Residual stress control of Cu film deposited using a pulsed direct current magnetron sputtering

Tae Hyung Kim\textsuperscript{a}, Soo Jung Lee\textsuperscript{b}, Do Han Kim\textsuperscript{a}, Dong Woo Kim\textsuperscript{c}, Jeong Woon Bae\textsuperscript{d}, Kyong Nam Kim\textsuperscript{c}, Yong Mo Kim\textsuperscript{d}, Geun Young Yeom\textsuperscript{a,b,}\textsuperscript{\*}

\textsuperscript{a} Department of Materials Science and Engineering, Sungkyunkwan University, Suwon, Gyeonggi-Do 440-746, South Korea
\textsuperscript{b} SKKU Advanced Institute of Nano Technology (SAINT), Sungkyunkwan University, Suwon, Gyeonggi-Do 440-746, South Korea
\textsuperscript{c} Department of Advanced Materials, Daejeon University, 62 Daehak-ro, Dong-gu, Daejeon 300-716, Republic of Korea
\textsuperscript{d} STC Division, Korea Instrument Co., Ltd., Ibhaseong-4, Geongjeo-Do 18487, Republic of Korea

Abstract

Residual stress of 10 \( \mu \)m thick copper (Cu) film deposited using direct current (DC) magnetron sputtering system was analyzed for various pulsed DC power conditions. It is found that the increase of pulse frequency at a fixed duty ratio of 80\% from continuous wave (CW) to 100 kHz while maintaining the time average power significantly decreased the residual stress of Cu film from 21.75 MPa to 1.8 MPa. The decrease of pulse duty ratio further decreased the residual stress by showing 0.7 MPa at 60\% of duty ratio and 50 kHz pulse frequency. Therefore, compared to CW power, Cu film with \(-1/30\) lower residual stress could be deposited by using DC pulse power. The decrease of residual stress by pulsing and by decreasing the pulse duty ratio while keeping the same time average power was related to the denser film density and smaller grain size of deposited Cu film caused by increased instant sputter power during the pulsing.

1. Introduction

Heat dissipation of electronic devices is one of the most important reliability issues in manufacturing high performance electronics. Many researchers have investigated various methods in improving heat dissipation efficiency on various electric devices such as high power light emitting diodes and batteries because of the heat generated by high power consumption [1–3]. Also, many different methods have been proposed to remove the heat generated by electric devices [4,5]. However, due to the increased integration of the devices, more efficient cooling method is needed for better thermal management in using high power and highly integrated electronics, therefore, other techniques in improving the heat dissipation efficiency such as the application of carbon nanotube in polymer matrices [6–8], formation of a water-flow structure [9,10], formation of patterned fin or rod structure [11], etc. have been also studied.

As heat sink materials, copper (Cu) has been the most widely used material due to low cost and high heat dissipation efficiency. Generally, to deposit Cu at a high deposition rate on the substrate as the heat sink material, the electroplating method has been used. However, this method uses hazardous solutions, therefore, it can lead to environmental pollution during the waste water treatment. In addition, Cu deposited by the electroplating method tends to exhibit high residual stress, therefore, it may not be able to function as a heat sink material especially for a thick Cu film due to a high stress difference between Cu and substrate.

Magnetron sputter deposition has been used to deposit various thin and thick film materials including Cu films due to its high deposition rate, large area deposition, and ability to deposit most of materials. To improve the heat dissipation, a Cu film thicker than a few \( \mu \)m is required, and, when a thick Cu film is deposited by the magnetron sputtering, a failure of the devices such as bending of the substrate, detachment of Cu film from the substrate, etc. is observed due to the stress developed in the film. Recently, many researchers are investigating a pulsed power during the magnetron sputtering of various materials by varying pulse duty ratio or pulse frequency to control the plasma properties during the deposition on the substrates. By controlling the plasma properties through the pulsed magnetron sputtering, less target damage at the same power density and more uniform composition and higher step coverage of the deposited film have been obtained compared to the conventional direct current (DC) magnetron sputtering [12–18]. However, it is found that the control of residual stress of deposited metal film by using pulsed magnetron sputtering has not been investigated.

\* Corresponding author at: Department of Materials Science and Engineering, Sungkyunkwan University, Suwon, Gyeonggi-Do 440-746, South Korea.
E-mail address: gyyeom@skku.edu (G.Y. Yeom).

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In this study, pulsed DC magnetron sputtering has been used to investigate the possible management of the residual stress in the deposited Cu film. The pulse frequency and pulse duty ratio of DC magnetron sputter power supply were varied while maintaining the time average power to control the residual stress of the Cu film. To study the mechanism related to the change of residual stress, the temporal change of voltage and current of the DC magnetron power, the change of ion energy on the substrate surface, and the microstructure of the deposited Cu film were investigated.

2. Experimental set-up

Fig. 1 shows the pulsed DC magnetron sputtering system used in the experiment. The size of the magnetron target was 3 in. diameter and the distance between the target and the substrate was ~10 cm. The base pressure was maintained < 1.3 × 10⁻³ Pa by pumping using a turbo-molecular pump (OSAKA VACUUM · TH520). A pulsed DC power supply (ENI, RPG-100) which can operate at pulse frequency of 150 kHz and at 2 kW was connected to the magnetron sputter target.

The sputtering was conducted at 0.67 Pa Ar and 300 W of time average power and, before deposition on the substrate, the sputter target was pre-sputtered for 5 min to clean the target surface and, during the deposition, the substrate maintained at room temperature was rotated at 15 rpm. As the sputter target, 3 in. diameter 99.99% pure Cu target (oxygen-free high-conductivity copper) was used and, as the substrate, 300 μm thick silicon wafers were used. Before the deposition, the silicon wafers were cleaned in sequence with acetone, icosapentaenoic acid, and deionized water in a ultrasonic cleaner for 15 min each. The thickness of Cu film deposited on the silicon wafer was maintained at ~10 μm.

The residual stress formed on the Cu film deposited silicon wafer was measured using a residual stress tester (JLCST022, J & L tech). This residual stress measurement device is measured by the method of the radius curvature stress. The laser specification used in this measurement is 1.0 mW of output power, 632.8 nm of wavelength, and 0.59 mm of beam diameter. Also, the lens shapes a plano-convex lens, and the laser spot was moved 25 mm for stress measuring from the center to the end on deposited film. Since the substrate is much thicker than the deposited Cu thin film, the residual stress σ of the film can be obtained using the following equation [19].

\[
\sigma = \frac{1}{6R} \left[ \frac{E_s}{(1 - \nu_s)} \right] \frac{f^2}{s}
\]

where \(E_s\) is the Young's modulus of the substrate, \(\nu_s\) is the Poisson's ratio of the substrate, and \(1/R\) is the curvature of the substrate/deposited thin film composite, \(s\) is the thickness of substrate and \(f\) is the thickness of deposited thin film. This simple formula results from the following basic hypotheses: (a) strains and rotations are infinitesimal, (b) substrate and coating thicknesses are very small compared to the lateral dimensions of the plate and edges' effects are negligible, (c) coating thickness is small compared to substrate thickness \((f \ll s)\), and (d) substrate and coating materials are homogeneous, isotropic, and linear elastic. The experimental error of the residual stress measurement was within ±9.5%. And the morphology of the deposited Cu film was observed using a field emission SEM (S-4700, HITACHI) at 15 kV condition.

3. Results and discussion

3.1. Effect of pulse frequency

Cu film was deposited on silicon wafers as a function of pulse frequency (CW, 50 kHz, and 100 kHz) at 0.67 Pa Ar for 200 min while maintaining the time average power of 300 W. For the pulse frequency, the pulse duty ratio was kept at 80%. The residual stress and thickness of Cu film deposited on the silicon wafers as a function of pulse frequency are shown in Fig. 2. As shown in Fig. 2, due to the same time average power, the thickness of deposited Cu film was similar for different pulse frequency conditions including CW. However, as shown in Fig. 2, the increase of pulse frequency from CW to 100 kHz significantly decreased the residual stress of Cu film from 21.75 MPa to 1.8 MPa.

To understand the reason for the decrease of residual stress of Cu film with increasing the pulse frequency, the characteristics of temporal voltage and current on the Cu target during the DC pulse magnetron sputtering were investigated and the results are shown in Fig. 3a and b, respectively, for the same conditions in Fig. 2. When the pulse frequency is varied from DC (0 Hz) to 100 kHz at the same time average power, total ion flux (that is, dose) to the Cu target is not significantly changed, and, as shown in Fig. 3b, the time average current is similar for DC, 50 kHz and 100 kHz indicating the total ion flux to the target is similar for different pulse frequency conditions. However, as shown in Fig. 3a and b, the peak voltage/peak current was varied from ~330 V/0.91 A for DC to ~670 V/3.2 A for 100 kHz, therefore, higher peak voltage/peak current was observed for the higher pulse frequency condition. The higher peak voltage with the higher peak current indicates higher instant Cu flux to the substrate for the higher pulse...
frequency condition. The higher sputtering voltage is also known to increase the energy of Ar\textsuperscript+ bombardment during the deposition \cite{20,21}.

The Ar\textsuperscript+ ion energy distribution on the substrate during the sputtering conditions in Fig. 2 was measured using the PSM located near the substrate location as a function of pulse frequency and the results are shown in Fig. 4. As shown, with the increasing the pulse frequency from CW to 100 kHz, the high energy tail was shifted toward higher ion energy indicating higher Ar\textsuperscript+ bombardment to the substrate for higher pulse frequency condition. The increased instant atom flux from the target and the higher Ar\textsuperscript+ ion bombardment energy during the deposition are known to form smaller grains on the substrate \cite{22–25} and to increase the density of the deposited film by compacting effect \cite{26}, respectively.

3.2. Effect of pulse duty ratio

The effect of pulse duty ratio on the residual stress of Cu film deposited on the silicon substrate was investigated at 50 kHz pulse frequency and the results are shown in Fig. 5. The pulse duty ratio was varied from 60 to 90\% while maintaining other conditions are the same as in Fig. 2. As shown in Fig. 5, due to the same average power of 300 W to the target during the sputtering, the thickness of Cu on the substrate was similar in the range of 9.18–9.74 μm after the deposition of
200 min. However, the decrease of pulse duty ratio at the time same average power from 90 to 60% decreased the residual stress of the Cu film from 6.1 MPa to 0.7 MPa.

The characteristics of temporal voltage and current on the Cu target during the DC pulse magnetron sputtering were also investigated as a function of duty ratio at 50 kHz of pulse frequency and at 300 W of time average power and the results are shown in Fig. 5. As shown in Fig. 5, even though the decrease of grain size is not linearly proportional to the pulse frequency, the increase of pulse frequency decreased the grain size of Cu film deposited on the silicon substrate. The decrease of Cu grain size deposited on the substrate with increasing the pulse frequency at the same time average power is believed to be related to the instant high flux of Cu atoms by the instantly increased peak power voltage and current as shown in Fig. 3. The increased Ar+ ion bombardment on the substrate with increasing the pulse frequency also appears to increase the Cu film density deposited on the substrate.

### 3.3. Effect on Cu film growth

Fig. 8a, b, and c show the Cu film morphology observed using a field emission SEM for different pulse frequency of DC, 50 kHz, and 100 kHz, respectively, for the condition shown in Fig. 2. As shown in Fig. 8, even though the decrease of grain size is not linearly proportional to the pulse frequency, the increase of pulse frequency decreased the grain size of Cu film deposited on the silicon substrate. The decrease of Cu grain size deposited on the substrate with increasing the pulse frequency at the same time average power is believed to be related to the instant high flux of Cu atoms by the instantly increased peak power voltage and current as shown in Fig. 3. The increased Ar+ ion bombardment on the substrate with increasing the pulse frequency also appears to increase the Cu film density deposited on the substrate.

It is investigated that the smaller grain size tends to exhibit lower residual stress of the film due to smaller differences in the growing grains in the film [27,28]. Also, the high flux and the high ion energy generated by pulsed power are expected to suppress tensile stress and lead to compressive stress in the film. These causes may also have
affected pulsed plasma lowering stress [29]. Therefore, it is believed that the lower residual stress obtained by the higher pulse frequency and lower pulse duty ratio in Figs. 2 and 5, respectively, is related to the smaller grain size of Cu film and the increased film density by the higher Ar+ ion bombardment. In some cases, however, research has also been reported on the tendency of stress to decrease as the grain size increases. They reported that diffusion of atoms into grain boundaries and grain boundary densification are reasons for this phenomenon [29,30].

4. Conclusion

10μm thick Cu film was deposited on the silicon substrate by DC pulse magnetron sputtering, and the effect of pulse frequency and pulse duty ratio on the residual stress of the deposited Cu film was investigated. The higher pulse frequency and the lower pulse duty ratio of the pulse power at the same average power decreased the residual stress of the deposited Cu film. It is believed that the lower residual stress of the deposited Cu film is related to the smaller grain size and higher film density of the deposited Cu film caused by the increased nucleation and increased Ar+ ion bombardment on the substrate, respectively. The increased Cu nucleation at the higher pulse frequency and lower pulse duty ratio is believed to be from higher Cu atom flux to the substrate by instant higher voltage and higher current of sputtering power. By depositing Cu film with the pulse power composed of 50 kHz of pulse frequency and 60% pulse duty ratio, the significantly lower Cu residual stress of 0.7 MPa compared to 21.7 MPa of the Cu film deposited with DC power could be obtained. It is believed that, by using the higher pulse frequency with the lower pulse duty ratio, the further decreased residual film stress film can be obtained not only for Cu film but also for other metal films.

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References