Physical Vapor Deposition(PVD) technique. Water droplet test was done using a prepared test chamber. The surface was treated using plasma parameter, whereby the plasma parameter and roughness were correlated. Plasma parameter was manipulated in two different experiment approaches. First experiment was using a combination of oxygen gas flow, argon gas flow, ICP power and Bias power. Second experiment consist of Carbon Tetrafluoride(CF_4) gas flow, Argon gas flow, ICP power and BIAS power. Results of surface characterization were analyzed using statistical analysis comprising main effect analysis and interaction between factor analyses. This provides the effect of each factor and combination of factors which affects the outcome of the experiment. This correlation of plasma parameter to surface roughness and water droplet test will enable us to understand aluminium surface manipulation for bonding and other adhesion purposes. Final results shows that the experiment comprising of Carbon Tetrafluoride(CF_4)gas flow, Argon gas flow, ICP power and BIAS power produces surface with a higher hydrophilicity characteristics proven by the measured contact angle which averages at 56°. The surface roughness value range from 2.46nm to 19.53nm Whereas, experiment comprising oxygen gas flow, argon gas flow, ICP power and BIAS power produces lower hydrophilic character surfaces which has a higher average contact angle which is 70°.This contact angles are produced by surface roughness ranging from 4.09nm to 20.09nm.

CHAPTER 1

INTRODUCTION

1.1 Overview of Surface Science

Foundation of surface science, which is regarded as one of the famous interdisciplinary field in modern science and technology has been evolving since the past five decades and has now steadied itself at an atomic and molecular scale. With regards to that, a large number of techniques have been technically developed to review the different characters of surfaces in terms of its physics and chemistry. However, only a few have found extensive application in basic science and applied surface analysis. The choice of technique depends upon the type of characterization to be made. Among the most widely used methods are X-ray Photoelectron Spectroscopy (XPS) and Fourier Transform Infra-Red Spectroscopy (FTIR) which are used to study the surface chemical composition. Similarly, Scanning Electron Microscopy(SEM), Stylus profilometer and Atomic Force Microscopy (AFM) are used to investigate the surface morphology of material in atomic scale.

The significance of surface science is widely recognized in various fields. The application and contribution has a very extensive range from agriculture, food and technology till engineering industry. The importance of surface science can be classified according to a few major fields. In the area of agronomy and food technology, the usefulness of most herbicides and pesticides in agricultural sprays are decided depending on the wettability of leaves and fruits. An agricultural spray which contains wetting agent highly increases the quality of the sprays and offers better crops production. This also happens in the food products such as margarine, puddings, salad dressings, whipped cream and other related food products. The chemistry of the surface is also important to determine the quality of food such as cakes and pastries.

In the energy field, the surfactant solutions and microemulsions are important in improving oil recovery from petroleum reservoirs. Another interesting application is in the area of combustion efficiency of various oils. Recently, it has been shown that if one injects a fine dispersion or emulsion of water and oil in furnaces, the efficiency of conversion of oil into heat is improved considerably. Although the exact mechanism is not established, the fact still remains that emulsification of oil and water improves the combustion efficiency.

Principles and techniques of surface science also find many applications in environmental problems. The dewatering of phosphate slimes, sludge formation, coagulation, and flocculation in many waste-water treatment plants rely on the surface interactions. The surface reactions and adsorption on activated carbon are very effective methods for removal of trace contaminants. Fibrous coalesces also are used for the removal of oil droplets from a few parts per million concentrations in the effluent streams of many industries. Here the attachment of oil drops to the fiber and their subsequent coalescence play an important role in the separation of oil. The use of surface films as oil herder for the contraction of oil-spills has been discussed. The presence of films at the air or water interface also causes wave damping of small ripples. This observation has been used in developing the instrumentation for remote sensing of oil spills. In all these systems and processes, the principles of physics and chemistry of surfaces and surface-active agents are involved.

In industries and engineering the contribution of surface science is enormous. For example, the production of magnetic tapes in which a dispersion of magnetic oxide is coated on polyester tapes. The stability of the dispersion and the strength of adhesion depend on the surface interactions. Other applications of surface science are found in the manufacture of inks, paints, pigments, nonstick cooking wares and so forth. The textile industry also utilizes considerable quantities of surface-active substances in the form of wetting agents, emulsions, dyesolubilization and other processes. The contact angle and wettability also enters into water-proofing of textiles, roofing material and similar systems. Many lubricants also involve the use of hydrocarbon oils and various surface-active agents as additives. The physics and chemistry of thin films are used extensively in the electronics industry. As discussed previously, the production of petroleum and petrochemicals also utilizes many processes which are in the general domain of surface science.

The field of catalysis is based on surface interactions between the substrate molecules and the catalyst surface. The formulations of soaps and detergents for household uses also are based on surface properties of surfactants. In the world about 109 tons of minerals are processed every year by the use of flotation technology which again relies on the adsorption of surfactant on mineral particles. Many office stationeries such as no carbon required papers (NCR) use microencapsulation powders to coat these papers. It is considered as an important application contributed by surface and colloid science to industrial processes.

Surface science also plays a major role in biology and medicine field. Many principles and techniques of surface science are relevant to the understanding of the properties and functions of biological membranes. It has been suggested that the spontaneous formation of membranes played an important role in the origin of prebiological cells during the chemical evolution which was followed by the biological evolution. These techniques are being used to elucidate the mechanism of action of many drugs, anesthetic agents, and pharmacological agents on membrane properties. It has been established during recent years that conduction of electrical signal along a nerve fiber is strictly a surface phenomenon occurring in the nerve cell membrane. A previous discussion shows that the surface properties of polymers are also relevant to the performance of tear substitutes in the eye.

These concepts can be also extended to the wetting of contact lenses and the comfort for the eye. The solubilization of oil soluble vitamins in micelles, that finds so many diverse and wide-ranging applications in engineering, biology and medicine. It is only during the past few years that we have seen rapid advances in understanding the complexities and unique properties of surfaces. Focusing to the topic of interest in this study, surface science has been an important agenda in the world of engineering, specifically in the field of electronics. A small segment of surface science is surface roughness and it plays a major role in the world of electronics, specifically microelectronic fabrication. Its crucial role determines the functional performance of many devices which is manufactured or researched. This effect of form on function is also present at nanoscale and below. Understanding and characterizing nanoscale and even sub-angstrom roughness is becoming increasingly important to our ability to continue exploring and building devices at even smaller length scales (Valipa, Aydil, & Maroudas 2004).

In the research world pioneered by Tomlinson (1919), Coulomb (1785) and Amontons (1699), the importance of surface texture has been initiated and has numerous usages. The multi discipline nature of the subject has created various problems. To provide solutions, manufacturers need to manufacture products that are incorporated with other systems. This is where the study on surface metrology comes in. This is because the real engineering surfaces are basically rough at the macro level and the interaction between surfaces involves the smallest level of connection.

There are many examples illustrating the importance of nanoscale surface roughness. In the industry of data storage, with areal bit densities surpassing 1 terrabyte per square inch, and read/write heads flying mere nanometers above the surface, hard disk platters are now being polished to a roughness of about 1Å. Likewise, as transistors scale down in accordance with Moore's Law, the ability to measure sub-angstrong surface roughness is becoming increasingly important for the semiconductor industry. Atomically flat silicon has shown great decrease in the transistor noise while increasing reliability and device performance.

Surface roughness also contributes deeply in microfluidics. The study of surface roughness or surface characterization is continuously related with the wettability of the material utilized. This is where the important chapter of this study is considered. Plasma has been used for a very long period of time to alter the surface roughness of materials in semiconductor fabrication. It plays an important role to modify the surface used to accomplish the function of the device generally (San Paulo & Garcia 2000 ; Bhushan 2001).

1.2 Problem Statement

Aluminium is widely used as acontact surface in electronics. The reputation of aluminium as contact is recognized at the level of micro and nano. Although micro and nano is a very minor in terms of size, but the impact towards the world of fabrication is huge. With regards to that, solder wetting is a vital factor for electronic connection and also bonding quality. This is proven by the number of studies focusing on the surface characterization and wettability of various materials used in electronics.

Therefore, this study is done to emphasize on the surface characterization and wettability of aluminium bond pad which is treated with Reactive Ion Etch (RIE). Past researchers have done experiments using plasma on various materials but studies focusing on aluminium are very minimal to the best of the author's knowledge. This is the recognized problem which is initialized in this study. Surface characterization on aluminium using RIE or plasma will improve the adhesion of the connection in many devices which will indirectly improve the reliability and functionality of devices generally.

1.3 Research Objectives

In this experimental study, there are few objectives that must be achieved. The primary objectives are;-

i. To study the surface roughness and contact angle of RIE etched aluminium bond pad
ii. To characterize each respective sample from the DOE by using Atomic Force Microscopy (AFM)
iii. To complete a wettability test on each sample and gather the contact angle data
iv. To compare and analyze the effect of RIE on the surface roughness and the wettability

1.4 Research Scope

The study is done according to a boundary which is set as the scope. The study is done to characterize the aluminium deposited wafer that is treated using Reactive Ion Etch. Surface characterization is done specifically on the surface roughness of the sample. The tools used in this experimental study consist of Reactive Ion Etch, Atomic Force Microscopy and a tool which was setup specifically for contact angle or wettability measurement. Therefore the scopes are the surface study mainly on the roughness study of aluminium bond pad and the wettability character of the pad.

1.5 Research Approach

This study was initialized by reviewing the past studies that had been done. This process is done by categorizing the important study that need to be focused. By categorizing, the study can be done in a more structured way and provides advantage to the knowledge that is gained from past years.

Next, the experimental design is planned according to the parameters and number of samples. Full factorial design is used in the study because the design suits the number of parameters that are available. Then, sample preparation is initiated. Two full size wafers are deposited with aluminium before dicing it into the number of samples required.

After the sample is diced, all the samples are cleaned and characterized using Atomic Force Microscopy (AFM). This is done to gather the surface character of the pad before it is treated with Reactive Ion Etch (RIE). Then, the samples are treated using RIE. This is the main part of the study, where the samples treated are characterized using AFM again. The results will be compared to obtain the effect of RIE on aluminium bond pad.

As the last experimental procedure, all the samples are put into the droplet test where a standard setup is set to measure the contact angle. This will provide the wettability results. All the gathered data will be analyzed and compared to study the effect of RIE on the aluminium bond pad.

1.6 Thesis Outline

This thesis is divided into five chapters. This chapter which is the Chapter 1 and Introduction provides a brief summary about the whole study. It includes overview, problem statement, objective, scope of the study, research approach and also the thesis outline.

Chapter 2 discusses about the past study which is an important part of the study. The past research that relates to this study is focused. Importance is given to the publications that were done on the aluminium material.

Chapter 3 explains the path this study has gone through. It presents the methodology and experimental process of the whole study.

Chapter 4 is the most important chapter in this thesis where all the results are discussed to provide evidence on the contribution of this study towards the field related. The results are analyzed compared and justified here.

Finally Chapter 5 presents the summary of research contributions and the suggestion for future work is also presented.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

In this chapter, the theoretical fundamentals of the study are discussed briefly. Reviews are done on the publications done in the past by many researchers. This chapter is divided into few sections to specifically review the related topics for a better understanding and to provide a structural study. Detailed review was done for aluminium deposition, surface characterization, and wettability. Last section provides the reviews done on many other related articles that provided valuable information to assist this experimental study.

2.2 Aluminum Deposition

PVD stands for physical vapor deposition. It works by vaporizing the solid materials, either by heating or by sputtering, and recondensing the vapor on the substrate surface to form the solid thin film. PVD plays an important role in the metallization of processes in semiconductor manufacturing.

Green, Levy, Nuzzo and Coleman (1984), have used chemical vapor deposition to deposit aluminium utilizing pyrolysis of triisobutyl aluminum which is an alkyl of aluminium. Temperature ranging from 220-300°C was used on Si, SiO_2 and device wafer substrates. It was a good step coverage of film where the ability to deposit in

2.5μm windows of a typical device. Surface roughness between 1000-1500 Å produces low optical reflectivity from the film. This also produces a frosty appearance. The films deposited were high in purity producing resistance value between 2.8 and 3.5 $\mu m\Omega$. TEM investigation shows that the films consist of columnar grains with 1-3 μm width. This may depend on the orientation or the treatment done on the surface. Deposition using CVD for metallization has a strong future in the VLSI devices. Main difference among CVD and evaporated aluminium films is the surface metrology features. CVD film is rougher and has a smaller reflectiveness but has a tremendous conformal coverage if compared to the films that were prepared using line-of-sight, sputtering or evaporation method.

According to Hirata, Nagakubo and Naoe (1991), thin Al films used in electrodes and wires in the LSI are mostly developed using DC magnetron sputtering and CVD. The sputtering method produces unsatisfactory results but it is the more convenient method for production line. At the same time, the substrates and the growing of films in the Facing Target Sputtering(FTS) system are rarely plasma exposed during the deposition in sputtering. Hence, presented in this study is the investigation on the Al properties and Al structure which is prepared using FTS system. FTS is a plasma free sputtering system which is used in Al thin film deposition producing smooth surface, fine grains, harder and low in resistivity. Low argon gas pressure and bias voltage at -40V produces films that are smother, with higher reflectivity and hardness. Films prepared at bias voltage of between -100 ~ -160 V produces a definite <111> orientation of the aluminum crystallites parallel to the film plane. Resistivity, ρ of the films deposited at argon pressure below 10^{-1} Pa was almost similar to that of bulk aluminium. Therefore, the structure and properties of thin aluminium films may be improved by using plasma free sputtering systems such as FTS.

Novoselova, Fox,Morgan, and O'Neill (2006), carried out experiments to show the results investigated using optical microscopy, SEM, X-ray diffraction, and porosity and micro hardness tests. Investigations are presented for the co-deposited Ti/Al prepared using Cold Gas Dynamic Spray Deposition(CGDS). CGDS which is a technique that is adapted recently in the production of coatings and large structures is used in manufacturing and prototyping process. The difference in this method is that it does not produce heat on the feedstock while spraying. It is also a method that is growing rapidly where the deposition of spraying particles are done using plastic impact with a velocity of 700m/s and in room temperature. The results were denser mixture of material at Al and Ti regions. This is because at low temperature it has not alloyed together. It also presents the effect of deposition parameters on the deposited structure besides producing ready intermetallic materials.

2.3 Surface Roughness Characterization

Lindseth and Bardal (1999), has presented the comparison results of rolled and etched aluminium surfaces used in industry. Three methods were disclosed with the ability to measure surface feature size of 10mm to sub-micrometer range. The methods were AFM, white-light interferometry and confocal laser scanning microscopy (LSM). Presented results show that similar results were produced by AFM and white-light interferometry although both use different mechanism. The two methods have a good agreement in terms of results. AFM has a better resolution whereas white light-interferometry is faster and has a larger scan area. Furthermore, AFM can measure steeper slopes compared to white-light interferometry. Samples used in this study were aluminium and the effect of optical measurement on the surface topography is yet to be determined. The LSM using highly reflecting metal surfaces that have been studied yields images with strong characteristics. However, there are reasons to believe that the artifacts in LSM might be particularly strong for this type of metal surface.

Zhu, Misawa and Tsukahara (1995), have presented an investigation on the phases of Al adsorbed on Si <100>-2 X 1 surfaces by STM. Experiment was done using UHV STM and observation was made using Al covering up to 0.5 to 9 monolayers. Results presented shows a well-structured A1-2 X 2 adlayer is completed at 0.5 ML at the beginning and further deposition produces roughness and surface disappearance. It is because of the Al clusters forming and adsorbs Al ad-dimers without interrupting Si-2 X 1 structure. Phase structure of A1-2 x 1 was not observed at coverage above 0.5 ML. As the growing process continues, the pseudomorphic layer forms islands at 2 ML. Islands formed are highly oriented and have a noticeable epitaxial connection between Al <110> and Si <100> which is Al <110> || Si <100> and Al <111> || Si <100>. This shows that Si-2 X 1 configuration produces large results on the epitaxial growth of the Al islands.

Revel, Khanfir and Fillit (2006), has presented his proposal of an experiment using two methods which were interferometry microscopy and grazing incidence X-ray diffraction. Surface conditions caused by manufacturing process have a clear impact on the performance of the product. There are few strict condition imposed on the dimension and surface quality products. Few processes are run to achieve these objectives such as turning, grinding and polishing. In this article, presented is a simple review of these techniques and followed by an experimental study of pure Al and Al alloys in terms of surface roughness and surface structure evolution. Both the pure Al and Al alloys are machined using single point diamond turning. A study was also conducted to optimize the turning parameters for various Al alloys. This will allow