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Adhesion and Failure Analysis of Metal-Polymer Interface in Flexible Printed Circuits Boards

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As device miniaturization in microelectronics is currently requested in the development of high performance device, which usually include highly-integrated metal-polyimide multilayer structures. A redistribution layer (RDL) process is currently emerging as one of the most advance fabrication techniques for on-chip interconnect and packaging. One of the major issues in this process is the poor adhesion of the metal-polyimide interfaces particularly in flexible circuit boards due to the flexibility and bendability of devices. In this study, low pressure O_2 plasma treatment was investigated to improve the adhesion of metal-polyimide interfaces, using inductively coupled plasma (ICP) treatment. We identified that the adhesion of metal-polyimide interfaces was greatly improved by the surface roughness control providing 46.1 MPa of shear force in the ball shear test after O_2 plasma treatment, compared 14.2 MPa without O_2 plasma treatment. It was seemingly due to the fact that the adhesion in metal-polyimide interfaces was improved by a chemical conversion of C=O to C−O bonds and by a ring opening reaction of imide groups, which was confirmed with FT-IR analysis. In the finite element numerical analysis of metal-polyimide interfaces, the O_2 plasma treated interface showed that the in-plane stress distribution and the vertical directional deformation agreed well with real failure modes in flexible circuits manufacturing.

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I. INTRODUCTION

Wafer-level packaging technology (WLP) has evolved from the solder bumping technology. Then, two trends have continued to drive the implementation of solder bump interconnect: (1) the continuing drive to higher densities on chip, resulting in the need for more I/O; and (2) consumer demand for continued miniaturization and increased functionality in handheld and portable products [1]. The redistribution requires thin-film polymers and metalization to reroute the peripheral pads to an area array configuration, and under bump metallurgy (UBM) to create reliable solder joints [2]. In the early 2000s, the question became how to use the WLP concept when larger numbers of I/O were required or how to maintain the same I/O and pitch during a die shrink [3]. Fan-out WLP (FOWLP) technology is an enhancement

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of standard wafer-level packages developed to provide a solution for semiconductor devices requiring a higher integration level and a greater number of external contacts. It provides a smaller package footprint with higher I/O along with improved thermal and electrical performance. Redistribution layers are formed using PVD seed deposition and subsequent electroplating / patterning to reroute I/O connections on the die to the mold compound regions in the periphery [4].

Flexible circuits are forms of printed wiring interconnect structure built on thin, flexible substrates. Because of this flexibility, flexible circuits have many advantages over other wiring methods and have many applications in electronic equipment that requires high density wiring in a small space. The basic advantages of the flexible circuits are its thinness and ability to bend. However, a thin flexible circuit generates many supplemental advantages not available with other wiring methods. A 3-D wiring with component assembling in a small space and a long-term dynamic flexing with small radius are typical examples of what other wiring materials cannot replace. On the other hand, flexible circuits also have many disadvantages due to unstable thin constructions. It is necessary to consider how to avoid the disadvantages when using a flexible circuit in a packaging system [5].

Miniaturization of devices in microelectronics has achieved significant progress in device density and performance. To leverage these advances to improve system performance, complex metal-polymer multilayered structures have been developed for on-chip interconnection and packaging. The polyimides, a class of high-temperature polymers, are widely used for such applications due to their low dielectric constant, easy processability, and good thermal stability [6–8]. For the minimization of the line-width and resistivity of metal lines on the flexible polyimide substrate, an additive process employing the electroplating of Cu into the patterned photoresist mask needs to be developed. One difficult problem during the metallization of conductive metals such as Cu on the polyimide substrate is that these metals do not adhere well to the polyimide surface [9–14]. Therefore, poor adhesion property of metals to polyimide has to be overcome to achieve the flexibility of fabricated devices because stress applied during bending is focused more and more on the interface between metal lines and polymer substrate with the line width decreased.

Weak adhesion of metals to polyimide is attributed to the intrinsic nature of polyimide surface [15]. Various researches using plasma treatments have been tried in order to modify the chemical and physical structures of the polyimide surfaces. The influence of low pressure oxygen and argon gas plasma modification of polyimide film on the adhesion to an alloy plating layer made from chromium and nickel was investigated. Changes in the roughness and an increase in the percentage of hydrophilic groups improved the adhesive strength between metal and polyimide [16]. On the other hand, to enhance peel strength, inductively coupled plasma (ICP) was treated on the polyimide surface using N_2 gas with Ar as a function of radio frequency (RF) power. A dramatic enhancement of the peel strength was achieved. The surface roughness of polyimide exposed to plasma treatments was rarely changed, while X-ray photoelectron spectroscopy (XPS) showed the substantial increase of C−N functional groups [17]. According to T. Woo, a plasma process using N_2, O_2 and Ar gases increases the peel strength between Ni and polyimide. In the case of Ar and O_2 atmospheres, it showed a tendency where C−O binding and C−N binding increased with increasing preprocessing time. Preprocessing under an O_2 atmosphere showed the largest peel strength by increasing the mechanical binding force with increasing polyimide surface roughness based on plasma preprocessing and changing its chemical binding structure [18].

In this study, low pressure O_2 plasma treatment was chosen as a method for enhancing adhesion of metal-polyimide interface by controlling the surface roughness of polyimide. C−O bonding changes were identified by chemical quantitative analysis for the modified surface of polyimide using O_2 plasma treatment. The effect of O_2 plasma treatment on the phase transformation was investigated through a Cole-Cole plot. The failure mode of polyimide was studied through finite element method (FEM) simulation for thermal stress and vertical directional deformation of polyimide and verified by actual failure phenomenon.

II. EXPERIMENTS

The material investigated in this study was a widely used commercial poly amic acid resin (HD-4110 Series, HD MicroSystems^{TM}), which is a negative-tone, solvent developed, photo-definable polyimide precursor for stress buffer and flip chip bonding applications. As reported in the material safety data sheet, the polyimide precursor is composed of mixture of *N*-Methyl-2-pyrrolidine, 3,6,9-trioxanudecamethylene dimethacrylate, methanol, and non-regulated ingredients at 40 - 50, 1 - 10, 1 - 10, and 40 - 50 wt%, respectively.

To investigate the property of polyimide surface, the specimens were prepared with 300 °C, 350 °C, and 375 °C curing, respectively. The surface roughness of polyimide was controlled by ICP equipment. The process pressure of the chamber was approximately 1.33 Pa. The source power of chamber was 1500 W and the RF was 13.56 MHz. The bias power was 200 V, controlled by voltage values. The flow rate of O_2 gas was 300 cm^3/min by using an electronic mass flow controller. The surface morphology of polyimide treated by O_2 plasma was obtained using atomic force microscopy (AFM, PSIA Inc. XE-100) by 30×30 um^2 scanning area size and scanning electron microscopy (SEM, TESCAN VEGA3). The adhesion of metal and polyimide in-

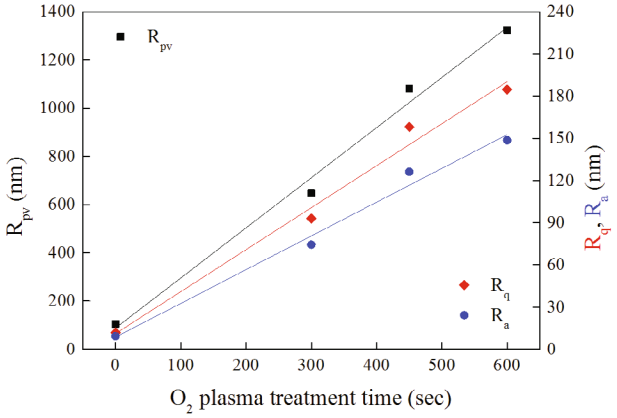

Fig. 1. (Color online) Relationship of surface roughness of polyimide and O_2 plasma treatment time.

terfaces was measured by ball shear tester (NORDSON Dage-4000HS) with JEDEC JESD22-B117A specification. The chemical property of polyimide treated by O_2 plasma was identified using Fourier transform infrared spectroscopy (FT-IR, BRUKER IFS-66/S) using attenuated total reflectance (ATR) technique. The mechanical property of polyimide was investigated by dynamic mechanical analysis (DMA, SEIKO EXSTAR 6000). The dimension of specimen was 7 mm width, 100 mm length, and 30 um thickness. The rate of temperature rise is 3 °C/minute, and the evaluation temperature range was 100 °C to 400 °C. The evaluation frequency was 1 Hz. The simulation for thermal stress and vertical directional deformation of polyimide was used by FEM tool (ANSYS 18.0).

III. RESULTS AND DISCUSSION

1. Quantitative analysis of surface roughness control

The major issue in the metallization of conductive metal such as Cu on the polyimide substrate is that the metal does not adhere well to the polyimide surface. In this case, low pressure O_2 plasma using ICP treatment can be as a method for improving adhesion of metal-polyimide interface by controlling the surface roughness of polyimide. ICP treatment has many advantages, suitable for surface roughness control, as high-density plasma, low-pressure etch reactor, longer mean free path, and both chemical and physical etching.

Figure 1 shows the relationship of surface roughness of polyimide and O_2 plasma treatment times, which are 0 second (as cured), 300 seconds, 450 seconds, and 600 seconds, respectively. There is a linear relationship between R_{pv}, R_q, R_a and O_2 plasma treatment times, where R_{pv} is the value of peak to valley, R_q is the value of standard deviation of the height value, and R_a is the value

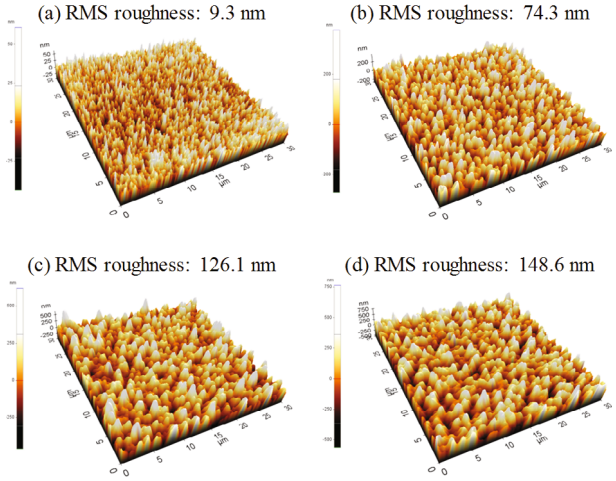

Fig. 2. (Color online) AFM scan images of polyimide surface with O_2 plasma treatment. O_2 plasma treatment time: (a) 0 second, (b) 300 seconds, (c) 450 seconds, (d) 600 seconds.

roughness average. The surface roughness of polyimide increases with increasing O_2 plasma treatment time, and the etching rate of polyimide is 0.24 nm/sec. The oxygen ions are bombarded into the polyimide backbone and the volatile byproducts are removed in the form of carbon oxide derivatives. The surface roughness of polyimide is caused by difference in etching rate on the polyimide surface.

Figure 2 makes it evident that the O_2 plasma treatment can control the surface roughness of polyimide. The surface of polyimide as cured has a small roughness despite of using the spin coating method. The reason for phenomenon is the fine surface movement during polyimide curing. As the O_2 plasma treatment time increases, the size and height of the polyimide surface peaks increase gradually.

SEM images show that surface roughness increases as the O_2 plasma treatment time increases, shown in Fig. 3. However, the bottom shape of the surface is slightly different from that of the AFM analysis. It is observed that the bottom shape of surface is flat. It is confirmed that the surface roughness controlled by O_2 plasma treatment physically widens the area of the metal-polyimide interfaces and the adhesion of interface increases.

2. Analysis of adhesion of metal and polyimide interface

In order to study the phenomena caused by poor adhesion of metal and polyimide in flexible circuits, the ball shear evaluation method using JEDEC JESD22-B117B standard was used. The tensile stress occurs on the side of the metal trace in the direction of applied load and the maximum compressive stress occurs on the opposite

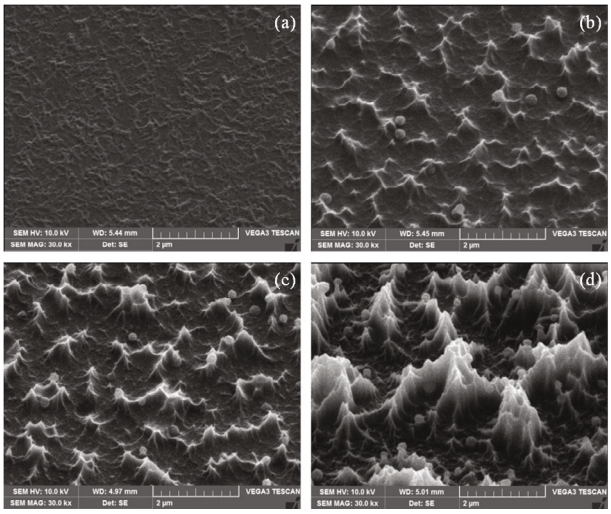

Fig. 3. SEM images of polyimide surface with O_2 plasma treatment. O_2 plasma treatment time: (a) 0 second, (b) 300 seconds, (c) 450 seconds, (d) 600 seconds.

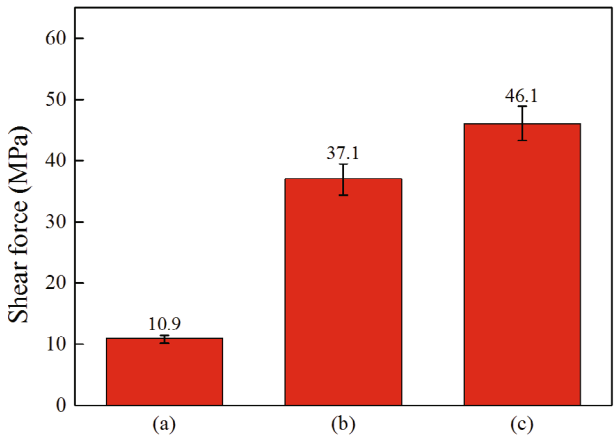

Fig. 4. (Color online) Ultimate shear force vs. plasma types. (a) O_2 plasma treatment with only source power (chemical effect), (b) Ar plasma treatment with source and bias power (physical effect), (c) O_2 plasma treatment with source and bias power (chemical and physical effect).

end of the pattern. The ultimate shear force is greater as the adhesion of the metal-polyimide interfaces is greater.

Figure 4 shows the relationship of the chemical / physical effects and the maximum shear force values under various plasma treatment conditions. The O_2 plasma treatment with source power condition indicates how the chemical reaction of the plasma affects the shear force value. The case of Ar plasma treatment with source power and bias power indicates how the physical reaction of the plasma affects the shear force value. The case of O_2 plasma treatment with source and bias powers indicates how the chemical and physical reactions of the plasma treatment affect the shear force value. The effect of the chemical reaction on the maximum shear force is 10.9 MPa, which is about 24% of the O_2 plasma

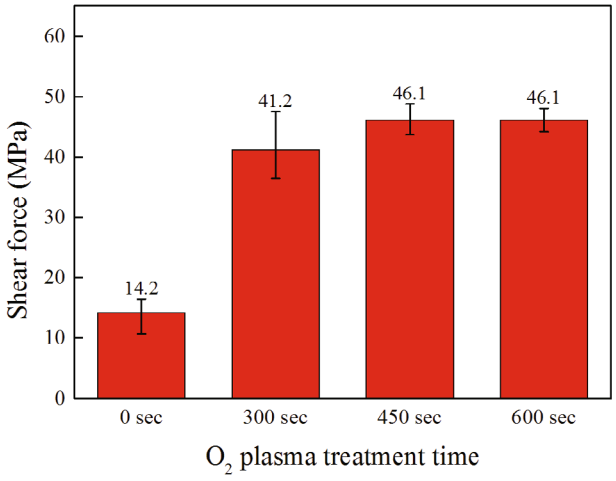

Fig. 5. (Color online) Ultimate shear force vs. O_2 plasma treatment time.

treatment with source and bias powers. The effect of the physical response on the maximum shear force is 37.1 MPa, which is about 80% of that. Therefore, it is confirmed that improvement of adhesion by physical reaction is much larger than that of adhesion by chemical reaction. In addition, the O_2 plasma treatment with source and bias powers confirms that chemical and physical effects can be obtained at once.

Figure 5 plots the measured ultimate shear force against O_2 plasma treatment times. The ultimate shear force values are 14.1 MPa, 41.2 MPa, 46.1 MPa, and 46.1 MPa at O_2 plasma 0 second, 300 seconds, 450 seconds, and 600 seconds, respectively. The values of ultimate shear force are saturated after O_2 plasma 450 seconds. The values of variation decrease as increasing O_2 plasma treatment time, which are 5.8 MPa, 11.0 MPa, 5.1 MPa, and 3.9 MPa, respectively. The trend of ultimate shear force indicates that the adhesion of metal-polyimide interfaces increases as increasing O_2 plasma treatment time.

The plot of the measured the ultimate shear force against the surface roughness of polyimide is shown in Fig. 6. The values of ultimate shear force are saturated over the 126.1 nm roughness. When the R_a values of the polyimide surface are 9.3 nm, 74.3 nm, 126.1 nm, and 148.6 nm, the adhesion values are 14.1 MPa, 41.2 MPa, 46.1 MPa, and 46.1 MPa, respectively. The fact that the shear force values of the metal and the polyimide interface is saturated when the surface roughness becomes more than a certain level indicates that the physical increase of the surface area has a limit to increase the adhesion of the interface. It also means that when the metal-polyimide bonding strength is maintained, the polyimide roughness acts as an anchor to the metal. That is, the roughness of the polyimide surface is one of the major factors of increasing the physical bonding strength.

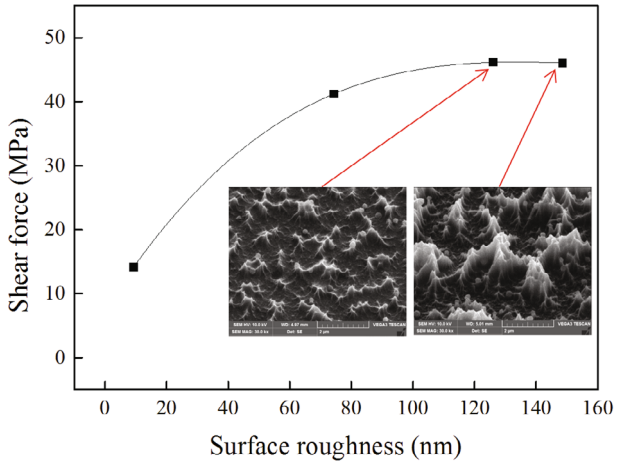

Fig. 6. (Color online) Ultimate shear force vs. surface roughness of polyimide. Inserted SEM images show the peak shape of surface at that roughness.

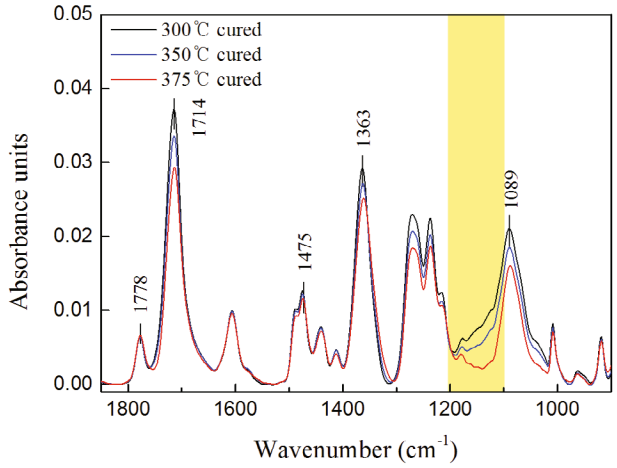

Fig. 7. (Color online) FT-IR spectra of various polyimide curing temperatures.

3. Chemical analysis for surface modification

When the polyamic acid was cured at high temperature, the solvents in the polyamic acid were removed and water molecules were removed through the condensation reaction. At this time, the specific amide FTIR peak disappears and the specific imide FTIR peak appears. Observation of these peak changes can qualitatively analyze and compare the imidaization rate of the sample.

Figure 7 shows the absorption peaks of polyimide after various curing temperatures. The absorption peak at 1778 cm^{-1} indicates the C=O symmetrical stretching of polyimide (imide I). The absorption peaks at 1714 cm^{-1} indicates the C=O asymmetrical stretching of polyimide (imide I). The absorption peaks at 1363 cm^{-1} indicates the C−N stretching vibration (imide II). Around 730 cm^{-1} bending vibration of cyclic C=O (imide IV) is not shown. It can be seen that the intensity of the

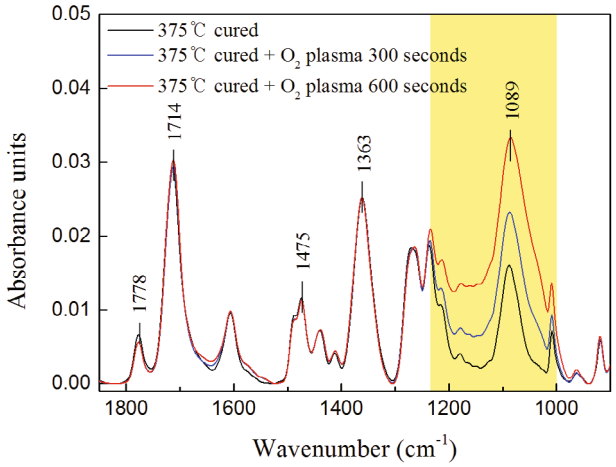

Fig. 8. (Color online) FT-IR spectra after O_2 plasma treatment.

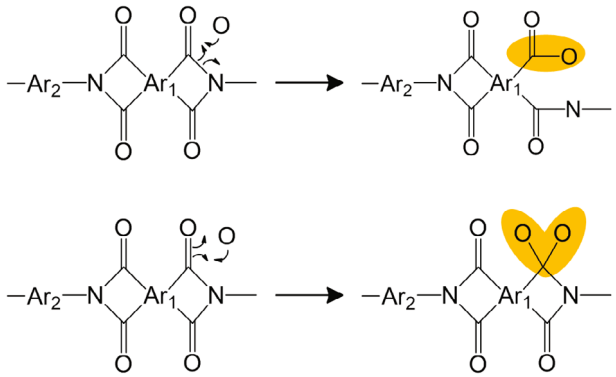

Fig. 9. (Color online) Mechanism of O_2 plasma reaction at polyimide surface.

absorption peak around 1100 - 1200 cm^{-1} changes as the polyimide curing temperature rises from 300 °C to 375 °C. The peak in this area corresponds to the C−O stretch peak of the carbonyl functional group. The imidization rate is higher, as the polyimide curing temperature is higher. As the curing temperature increases, the carbonyl group of polyamic acid is more reactive and the intensity of this area peak decreases.

When the polyimide is subjected to O_2 plasma treatment, the oxygen ions attack the backbone of the polyimide molecular chain to break bonds and form new bonds. It is possible to qualitatively compare and analyze the intensity of the FTIR peaks. Figure 8 shows that only peaks around 1000 - 1230 cm^{-1} are affected. This indicates that the O_2 plasma reaction has selectivity to certain bonds.

The peak intensity around 1089 cm^{-1} shows a very large change. This peak represents generally the C−O stretch peak of the terminal alcohol. It means that C−O bonds which are the same structure as terminal alcohol have been generated after the O_2 plasma treatment. It is presumed that the oxygen ion, in the mechanism of Fig. 9, attacked the imide ring and the carbonyl group

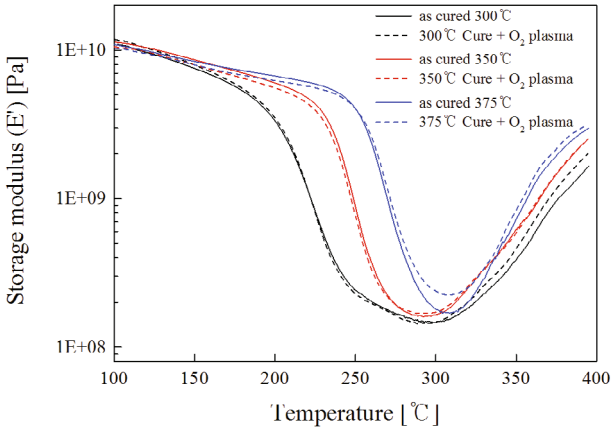

Fig. 10. (Color online) Comparison of storage modulus (E') of samples between as cured and after O_2 plasma.

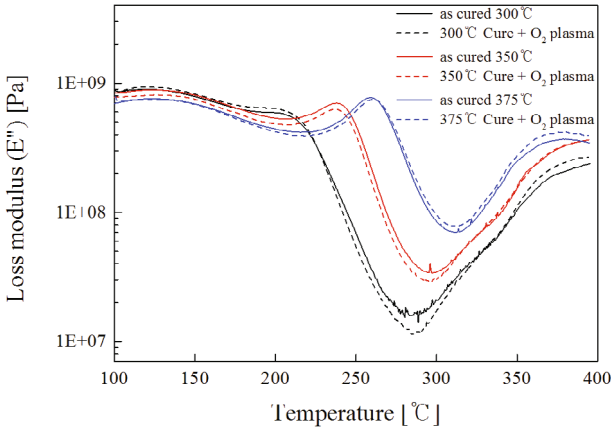

Fig. 11. (Color online) Comparison of loss modulus (E'') of samples between as cured and after O_2 plasma.

changed into terminal alcohol C−O group. The resulting C−O bond serves to increase the adhesion of the metal and polyimide by forming a C−O−Ti bond with titanium which generally is used as a seed layer of the micro electro mechanical systems (MEMS) structure.

Also, it can be confirmed that the peak intensity near 1160 cm^{-1} is changed. This peak corresponds to the C−O stretch peak of the carbonyl group, as mentioned. This is the result of oxygen ion attacking to the imide groups. As shown in Fig. 8, this C−O bond is created simultaneously with a ring opening reaction of imide groups. This bond also serves to increase the adhesion of the metal and polyimide by forming a C−O−Ti bond.

When O_2 plasma treatment is carried out, a large amount of C−O bond is generated and the sites capable of chemical bonding with titanium increase, and the bonding strength of metal and polyimide increases.

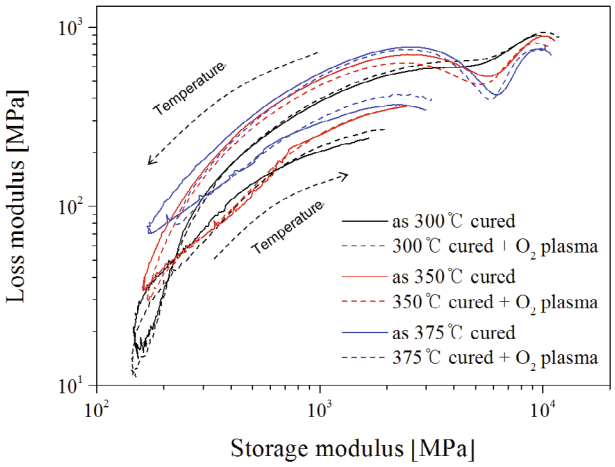

Fig. 12. (Color online) Cole-Cole plot after O_2 plasma treatment.

4. Mechanical analysis to surface modification

Dynamic mechanical analysis (DMA) can be simply described as applying an oscillating force to a sample and analyzing the material's response to that force. From this, one calculates properties like the tendency to flow, called viscosity, from the phase lag and the stiffness from the sample recovery. These properties are often described as the ability to lose energy as heat damping and the ability to recover from deformation, elasticity. DMA analysis can define the mechanical properties of polymers and the change of properties from the O_2 plasma effect.

Figure 10 shows that the shape of storage modulus (E') curve is very similar to each other. It seems that the surface change of polyimide by O_2 plasma treatment does not affect the storage modulus property. The onset temperature of the sample as cured at 300 °C is shifted −0.3 °C after O_2 plasma treatment. That of the sample as cured at 350 °C is shifted −0.2 °C in same process. That of the sample as cured at 375 °C is shifted −1.2 °C. The shape of the pair of two curves, which are as cured state and after O_2 plasma treatment, is very similar each other at different temperatures. It is confirmed that the effect of O_2 plasma treatment does not give a great effect to the storage modulus.

Figure 11 shows that the peak loss modulus (E'') as cured is similar to that after O_2 plasma treatment in the case of curing at 300 °C, 350 °C, and 375 °C. The gaps of two peaks, which are as cured and after O_2 plasma treatment cured at 300 °C, 350 °C, and 375 °C, are −0.1 °C, +1.1 °C, and −1.8 °C, respectively. The shape of the pair of two curves is very similar each other at different temperatures, which is similar to the case of storage modulus. It is also confirmed that the effect of O_2 plasma treatment does not give a great effect to the loss modulus.

Cole-Cole plot represents the storage modulus and loss modulus at the same time and confirms the information of phase transformation. In Fig. 12, the increase in the evaluation temperature is indicated by arrows. The individual curves plot the values for the cured tempera-

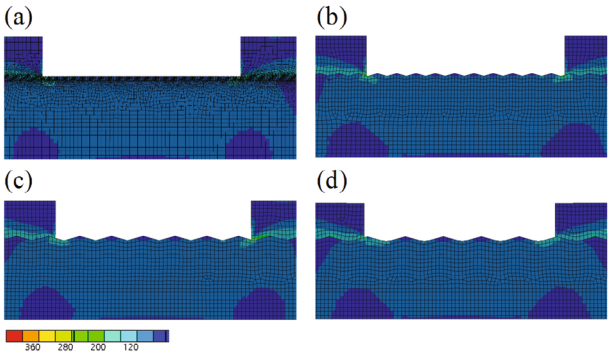

Fig. 13. (Color online) Total stress distribution after various O_2 plasma treatment times. Surface roughness: (a) 100 nm, (b) 650 nm, (c) 1080 nm, (d) 1325 nm.

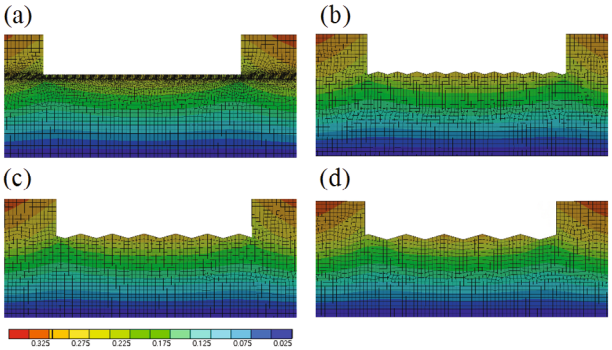

Fig. 14. (Color online) The vertical directional deformation after various O_2 plasma treatment times. Surface roughness: (a) 100 nm, (b) 650 nm, (c) 1080 nm, (d) 1325 nm.

ture and the O_2 plasma treated at that temperature, respectively. The hemispherical shape of the Cole-Cole plot means T_g, and all series have asymmetrical hemisphere shapes. The phase transformation, which is T_g, is found in all curve shapes. By comparing curves, it can be concluded that O_2 plasma treatment does not affect mechanical properties such as storage modulus and loss modulus. In other words, the O_2 plasma treatment affects the surface but does not affect the overall properties of the entire polyimide film. The singularity is that the hemispherical graph progression direction has the minimum value and the hemispherical shape is reversed at the minimum point. That is, T_g appears twice. The cause of second T_g is due to the additional curing or branching of the polyimide structure.

5. Simulation for polyimide film treated by O_2 plasma

To increase the bond strength of metal and polyimide, the stress distribution of interface with polyimide film treated by O_2 plasma was proceeded to ANSYS 18.0 version which is the FEM tool. Young's modulus of the polyimide film was 4.1 GPa measured by tensile test, Poisson's ratio was 0.34, CTE was 35 ppm/°C, and thermal conductivity was 0.2 W/mK. The metal material used was Cu, Young's modulus was 110 GPa, Poisson's ratio was 0.34, CTE was 18 ppm/°C, and thermal conductivity was 401 W/mK. The mesh size of the drawings used in simulation was 100 nm at the interfaces and under 1 um at the bulk. If the value of dividing one side by 3 was greater than 100 nm, the side was divided into a larger number. The limitations were that temperature was applied to the top of surface, both of ends were infinitely long, each layer was bonded, and contact surface was saw-edged shape.

Figure 13 shows the analysis of stress distribution of polyimide to surface roughness after O_2 plasma treatment. The highest stress value is located near the edge

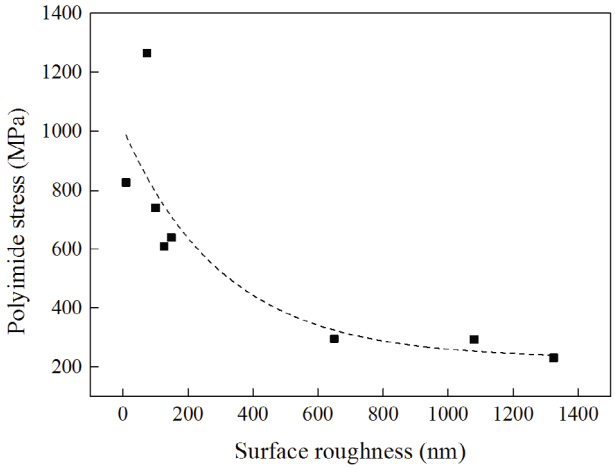

Fig. 15. The change of polyimide stress vs. surface roughness.

of the metals. This is a result of applying the limitation that the adhesion is strong enough and that the failure does not occur between the metal and the polyimide. As the roughness of the polyimide surface increases, the phenomenon of spreading of the stress concentrated area is observed. The maximum stress values are 741 MPa, 296 MPa, 293 MPa, and 232 MPa at surface roughness of 100 nm, 650 nm, 1080 nm, and 1325 nm, respectively. This is an effect of increase in surface area where stress is induced.

Figure 14 shows the analysis of vertical directional deformation of polyimide to surface roughness after O_2 plasma treatment. The highest stress value is located in middle between the metals. In particular, it is observed that the maximum deformation is concentrated on the top of the polyimide peaks. As the surface roughness of the polyimide increases, the vertical directional deformation of polyimide increases. The maximum stress values are 1.57 GPa, 0.55 GPa, 0.35 GPa, and 0.33 GPa at surface roughness of 100 nm, 650 nm, 1080 nm, and 1325 nm, respectively.

Figure 15 plots the stress distribution for various sur-

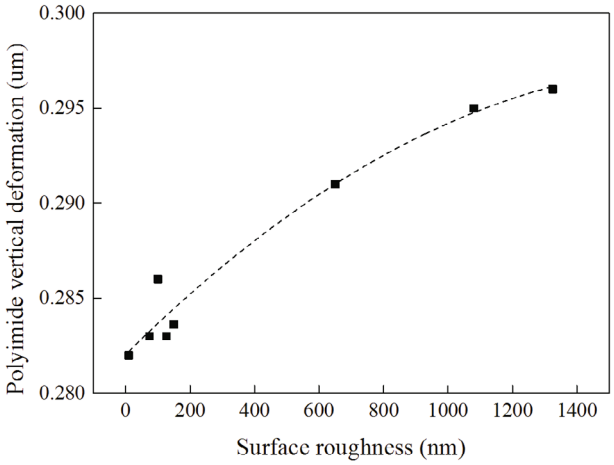

Fig. 16. The change of vertical directional deformation of polyimide vs. surface roughness.

face roughness of polyimide. The stress values represent an exponential decay relationship for surface roughness. The same tendency is observed in the area where the actual O_2 plasma treatment is evaluated, under 600 seconds. According to the simulation results, it may be advantageous to increase the surface roughness.

Figure 16 shows the vertical directional deformation of the polyimide at various surface roughness. The vertical directional deformation value represents a quadratic polynomial relationship to the surface roughness. In low surface roughness area, there are also outliers, but overall trend matches. The larger the vertical directional deformation value means that failure due to deformation may occur, so the longer the O_2 plasma processing time can be the disadvantage.

IV. CONCLUSION

Low pressure O_2 plasma treatment was conducted to investigate the increasing adhesion of metal-polymer interfaces which is one of the major issues in flexible printed circuit boards. It was confirmed that O_2 plasma treatment could control the roughness of the polyimide surface and that the adhesion of the metal and polyimide interfaces was improved through the ball shear test. Interpreting the FT-IR spectra for the C−O bonging accurately described the mechanism for the chemically enhancing adhesion of metal-polymer interface. The Cole-Cole plot showed that the change of polyimide by O_2 plasma treatment did not significantly affect the mechanical properties of the polyimide. The finite element method was used to confirm that the improvement of adhesion using low pressure O_2 plasma treatment could give simultaneously different advantages and disadvantages to polyimide stress and vertical directional deformation of polyimide.

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