Effect of Plasma Surface Treatment for Peel Strength of Metallization Based on Polyimide

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Abstract

The purpose of this study is to investigate the optimum plasma processing as a pre-treatment for the surface of Polyimide (PI) in order to increase adhesion strength of electroless copper (Cu) plating to PI directly. Two kinds of oxygen (O_2) plasma processing were applied and compared in the experiment to investigate the optimum plasma processing. One method was surface wave plasma (SWP) processing generated by the microwave plasma reactor with the 2450MHz microwave power source. The other processing method was reactive ion etching processing generated by the capacitively coupled plasma reactor with the 13.56MHz radio frequency (RF) power source. The processing characteristic of microwave plasma which has comparatively higher electron density than that of RF plasma is anisotropic chemical etching processing without collision of electrically-accelerated atoms to processing objects. On the other hand, the processing feature of RF plasma is isotropic processing with ion bombardment and reactive etching to processing objects. The first part of this paper described the result of the surfaces roughness formed by two types of plasma processing under various processing time. The surface roughness was evaluated by atomic force microscope (AFM) measurement. The AFM measurement showed that the surface roughness formed by the SWP processing was much smaller than the surface roughness formed by the RF plasma processing. In the second part of the paper, chemical conditions of the PI surface measured by X-ray photoelectron spectroscopy (XPS) were investigated after SWP and RF plasma treatments. The measurements of XPS showed that large number of hydroxyl groups as well as the functional groups -C=0 were observed on the surface of PI treated by SWP. In the case of RF plasma treatment, hydroxyl as well as the functional groups -COO was observed on the surface of PI. The functional groups -C=O was not observed on the surface of PI treated by RF plasma. The final part of this paper focused on the relationship between peel strength of electroless Cu based on treated PI measured by T-peel test. T-Peel adhesion strength measurement proved that PI surface treated by the SWP processing was greatly improved in adhesion strength without forming surface roughness or seed layers. On the other hand, electroless Cu adhesion strength after the RF plasma treatment was comparatively weak due to forming the excess surface roughness of PI due to oxygen ion bombardment and isotropic reactive etching.

Introduction

In recent years, improvement of micro-fabrication technology has been a prime task for printed circuit board (PCB) manufacturers because electronics devices more and more require intense minimization and tremendous increases in processing capacity. In some regards, flexible printed circuit boards (FPCB) have some advantages compared to rigid PCB because of its thickness and handiness, especially in mobile phone, or liquid crystal display manufacturing industries. In order to take advantages of its characteristics, FPCB needs further improvement in its manufacturing process. Copper-metalized polymer film is commonly used for the base material of FPCB. Ordinary polyimide (PI) is applied for the polymer film as a layer of dielectric material because of its appropriate physical and chemical characteristics for FPCB. PI has outstanding characteristics for the usage of micro-electronic devices and its fabrication in terms of heat resistance, mechanical strength, dimensional stability, and low dielectric constants [1, 2]. The characteristics of PI are important for manufacturing applications in the integrated circuit technology. Even though PI has good characteristics for the base material of FPCB, there is the major concern about Cu-metalized PI during FPCB manufacturing. The weak adhesion property of metalized copper (Cu) on PI film is serious problem. As a result of this problem, disconnection failure between Cu and PI is sometimes come up under stress of bending or heating. The reason of this adhesion weakness of Cu is thought to be an attribution of the intrinsic characteristics of the surface of PI [3]. The surface of PI is generally hydrophobic which results in poor wettability. The poor wettability disturbs Cu adhesion on the surface of PI [4]. So as to overcome insufficient adhesion strength between Cu and PI, pre-treatment techniques for the surface of PI film is often employed during FPCB manufacturing process. For example, employment of the adhesion seed layers of nickel, chrome, or both on the surface of PI film before Cu metallization could improve adhesion property due to their high chemical reactivity to the Cu [5]. However, this method requires additional processes for building and etching the adhesion seed layers. The additional process easily connects to a decrease in productivity and an increase in production cost in FPCB manufacturing. In addition, the additional processes might not be friendly for the global environment. Plasma treatment for the surface of PI is another pre-treatment method for improving adhesion strength between Cu and PI. In this case, $oxygen (O_2)$ is often used as plasma process gas. Some researchers have shown that exposure of PI surface to dielectric barrier discharge plasma with O_2 under vacuum or atmospheric condition could improve adhesion strength of Cu to PI [6, 7, 8, 9]. O₂ plasma could carry out chemical and physical modifications which strongly influence for Cu adhesion characteristics on the surface of PI. In terms of chemical effect, the O_2 plasma treatments for the PI surface are effective in generating hydroxyl as the functional groups of

C-OH, or C-O-O. Because of the result of the generating functional group on the surface of PI, the PI surface which is naturally hydrophobic in characteristic could be modified to have hydrophilic properties. Modification of the PI surface to be hydrophilic could improve adhesion strength between Cu and PI. Also, the O_2 plasma process makes the surface of PI rough due to the chemical reaction and ion bombardment. Various studies show that the surface roughness of PI formed by O₂ plasma treatment helps PI increase adhesion strength as a physical effect [6, 7, 8, 9]. However, forming roughness on the surface of PI to improve adhesion strength of Cu might connect to a new concern for micro-scale FPCB fabrication. In the future, the thickness of metalized Cu on the PI film will become thinner and thinner because the thickness of the Cu needs to reach a satisfactory level to respond to micro strip lines formed by etching process. Surface roughness of the base PI would become an obstacle for micro fabrication with thin Cu layer of FPCB which requires narrow and thin line patterns on it. In order to improve adhesion strength between Cu and PI without any difficulty for micro-scale FPCB fabrication, improvements in the PI surface for hydrophilic properties by chemically functional groups without large surface roughness is one solution for the problem. In this study, experiments of the O_2 plasma surface treatments for the PI to improve adhesion strength of electroless Cu plating without adhesion seed layers or large surface roughness was demonstrated. Two plasma treatment systems were applied for the experiment. One was a microwave plasma system with a microwave power source. The other is a radio frequency (RF) plasma system. Surface physical and chemical properties were investigated and compared after the two types of plasma treatments done by microwave and RF plasma system with various process times. Surface roughness of the treated PI was investigated by atomic force microprobe (AFM). Chemical property of the surfaces was measured by X-ray photoelectron spectroscopy (XPS). In addition, the adhesion strengths of Cu plating on PI after two types of plasma treatments were measured using T-peel test.

Experiment

Chemical structure of the PI film used in this study was pyromellitic dianhydride-oxydianiline (PMDA-ODA, Toray-Du Pont Chemical; Kapton-V[®]) with the thickness of 125 µm. Fig. 1 shows schematic diagram of the microwave plasma apparatus used to surface treat the PI in this study. In the case of microwave plasma, plasma was generated by the 2450MHz microwave power source. Microwaves were led to the vacuum chamber through the wave guide and the quartz window as a dielectric material set on the top of the chamber. The microwave which was led inside the chamber excited oxygen molecules, and then O_2 plasma was generated on the surface of the quartz window. This type of the plasma is generally called surface wave plasma (SWP) because the plasma is generated just on the surface of the quarts window by propagating microwaves on the surface of dielectric. Excited oxygen atoms generated by SWP diffused downward toward the surface of PI without impressed electromotive force, so that they simply fell downstream and initiated chemical reaction on the surface of PI without physical impact. In the experiment with SWP, O_2 flow rate was 1600sccm, and input microwave power was 2.6kW constant. O_2 flow rate was controlled by a mass flow controller. In order to understand tendency of the PI surface reaction conditions with regard to plasma treatment time, the experiment with the microwave plasma was done under plasma treatment times of 15, 30, 60 and 90sec. respectively. During the experiment with SWP, pressure in the vacuum chamber was set at 80Pa constant. Fig. 2 shows the RF plasma experimental setup. In the case of RF plasma, the plasma was generated between the electrodes by impressed electromotive force. The RF power source of 13.56MHz was applied. In case of the RF plasma treatment, reactive etching due to chemical reaction as well as energetic bombardment by oxygen ions interacted on the surface of the PI. Bias power was set 600V constant. O₂ flow rate was 30sccm controlled by the mass flow controller, and input RF power was 200W constant. RF plasma treatment times were set at 3, 6, 12 and 20min. respectively. During the RF plasma treatments a working pressure of 45Pa wasconstant at each condition. In comparison to the RF plasma whose electron density is about 10^8 /cm⁻³, electron density of the microwave plasma is commonly known as about $10^{12}/\text{cm}^{-3}$ [10]. Treatment times for the SWP and the RF plasma were decided from the etching rate of photo-resist. Etching rate of the





Fig. 1. Schematic diagram of microwave plasma (SWP)



Input frequency: 13.56MHz Input power: 200W Process gas: O₂ Flow rate: 30sccm Pressure: 45Pa Processing time: 3, 6, 12, 20min..

Fig. 2. Schematic diagram of radio frequency (RF) plasma

photo-resist by SWP processing is about 12 to 15 times larger than that of the RF plasma treatment because plasma density of the SWP is much higher than that of RF plasma. Therefore, the treatment time of the RF plasma was set about 13 times longer than treatment time of the SWP. The topography of the PI surface after different types of plasma treatments was characterized by atomic force microscopy (AFM). Areas of $20 \times 20 \mu m^2$ were scanned using contact mode and evaluated for surface roughness (Ra). In order to examine the surface chemistry of the PI before and after the two types of plasma treatments, X-ray photoelectron spectroscopy (XPS) measurements with an AlK α X-ray source were applied. The pressure in the chamber during the XPS measurement was maintained at 2×10^{-8} Torr.



Treatment time=3min. Ra=100nm



Treatment time=6min. Ra=350nm







Treatment time=20min. Ra=510nm

Fig. 3. 20×20µm²AFM images treated by RF plasma



Treatment time=15sec. Ra=0.4nm



Treatment time=30sec. Ra=0.6nm



Treatment time=60sec. Ra=1.1nm



Treatment time=90sec. Ra=1.7nm



After the plasma treatments and investigations of the surface of PI, electroless plating of Cu of $0.3 \mu m$ was carried out for each sample without any adhesion seed layers in order to investigate adhesion strength of electroless Cu plating on treated PI directly. The adhesion strength of the electroless plating of Cu on PI after two types of plasma treatments under various processing time were determined by 90 degree T-peel adhesion strength measurement. In order to measure the adhesion strengths, electrolytic Cu plating of $3 \mu m$ was implemented on the surface of electroless Cu whose thickness was $0.3 \mu m$.

Result and Discussion

In order to investigate topographical change of the PI surface after two types of plasma treatment, atomic force microprobe (AFM) images was compared. Fig. 3 shows area of $20 \times 20 \mu m^2$ AFM images of the surface of PI after RF plasma treatment. Surface roughness (Ra) of untreated PI was 0.7nm. As seen the figure, Ra values of PI treated by the RF plasma with treatment time 3, 6, 12, 20min. under constant input power condition were increased to 100, 350, 420, and 510nm, respectively. In the RF plasma treatment, it is estimated that the bombardment energy of oxygen (O₂) ion and reactive etching to the surface of PI leads to this surface topographical change in each experiment. It is thought that increased treatment time roughened the surface more due to the acceleration of reactive etching and oxygen ion bombardment of the surface of the PI.



The PI surface 20×20µm² AFM measurement results after SWP treatment were shown in Fig. 4. Ra values of PI treated by SWP with treatment time 15, 30, 60, 90sec. were 0.4, 0.6, 1.1, and 1.7nm, respectively. The result shows that the surface roughness of PI after the SWP treatment is very small compared to Ra of the PI surface treated by the RF plasma even though treatment time was much shorter in the SWP plasma treatment than in that of the RF plasma treatment. The surface roughness of PI treated by the SWP was almost unchanged even in the longest treatment time of SWP in the experiment. The figure shows that Ra values of PI treated by the SWP were increased as treatment time increased. However the value of Ra was tiny. It is thought that the difference of PI surface treatments results between the RE plasma and the SWP came from the characteristics of the plasma processing mode. In case of the RF plasma, the roughness of PI surface was formed by ion bombardment and reactive etching simultaneously in the surface of PI. The surface of PI was exposed to reactive etching and ion bombardment of oxygen generated in the RF plasma. In addition, treatment by the RF plasma was an isotropic processing because oxygen atoms generated in the RF plasma were accelerated by impressed electromotive force to the vertical direction against the electrodes. In case of the isotropic plasma treatment, oxygen atoms collide vertically on the surface of PI, and then reactive etching and physical etching occurred. As the result of vertical collision of oxygen ion and reactive etching, depth of the surface roughness in the RF plasma treatment became large in a longitudinal direction. On the other hand, the PI surface treated by the SWP was almost flat. In the case of SWP processing, only chemical etching occurred without physical impact by accelerations of oxygen atoms. Also, the electron density of SWP was much higher than that of RF plasma. As a result of the high density anisotropic plasma treatment, chemical etching proceeded delicately against the surface of PI. Conceivably, there might be very fine roughness which is difficult to observe from the chemical etching. The result of SWP treatment showed that anisotropic plasma process with no ion bombardment could treat the PI surface without large roughness formation. In order to understand the change in the chemical binding states of PI surface after two kinds of plasma treatments, X-ray photoelectron spectroscopy (XPS) spectra of C1s were examined. Fig. 5 shows the narrow scan C1s XPS spectra measured from the surface of two kinds of plasma treatment of the PI with treatment time of 90sec. for the SWP and 20min. for the RF plasma treatment. The figure on the left side shows that the component ratio of -C=O bonding on the surface of PI after the SWP plasma treatments was observed with treatment time 90sec. The -C=O bond structure is not found in the original PI surface. It is thought that chemical structure on the surface of PI was re-arranged by the plasma treatment. -C=O bonding is known to give improved wettability. However, -COO bonding which is also known to be one of the important chemical bonds for wettability improvement was not observed on the surface of PI after the SWP treatment. Alternatively, there was no -C=O bond structure on the surface of PI after the RF plasma treatment even though -COO bonding was observed as shown in right side of the Fig. 5. The results might come from the difference of the plasma treatment processing. While the RF plasma processing is isotropic treatment, the SWP plasma treatment processing is anisotropic. In the case of SWP process, oxygen atoms simply fell downstream and initiated chemical modification on the surface of PI without ion bombardment. This characteristic might cause the difference in chemical properties of PI surfaces after each plasma treatments. In order to understand this phenomenon, the details will be treated as a future direction of this study. Fig. 6 (a) shows the ratio of hydroxyl as -COO on the surface of PI after the RF plasma treatment. The ratio of chemical composition -COO bond after the treatment of RF with treatment time 3, 6, 12, 20min. were 0.1, 0.066, 0.055, and 0.035%, respectively. In the case of RF plasma, the ratio of -COO bonding decreased for treatment time longer than 3min. It is believed that chemical modification of the surface of PI was disturbed by forming surface roughness. The situation could be that chemical surface modification and physical changing remained in an equilibrium condition for treatment times of less than 3min. After the RF plasma treatment of 3min., ion bombardment might decrease hydroxyl of -COO bonding. Fig. 6 (b) shows the ratio of hydroxyl as -C=O on the surface of PI after the SWP treatment in various treatment times. The ratio of chemical composition -C=0 bond after the treatment of SWP with treatment time 15, 30, 60, 90sec. were 1.3, 1.5, 1.8, and 2.0%, respectively.



(a) Chemical composition of -C=O after SWP treatment







The component ratio of hydroxyl as -C=0 bonding treated by SWP was increased as treatment time was longer. However, the increase rate of the ratio of -C=0 bonding was gradually reduced as treatment time increased. The increase rate of in the ratio of -C=0 bonding looks almost saturated after treatment times of 60sec. It may be presumed that the chemical modification of the surface of PI was almost completed at treatment times of 90sec. Based on the results of AFM and XPS measurements, the surface of PI could be improved without forming surface roughness by the SWP processing. To investigate the effect of plasma treatment for the adhesion strength of direct electroless Cu plating, T-peel test was carried out. In order to carry out the adhesion strengths measurement test, electrolytic Cu platings of 3μ m were implemented on the surface of electroless Cu of 0.3μ m thickness. Fig. 7 shows the results of T-peel strength measurement after SWP treatment. The peel strengths of PI treated by the RF plasma for all treatment times could not be measured because the adhesion peel strengths were less than 0.1kg/cm which was the submarginal value of the measurement's limitations. It is estimated that large surface roughness by excess RF plasma treatment generated a weak layer between Cu and the PI and surface chemical modification was not enough to improve adhesion strength. The peel strengths after the treatment of SWP with treatment time 15, 30, 60, 90sec. were 0.31, 0.32, 0.48, and 0.54kg/cm, respectively. As SWP treatment time was longer, the peel strength became stronger. The result shows that the adhesion strength of Cu was improved by the SWP plasma treatment.

Conclusion

In this study, the optimum plasma processing for improvement of adhesion strength of direct electroless copper (Cu) plating for polyimide (PI) surface was investigated. Two kinds of oxygen (O_2) plasma were applied for the experiment. One was the radio frequency (RF) plasma which was generated by 13.56MHz RF power source. The other was the surface wave plasma (SWP) which was generated by 2450MHz microwave power source. The feature of the RF plasma treatment was isotropic treatment with ion bombardment and reactive etching process.

On the other hand, the SWP which was comparatively higher electron density than that of the RF plasma was anisotropic chemical etching processing without ion bombardment. Chemical and physical characteristics of the PI surface after two kind of plasma treatment were investigated by atomic force microprobe (AFM) and X-ray photoelectron spectroscopy (XPS). The result of the AFM measurements showed that the PI surface treated by the SWP was almost flat compared to the RF plasma treatment. The surface roughness of PI treated by the RF plasma increased with treatment time. In the XPS experiment, hydroxyl -C=O was observed on the surface of PI after the SWP treatment. The chemical structure of -C=O could improve surface wettability of the PI. However, -COO which can also improve surface wettability was not observed after the SWP treatment on the surface of PI. On the contrary, there was no chemical formation of -C=0 on the surface of PI after treatment by the RF plasma even though chemical formation of -COO was observed. This might be the result due to the difference of plasma treatment features. In the case of RF isotropic plasma treatment, the surface of the PI was exposed to reactive etching and ion bombardment. In the case of SWP anisotropic treatment oxygen atoms simply fell downstream and initiated chemical reaction on the surface of PI without physical impact. These characteristics of the each plasma process might cause of the difference of chemical properties of the PI surfaces after the SWP treatment and the RF plasma treatment. Adhesion strength was measured by using the T-peel test after Cu plating without any adhesion seed layers. The result of the T-peel measurement showed that the adhesion strength of the electroless direct Cu plating to the PI after the SWP treatment was greatly improved. The peel strength value of 0.31 to 0.54kg/cm after the SWP treatments was measured with treatment times of 15 to 90sec. The peel strengths of PI treated by the RF plasma of all treatment time could not be measured because the adhesion peel strengths were less than 0.1kg/cm which was the submarginal value of the measurement's limitations. The result indicates that the SWP could improve adhesion strength of electroless Cu plating without adhesion seed layers or surface roughness. The chemical surface modification with -C=0 bond structure effected a greatly improved adhesion strength.

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Effect of Plasma Surface Treatment for Peel Strength of Metallization Based on Polyimide

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Back Ground

Current Condition

- Electric devices are required minimization and increase in process capacity
- Improvement of micro-fabrication technology in PCB is pressing need
- Polyimide has ideal properties as a flexible substrate in FPCB manufacturing

<u>Problem</u>

- The surface of PI is hydrophobic characteristic
- •The adhesion strength of metal to PI is generally poor (in electroless Cu plating)

Solutions

- Building adhesion seed layer on the surface of PI before Cu plating →Additional processes connects to increase cost and decrease productivity
- Pre-treatment by plasma
 - \rightarrow Surface roughness of PI might be an obstacle for micro-fabrication



Purpose

Purpose of the Experiment

Investigate the optimum plasma processing for electroless Cu plating on PI directly w/o surface roughness formation

Evaluation



- Compare surface treatments of RF plasma and microwave plasma
- Investigate surface roughness of PI after RF and microwave plasma treatment
- Examine surface chemistry of PI after RF and microwave plasma treatment
- Adhesion strength measurement of Cu plating to PI without adhesion seed layer



Experimental Apparatus

RF plasma equipment





Experimental Apparatus

Microwave plasma equipment





Surface wave plasma (SWP)





 μ -wave generated by magnetron is led into the wave guide.

μ-wave leaks from slots and propagates into the chamber through the quartz window.



Plasma is generated on the surface of quartz window.

Electron density

Electron density of microwave plasma is much higher than that of RF plasma.

- Electron density of microwave plasma : 10¹²/cm⁻³~
- Electron density of RF plasma : 10⁸/cm⁻³ ~

Process mechanism

Oxygen atoms generated in microwave plasma are diffused and fall to the sample slowly.

- Microwave plasma process: No physical impact, only chemical reaction occurred
- •RF plasma process: Physical impact of ion bombardment assists chemical reaction

Isotropic and anisotropic process

- Direction of etching processing with microwave plasma : Isotropic process
- Direction of etching process with RF plasma : Anisotropic process



Experimental Overview

Type of plasma treatment for PI surface

- Microwave plasma (SWP) (Treatment time: 15, 30, 60, 90sec.)
- •RF plasma
 - (Treatment time: 3, 6, 12, 20min.)

*Treatment time of microwave plasma and RF plasma was decided based on resist stripping amount.

Measurement method

- Surface physical property Atomic force microprobe(AFM)
- Surface chemical property X-ray photoelectron spectroscopy(XPS)
- Adhesion strength between electroless Cu plating and polyimide T-Peel test
- *Electrolytic Cu plating of $10\mu m$ were conducted on the surface of electroless Cu for T-Peel measurement.



AFM images

Measurement area of $20\times 20 \mu m^2$



<figure>

Untreated Ra=0.7nm

*Ra is arithmetic average of the absolute value of the roughness

RF plasma 20min. Ra=510nm



Microwave plasma 90sec. Ra=1.7nm



AFM images as a function of plasma treatment time

RF plasma treatment Measurement area of $20 \times 20 \mu m^2$



3min. Ra=100nm



6min. Ra=350nm



12min. Ra=420nm



20min. Ra=510nm

Microwave plasma treatment



15sec. Ra=0.4nm



30sec. Ra=0.6nm



60sec. Ra=1.1nm



90sec. Ra=1.7nm



Function of RF plasma treatment time





Function of microwave plasma treatment time













Function of RF plasma treatment time





Function of microwave plasma treatment time





Peel Strength

Function of microwave plasma treatment time





Summary

<u>Purpose</u>

Investigate the optimum plasma processing for electroless Cu plating on PI directly w/o surface roughness formation

Experiment

Investigate surface roughness formation, chemical composition and peel strength between electroless copper plating and polyimide after two types plasma treatment, RF plasma and microwave plasma.

<u>Result</u>

•RF plasma : Large surface roughness was formed Surface was chemically modified but decreased as processing time

Peel strength was too weak to measure absolute value

•Microwave plasma

: There is no surface roughness after plasma treatment Surface was chemically modified and increased as processing time Peel strength increased as processing time is longer

The result shows that microwave plasma process can increase adhesion strength between electroless Cu plating and PI without surface roughness formation, but the adhesion strength is not enough for practical use.



Thank you for listening