

Improved Adhesion Between Copper and Prepreg Using Plasma Press Process

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ABSTRACT

For the various next generation flexible devices such as wearable devices, implantable devices, etc., adhesion strength between different surfaces is very important for the reliable operation of the devices. In this study, to improve the adhesion strength between metal (Cu) and polymer substrate (prepreg), a plasma press process, where two surfaces are hot pressed together while generating a plasma between them, were used and the effect of plasma press method on the adhesion strength between prepreg and Cu were investigated and the results were compared with a conventional hot press method. When the prepreg surface was treated with a plasma using He/N₂/O₂ or He/O₂ before hot press, the adhesion strength was decreased compared to the hot press without previous plasma treatment due to the hardening of the prepreg surface before the hot press. In contrast, when the Cu surface was treated with a plasma using He/N₂/O₂ or He/O₂ before hot press, the adhesion strength was increased about 20% compared to the hot press only due to the formation hydroxyl group and Cu²⁺ on the Cu surface before the hot press. However, when the prepreg and Cu were plasma pressed together, the use of He/O₂ and He/N₂/O₂ improved the adhesion strength significantly about 35% and 75%, respectively, compared to the hot press only due to the activation of the prepreg surface and Cu surface during the hot press by forming dangling bonds on the surfaces in addition to the formation of hydroxyl group and oxygen bonding on the surfaces.

KEYWORDS: Plasma Press, Prepreg, Adhesion, He/O₂/N₂.

1. INTRODUCTION

For the next generation flexible electronics such as wearable and attachable electronics, one of the important issues to be resolved is the strong adhesion among different component layers attached in the multilayer flexible printed circuit board (PCB), where, various semiconductor/functional devices are embedded and connected in the multilayer flexible substrates.^{1,2} For the adhesion of component layers on the multilayer flexible PCB, various methods such as hot pressing method, thermal curing method, and ultraviolet (UV) curing method are currently employed.^{3–5} However, due to the scaling down of the components, more improved adhesion properties between the interfaces are required. In addition, for the hot press method and hot curing methods, processing rate is slow and materials can be easily deformed when a high temperature is used during the processing, therefore, a low temperature

press processing has to be investigated especially for flexible electronics. In the case of the UV curing method, a low temperature processing and high rate processing are possible but the high cost of UV hardening materials and high cost of ownership for UV curing equipment need to be resolved. Among these methods, the hot press method is the most widely used for the multilayer flexible PCB.

For the formation of flexible multilayer PCB by the hot press method, prepreg, which is a flexible weaved fiber (such as glass fiber) containing polymer substrate with an adhesive material on the surface, is generally used. It is used as an insulating layer between two component layers and to provide the adhesion between the microelectronic component layers composing the multilayer PCB by the curing of the resin during the hot press.^{6–10} On the surfaces of the component layers, a patterned copper foil is located for the internal wiring of the microelectronic devices attached on the component layer and a several component layers can be pressed together with the prepreg layers located between the component layers for the required flexible multilayer PCB. However, after the hot press of the multilayer PCB, due to the different properties of the

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material surfaces and/or due to the impurities on the materials, strong adhesion between the component layers may not be obtained or, in some areas, the interface can be easily peeled off.^{11–13}

In this study, to improve the adhesion properties between prepreg and a component layer during the hot press, a plasma press method, where, two surfaces are hot pressed together while a plasma is turned on between the two surfaces, was investigated. Generally, to improve the adhesion between different materials further, a plasma treatment method has been generally applied before the adhesion processing for activation of surfaces as pretreatment methods not during the processing.^{14–17} The difference between the plasma press and the previous plasma treatment method is that adhesion process is conducted with the plasma-on for the plasma press not after the plasma treatment. The adhesion process after the plasma treatment can contaminate the plasma treated material surfaces during the transfer from the plasma chamber to hot pressing chamber for adhesion process and the material surfaces to be bonded contain no dangling bonds. By using the plasma press method, the adhesion process can be

conducted with activated surfaces with dangling bonds and at variously activated gas conditions which could change the interface properties significantly. Therefore, in this study, the effect of plasma press was also compared with a conventional hot press after the plasma treatment. Especially, the effects of plasma press using different plasma gas combinations on the adhesion properties between the Prepreg and the copper foil on the component layer were investigated and the results were compared with the conventional hot press with/without a post plasma treatment conducted at the same temperature.

2. EXPERIMENTAL DETAILS

2.1. Plasma Press Module

The plasma press system used in the experiment is shown in Figure 1. The plasma press module was composed of a top electrode and a bottom electrode made of 300 μm thick Al_2O_3 covered aluminum and was located in a sealed vacuum chamber. Both electrodes were heated to 180 $^\circ\text{C}$ using an oil heater (LAUDA PROLINE P5) to press two materials at a heated temperature. The top electrode was

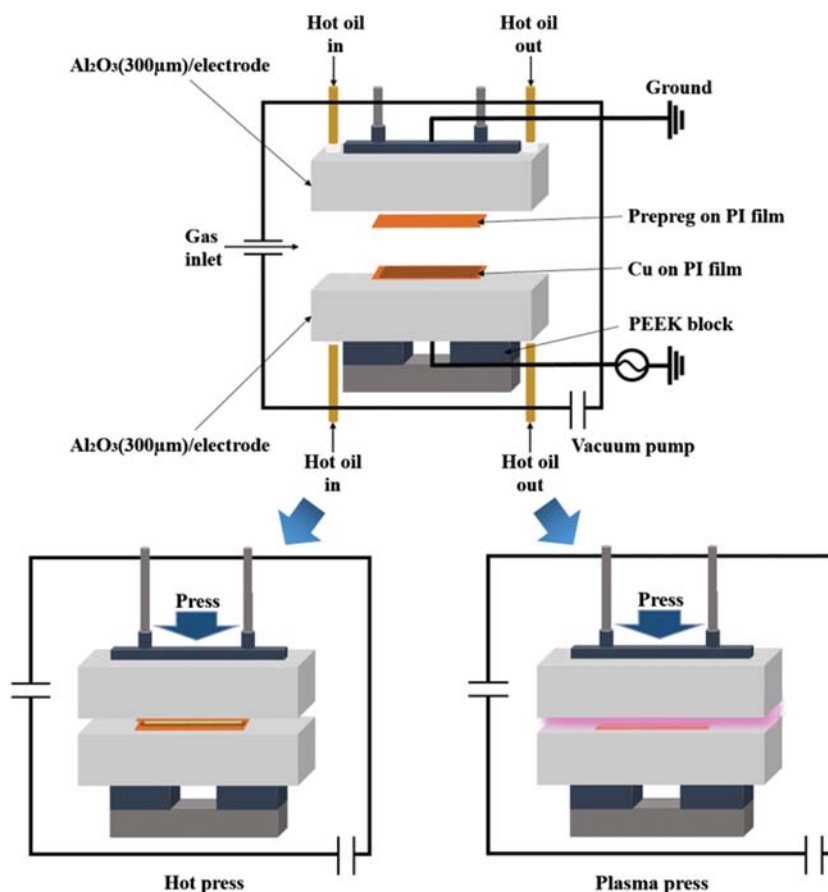


Fig. 1. Schematic diagrams of the plasma press system used in the experiment and the operation as a hot press process or a plasma press process.

connected to a pressing machine while the bottom electrode was located on a load cell to press and measure the load during the pressing. Polyether ether ketone (PEEK) block was used to connect the pressing machine with the top electrode, and also to connect the bottom electrode with the load cell while insulating the pressing machine and load cell from the electrodes and to prevent arcing during the plasma pressing. An additional 1 mm thick Al_2O_3 plate was located on the bottom electrode. Before the plasma pressing, the gap between the top electrode and bottom electrode was kept at 2.5 mm. Prepreg film located on a polyimide (PI) film was attached on the top electrode surface and a copper film located on a PI film was located on the bottom electrode surface while flowing hot oil on both electrodes.

2.2. Hot Press and Plasma Press

For the hot press process, as shown in the lower left figure of Figure 1, two surfaces were pressed by pressing the top electrode using the pressing machine until the pressure is reached to 2.5 kg/cm^2 . The samples were hot pressed for 450 sec. For the comparison with the plasma press process, the pressure in the chamber during hot pressing was maintained at 200 Torr.

For the plasma press process, a similar condition as the hot press process condition was used except for a plasma generation between the two electrodes during the hot pressing. The pressure of the system was maintained at 200 Torr and, as the process gas combination, gas mixtures of He, O_2 , N_2 , SF_6 were used. As the process gas mixtures, $\text{He}(7\text{slm})/\text{O}_2(2\text{slm})$, $\text{He}(7\text{slm})/\text{N}_2(2\text{slm})$, $\text{He}(7\text{slm})/\text{N}_2(2\text{slm})/\text{O}_2(2\text{slm})$, and $\text{He}(7\text{slm})/\text{SF}_6(1\text{slm})$ were used. For plasma generation, the AC voltage of 60 kHz 2.7 kV was connected to the bottom electrode while the top electrode was grounded. Total plasma on-time was maintained for 25 sec.; the plasma was turned-on for 10 sec. while keeping the electrode gap at 2.5 mm and, after the 10 sec., the gap was continuously decreased to the contact position for 5 sec. while the plasma was turned on. After the two surfaces were in contact, the voltage to the bottom electrode was turned off and 2.5 kg/cm^2 of pressure was applied to the sample for 450 sec.

2.3. Analysis and Measurements

To measure the adhesion strength between the two surfaces after the press, a tensile strength tester (MECMESIN, Multi test 1-i) was used. A peel-off method was used to measure the adhesion strength by preparing the pressed sample with 25 mm width and by peeling off the two surfaces at the speed of 1 inch/min. The surfaces of prepreg film and Cu film before and after the plasma treatments were observed with a scanning electron microscope (FE-SEM: HITACHI, s-4700). The contact angles of prepreg film surface and Cu film surface before and

after the plasma treatments were measured using a contact angle analyzer (SEO, phoenix 450). The surface compositions and chemical binding states were investigated using X-ray photoelectron spectroscopy (XPS: thermo VG SIGMA PROBE).

3. RESULTS AND DISCUSSION

To investigate the effect of the plasma treatment during the pressing for bonding, the surfaces of Cu film and prepreg film were studied using XPS, SEM, and contact angle measurement before and after the plasma treatment using various gas mixtures composed of, $\text{He}(7\text{slm})/\text{N}_2(2\text{slm})$, $\text{He}(7\text{slm})/\text{N}_2(2\text{slm})/\text{O}_2(2\text{slm})$, $\text{He}(7\text{slm})/\text{O}_2(2\text{slm})$, and $\text{He}(7\text{slm})/\text{SF}_6(1\text{slm})$ before the hot pressing.

Table I shows the surface compositions of the prepreg film measured using XPS before and after various plasma treatments using He/N_2 , He/O_2 , $\text{He}/\text{N}_2/\text{O}_2$, He/SF_6 . For the plasma treatments, the AC voltage of 60 kHz 2.7 kV was applied to the bottom electrode and the surface was plasma treated for 25 sec. As shown in the table, after the He/O_2 plasma treatment, the oxygen percentage was increased from 16.85 to 49.16%. When the prepreg was plasma treated with He/N_2 , the nitrogen percentage was increased from 3.19 to 6.19% but oxygen percentage was also increased to 44.64% due to the air exposure after the He/N_2 plasma treatment at the temperature of 180°C .¹⁸ The use of $\text{He}/\text{N}_2/\text{O}_2$ plasma exhibited the middle range of oxygen and nitrogen percentages by showing the oxygen percentage of 46.12% and the nitrogen percentage of 4.77%. The use of He/SF_6 plasma treatment showed the fluorine percentage of 29.59% due to fluorine plasma in addition to the lowest oxygen and nitrogen percentages among the prepreg surfaces treated with various plasmas.

We also investigated the surface composition of Cu film before and after the plasma treatments with various gas mixtures. When the Cu film surfaces were treated with various plasmas used in this experiment, no significant changes in the surface composition could be identified except for He/SF_6 due to fluorine in the plasma (not shown). However, the chemical binding states of Cu film

Table I. Surface compositions of the prepreg film after the various plasma treatment using He/N_2 , He/O_2 , $\text{He}/\text{N}_2/\text{O}_2$, and He/SF_6 measured by XPS. The surface composition of the prepreg film before the plasma treatment was also shown. For the plasma treatments, the AC voltage of 60 kHz 2.7 kV was applied to the bottom electrode and the surface was plasma treated for 25 sec.

| | Atm. % | | | | |
|---|--------|------------------------|-----------------------------------|------------------------|-------------------------|
| | As-is | He/N_2 | $\text{He}/\text{N}_2/\text{O}_2$ | He/O_2 | He/SF_6 |
| C | 79.96 | 48.44 | 49.11 | 46.41 | 39.6 |
| N | 3.19 | 6.29 | 4.77 | 4.43 | 2.61 |
| O | 16.85 | 44.64 | 46.12 | 49.16 | 28.21 |
| F | – | – | – | – | 29.59 |

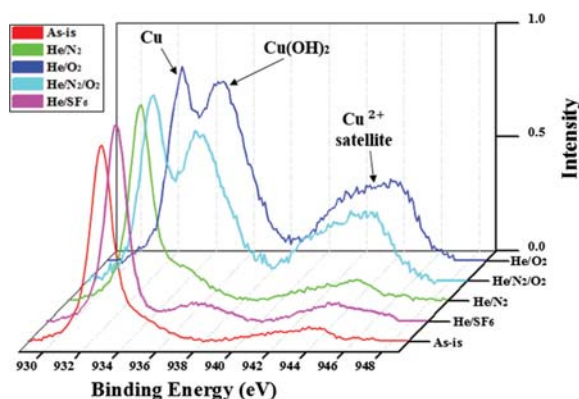


Fig. 2. XPS narrow scan data of Cu film surface before and after the various plasma treatments using He/N₂, He/O₂, He/N₂/O₂, and He/SF₆. The plasma treatment conditions are as in Table I.

surfaces were varied after the various plasma treatments and the results are shown in Figure 2 and the percentages of the chemical binding states of Cu in Figure 2 were summarized in Table II. The plasma treatment conditions are as in Table I. As shown in Figure 2, after the plasma treatments with He/O₂ and He/N₂/O₂, significant chemical binding peaks of Cu at 934.6 eV related to Cu(OH)₂ and at 942~946 eV related to Cu²⁺ (CuO_x, CuF_x, etc.) were observed in addition to the peak at 932 eV related to Cu metal itself.^{19–20} For the He/SF₆ plasma treated Cu, the chemical binding states of Cu were similar to the as-is. As shown in Table II, after the plasma treatment with He/N₂/O₂, the Cu surface exhibited the highest Cu(OH)₂ binding state while the Cu surface treated with He/O₂ plasma exhibited the highest Cu²⁺ binding states related to CuO_x on the Cu surface. The Cu surface treated with He/SF₆ exhibited higher Cu²⁺ but it was related to CuF_x not CuO_x. It is known that, in general, Cu exhibits higher adhesion with other materials when the Cu surface contains chemical bindings of Cu(OH)₂ and CuO_x.²¹

Contact angles on prepreg surface and Cu surface measured before and after the various plasma treatments are shown in Figures 3(a and b), respectively. The plasma treatment conditions are as in Table I. The contact angles on the as-is prepreg and as-is Cu were as high as 73~79°, but after the plasma treatments using He/N₂, He/N₂/O₂,

Table II. Percentages of chemical binding states with Cu on Cu film surfaces measured by XPS before and after the various plasma treatments. The plasma treatment conditions are as in Table I.

| | % of chemical binding states | | |
|-----------------------------------|------------------------------|---------------------|----------------------------|
| | Cu | Cu(OH) ₂ | Cu ²⁺ satellite |
| He/O ₂ | 30.73 | 34.20 | 35.07 |
| He/N ₂ /O ₂ | 23.09 | 45.96 | 30.95 |
| He/N ₂ | 73.52 | 13.24 | 13.24 |
| He/SF ₆ | 78.55 | – | 21.45 |
| As-is | 75.65 | 16.39 | 7.96 |

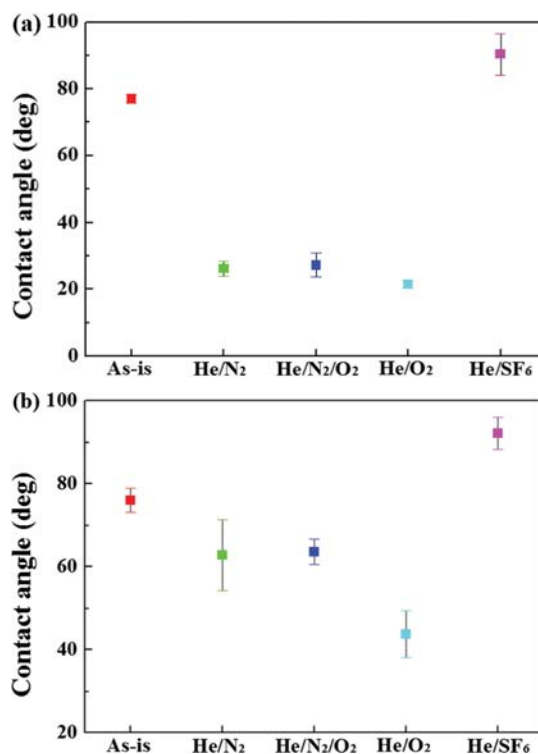


Fig. 3. Contact angle measured on (a) prepreg surface and (b) Cu surface before and after the plasma treatments using He/N₂, He/O₂, He/N₂/O₂, and He/SF₆. The plasma treatment conditions are as in Table I.

and He/O₂, the contact angles were decreased due to the formation of hydroxyl group and oxygen bonding on the surface, therefore, hydrophilic surfaces were obtained. Among the plasma treatments using He/N₂, He/N₂/O₂, and He/O₂, He/O₂, the prepreg surface and Cu surface treated by He/O₂ plasma exhibited the lowest contact angle. In addition, the prepreg surface showed the lower contact angle (21~27°) after the plasma treatments by He/N₂, He/N₂/O₂, and He/O₂ compared to the Cu surface (42~62°). However, when the prepreg surface and Cu surface were treated with a He/SF₆ plasma, the contact angles were increased to 90~92°, therefore, hydrophobic surfaces were obtained for both prepreg and Cu possibly due to the fluorization of material surfaces. In general, the contact angle is related to the adhesion strength and the surfaces with lower contact angle exhibit the higher adhesion strength after the bonding.^{22–25}

The surfaces of prepreg and Cu before and after the plasma treatments using He/N₂, He/O₂, He/N₂/O₂, He/SF₆ were observed by SEM and the results are shown in Figures 4 and 5, respectively. The plasma treatment conditions are as in Table I. As references, SEM images of the prepreg surface and Cu surface exposed to the hot press condition were included. Figure 4(a) is as-is prepreg surface and Figure 4(b) is the prepreg surface after exposing in the hot press condition. Figures 4(c–f) are the

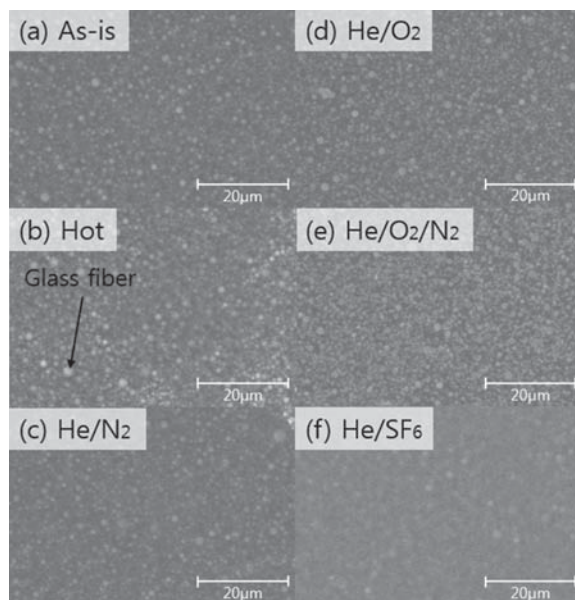


Fig. 4. SEM images of prepreg surfaces before and after the plasma treatments using He/N₂, He/O₂, He/N₂/O₂, and He/SF₆. As a reference, the prepreg surface exposed to the hot press condition was included. (a) As-is, (b) hot press condition, (c) He/N₂, (d) He/O₂, (e) He/N₂/O₂, and (f) He/SF₆. The plasma treatment conditions are as in Table I.

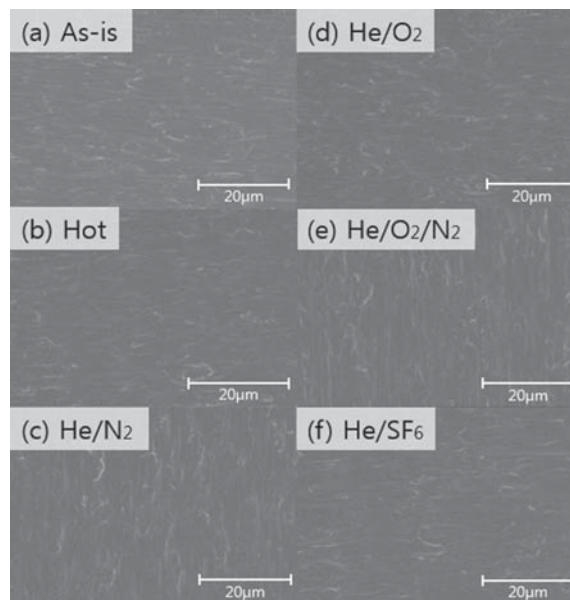


Fig. 5. SEM images of Cu surfaces before and after the plasma treatments using He/N₂, He/O₂, He/N₂/O₂, and He/SF₆. As a reference, the prepreg surface exposed to the hot press condition was included. (a) As-is, (b) hot press condition, (c) He/N₂, (d) He/O₂, (e) He/N₂/O₂, and (f) He/SF₆. The plasma treatment conditions are as in Table I.

prepreg surfaces after the plasma treatment using He/N₂, He/O₂, He/N₂/O₂, and He/SF₆, respectively. As shown in the figures, after exposure to hot press condition and various plasmas using He/N₂/O₂, and He/O₂, the prepreg surfaces showed more white dots related to glass fibers in the prepreg than as-is surface which appeared to be related to the hardening of prepreg surface^{26–28} while the prepreg surface exposed to the He/SF₆ plasma and He/N₂ appeared to be similar to as-is surface. As shown in Figure 5, in the case of Cu surfaces, the surface morphologies observed (c~f) after the plasma treatments and (b) after exposing hot press condition were similar to (a) the as-is surface, therefore, no significant change of the Cu surface could be observed after the various treatments.

The adhesion strengths of prepreg/Cu interfaces after the bonding using hot press and plasma press using various gas mixtures were investigated and the results are shown in Figure 6. To compare the plasma press with the hot press after the plasma treatments, the prepreg and Cu were hot pressed after (not during) plasma treatments of prepreg or Cu using the various gases. The adhesion strengths of prepreg/Cu interfaces which were hot pressed after the plasma treatments of prepreg surface and Cu surface are shown in Figures 6(a and b), respectively. The adhesion strengths of prepreg/Cu interfaces after the bonding using plasma press using various gas mixtures are shown in Figure 6(c). Also, the adhesion strength of prepreg/Cu interface after the bonding using hot press only without plasma treatment was included. As shown

in Figure 6(a), the adhesion strengths of prepreg/Cu interfaces which were hot press after the plasma treatments of prepreg surfaces using various plasmas were lower than that of prepreg/Cu interface which was hot pressed without any plasma treatment. Even though the prepreg surfaces became hydrophilic surfaces after the plasma treatments using He/N₂, He/N₂/O₂, and He/O₂, due to the lower hydroxyl group and Cu²⁺ for He/N₂ and due to the prepreg surface hardening for He/N₂/O₂ and He/O₂, the adhesion strengths of the prepreg/Cu interfaces which were hot presses after the plasma treatments using He/N₂, He/N₂/O₂, and He/O₂ were lower than that were hot pressed without treatment. In the case of prepreg surface treated using a He/SF₆ plasma, due to the high contact angle caused by surface fluorination, the adhesion strength of prepreg/Cu interface was the lowest.

For the adhesion strengths of prepreg/Cu interfaces which were hot press after the plasma treatments of Cu surfaces using various plasmas, as shown in Figure 6(b), the adhesion strengths of prepreg/Cu interfaces treated with He/O₂ and He/N₂/O₂ were higher about 20% than that of prepreg/Cu interface which was hot pressed without any plasma treatment. The higher adhesion strengths of prepreg/Cu interfaces which were hot pressed after the plasma treatment of Cu using He/O₂ and He/N₂/O₂ are related to the high Cu(OH)₂ and Cu²⁺ formed on the Cu surface before the hot press. In the case of prepreg/Cu interface hot pressed after the plasma treatment of Cu surface with He/N₂, no significant change

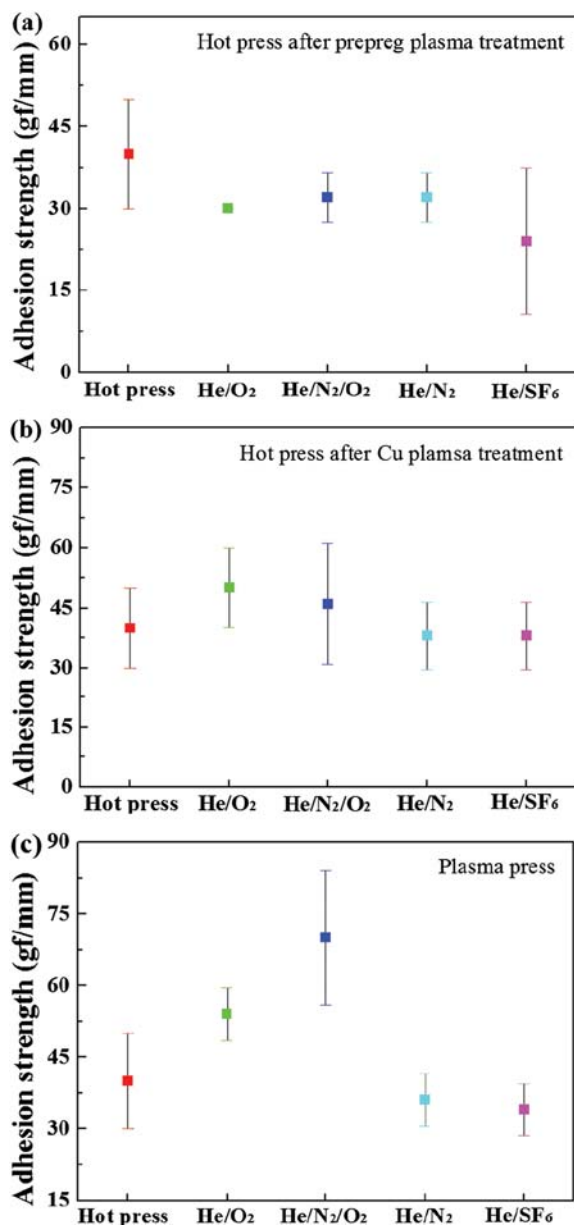


Fig. 6. Adhesion strengths of prepreg/Cu interface after bonding using hot press and plasma treatments using He/N₂, He/O₂, He/N₂/O₂, and He/SF₆. The prepreg and Cu were bonded (a) by hot press after the plasma treatments of prepreg surface, (b) by hot press after plasma treatments of Cu surface, and (c) by plasma press during plasma treatments.

of adhesion strength compared with that was hot pressed without any plasma treatment was obtained possibly due to no formation of Cu(OH)₂ and CuO_x on the surface. In the case of prepreg/Cu interface hot pressed after the plasma treatment of Cu surface with He/SF₆, due to the high contact angle of Cu caused by a fluorinated surface, no significant change of adhesion strength was also obtained.

As shown in Figure 6(c), in the case of plasma press with He/SF₆ and He/N₂, due to the fluorination of the surface and no change of Cu(OH)₂ and CuO_x on the Cu surface (in addition to no significant change in nitrogen content which could increase the adhesion, too as reported by other researchers^{29–31}), respectively, no improvement of adhesion strength was observed by plasma press similar to the hot pressed after plasma treatments with He/SF₆ and He/N₂. However, in the case of prepreg/Cu interfaces which were plasma pressed using various plasmas, the adhesion strengths of prepreg/Cu interfaces formed during plasma pressing using He/O₂ and He/N₂/O₂ were increased significantly about 35% and 75%, respectively, compared to that of prepreg/Cu interface which was hot pressed without any plasma treatment. For the plasma press, that is, when the plasma is on during the hot press of prepreg and Cu, the prepreg surface and Cu surface are bombarded by ions in addition to the modification of the surfaces by the reactive gas radicals formed by the plasma. The prepreg surface and Cu surface are activated by forming dangling bonds on the surfaces in addition to formation of hydroxyl group and oxygen bonding on the surfaces as observed after the plasma exposure using He/O₂ and He/N₂/O₂ without hardening or changing the surfaces. Therefore, due to the strong atomic bonding in addition to the hydroxyl group and oxygen bonding on the surfaces, very high adhesion strengths could be obtained for the plasma press.

4. CONCLUSION

In this study, to improve the adhesion strength of a multilayer flexible PCB formed by bonding of prepreg film and Cu film, the effect of plasma press method, where the prepreg and Cu are hot pressed together while generating a plasma between them, were investigated in addition to the conventional hot press method. Also, the effect of various gas mixtures such as He/N₂, He/N₂/O₂, and He/O₂ for the plasma press on the adhesion strength was investigated. When the prepreg surface was treated using various plasmas before hot press of prepreg and Cu, the use of He/N₂/O₂ and He/O₂ increased the oxygen percentage on the prepreg surface and the contact angle of prepreg surface was decreased significantly which are required conditions for high adhesion strength. However, due to the hardening of prepreg surface before the hot press, the adhesion strength was decreased compared to the hot press without previous plasma treatment. When the Cu surface was treated using various plasmas before hot press of prepreg and Cu, the use of He/O₂ and He/N₂/O₂ improved the adhesion strength about 20% due to the formation of Cu(OH)₂ and Cu²⁺ on the Cu surface and by decreasing the contact angle while showing no change of Cu surface before hot press. In contrast, when the prepreg and Cu were plasma pressed, the use of He/O₂ and He/N₂/O₂ improved the adhesion strength significantly about 35% and 75% compared to the hot press only.

In the case of He/N₂ and He/SF₆, due to the no significant reaction with the surface and due to the formation of hydrophobic fluorinated surfaces, no improvement of adhesion strengths were obtained for both plasma press and hot press after the plasma treatment. It is believed that, using the plasma press, significantly higher adhesion strength can be achieved between various materials by plasma press compared to conventional hot press if adequate plasma gas mixtures are used during the plasma press.

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