

Research Article

Effect of Annealing on Microstructure and Mechanical Properties of Magnetron Sputtered Cu Thin Films

Shiwen Du and Yongtang Li

School of Materials Science and Engineering, Shanxi Key Laboratory of Metallic Materials Forming Theory and Technology, Taiyuan University of Science and Technology, Taiyuan 030024, China

Correspondence should be addressed to Shiwen Du; tykddsw@126.com

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Cu thin films were deposited on Si substrates using direct current (DC) magnetron sputtering. Microstructure evolution and mechanical properties of Cu thin films with different annealing temperatures were investigated by atomic force microscopy (AFM), X-ray diffraction (XRD), and nanoindentation. The surface morphology, roughness, and grain size of the Cu films were characterized by AFM. The minimization of energy including surface energy, interface energy, and strain energy (elastic strain energy and plastic strain energy) controlled the microstructural evolution. A classical Hall-Petch relationship was exhibited between the yield stress and grain size. The residual stress depended on crystal orientation. The residual stress as-deposited was of tension and decreased with decreasing of (111) orientation. The ratio of texture coefficient of (111)/(220) can be used as a merit for the state of residual stress.

1. Introduction

With the rapid change of materials systems and decreased feature size, thin film microstructure and mechanical properties have become critical parameters for microelectronics reliability [1]. Copper is an attractive interconnecting material for current Si ultralarge scale integrated (ULSI) device due to its low resistivity and superior resistance to electromigration [2–4]. Cu film has attracted great attention worldwide due to its potential applications in replacing Al-based interconnects on silicon chips.

According to research object, film structure can be divided into crystalline form, crystallographic structure, and surface structure. Microstructure of materials here mainly refers to crystal orientation and grain size. The electrical resistivity and mechanical properties of Cu films are important factors for its use as interconnecting material. Magnetron sputtering has become one of commonly used techniques for industrial deposition of thin films and coatings, due to its simplicity and reliability [5]. Generally, structure and electrical qualities of films strongly depend on the deposition process [6]. On the other hand, the postprocessing such as

annealing can also change the microstructure and mechanical properties of the films [7, 8].

In sputter deposition, the nature of the substrate, the deposition temperature, the deposition pressure, and the vacuum quality are some of the parameters that influence the film properties. Properties such as stress, texture, and morphology are of key importance for predicting the reliability of thin film systems. The influence of the magnetron source operation mode (standard or self-sustained), as well as the type of power (DC, medium frequency, or pulsed DC) on the microstructure, and surface morphology of the copper thin films have been reported [5]. Sputtering DC power affected the structural features, electrical properties, and the nucleation and growth of Cu films during the initial stage of sputtering [6]. A comparative study of structural, electrical, and thermoelectric properties of nanocrystalline copper thin films deposited using anodic vacuum arc plasma deposition technique and DC magnetron sputtering was also conducted [9]. The effects of barrier layer and annealing temperature on texture variation, grain growth, and void formation of nanocrystalline Cu films were investigated [10]. The deposition pressure and the type and cleaning condition

of substrate had important role on the film properties. The substrate type and the substrate surface condition had marked influences on the texture of as-deposited Cu films [11]. Cu films were deposited on Si(001) substrates under various Ar deposition pressure by radio frequency (RF) magnetron sputtering. The intensities of Cu peaks changed with the Ar pressure systematically. Strong correlations were observed between optical emission, electron temperature, and the microstructure of Cu films [12].

For Cu nanocrystalline films to be widely used, it is necessary to explore its microstructure and mechanical properties. The physical properties and microstructure of Cu films, such as mechanical properties, grain boundary, and crystallographic texture can significantly influence the work reliability in microelectronic devices [10]. Mechanical properties of thin films often differ from those of the bulk materials [13–16]. The physical dimensions of thin film materials are generally comparable to the characteristic microstructural length scales that strongly influence their mechanical properties [17]. In general, two different size dependencies determine the properties of a material. One is the dimension characteristic of the physical phenomenon involved. The other is some microstructural dimension. Microstructural evolution during elevated temperature annealing of sputter deposited copper (Cu) films was investigated by electron backscatter diffraction (EBSD). Not only are the Cu film texture and grain size a function of film thickness, but also the fraction of twin boundaries present in the material is strongly dependent upon film thickness [18].

Generally, microstructure and mechanical properties of films depend on the deposition process and postproduction process such as annealing. However, most previous works have focused on the deposition process. As we know, physical properties of the sputtered films are controlled by its microstructure. In this paper, we report the microstructural evolution and mechanical properties of the magnetron sputtered Cu thin films with different thicknesses after annealing at several temperatures. Surface topography/roughness and grain size are measured by AFM. Residual stress (stresses that remain in the films after annealing) and textures of the Cu films are evaluated with X-ray diffraction. Furthermore, nanoindentation is also performed to study the mechanical properties of the Cu films, which include the hardness, yield stress (the stress at which a material begins to deform plastically), and elastic modulus. Discussions are made in terms of the mechanical properties with film microstructure.

2. Experimental Details

Three series of copper films were produced by magnetron sputtering deposition on commercial Si(100) single-crystal wafer whose thickness was $525 \pm 25 \mu\text{m}$. Size of the copper target is $\Phi 60 \text{ mm}$, 3 mm thickness. The substrate temperature T_s is 473 K, and the substrate bias U_s is grounded. Before sputtering, wafer was ultrasonically cleaned in high purity acetone. After vacuum drying, wafer was reserved in a dry cylinder for use. Copper thin films were deposited by a magnetron sputter coater (FJL560II). Copper target material purity was 99.99 wt.%. Base vacuum pressure of the sputter

coater was $2 \times 10^{-4} \text{ Pa}$, and 99.9% argon was used as a working gas whose pressure was 1.5 Pa. The distance between the sputter target and the Si wafer was 60 mm. Sputtering voltage was 470 V and power was 36 W. Thickness of the films is tested by SEM. Deposition rate is calculated by films thickness and deposition time. Different thickness films are deposited by controlling the deposition time. The deposition time was 60, 90, and 170 min, respectively, and these samples were labeled as A, B, and C accordingly. The film thickness was measured by S-4800 field emission scanning electron microscope (SEM). For samples A, B, and C, thickness was 1.0 μm , 1.6 μm , and 3.0 μm accordingly. Some samples were annealed in vacuum at 300, 400, and 500 °C, respectively. The base pressure during annealing is $4 \times 10^{-4} \text{ Pa}$.

Agilent 5420 atomic force microscope (AFM) was used to observe the sample surface morphology. Each sample was scanned at three different regions and then the typical region was utilized for analysis. Grazing incidence X-ray diffraction (GIXRD) was performed to study the texture in the annealed Cu films. Experiment was performed using the Philips X'Pert Pro XRD system with 0.5° grazing incidence. The stresses of film specimens have been studied by the $\sin^2\psi$. The mechanical properties of the annealed copper films were evaluated using a nanoindenter with a Berkovich tip [19]. The continuous stiffness modulation (CSM) technique was used, wherein the contact stiffness was measured continuously as a function of displacement under the load.

3. Results and Discussion

3.1. Surface Characterization by AFM. The representative AFM images of the coating samples with different thickness and after annealing at 300 °C are shown in Figure 1. All the samples have a columnar surface morphology. The images of the Cu films were acquired in a $1 \mu\text{m} \times 1 \mu\text{m}$ area. It is clear that as the annealing temperature increased, the surface morphology evolved and the grains grew larger in the Cu films.

S_q is the root mean square (RMS) height of the surface which is a statistical amplitude parameter representing the root mean square of surface roughness deviation from reference datum. It can be seen from Table 1 that the lowest S_q value is observed for the samples annealed at 400 °C. The roughness decreases when continuous and compact films with crystal structure are formed during the films deposited. When the annealing temperature exceeds 400 °C, recrystallization occurs. Due to agglomeration and coalescence of the grains, the roughness of Cu film surface increases.

The grain growth in the Cu films can be correlated to the annealing temperature as shown in Table 2, using the grain size data determined by AFM. The driving force for grain growth in thin films materials is the reduction of grain boundary energy that results from the reduction of the total grain boundary area. In grain growth, some grains already present in the matrix grow at the expense of the other grains. A few grains which consume the surrounding (stagnant) fine-grained matrix will grow until the large grains meet and the fine-grained matrix is completely consumed [19–22].

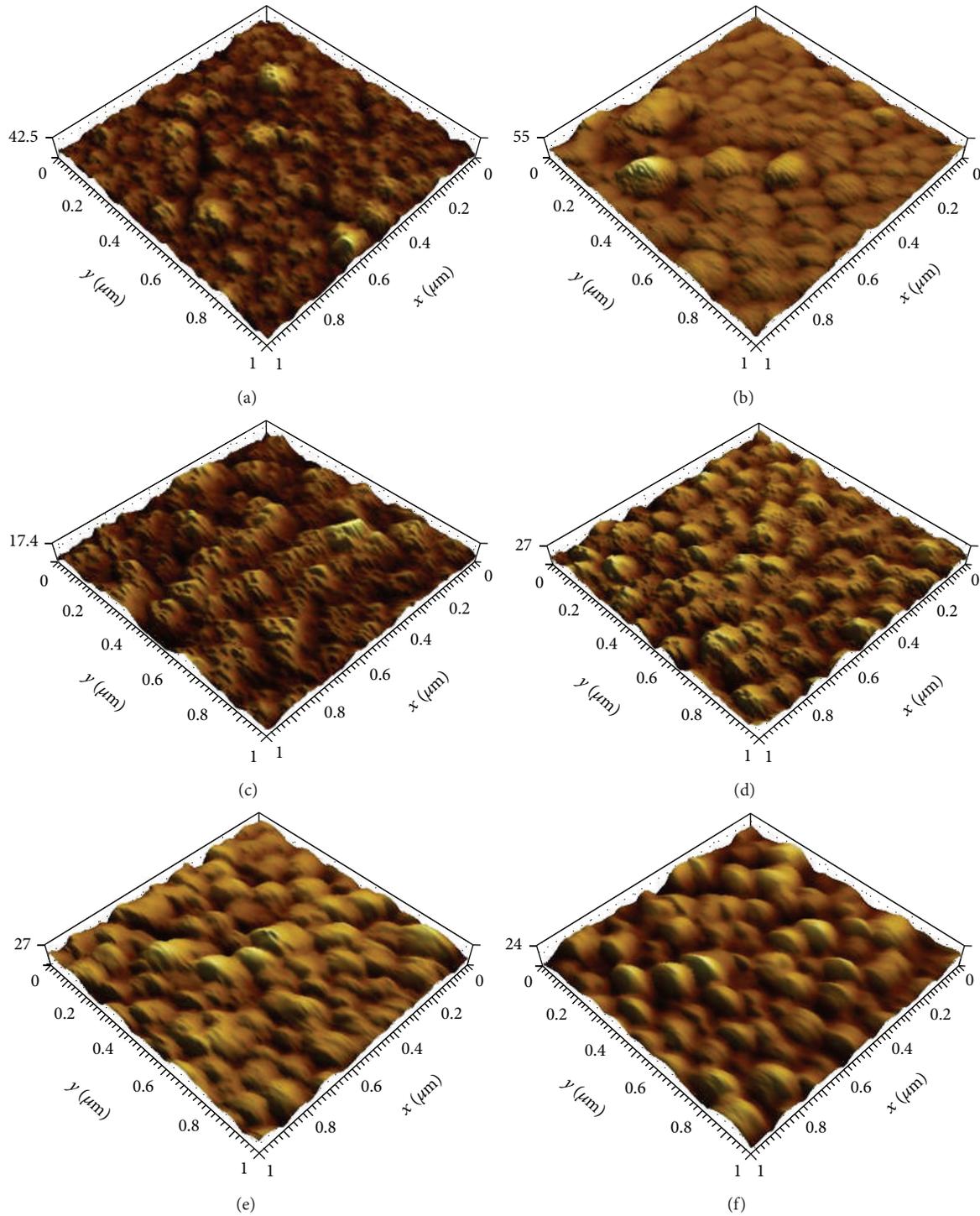


FIGURE 1: 3D surface topographies of the Cu films: (a) 1.0 μm thick, as-deposited; (b) 1.0 μm thick, annealed at 300°C; (c) 1.6 μm thick, as-deposited; (d) 1.6 μm thick, annealed at 300°C; (e) 3.0 μm thick, as-deposited; and (f) 3.0 μm thick, annealed at 300°C.

The higher the annealing temperature is, the larger the grain growth driving force is. If the annealing temperature exceeds 400°C, grains grow quickly and coalescence happens, which will cause grain boundary groove deepening and film agglomeration to occur.

3.2. Texture and Microstructure. Texture in materials has a large influence on many properties of thin films. XRD has been the primary method for the characterization of film texture for many years. The texture coefficient (TC) represents the texture of a particular plane, whose deviation

TABLE 1: Surface roughness Sq (nm) for the Cu films with different thicknesses annealing temperatures.

Annealing temperature (°C)	Thickness (μm)		
	1.0	1.6	3.0
As-deposited	3.88	3.4	2.98
300	3.66	3.09	2.61
400	1.63	2.91	2.16
500	2.88	4.08	9.48

TABLE 2: Grain size d (nm) for the Cu films with different thicknesses and annealing temperatures.

Annealing temperature (°C)	Thickness (μm)		
	1.0	1.6	3.0
As-deposited	66	88	91
300	100	124	143
400	120	165	170
500	375	405	550

from the ideal value implies the preferred growth. Quantitative information concerning the preferential crystallite orientation was obtained from the texture coefficient TC_{hkl} defined as [23]

$$TC_{hkl} = \frac{I_{(hkl)}/I_{0(hkl)}}{\sum_{i=1}^n I_{(hkl)}/I_{0(hkl)}} \times 100\%, \quad (1)$$

where $I_{(hkl)}$ is the measured relative intensity of a plane (hkl) and $I_{0(hkl)}$ is the standard intensity of the plane (hkl) taken from the JCPDS data. The value $TC_{hkl} = 1/n = 0.25$ (i.e., $n = 4$ for our case, as four planes are included in our XRD study) represents films with randomly oriented crystallites, while higher values indicate the abundance of grains oriented in a given (hkl) direction [23].

3.2.1. Effect of Thickness on Texture. From Figures 2 and 3, we can see that the preferred orientation and texture in the thin films change with the thickness. The strongest X-ray reflections are visible from Cu(111) planes. This indicates that the crystallization occurs preferentially in the (111) planes. While the polycrystalline films are thin, growth of grain with (111) texture is favored by surface and interface energy minimization, especially in very thin films [24, 25]. With the increase of the thickness, the (111) orientation decreases whereas the (220) orientation increases. Strain energy (the energy stored by a system undergoing deformation including elastic strain energy and plastic strain energy) controls the grain growth gradually as the thickness increases. Elastic strain energy is the potential mechanical energy stored in the configuration of a material as work is performed to distort its volume or shape. Plastic strain energy is the energy stored by a system undergoing plastic deformation.

3.2.2. Effect of Annealing Temperatures on Texture. Figure 4 shows the XRD patterns of the Cu films with a thickness of $1.0 \mu\text{m}$ under different annealing temperatures. All

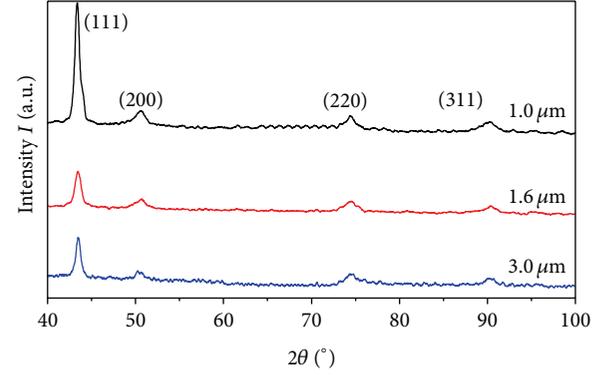


FIGURE 2: XRD patterns of the Cu films with different thicknesses.

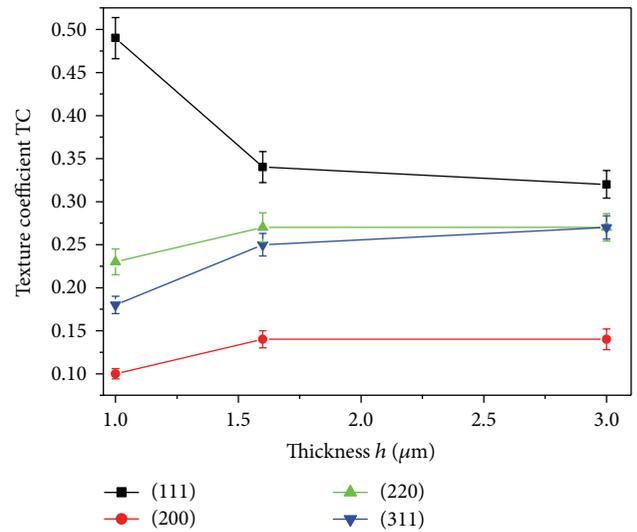


FIGURE 3: Texture coefficients of the Cu films with different thicknesses.

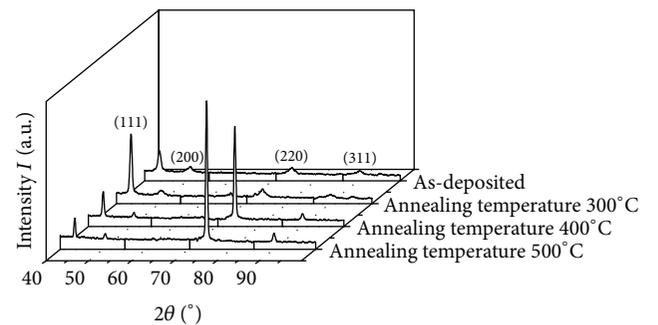


FIGURE 4: XRD patterns of the $1.0 \mu\text{m}$ Cu film annealed at different temperatures.

the films exhibit X-ray reflections from Cu (111), (200), (220), and (311). To investigate the evolution of crystallite orientations with different annealing temperatures, the texture coefficient under different annealing temperatures is shown in Figures 5–7 for the films with thickness of 1.0, 1.6, and $3.0 \mu\text{m}$, respectively.

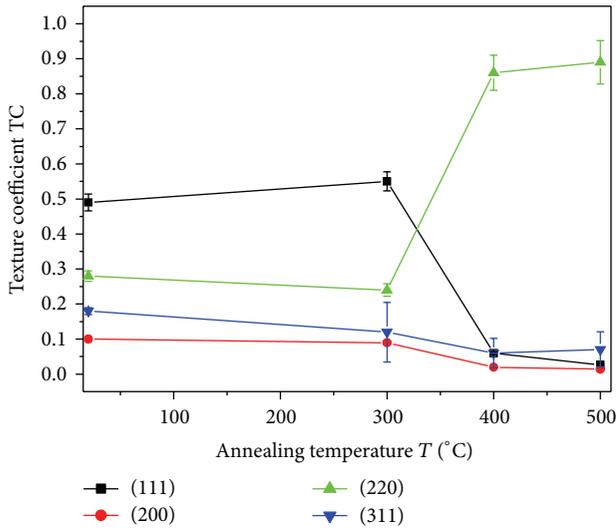


FIGURE 5: Texture coefficients of the 1.0 μm Cu film annealed at different temperatures.

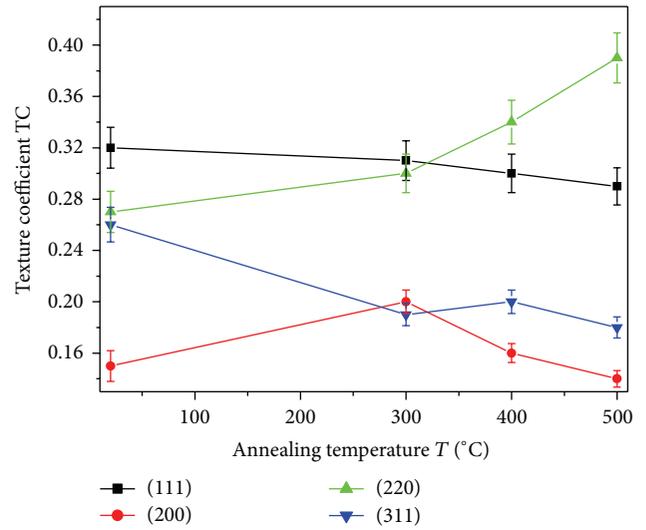


FIGURE 7: Texture coefficients of the 3.0 μm Cu film annealed at different temperatures.

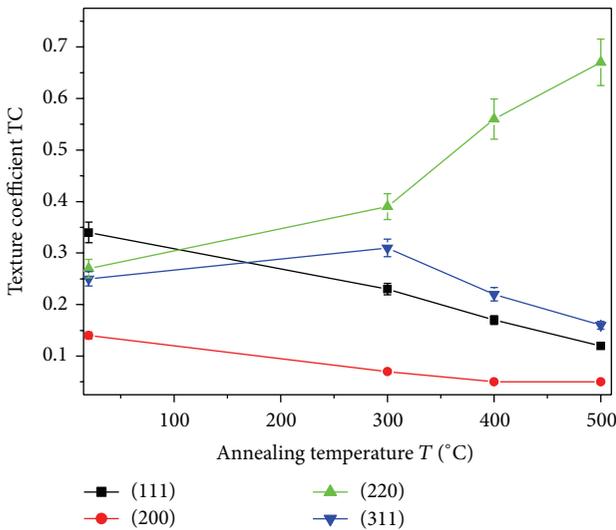


FIGURE 6: Texture coefficients of the 1.6 μm Cu film annealed at different temperatures.

The main driving force for subsequent grain growth is minimum total free energy (surface energy, grain interface energy, and film strain energy) [21]. For thinner films, grain growth is under the control of surface energy minimization. Only a few grains can grow whose surface energy is relatively lower than the others. Grains whose surface energy is higher will be merged into adjacent grains. Grain growth eliminates free surface and thus the total surface energy decreases accordingly. However, for thicker films, grain growth is under the control of strain energy minimization. For face-centered cubic (fcc) metals, grain orientation which has the lowest plastic strain energy is the (220) plane [24]. Development of strain energy minimizing textures does not minimize surface and interface energies [25, 26]. Therefore, surface structure of the copper films with different thicknesses reflects

a dynamic equilibrium between surface energy and strain energy. Surface energy is very important for the structure of thinner films, whereas strain energy is very important for the structure of thicker films.

During the annealing processes, the intensity of the (220) grain increases but that of the (111) grain decreases. A preferred grain orientation (220) after annealing is observed while (111) is the preferred orientation before annealing. The effect of yield in grain is proposed to explain the (220) preferred orientation during annealing. When the annealing temperatures increase, thin films will start to yield. Also, the minimum strain energy will control the grain growth. In-plane stress in a grain is a function of grain orientation factor C_{ijk} , and the yield stress of the grain also varies depending on its orientation. The orientation factor C_{ijk} of (220) has the smallest one of 1.42 while that of (111) has the largest one of 3.46 [27]. When the thin films start to yield, for grains of equal initial sizes, the (220) grains will yield before the (111) grains; thus, the (220) grains have an energetic advantage for further growth [28]. This yielding process also leads to strain energy minimization. This may explain why the (220) grains grow faster than other grains and become the final preferred orientation.

In addition, initial grain size also has an effect on the texture evolution. With a certain volume, the smaller and the more uniform the grain is, the more evenly the strain energy will disperse to all the grain. So the distribution of internal stress (the stress due to difference in the thermal expansion coefficients and thickness between films and substrates during annealing) will be more even which will make the grain with minimization strain energy grow easily and quickly. Grain size in thin films is smaller than thick films. We can see from Figures 5–7 that the final texture coefficient after annealing at 500°C in thick films is less than thin films.

3.3. Mechanical Properties. Nanoindentation was performed on all the Cu films annealed at different temperatures.

The typical load-indentation depth curves for the 3.0 μm film annealed at different annealing temperatures are shown in Figure 8. It can be seen from Figure 8 that the load/unload curves for all the samples are nearly similar, which may be attributed to the similar crystalline nature of Cu films. Figure 9 shows the hardness as a function of the annealing temperature for the Cu films with various thicknesses. More recent and systematic experiments indicate that both the grain size and the film thickness have a marked influence on the strength of thin films [29–32]. Relationship between the hardness and the yield stress can be expressed as $H = 3\sigma_y$. σ_y is the yield stress and H is the hardness.

Effect of different parts of microstructure on the yield stress can be expressed as [29]

$$\sigma_y = \sigma_0 + kd^{-n} + k't^{-m}, \quad (2)$$

where σ_0 is the bulk yield stress (large-grained polycrystal); kd^{-n} is the contribution from the grain boundaries (d , grain size); $k't^{-m}$ is the contribution from the film surface or interface (t , film thickness). The first two terms together form the well-known Hall-Petch relation, where $n = 0.5$ commonly. Combining the data in Table 2 and Figure 9, Figure 10 can be obtained, which shows that the grain size dependence of strength in Cu thin films on Si substrates followed a Hall-Petch type relation. This is the described Hall-Petch effect that establishes a linear dependency of the hardness with the reciprocal square root of grain size. Clearly, the strengthening of the sputtered copper films was mainly attained by grain refinement. The Hall-Petch effect is explained in terms of a restriction in the movement of grains, that is, strengthening due to the formation of pileups in the larger grain boundaries associated with low grain size. From Figure 8, we can see that the indentation depth increases with the annealing temperatures under the same load which may be a factor affecting the Hall-Petch type relation. In addition, grain size changes with the films thickness, which may be another reason for the yield stress variation.

Different load-indentation depth curves for the Cu films annealed at different temperatures imply different indentation plastic characteristics for these films. These curves can be separated into the following three stages: pure elastic deformation stage at the beginning of the load; elastic-plastic deformation stage after displacement jump; and elastic response during unload. It agrees with the Hertz contact theory well during the elastic deformation stage. The lower the annealing is, the less the elastic displacement is. Displacement jump caused by the dislocation pileup and increment on the plastic deformation region increase with the increase of the grain size. With the decrease of the grain size, the density of the grain boundaries increases. It cannot only act as the source of dislocation but also decrease the dislocation activation energy.

The values of elastic modulus can also be obtained by nanoindentation. The Young modulus decreased 20% compared to that of the traditional coarse-grained Cu. The elastic modulus is one of the intrinsic properties of a material [33]. Elastic modulus is an important indicator to reflect the bond strength between the atoms. Many factors can affect

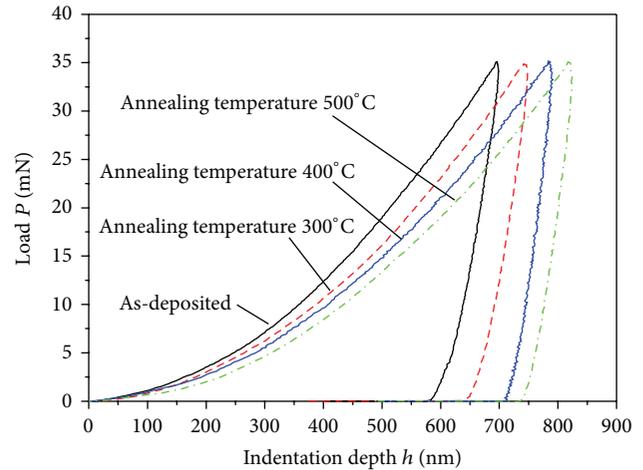


FIGURE 8: Load-indentation depth curves for the 3.0 μm Cu films annealed at different temperatures.

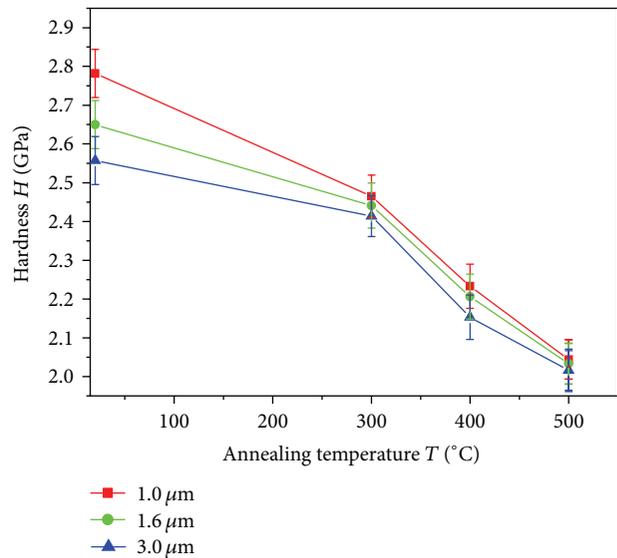


FIGURE 9: Hardness versus annealing temperature for the Cu films with different thicknesses.

the elastic modulus such as texture [33], grain coalescence, and microcrack [34]. Elastic modulus of the Cu thin films will decrease 20% when 1/3 of grain boundaries is destroyed based on the microcrack mechanism.

XRD diffraction technique was carried out to investigate the residual stress by the well-know $\sin^2\Psi$ method. Figure 11 shows the relationship between residual stress and TC(111)/TC(220) ratio in Cu films. The film with (111)-orientated grains had the highest tensile one and that with (220)-orientated grains had the lowest tensile one. The residual stress in as-deposited copper films reached a high value but decreased down to a minimum value after samples annealing. This was obviously due to the thermal relaxation of residual stresses and the annealing effect on microstructure defects. This may be due to the preferred growth of grains, which leads to a change of residual stress.

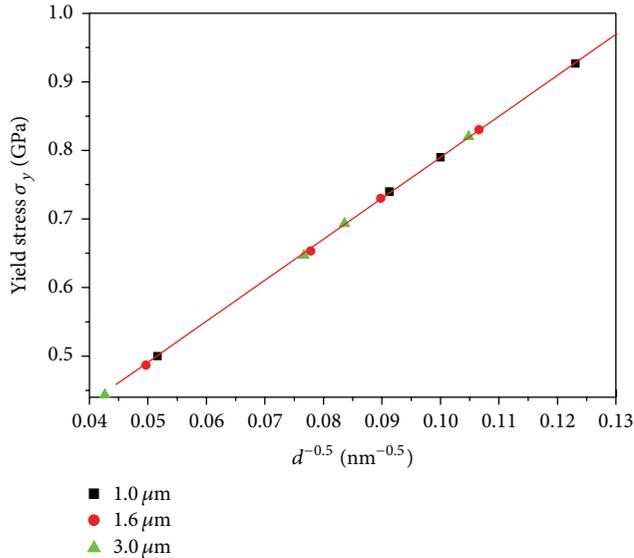


FIGURE 10: Trend of yield stress dependency on grain size for the Cu films.

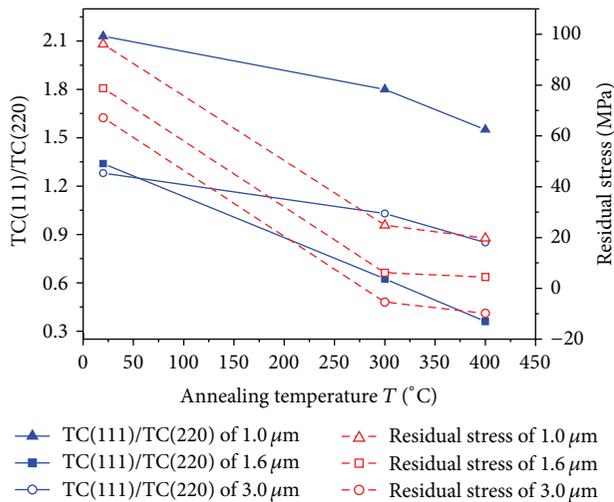


FIGURE 11: TC(111)/TC(220) ratio and residual stress versus annealing temperatures of 1.0 μm , 1.6 μm , and 3.0 μm .

4. Conclusions

The effect of annealing treatment on magnetron sputtered Cu films is investigated using AFM, XRD, and nanoindentation techniques. Surface topography and microstructural evolution after annealing is studied in detail. Relationship between the microstructure and the mechanical properties of the thin films is also proposed. The higher the texture of (111) is, the lower the resistivity is. With the increase of (111) texture, tensile stress increases. For microelectronic application, large residual stress will cause cavity, crack, and peeling of Cu films which will cause circuit deformation and even produce short circuit or open circuit. Annealing is usually taken during IC. Although the resistivity of Cu films decreased a little, the reliability of the system is greatly increased.

The following are our main conclusions.

- (1) Annealing treatment can provide enough energy for the grain to grow. When the annealing temperature is less than 400°C, the higher the annealing temperature is, the more the energy for the grain growth will be. With the grain growth, surface void is filled and the surface RMS decreases. However, when the annealing temperature is >400°C, grain grows abnormally and coalescence occurs. Surface void defects and microcracks increase and the surface RMS increases.
- (2) Films thickness, grain size, and annealing temperatures are the main factors that affect the microstructure of the annealed Cu films. The minimization of energy including surface energy, interface energy, and strain energy (elastic strain energy and plastic strain energy) controls the microstructural evolution.
- (3) The grain size dependence of strength in the Cu thin films on the Si substrates followed a Hall-Petch type relation. In addition, grain size changes with the films thickness, which may be another reason for the yield stress variation. The as-deposited Cu films are in tensile state and have strong (111) orientation. During the annealing, with the decreasing of (111) orientation, tensile stress decreased. The ratio of TC(111)/TC(220) can be used as a merit for the state of residual stress.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgments

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