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Microstructure and Residual Stress of Metallic Thin Films According to Deposition Parameters

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Abstract

In general, the microstructure in thin films was known to evolve in similar manner according to the energy striking the condensing film at similar homologous temperature, Th for the materials of the same crystal structure. The fundamental factors affecting particle energy are a function of processing parameters such as working pressure, bias voltage, target/sputtering gas mass ratio, cathode shape, and substrate orientation.

In this study, Al, Cu, Pt films of the same crystal structure of face centered cubic (FCC) have been prepared under various processing parameters. The influence of processing variables on the microstructures and residual stress states in the films has been studied.

Keywords : Thin film, Stress, Microstructure, Heat treatment

1. INTRODUCTION

Residual stresses may cause the problem of stability and reliability in the multi-layer structures. Therefore, it is important to understand the origin of residual stress in thin films and to restrain the residual stress during processing. The intrinsic stress can be tensile or compressive depending on the particle energy during the deposition process. The fundamental factors affecting on the particle energy are a function of processing parameters such as working pressure, bias voltage, target/sputtering gas mass ratio, cathode shape, and substrate orientation, which results in microstructure changes. When working pressure gets lower and bias power gets higher, the particle striking the condensing film gets more energy[1-3]. The microstructure translates from porous columnar to condense one as particle energy and temperature gets higher[1,4]. Thornton[5] illustrated this trend using four zone diagrams in terms of processing parameter and deposition temperature as shown in Fig. 1. Zone 1 structure consists of tapered crystallites with domed tops. The structure has many micro-void due to low particle energy and self-shadowing. Zone T structure has a fibrous morphology with smooth material surface and is considered to be a transition from Zone 1 to Zone 2. The formation of the Zone T is due to

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the energetic bombardment from reflected high-energy neutrals from the sputtering target at low gas pressures. In Zone 2, the growth process is dominated by adatom surface diffusion, which allows the densification of the intercolumnar boundaries. But the basic columnar morphology remains still in this stage. In Zone 3, bulk diffusion allows recrystallization, grain growth and densification[3,4,6]. This may suggest a link between microstructure and residual stress.

In this study, Al, Cu, Pt films were prepared by RF magnetron sputtering at various working pressures and RF powers. The influence of processing variables and materials on the microstructure and residual stress states in the films has been studied. The relationship between microstructure and residual stress changes in the films was also investigated for as-coated specimens as well as heat-treated specimens.

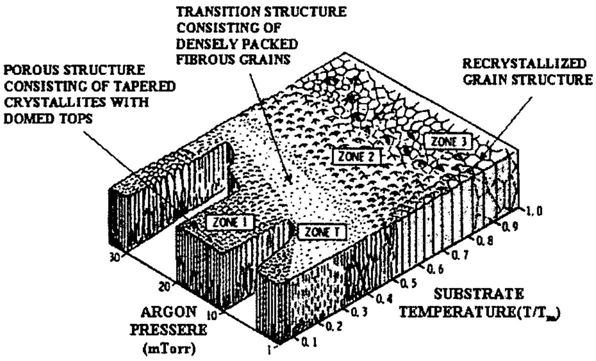


Fig. 1. Structure-Zone model as a function of growth temperature and Ar pressure proposed by Thornton.

2. EXPERIMENT PROCEDURE

The P-type <100> Si wafer ground to 520μm in thickness was cut into the dimensions of 6× 30mm using a dicing saw. The specimens were cleaned with acetone and alcohol for 5 minutes each, and with deionized water for 10 minutes before blow-drying using N_2 gas. Dried specimens were thermally oxidized to form SiO_2 layers of approximately 200nm thickness in the furnace at 900℃ using water vapor. Since Pt, Cu, Al layers are known to have problems in adhesion with SiO_2 insulation layers, the Ti layer of 10nm was deposited between the Pt, Cu, Al layers and SiO_2 insulation layer to improve adhesion and prevent the diffusion of Si atoms to the layers. A standard condition of RF magnetron sputtering for the film deposition is given in Table 1. The curvature of the specimen was measured using a laser scanning device (Fig. 2) before and after each layer was deposited. From the change in the curvature of a certain layer before and after the deposition, the residual stress in the layer was obtained using a modified Stoney's equation with the knowledge of Young's modulus and Poisson's ratio of substrate, thickness of substrate and film layer.

$$\sigma_f = \frac{E_s}{6(1-\nu_s)t_s}\frac{t_s^2}{t_f}\left(\frac{1}{r}-\frac{1}{r_0}\right) \qquad (1)$$

Table 1. Processing conditions of Al, Cu, Pt films using RF magnetron sputter

	Working pressure (mtorr)	Bias power (W)	Gas flow (SCCM)	Deposition Time (min)	Deposition temperature
Condition	4	60 80 100	10	10	Room temp.
	6				
	8				
	10				

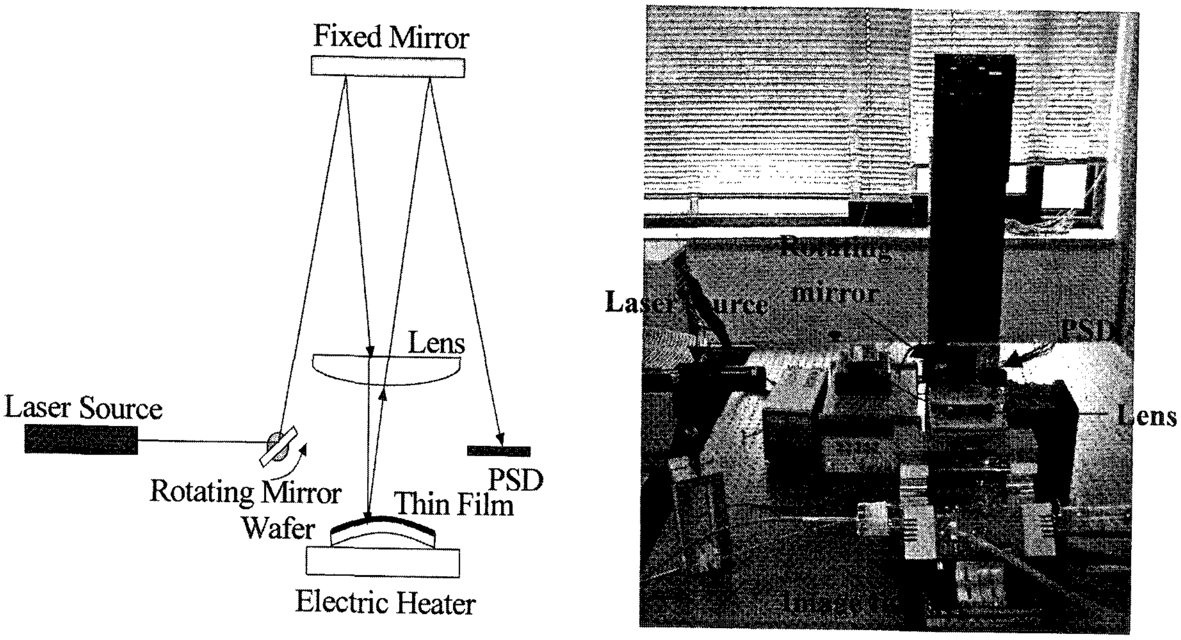


Fig. 2. Laser scanning device for measuring radius of curvature.

Where Es is Young's modulus of the substrate, ts, tf are the thickness of substrate and film, respectively. Also νs is Poisson's ratio of the substrate, r is the radius of curvature after the deposition of the thin film and r0 is the radius of curvature before the deposition of the thin film.

After the deposition of all the film layers was completed, the final heat treatment at 300, 500, 700℃ for 5 hours was carried out. The residual stresses in thin films were measured using in-situ curvature measurements during heat treatments at 1, 3, and 5 hour holding. The microstructure of each thin film was observed using SEM.

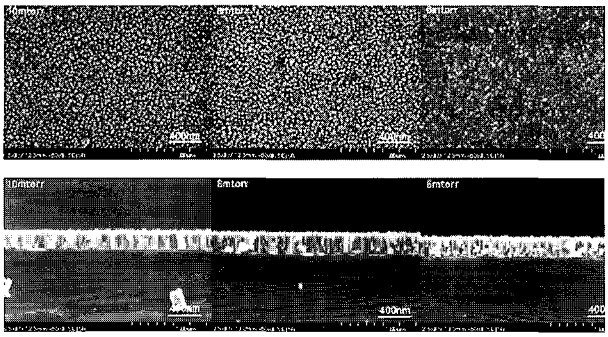


Fig. 3. Surface and cross section SEM images of Pt thin film processed at various working pressures with a fixed bias power of 80W (magnification 50,000).

3. RESULTS AND DISCUSSION

Fig. 3 shows the SEM images of Pt film processed at various working pressures with a fixed bias power of 80W. Condensed columnar structure was observed along the cross section of thin film. As the working pressures decreased, grain size decreased and domed tops structure changed to smooth surface. While the normal flux component and the particle energy striking the film increased, gas scattering reduced when the working pressure decreased. The microstructure might have been translated from Zone 1 to Zone T due to the reduction of the self-shadowing and the collapse of the micro-void with decreasing working pressure[1,8].

The residual stresses of Pt thin films are shown in Fig. 4 according to processing variables of working pressures and bias powers. In Pt thin films processed at RF power of 60W, the level of residual stress increased with decreasing working pressure. For the specimens processed at RF power of 80, 100W, the residual stress showed the highest level at 6mtorr, 8mtorr, respectively. As shown in Fig. 4, the microstructure of Pt film may be the origin of stress level change, where the microstructure translated from Zone 1 to Zone T. Generally, the grain boundary relaxation model, proposed by Finegan and Hoffman, are the one most popular way to explain the tensile stress in polycrystalline films[1,9]. This model is based on the interatomic attractive forces acting across the gaps between contiguous grains causing an elastic defomation of the grain walls. In this model, the decrease in grain size causes the increase in residual stress. When working pressure decreased until 6mtorr with a fixed bias power of 80W, it was observed that the grain size decreased as shown in Fig. 4.

The SEM images for Cu thin film processed at various working pressures with a fixed bias power of 80W are shown in Fig. 5. Columnar structure disappeared as working pressure decreased. The residual stresses of Cu thin films are shown in Fig. 6 according to processing variables where residual stress decreased with decreasing working pressure. Particle energy is known to increase due to lowing working pressure, intended inducing compressive energy. Since the mobility of Cu atoms is higher than that of Pt, Cu atoms have more probability of atomic displacement of surface atoms into the film interior by bombardment between particle and film[1,9]. Thus the columnar structure might have disappeared in Cu films.

Fig. 7 shows the SEM images of Al film processed at various working pressures with a fixed

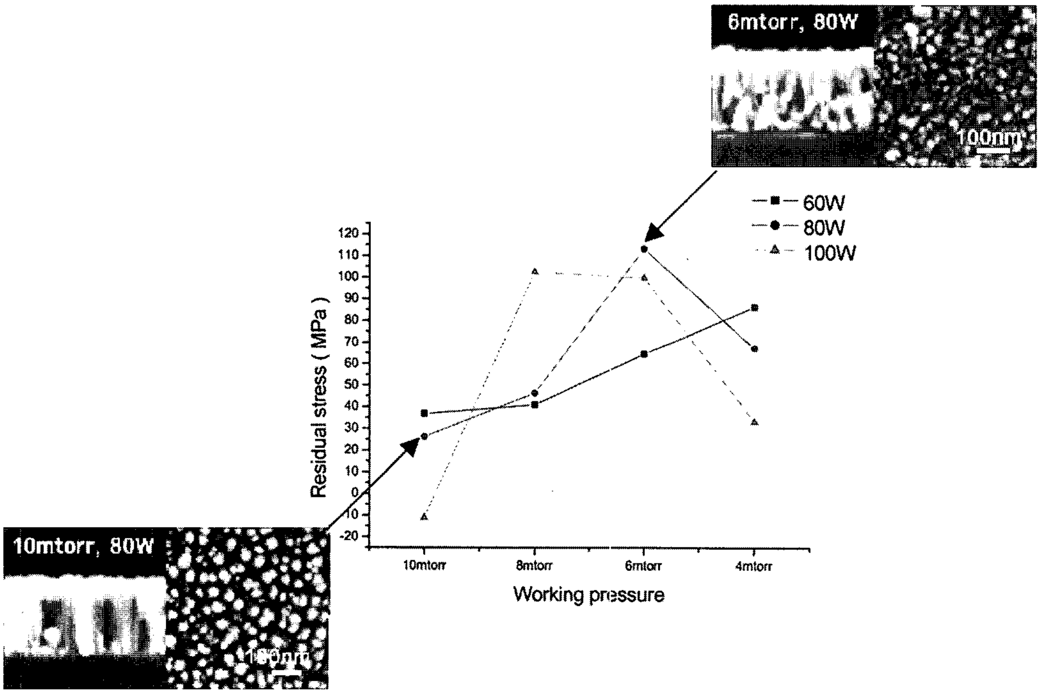


Fig. 4. Stress and surface SEM images of Pt thin films according to processing variables.

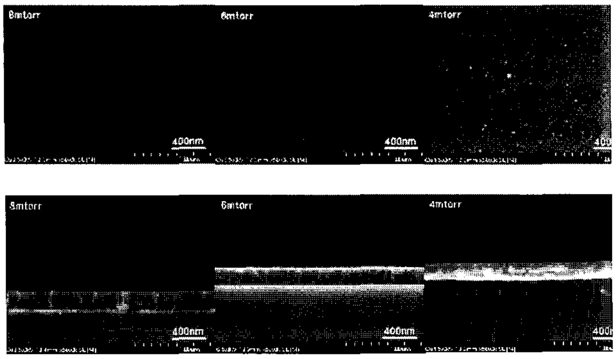


Fig. 5. Surface and cross section SEM images of Cu film processed at various working pressures with a fixed bias power of 80W (magnification 50,000).

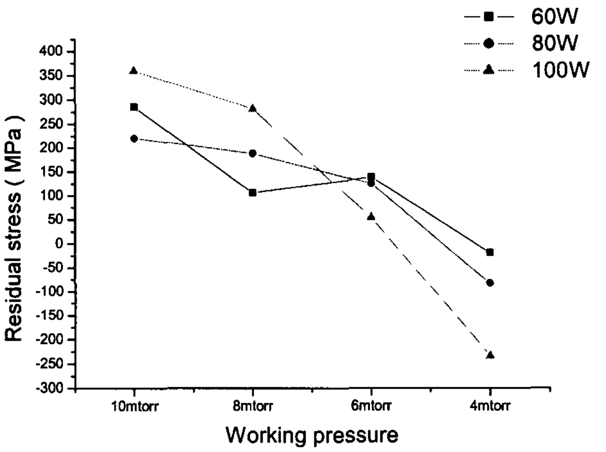


Fig. 6. Stress of Cu thin films according to processing variables.

bias power of 80W. The grain size became smaller and more uniform as decreasing working pressure. In addition columnar structure was not observed in the cross section of Al thin films. It is known that bulk grain boundary motion during coalescence and thickening of films is possible inducing to restrain columnar structure even at homologous temperatures as low as 0.2 in Al thin film[6]. The residual stress of Al thin films is shown in Fig. 8 according to processing variables. The stress levels in Al films were found to be compressive for all the specimens studied in this study. In general, atomic peening effect invoked to explain the origin of compressive. In Al thin film, the influence of atomic peening effect may be greater than Cu, Pt thin films because Al yield strength is lower than others. In our experiment, it was found that compressive stress increases with decreasing working pressure. It seems to be affected by longer mean free path and higher bombardment energy of incident particles[1,11].

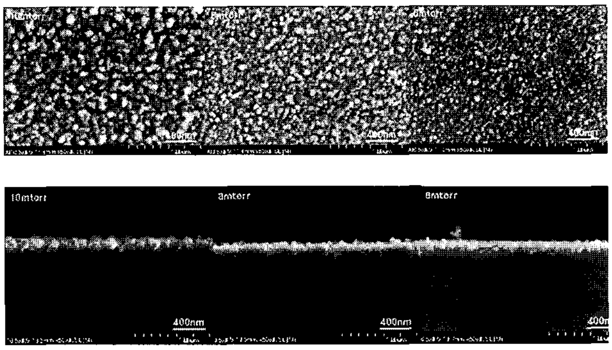


Fig. 7. Surface and cross section SEM images of Al film processed at various working pressures with a fixed bias power of 80W (magnification 50,000).

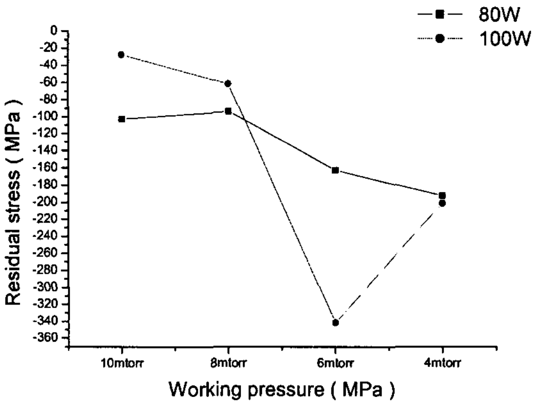
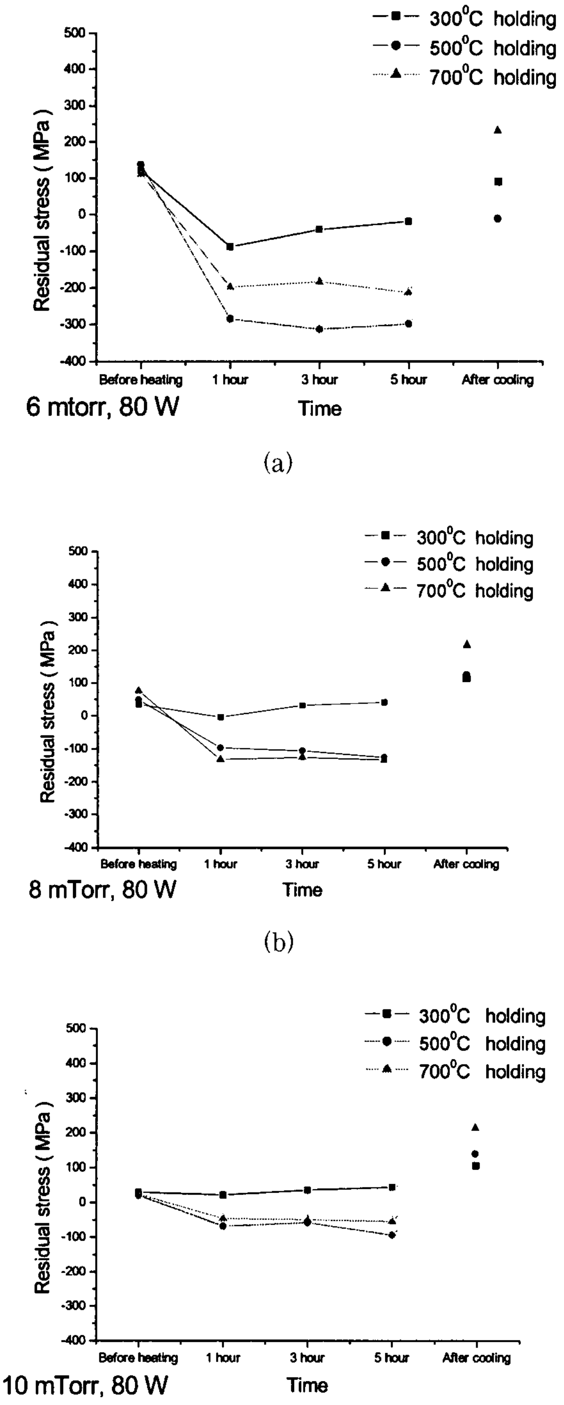


Fig. 8. Stress of Al thin films according to processing variables.

Although the Pt, Cu, Al thin films deposited at the same working pressure and bias power, but the microstructure and the stress were found to be different. This may be due to the difference of particle mobility on surface of films[6,8]. Adatom surface mobility increases with low energy of the atom, low atom-surface interaction, and high temperature of surface[8]. Adatom energy depends on mass ratio of target to sputtering gas, thus adatom energy is the lowest for Pt thin film. When Pt, Cu, Al films are deposited at the same temperature, homologous temperature is the lowest for Pt thin films. For the case of Pt thin films, the microstructure becomes porous columnar inducing tensile stress due to the low mobility of Pt atoms[12]. The materials like Al thin films that mobility is the higher can move easily to stable sites. In those materials, bulk grain boundary motion during coalescence and thickening of films is possible, where the growth mode restrains columnar structure inducing compressive stress[6,12].

Fig. 9 shows the residual stress change of Pt thin films during heat treatment. It was observed that stress was compressive during heating, but stress was tensile after cooling. The primary reason for tensile stress after cooling is known as the thermal stress from the difference in thermal expansion coefficient among layers[13].



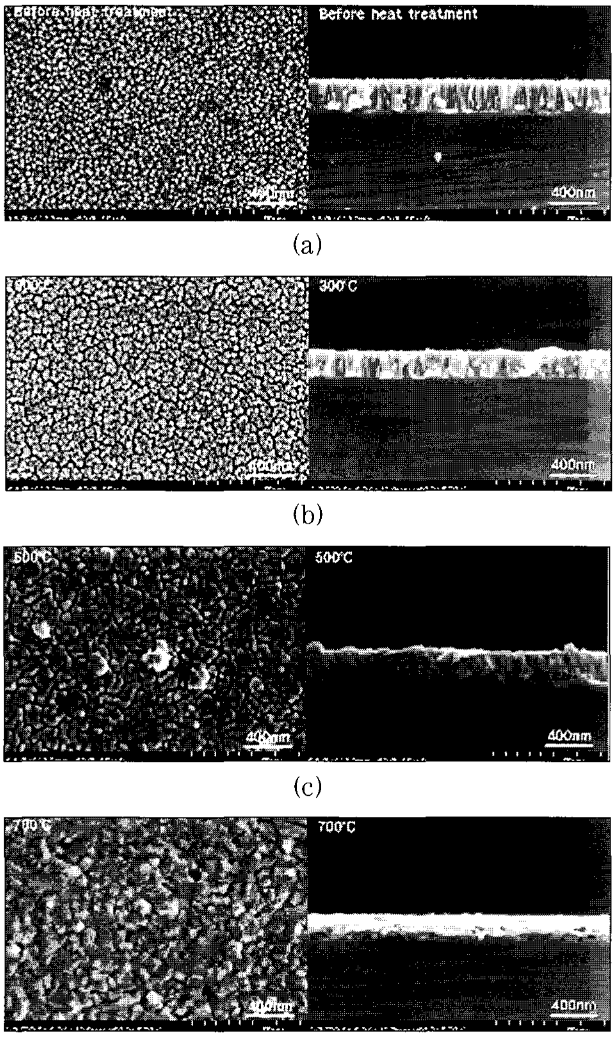
(a)

(b)

(c)

Fig. 9. Stress curves of Pt films processed with a fixed bias power 80W at a) 6mtorr b) 8mtorr c) 10mtorr during heat treatment.

If only thermal stress component is considered, the stress levels for 500℃, 700℃ holding specimens in Fig. 9 are not easily explained. The lager temperature changes results in the more stress changes. Thus microstructural changes are considered as other possible origin for stress changes during heat treatment. The lowest compressive stress measured during heat treatment was observed for the specimen heat treated at 300℃. Since Pt has a high melting temperature of 1760℃, recrystallization and grain growth are not expected to happen in serious amount at 300℃. For the Pt films processed at 8, 10 mtorr working pressures and at 80 W bias power subsequently heat-treated at 500℃, 700℃, the stress behavior was similar as shown in Fig. 9 (b) and (c). The stress change at the 700℃ might have exceeded the elastic range and translated plastic flow like the results proposed by Flinn et. al.[14] and Proost et. al.[15]. The stress drop at 700℃ may be due to recrystallization and grain growth as observed in Fig. 10, where microstructural change in the specimen was observed to be more severe for 700℃ heat treated specimen than 500℃ heat treated one. The microstructure changes may have compensated the influence of thermal stress.



(a)

(b)

(c)

(d)

Fig. 10. Surface and cross section SEM images of Pt film processed at 8mtorr, 80W (magnification 50,000). a) before heat treatment b) 300(C holding for 5hr c) 500(C holding for 5hr d) 700(C holding for 5hr.

4. CONCLUSION

In this study, Al, Cu, Pt films have been prepared by RF magnetron sputtering at various working pressure and RF power. The influence of processing variables on the microstructure and residual stress states in the films has been studied.

The microstructure of Pt thin films translated porous columnar to collapsed columnar as decreasing working pressure. In Pt thin films processed at RF power of 60W, the level of residual stress increased with decreasing working pressure. For the specimens processed at RF power of 80, 100W, the residual stress showed the highest level at 6mtorr, 8mtorr, respectively. For Cu thin films, columnar microstructure disappeared and residual stress level decreased with decreasing working pressure. Columnar structure was not observed in the cross section of Al thin films. The stress levels in Al films were found to be compressive for all the specimens studied in this study. The differences of the microstructure and stress with same processing variables may be due to difference of particle mobility on surface of films.

The compressive stress was observed during heating but tensile stress was observed after cooling. If only thermal stress component is considered, the stress levels for heat treated specimens are not easily explained. Thus microstructure changes are considered as other possible origin for stress changes during heat treatment. This changes may be resulted from the interaction between thermal stress and microstructure changes.

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