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## Research Article

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# Mechanical and Interfacial Properties Characterization of Physical Vapor Deposited Metallic Films after Rapid Thermal Annealing

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**Abstract** – Physical vapor deposited metallic films have been widely used in semiconductor and microsystem applications. Their mechanical properties and interfacial behavior are vital for device longevity assessment and are strongly related to related processing and subsequent annealing parameters. In this work, the influence of RTA temperature on material properties and interface behavior of both aluminum and copper films with various deposition parameters are investigated through nanoindentation, nanoscratch, and adhesion tests, as well as XRD. Through these characterizations, a general assessment on processing parameters could be mapped and led to processing structural integrity optimization. Technically, it shows that RTA temperature indeed has strong influence on the mechanical behavior and strengths. Apparently, both elastic modulus and hardness decrease with RTA temperatures. On the other hand, the adhesion strength could be improved with proper RTA processing. Microscopically, RTA processing also effectively changes the microstructure and surface morphology of deposited film and the observed grain growth and surface hillocks are believed to be highly correlated with the measured properties and strength variations. It is believed that by integrating all characterization results, trade-offs on various design and performance concerns of metallic film deposition could be drawn can elucidated for future reliability engineering applications.

**Keywords** – metallic films, physical vapor deposition, rapid thermal annealing, mechanical characterization, interfacial behavior

## I. INTRODUCTION

Semiconductor devices are formed by a series of lithography, etching, and deposition cycles. Amonging the deposition processes involved, physical vapor deposition (PVD) for both evaporating and sputtering are vital for forming conductive metallic layers [1] such as aluminums and coppers. In addition, in recent years, dielectric films such as alumina ( $\text{Al}_2\text{O}_3$ ) and yttrium oxide ( $\text{Y}_2\text{O}_3$ ) have been widely used as coating layers of IC processing equipments such as CVD chambers [2], a major coating procedure is to perform highly vacuumed sputtering by simultaneous introducing metallic and oxygen sources under elevated temperatures. The reliability of coatings are thus critical for both device performance and equipment integrity.

From the device reliability perspective, the major concerns of the above films are the residual stress and interfacial toughness between themselves and their corresponding

substrates during fabrication and subsequent service. For example, defects such as hillocks, cracking, delamination, and device warping are direct consequences. In aluminum wires, microstructure migrations and internal defect evolutions are reported ranges from traditional IC devices [3,4,5] to the state-of-the-art IGBT power electronics [6]. On the other hand, for copper films, the current conductor layer materials for ICs, usually suffers from electromigration itself and adhesion with its associated substrates [5]. In addition, particulate forming on coating materials could also be attributed to similar causes and such circumstances are no doubt fatal for semiconductor processing equipment such as highly vacuumed chambers. In short, in addition to the main functionality such as electrical performance, mechanical characterization of those films should also be conducted for device longevity concerns. Furthermore, since those concerns are highly process parameter dependent, by means of such characterizations, its results could also serve as key consideration of process design optimization in coating process. To concentrate on focused themes, this work mainly concerns reliability issues related to PVD metallic films. For the concerns raised for dielectric coating, they are currently investigated. Similar investigations aim for dielectrics as thermal and chemical inert barriers of processing chamber coatings are currently underway.

The above thermo-mechanical concerns are mainly determined by deposition parameters. For example, the effect of deposition temperature of aluminum film to the resulting tensile stress and strength has been reported by Eiper et al [7]. The deposition thickness dependent residual stress on aluminum and copper films has also been frequently reported [8,9,10]. Meanwhile, post-deposition heat treatments could also partially adjust the results by means of modifying the microstructures and the resulted mechanical behavior such as stress state are thus changed due to stress relaxation. Effect of annealing temperature [11] and history [12] on the stress behavior of aluminum films has also been discussed.

For recovering desired electric performance, rapid thermal annealing (RTA) is frequently applied in modern semiconductor fabrication with the ability to provide necessary thermal annealing with minimum thermal budget [13]. However, just like other thermal processes, RTA process would certainly influence the microstructure of deposited films and consequently, key material behaviors and mechanical performance such as modulus, strength, toughness, residual stresses, and interfacial strength and the influence should be evaluated. Previously, the behavior of PECVD dielectric films

such as oxides and nitride after RTA have been systematically investigated [13,14]. However, such investigations are less reported in metallic conductive films.

Inspiring by the needs of post-deposition thermal processing and understanding the resulted mechanical behavior variation, it is desired to perform essential characterization to clarify the concern for process parameter optimization, the goal of this work is to perform essential mechanical characterization on interfacial properties of thin film structures after rapid thermal processing. To elucidate the possible applications of the characterization, both in flow and results, conventional aluminum and cooper films, would be hired to serve as the target materials for this investigation. Through the characterization procedure with domain knowledge integration, the observed results should provide technical insights, both for the materials currently examined or for future applications, in their related deposition and thermal annealing processing.

The rest of this paper presents the technical issues of the proposed accelerometer module in detail. In Section II, the specimen preparation and characterization flow are presented. The material properties such as modulus, hardness, and microstructures are characterized in Section III, followed by the residual stress characterization and interface behaviors addressed in Section IV and V, respectively. Essential discussions on the characterization results for elucidating the possible engineering applications are presented in Section VI and finally, Section VII concludes this work.

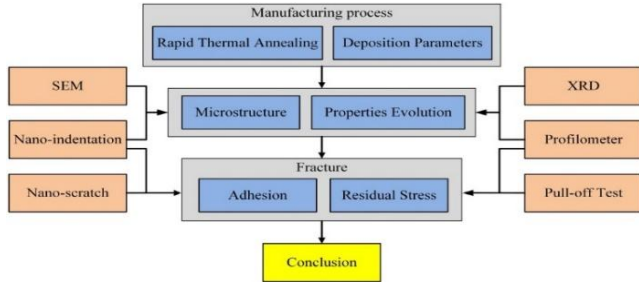


Fig. 1. Overall characterization flow

## II. SPECIMEN PREPARATION AND CHARACTERIZATION FLOW

Here the fabrication and the subsequent characterization flow is briefly addressed.

### A. Specimen prerapation

For conducting the planned investigations, metallic films are deposited on silicon wafers by NYCU nano-fabrication center (Hsin-Chu, Taiwan). Aluminum films deposited evaporation (ULVAC EBX-6D) with deposition rates from 4-8 (Å/s) and the achieved final thickness between 500 and 700 nm. Meanwhile, copper films are realized by using sputtering (Ion Tech Microvac 450CB) with rates of 0.4 to 0.8(Å/s) and thickness of 300-500 nm. After deposition, the films are then treated with additional RTA using different temperatures and

durations based on the experimental plan. Table 1 lists the essential parameters for deposition and specimens. The essential reported material properties of all materials involved in this work is then summarized in Table 2.

After deposition, all wafers are then sliced into strips with a width of 1 cm (mainly for curvature-based residual stress measurement) and die with a dimension of  $1 \times 1 \text{ cm}^2$  (for other destructive characterizations).

TABLE 1. DEPOSITION PARAMETERS OF FILMS

| Specimens | Vacuum (Torr)        | Temp. (°C) | Rate (Å/s) | Thickness (nm) | Power (W) |
|-----------|----------------------|------------|------------|----------------|-----------|
| Al-1      | $4 \times 10^{-6}$   | 45         | 4          | 500            | -         |
| Al-2      | $4 \times 10^{-6}$   | 45         | 6          | 500            | -         |
| Al-3      | $4 \times 10^{-6}$   | 45         | 8          | 500            | -         |
| Al-4      | $4 \times 10^{-6}$   | 45         | 6          | 600            | -         |
| Al-5      | $4 \times 10^{-6}$   | 45         | 6          | 700            | -         |
| Cu-1      | $7.6 \times 10^{-3}$ | 30         | 0.4        | 500            | -         |
| Cu-2      | $7.6 \times 10^{-3}$ | 30         | 0.6        | 500            | -         |
| Cu-3      | $7.6 \times 10^{-3}$ | 30         | 0.8        | 500            | -         |
| Cu-4      | $7.6 \times 10^{-3}$ | 30         | 0.6        | 300            | -         |
| Cu-5      | $7.6 \times 10^{-3}$ | 30         | 0.6        | 400            | -         |

TABLE 2. ESSENTIAL BUCK MECHANICAL PROPERTIES OF MATERIALS USED

| Material     | Poisson's Ratio | Young's Modulus (GPa) | Hardness (HV) | CTE ppm K <sup>-1</sup> | Melting Temperature (°C) |
|--------------|-----------------|-----------------------|---------------|-------------------------|--------------------------|
| Al           | 0.36            | 68                    | 15            | 24                      | 660                      |
| Cu           | 0.343           | 110                   | 50            | 16.4                    | 1083                     |
| Si           | 0.22            | 180                   | 1280          | 2.49                    | 1412                     |
| Fused silica | 0.17            | 74                    | 1000          | 0.57                    | 1610                     |

### B. Characterization flow

After specimens being fabricated, they then experienced a series of rapid thermal annealing. Detail processing parameters have already been shown in Table 1 using an AS-One RTA system. After heat treatment, those specimens are then used for various mechanical characterization as outlined in Fig. 1 and is briefly addressed below. The temperature ramp up rate is set as 5 °C/s and the peak annealing temperature is set between 200 - 500 °C with a 20 second annealing period.

**Residual stress:** Residual stress is characterized by curvature measurement. The 1cm wide strips are scanned by a Surfcoorder ET-3000 ( $\alpha$ -step) to obtain the profile and subsequently, the associated warpages and curvatures. Stoney equation is then hired to convert the measured curvature into film stress [15].

**Microstructure evaluation:** The microstructure evolution of films subjected to different deposition and RTA conditions is then characterized using X-Ray Diffraction (XRD) technique. By utilizing a D2 Phaser XRD system and Scherrer equation, it is possible to find the crystal orientation and average grain size. In addition, associated surface morphologies are also observed by field SEM (JEOL JSM-7001F).

**Nanoindentation:** the goal is to obtain load-penetration ( $P$ - $h$ ) curves and hire Oliver and Pharr model[16] to convert  $P$ - $h$  curves to their associate elastic modulus and hardness

information for correlating with processing parameters. A Nanoindenter XP is used to perform the indentation. Notice that possible substrate effect might cause deviation in quantitative results based on the ideal Oliver and Pharr model. However, this might not be a concern for qualitative comparison between different processing conditions based on the same model. For the quantitative correction, please refer to elsewhere [17].

**Nano-scratching:** The same Nanoindenter XP is also hired to perform scratching test. The scratching distance is set as 500  $\mu\text{m}$  with a speed approximately 100  $\mu\text{m}/\text{min}$  and a contact load linearly increase with scratching distance. The surface roughness and indentation depth data are then collected. From the test results, it is possible to correlate the frictional behavior and hardness variation [18] due to RTA through this test. Meanwhile, the surface morphologies of these scratched sample are also observed with the SEM for correlated scratching data and surface damage observed.

**Adhesion Characterization:** A self-designed pull-off test system, shown in Fig. 2 is hired to characterize the normal adhesion strength of these films with their corresponding substrates. Like the industrial cross-cut test, the test system records the fracture load, and the failure surfaces are examined to determine the effective pull-off area to calculate the adhesion strength [19].

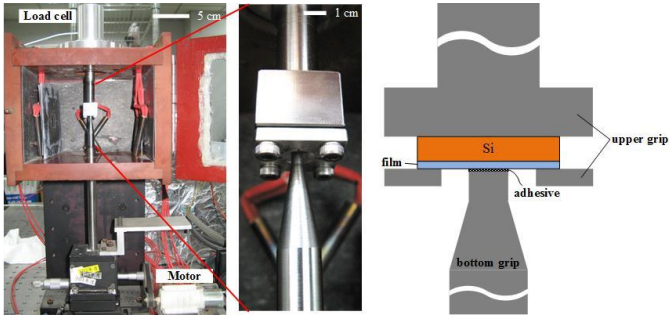


Fig. 2. The self-designed pull testing stage for adhesion characterization

### III. MECHANICAL PROPERTIES OF METALLIC FILMS

Metallic films are characterized first to elucidate focused mechanical behaviors *w.r.t.* deposition and annealing conditions and are addressed below.

#### A. Modulus and hardness

Both aluminum and copper films are indented to the designated depth and hold for 30 seconds before unload. The indentation depths are between 20% and 125% of the film thickness. Notice that the modulus  $E_f$  and hardness  $H_f$  would then be converted using Oliver and Pharr's model for those shallow penetration situations. For those indentation depths higher than 50% film thickness, the purpose is to observe the resulted fracture.

For films without RTA processing, as shown in Fig. 3, for a

maximum indentation depth as 20% of film thickness, the load-penetration curves are shown. From that,  $E_f$  and  $H_f$  of are estimated as 115 GPa and 1.6 GPa for aluminum films and 135 GPa and 2.2 GPa for copper films. Notice that those values are much higher than those obtained reported in bulk material state, and such a trend has been observed in microscale aluminum characterization by Malhaire et al [20]. However, the discrepancies are much larger. It is believed that a major reason could be attributed to the substrate effect and a more sophisticated model should be able to obtain more accurate results. Nevertheless, the major purpose of this work is to examine the effects of deposition and annealing parameters on the resulting mechanical properties. As a result, instead of seeking a more comprehensive model for conversion, we prefer to use the results obtained from the "improper" model as the benchmarks to evaluate the influence of processing parameters on the properties evolution.

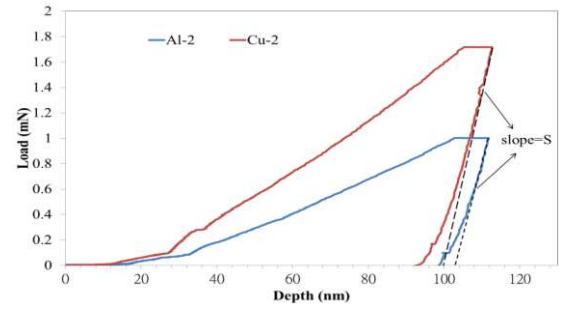


Fig. 3. Nanoindentation behavior of aluminum and copper films without experiencing RTA processing.

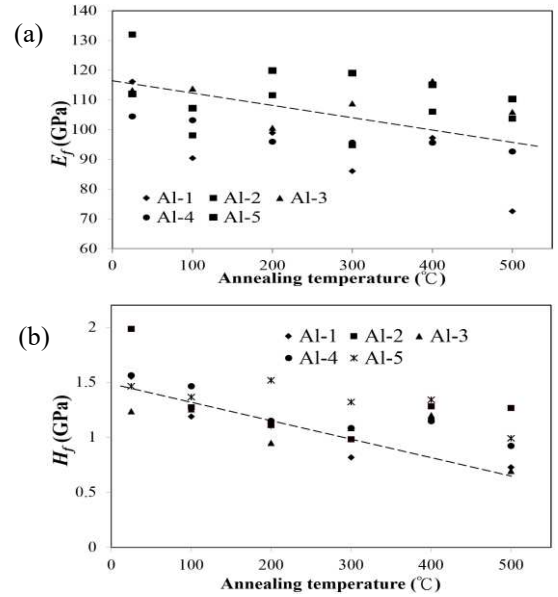


Fig. 4. Nano-indentation characterization results of (a) elastic modulus and (b) hardness results for aluminum films subjected to RTA processing

The effects of RTA temperature to the elastic modulus and hardness of aluminum films are shown in Fig. 4. Although the results are scattering, both the modulus and hardness decrease clearly as the RTA temperature increases. Specifically, at a RTA temperature of 500°C, the elastic modulus and hardness reduced from its original values to 90 GPa (reduced by 20%)

and 0.9GPa (reduced by 45%). On the other hand, as shown in Fig.5, similar trends are also observed in copper films but with less sensitivity. The reduction ratios are 9%(i.e., 135 to 120 GPa) for modulus and 13% (i.e., 2.3 to 2 GPa). One possible explanation of the tendency could be attributed to the effect of increased tensile residual stress shown in Section IV and this issue would be further discussed in Section VI.

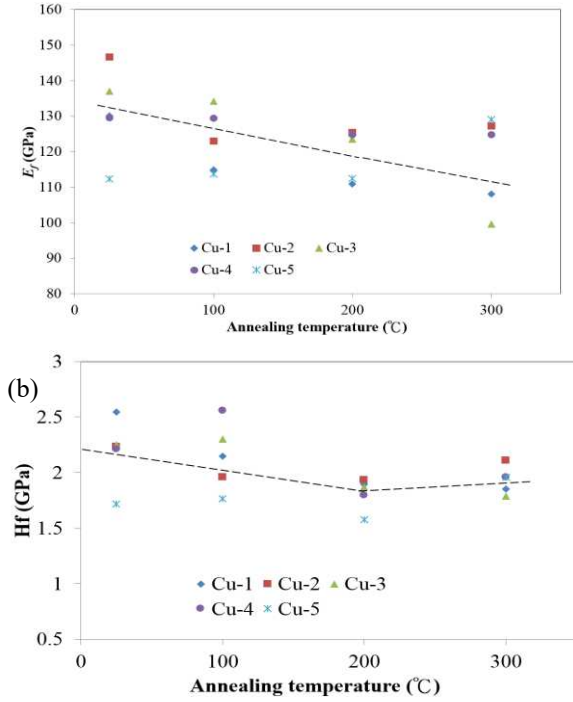


Fig. 5. Nano-indentation characterization results of (a) elastic modulus and (b) hardness results for copper films subjected to RTA processing

### B. Microstructures

To estimate the microstructure evolution of films after annealing, XRD measurements are then performed. Here, by considering the effect of residual stress and larger grain size (i.e., greater than 100nm), the fundamental Scherrer equation [21] is hired to convert the measured Bragg angle  $\theta$  and the full width at half maximum (FWHM)  $\beta$  of the diffraction peak to the average grain size  $D$  and is expressed as

$$\beta \cos \theta = \frac{k\lambda}{D}, \quad (1)$$

where  $\lambda$  is the wavelength of the used X-ray, residual stress, and elastic modulus of the film, respectively.  $k$  is a dimensionless factor usually around 0.9. The effects of RTA temperatures, deposition rate, and film thickness on the grain size are shown in Fig. 6 and 7, for aluminum and copper films, respectively. It is observed that although it is observed that the deposition rate does influence the grain size, the relation is not clear. However, the grain size increases monotonically with film thickness for both aluminum and copper films. It is believed that the above results could be attributed to the total thermal budget resulting in the deposition and annealing periods. For example, for

achieving a 100nm thickness deposition at high temperature, it requires approximately 200 seconds or aluminum and 2000 seconds for coppers and this implies that it takes a much longer period for a thicker film to stay in processing temperature and possible microstructure evolution tends to occur apparently.

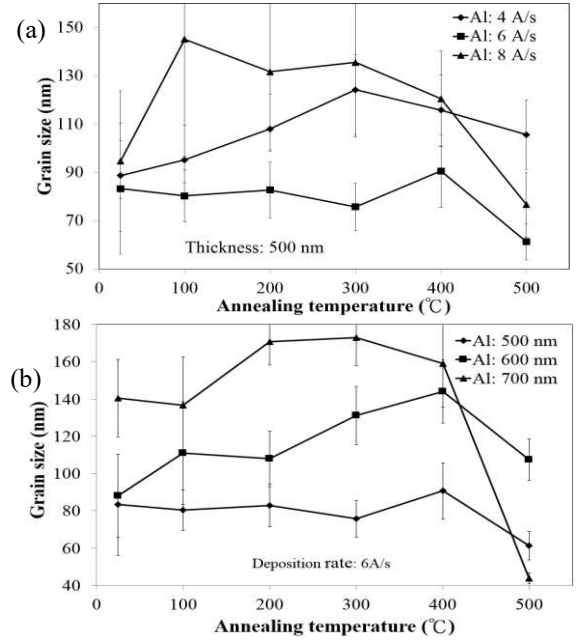


Fig. 6. The effect of (a) deposition rate and (b) film thickness on the grain size of aluminum films subjected to different RTA temperatures.

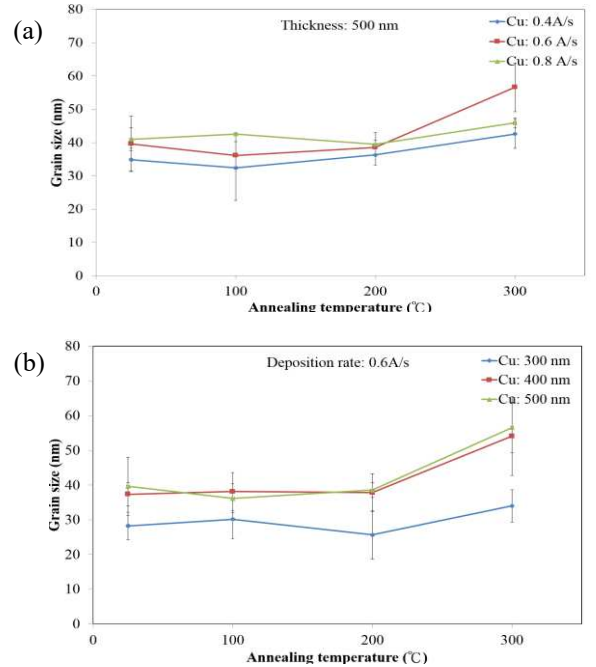


Fig. 7. The effect of (a) deposition rate and (b) film thickness on the grain size of aluminum films subjected to different RTA temperatures.

However, for RTA processing, the total ramping up and annealing period is only approximately 50 seconds. The relatively less thermal budget implies that for thin films, it is

less chance to generate sufficient microstructure evolution. On the other hand, for a thicker film, it requires a longer deposition period, and this allows extra times for microstructure evolution. This explains that the grain size of both films is increasing slightly with thickness and has no remarkable changes with RTA temperature. In addition, it is also interesting to point out that physically, by thermodynamics evolution, the grain size should be monotonically increased. However, as seen in aluminum, a sudden drop in grain size is observed as RTA temperature exceed 400°C. It is believed that this phenomenon is attributed to the residual stress and change in elastic modulus at different RTA temperatures and should be better computed with a better model [22] in the future.

### C. Surface Morphologies

It is also interesting to observe the surface morphologies of metallic films subjected to different deposition and RTA parameters. The effect of deposition rate of 500nm copper is presented in Fig. 8. Surface micro defects are observed and the defect density and size depends on the deposition rate. Specifically, a 0.6A/s deposition rate could yield the optimal result. On the other hand, the effect of RTA processing is investigated with a 500nm aluminum film. It can be seen that a relative loose granular structure is observed for as-deposited film. However, with the increase of RTA temperature, such a loose structure tends to be packed more closely possibly due to grain coalescence effect. Possible residual tensile stress could also be accompanied during such an evolution period.

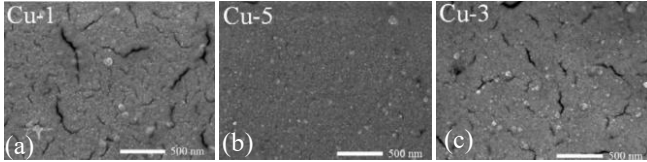


Fig. 8. Surface morphologies of as-deposited 500nm copper films with different deposition rates: (a) 0.4A/s, (b) 0.6A/s, and (c) 0.8A/s.

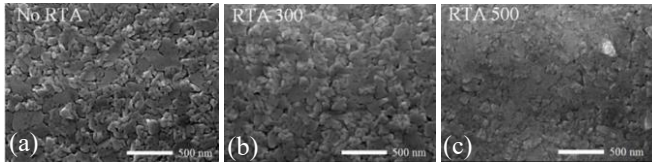


Fig. 9. Surface morphologies of 500nm aluminum films after different RTA processing temperatures. (a) No RTA, (b) 300°C, and (c) 500°C

## IV. RESIDUAL STRESSES OF METALLIC FILMS AFTER THERMAL PROCESSING

With the elastic properties characterization results in hand, it is possible to further investigate the deposition and thermal annealing induced residual stresses and interfacial adhesion behaviors of both metallic films. In associated with properties characterization results addressed in Section III, it is hoped to provide practical process parameter optimization guidance.

The first issue to be inspected is the influence of film thickness on the residual stress level. Noticed that all measurements are performed at room temperature and thus thermal stress due to temperature difference is not considered and only intrinsic stress variation due to deposition processing is revealed in the characterization results. As shown in Fig. 10a, residual tensile stress decreases with deposition thickness in both aluminum and copper films although the dependency on copper film is not so sensitive. The reason could possibly be attributed to the rigidity of deposited film. As the film thickness increases, the accompanied growth in film rigidity could cause deformation in substrate and thus partially relieves the film stress. Meanwhile, as indicated in Fig. 10b, for aluminum films the resulting stress also increases with deposition rate. On the other hand, the residual stress of copper films exhibits less sensitivity to the deposition rate. It is believed that the relative importance on the cohesive energy density between metal atoms themselves and the surface energy density between film and substrate materials are different and this results in different microstructures. For example, once cohesive energy is more dominant, a more island-like structure is form [1, 23, 24]. Such a structure would tend to generate higher tensile stress due to subsequent coalescence process. On the other hand, for copper film deposition with surface energy dominated, a layer-like structure is more stable and less microstructure evolution-induced stress generation consequently.

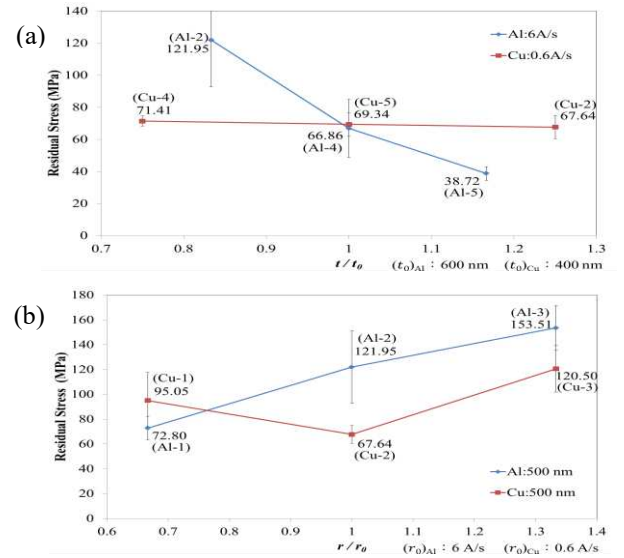


Fig. 10. The effect of (a) thickness and (b) deposition rate on the resulted residual stresses of both aluminum and copper as-deposited films.

For the influence of RTA annealing temperature, as shown in Fig. 11 and 12, as the RTA temperature increases, the post-anneal stress also increases. However, once the annealing temperature above a threshold level, the final stress reduces due to possible stress relaxation. For aluminum film, the higher the deposition rate, the larger the resulting residual stress after RTA. However, for copper film, the tendency is so not obvious, and additional experiments could be implemented for further clarification. On the other hand, for copper film, the post RTA residual stress reduces as film thickness increases. In contrast, there is no obvious correlation between film thickness and after RTA residual stress for aluminum films.

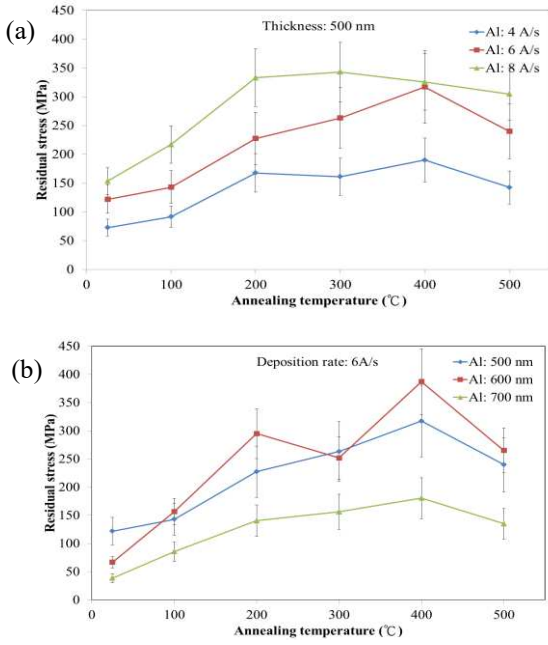


Fig. 11. The effect of (a) deposition rate and (b) film thickness on the resulted residual stress of aluminum film at different RTA temperatures.

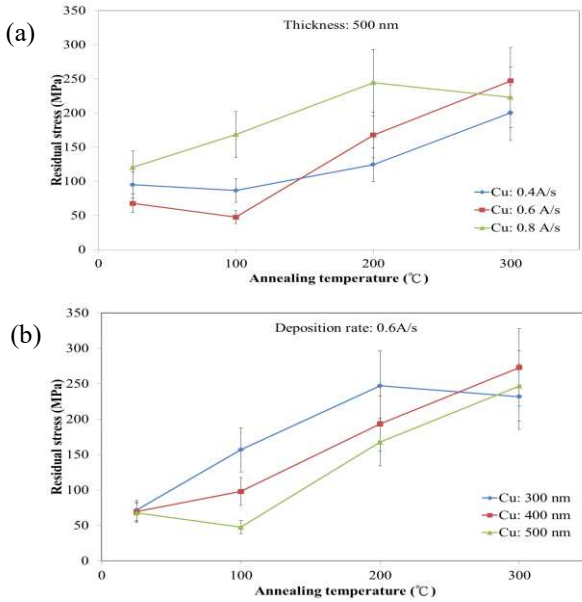


Fig. 12. The effect of (a) deposition rate and (b) film thickness on the resulted residual stress of copper film at different RTA temperatures.

In summary, the residual stress of aluminum films is more sensitive to deposition and annealing parameters than that observed in copper films. In addition to the differences in molecular material nature, the mechanism of PVD could not be neglected. That is, evaporation process lacks stress control mechanism, and the resulting microstructure is naturally sensitive to the processing parameters. On the other hand, sputtering process usually involves inert gas atoms bombardments and thus the microstructure and resulted residual stress are less sensitive to the process parameters.

## V. INTERFACIAL BEHAVIOR OF METALLIC FILMS

For characterizing the interfacial strength, both scratching and pulling tests are performed for finding the relationship between interface strength and RTA processing parameters and are addressed below.

### A. Scratching test

Nanoscratch test has been identified as one of the important tools for evaluating the mechanical and tribological properties of materials [25]. Previously, Wang et al [26] performed nanoscratch testing for aluminum subjected to different environments for CMP applications. In this work, the scratching tests are performed by the same nanoindentation testing system described above. A 500  $\mu\text{m}$  scratching distance with a compression force with a ramping rate of 0.2 mN/ $\mu\text{m}$  (for Al) and 0.1 mN/ $\mu\text{m}$  (for Cu) are used for 500 nm thick films with different RTA temperatures. Typical scratch results of as-deposited copper and aluminum films are shown on Fig. 13. Both films exhibit essentially spallation with partially buckling [27] induced by excessive compression stresses commonly observed in ductile metals with sufficient adhesion.

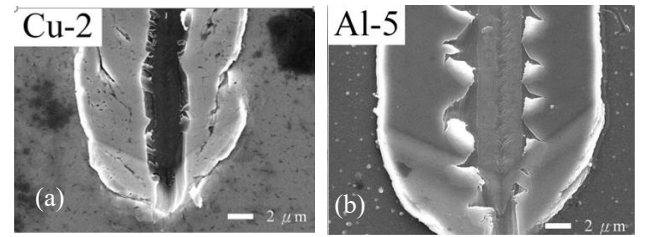


Fig. 13. SEM micrograph on nanoscratch testing of (a) copper and (b) aluminum films.

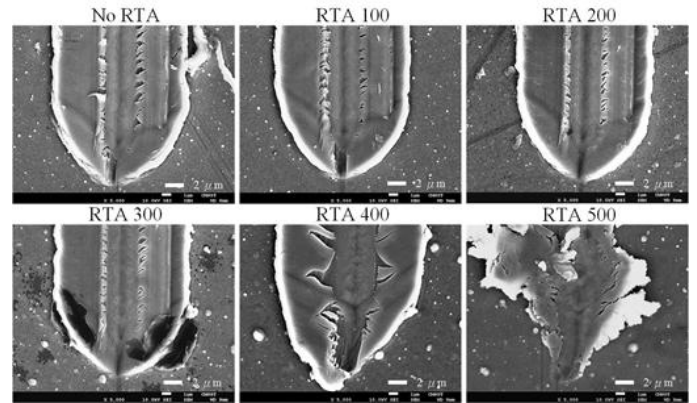


Fig. 14. SEM micrograph on nanoscratch testing of aluminum films after subjecting different RTA temperatures.

Fig. 14 shows typical SEM micrographs of scratch results of aluminum films subjected to different RTA temperatures. It shows the material responses vary significantly with different processing temperatures. Specifically, Fig. 15a shows the depth profile before, during, and after scratching tests of as-deposited aluminum films. Plastic deformation is observed after

removing the indenter. However, the depth curve is relatively smooth, which implies there are no brittle fractures that occur. However, for aluminum films, as the RTA temperature increased over 300°C, the failure mode gradually changes to tensile chipping failure, which indicates the material becomes more brittle. This fact can be observed from the SEM micrographs shown in Fig. 14 and the scratch profiles provided in Fig. 15b, where for RTA temperature below 300°C, the behavior keeps the same essentially. However, for RTA temperature at 400°C, more indentation depth is observed but it remains smooth, which implies that the film is still ductile but become more compliant. For RTA temperature being increased to 500°C, the response becomes completely different. The observed severe fluctuations imply that the major failure mode is brittle fracture. Each spiking result implies a possible fracture or delamination occurred. On the other hand, for scratching length less than 300μm, the mean indentation depth is close to those observed on the specimens with RTA temperatures below 300°C. However, for scratching distance further increases, the observed film compliance increases. This implies that significant material behavior changes for aluminum experiencing RTA temperature 300°C or above.

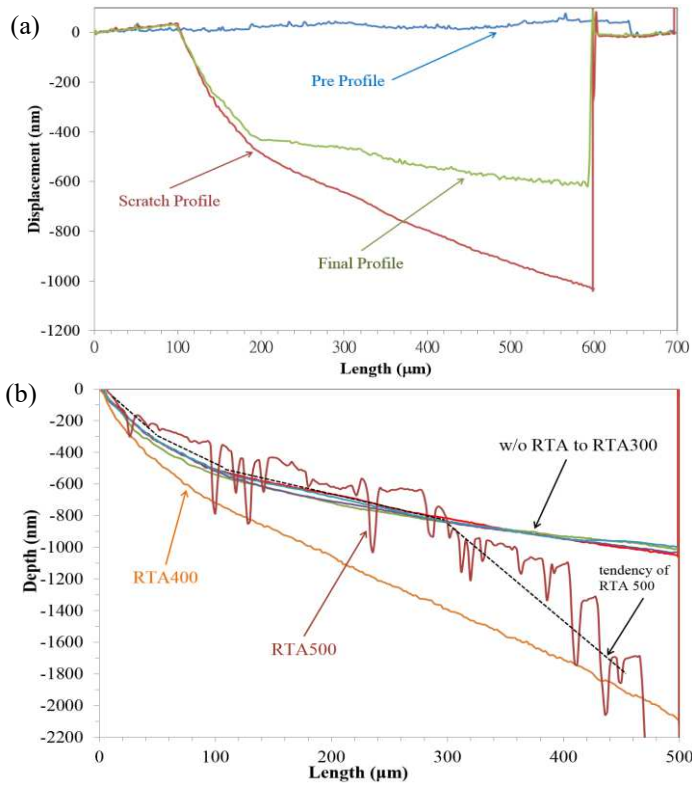


Fig. 15. The scratching profile of aluminum films: (a) as-deposited state and (b) subjected to different RTA temperatures.

### B. Pull test

Pull tests are performed for both aluminum and copper films. The testing system has been shown in Fig. 2 already and a typical test specimen for copper films is shown in Fig. 16. The obtained breaking forces are converted to adhesion strength. Samples with different thickness, deposition rates, and RTA

processing temperatures are investigated and are shown in Fig. 17 for copper films. The tested adhesion strength exhibits scattered results with different deposition rates and different film thickness. Nevertheless, the adhesion strength tends to be enhanced with a higher RTA temperature. That is, the adhesion strength could be improved from approximately 1 MPa to 5 MPa with proper deposition and RTA processing parameters.

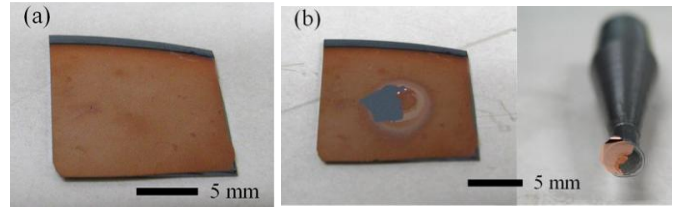


Fig. 16. Copper/Silicon adhesion test specimen (a) before and (b) after pulling.

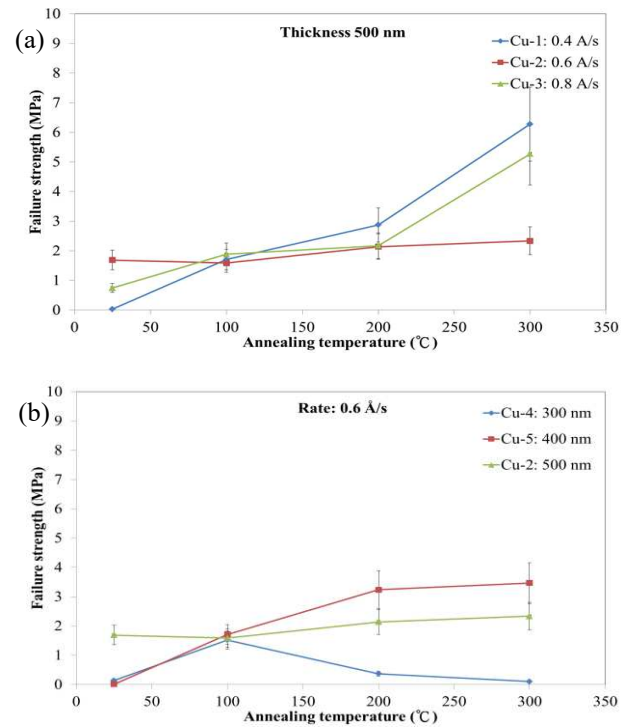


Fig. 17. The effect of (a) deposition rate and (b) film thickness on copper/silicon interfacial strength after different RTA processing temperature

It is also interesting to find the correlation between surface morphology and adhesion strength. As shown in Fig. 18, four as-deposited copper specimens with different surface conditions although they were fabricated based on the same processing parameters. Specimens with more surface hillocks exhibit less interfacial strength, possibly due to less effective contact area. It is believed that the RTA processing could improve surface roughness and consequently effectively increase the contact area and thus improve the strength. Notice that the delamination behavior of copper could be a vital factor in CMP processing of modern interconnection fabrication to withstand sufficient polishing shear force as depicted by Yuan et al [28].

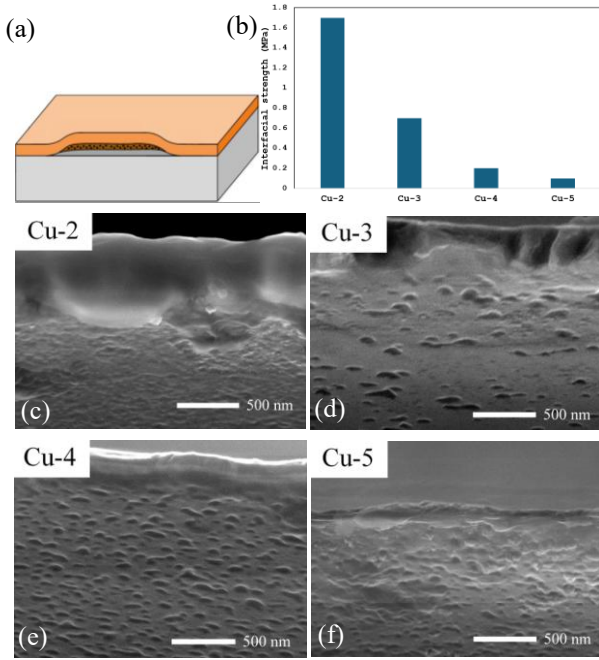


Fig. 18. The correlation between surface roughness and the tested copper/silicon interface strength of as-deposited copper films: (a) schematic plot, (b) strength of various copper specimen, and (c)-(f) surface morphology of those specimens.

## VI. DISCUSSIONS

### A. Summary

In this work, the influence of deposition rate, thickness and RTA temperatures on material properties and interface behavior of both aluminum and copper films are investigated through nanoindentation, nanoscratch, and adhesion tests, as well as XRD. The characterization results indicated that the RTA processing temperatures could have strong effects in various concerned properties and are listed below.

The elastic modulus and hardness of both films reduce as RTA temperature increases. But the dependence of grain size on RTA temperature is not obvious. On the other hand, residual stress changes significantly with RTA temperature. A higher RTA temperature would result in higher residual stress levels. In addition, through nanoscratch tests, the deformation mechanism of both metals changes from ductile plowing toward brittle failure at higher RTA temperature, which reflects changing in both the material properties and adhesion strength. Finally, increasing in RTA temperature could improve adhesion strength between copper and silicon, possibly due to surface morphology variation with surface hillock reductions to effectively increase micro contact areas. This information could be further integrated for providing basis of annealing parameter optimization toward interfacial strength enhancement.

### B. Key discussions

There are several issues discovered or learned based on this

investigation. First, the hardness and modulus obtained in this study is based on the traditional Oliver and Pharr model [16] embedded in the commercial testing system, which ignores the possible substrate and residual stress effects. Previous investigations [17] indicated that such effects could be substantial for thinner films and the existence of tensile residual stress could effectively reduce the unload slope and the curvature of loading curve. Both are the key influence factors for estimating the resulted modulus and hardness. This could possibly explain why the apparent modulus and hardness of both films decrease with RTA temperature.

For the grain size issue, the results presented in Fig. 6 show that the grain size of aluminum films is reduced after 500°C RTA. This is unreasonable from thermodynamics. However, all aluminum specimens show similar trends, thus such a correlation implies that the experimental results should be meaningful. One possible explanation is that the hired Scherrer relation also does not consider the effect of residual stress, which has been pointed out as one factor to influence XRD results. As shown in Fig. 11, the residual stress increases with RTA temperature and this could partially explain such abnormal data. Further investigations should be proceeded to clarify this issue.

In summary, strong correlations are observed between mechanical properties, residual stress, and microstructures during RTA. By integrating the information presented in Figures 4, 5, 6, and 7, it is possible to further elucidate the complicated coupling between them and better mechanics models should be hired in the future for better explaining and quantifying the experimental data. Meanwhile, for the effect of deposition rate and deposition thickness, it is possible to translate the results in terms of deposition time, which could be analogous to thermal budget. That is, for less deposition rate and higher deposition thickness, the deposition time duration would be longer, and it allows more time for reaching thermodynamics equilibrium and thus alter the resulted microstructure and residual stress, which also lead to the variation in observed mechanical properties.

For the interfacial behavior, the scratch test for aluminum shown in Fig. 14 and Fig. 15 indicated that RTA temperature above 400°C could result in significant change in interfacial strength. Specifically, after 400°C RTA, the scratching depth increases significantly, and this implies that the hardness is reduced, and the tendency agree with that observed from nanoindentation tests. However, further increase in RTA temperature might reduce the toughness and more brittle debris are observed from the plowing process. The brittle fracture also causes strong fluctuation of the scratch response shown in Fig. 15. The results imply that annealing process parameters should be carefully selected to avoid undesirable changes in material responses.

From the review article provided by Drener and Nix[23], the mechanisms of structure evolution during deposition has been clearly described. Base on material thermodynamics and energy minimization, the microstructure evolution during deposition and subsequent annealing tends to reduce the free energy by means of grain growth but the coalescence process could also induce residual stress. Such a phenomenon has been

observed in low temperature deposited oxides [29,30] and nitride [31] during its post deposition annealing.

Furthermore, as has already been shown in Fig. 17, RTA processing could effectively enhance the interfacial strength but with considerable scattering and a highly correlated relationship is observed between surface roughness and adhesion strength as being depicted in Fig. 18. Specimens with less hillocks exhibit higher strength and this could be possibly attributed to more contact areas by reducing contact with asperities. The observation is highly qualitative but with certain physical insight. In the future, more investigations and parametric studies should be performed to address the influence of deposition and annealing parameters on surface morphology and adhesion behavior. Nevertheless, for copper films, the current investigation results should be helpful for copper CMP processing where the film compliance and interfacial strength are critical [28] and the test results should be applicable for enhancing the reliability in copper CMP.

### C. Contributions

Although there are many reported thin film characterization researches, to the best of our knowledge, less reported studies focused on discussing the effect of processing and annealing parameters on the mechanical behavior and the subsequent structure reliability assessment. The results presented should be useful for interconnection longevity concern of both IC and MEMS. Specifically, as addressed by Yuan et al [28], an enhanced interfacial strength of copper film is critical for CMP performance and the observation by this work could provide a qualitative guide for improvement. In particular, the strong correlation between surface morphology and adhesion strength observed from copper film provides a rational explanation and possible engineering solution on adhesion strength improvement through surface roughness modifications.

Finally, for aluminum film applications, this study provides insightful guides to related applications such as interconnection for semiconductor devices, where the signal transmission could be affected, and MEMS thermal actuators, where the structure suffers from high temperature during service and the adhesion strength could be a concern for actuation reliability. On the other hand, for copper related application, such as CMP, the presented characterization procedure could serve as a guideline on determining the adhesion behavior of copper with other materials such as low-k oxide and nitrides, commonly used in copper interconnections and possible delamination could be a problem in CMP processing [32].

### D. Future perspective

This work provides primitive conclusion to address the influence of deposition rate and thickness, as well as the RTA temperature, on key material and interfacial behaviors of PVD metallic films. Currently, more detail works are carried to further explore and to improve the performance of the characterization results: including adding finite element simulation, hiring better analytical models to convert nanoindentation and XRD data to the final desired mechanical

behavior, and a more integrated data processing to correlate the material behavior, microstructures, residual stress, and processing parameters simultaneously. In addition, similar characterization approaches are also currently proceeded for barrier dielectric material coatings such as Yttria and Yttrium films, which are commonly used in semiconductor processing chamber coatings and thus the adhesion behavior is crucial for device longevity.

## VII. CONCLUSIONS

The mechanical properties and interfacial behavior of PVD metallic films are vital for device longevity assessment and are strongly related to related processing and subsequent annealing parameters. Investigations on characterizing the stress states, interfacial strength and elastic properties of both aluminum and copper films with different RTA temperatures have been performed. From these studies, stress and strength behaviors after annealing are observed and have been correlated with microstructure evolution. Technically, it shows that RTA temperatures indeed have strong influences on the achieved properties and strengths. Apparently, both elastic modulus and hardness decrease with RTA temperatures. On the other hand, the adhesion strength could be improved with proper RTA processing. Microscopically, RTA processing also effectively changes the microstructure and surface morphology of deposited film, and the observed grain growth and surface hillocks are believed to be highly correlated with the measured properties and strength variations. All of these results would be invaluable for guiding the longevity evaluation for related semiconductor devices.

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