Research Article

Agglomeration and Dendritic Growth of Cu/Ti/Si Thin Film

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Agglomeration and the transformation from random fractal to dendritic growth have been observed during Cu/Ti/Si thin film annealing. The experimental results show that the annealing temperature, film thickness, and substrate thickness influenced the agglomeration and dendritic growth. Multifractal spectrum is used to characterize the surface morphology quantitatively. The shapes of the multifractal spectra are hook-like to the left. Value of $\Delta\alpha$ increases with the annealing temperature rising, and Δf increases from 500°C to 700°C but reduces from 700°C to 800°C. The dendritic patterns with symmetrical branches are generated in the surfaces when the thin films were annealed at 800°C.

1. Introduction

Cu is widely used in microelectronics and ultralarge scale integration [1]. Ti is resistant to external influences and has good adhesive and gettering properties [2]. In the past two decades, Cu/Ti thin film system due to its potential applications has been studied, such as the kinetics of solid-phase interactions [3, 4], microstructure [5], metallurgical [6], the formation of interface layers [7, 8], thermal oxidation [9], mechanical behavior [5, 6, 10], and electrical properties [11]. In some Cu film systems [12–18], remarkable agglomeration occurs at the interfaces on annealing. This morphology evolution make the device performance be degraded. As film thickness decreases, the formation of holes and hillocks occurring in thin film agglomeration has become less desirable. There are rare reports about the investigations on morphology evolution of Cu/Ti thin film at different temperature, especially on the agglomeration and dendritic growth of Cu/Ti thin film.

The film growth on thermal annealing is a nonequilibrium process in which fractal pattern can be formed. For example, Roy et al. [19] reported on a fractal pattern formation due to thermal grooving in a grainy epitaxial thin film. The dendritic patterns symmetrically branched such as snowflakes are one type of the fractal patterns [20].

Multifractal analysis can depict the system characteristics in its multifractal spectra. It shows the variation in fractal characteristics of a system for a wide range of scales affecting the system behavior [21]. Compared to simple fractal, multifractal spectrum gives much more information. Thin film surfaces whose nonequilibrium growth fronts exhibit self-affine behavior can be quantitatively characterized using multifractal spectra. It is a promising technique in the study of surface characteristics in different disciplines [22–26].

In our previous research [27], two Cu/Ti thin film systems (70 nmCu/20 nmTi/0.4 mmSi <100> and 20 nmCu/70 nmTi/0.6 mmSi <100>) were deposited. The surface morphology evolution of the two thin films was different. The agglomeration processes from continuous to agglomerated islands and the transformation from random fractal to dendritic growth were not obvious. In this paper, Cu/Ti thin films were fabricated on single-sided polishing Si(100) substrate with thickness of 0.6 mm and then were annealed at the temperature from 500°C to 800°C using rapid thermal annealing (RTA) method. The phase composition and surface topography after different annealing temperature were studied. The agglomeration of Cu and dendritic growth at high temperature were obtained. Multifractal spectrum was used to characterize the surface morphology after annealing.

FIGURE 1: X-ray diffraction spectra of 90 nmCu/20 nmTi/Si thin film after annealing at various temperatures.

2. Experiment

Cu/Ti thin films were deposited on single-sided polishing Si(100) substrate with the thickness of 0.6 mm at room temperature using DC magnetron sputtering technique. Cu and Ti (purity grade 99.99%) targets of 75 mm in diameter were used. The experiments were carried out in an Ar atmosphere with a flow rate 20 sccm under the condition of a base pressure of 2×10^{-5} Pa. The substrates were cleaned with successive ultrasonic baths of acetone and absolute ethyl alcohol, rinsed with deionized water, and dried. The sputtering power was 99 W for Cu and 105 W for Ti, and the deposition rate was 4 Å/s and 1.2 Å/s, respectively. The two thin films were 90 nmCu/20 nmTi/Si and 50 nmCu/20 nmTi/Si. They were controlled by adjusting the deposition time. The deposited Cu/Ti films were annealed for 30 min at 500°C, 600°C, 700°C, and 800°C, respectively, using a RTA system (RTP-500) at Ar atmosphere with the ramping rate of 20°C/s and then furnace cooling down to room temperature. The crystallization structures of the Cu/Ti thin films annealed at different temperatures were measured using XRD technique at room temperatures, with a monochromatic Cu K α ($\lambda = 1.54$ Å), in the scan range of 2θ from 15° to 65° with a step size of 0.06 (2θ /s). The surface of the films was measured using SEM and AFM. Multifractal spectra $f(\alpha) - \alpha$ were used to determine the SEM images quantitatively.

3. Results and Discussion

The microstructure and the formation of compounds of the Cu/Ti thin film annealed at different temperature were investigated using XRD technique. Figure 1 shows the XRD patterns of 90 nmCu/20 nmTi/Si thin film annealed at 500°C, 600°C, 700°C, and 800°C for 30 min. There is no obvious reflection peak of Ti for the Cu/Ti films. It reveals that

the Ti film has an amorphous or nanocrystalline structure. Weak TiO₂ peaks can be found in all annealing thin film. It shows that parts of Ti have oxidized. Ti/Si peaks indicating the interdiffusion of Ti and Si to induce the formation of TiSi compounds can be observed after 800°C. The intensity of the TiSi peak is not obvious at temperatures from 500°C to 700°C. Strong Cu(111) and weak Cu(200) peaks are observed in all annealed thin films. It implies that the Cu films have [111] crystal orientation. The diffraction peak intensity of Cu(111) will wear off with the annealing temperature increasing. The Cu/Ti intermetallic compounds are not observed in the Cu/Ti thin film annealed at 500°C. The Cu₃Ti phase begins to appear at annealing temperature of 600°C and the intensity of diffraction peak increases with the annealing temperature increasing. The appearance of a reflection peak of Cu₃Ti phase indicates the formation of Cu-Ti compounds induced by the interdiffusion of Cu and Ti. The solid-phase reaction on the interface of Cu/Ti starts at the annealing temperature upon 600°C. After the thin film is annealed at 800°C, a new phase (Cu₃Si) is formed.

The SEM micrographs of Cu/Ti thin film annealed at temperature from 500°C to 800°C for 30 min are shown in Figure 2. It is noted that the surface is compact and void-free after annealing at 500°C. The grains are well crystallized and some anomalously large grains are distributed in the surface. After annealing at 600°C, the Cu film begins to agglomerate and the surface becomes discontinuous, and there appear a large number of voids. Some large elliptic or round particles are generated in the surface. They are the Cu/Ti intermetallic compounds (Cu₃Ti) formed upon annealing at 600°C indicated by the XRD. With the annealing temperature rising up to 700°C, the voids in the thin film enlarge and the surface becomes discontinuous. Large agglomerated islands connected with each other are observed. But the agglomerated Cu islands would not be connected any longer and were fully isolated after annealing at 800°C for 30 min. Large rectangular crystallites appear on the surface of the Cu/Ti thin film system (shown in Figure 3). These rectangular crystallites are often obtained in the Cu/Si metallization systems and correspond to the Cu/Si reaction products. It is consistent with the observation of the Cu₃Si phase in the XRD patterns of Cu/Ti thin film after annealing at 800°C. It can also be found in the Cu/Ta/TaN/Ta/Si [12] and Cu/TiB₂/Si [13] systems after annealing at 800°C for 30 min.

Figure 4 shows the AFM topographic images of Cu/Ti/Si thin film after annealing at temperatures from 500°C to 800°C. The scanning area is 4 $\mu\text{m} \times 4 \mu\text{m}$. Similar to the results of SEM images, it can be obtained that the surface is compact annealed after 500°C and some particles and voids appear on the surface annealed after 600°C. The particles are grown in size to form islands and some of the islands still connected to each other after annealing at 700°C. The agglomerated Cu islands were fully isolated after annealing at 800°C. The surface roughness was measured to study the morphology evolution. The roughness values (root mean square, Rq) are listed in Table 1. It can be seen that Rq values increase with the annealing temperature increasing.

The agglomeration of Cu can also be formed in many other Cu film or alloy systems after high temperature

FIGURE 2: Scanning electron micrograph on the surfaces of 90 nmCu/20 nmTi/Si thin film after annealing at temperature from 500°C to 800°C, (a) 500°C, (b) 600°C, (c) 700°C, and (d) 800°C.

TABLE 1: Rq and the parameters of the multifractal spectra of the samples.

	Rq (nm)	$\alpha_{\min}$	$\alpha_{\max}$	$\Delta\alpha$	$f(\alpha_{\min})$	$f(\alpha_{\max})$	$\Delta f = f(\alpha_{\min}) - f(\alpha_{\max})$
500°C	8.68	1.8147	2.1793	0.3646	0.3604	-0.0781	0.4386
600°C	10.6	1.8812	2.2658	0.3846	1.2161	0.0777	1.1383
700°C	87.1	1.7828	2.3919	0.6090	1.4912	0.0717	1.4195
800°C	92.9	1.7534	2.6913	0.9379	0.9702	0.3707	0.5995

FIGURE 3: Cu_3Si rectangular crystallites occurring after annealing at 800°C.

annealing [12–15]. The agglomeration of polycrystalline films were modeled into a three-step process: the grain growth, the formation of intergrain voids, and the formation of agglomerated islands. This process was reflected in the appearance of Cu/Ti/Si thin film after annealing at various

temperatures from 500°C to 800°C. As the SEM micrographs of Cu/Ti/Si thin film show in Figure 2, the surface of Cu thin film was compact and void-free at the beginning. With the annealing temperature increasing, the voids began to appear and enlarge in the continuous Cu film and then the Cu film became discontinuous. So, the agglomeration islands are formed and connected to each other, finally completely isolated. Film thickness significantly influences the progress of agglomeration [16–18]. Annealed after the same temperature, the film thickness becomes thinner, and agglomeration occurs easier. Figure 5 shows the SEM micrographs of 50 nmCu/20 nmTi/Si thin film annealed at temperature of 700°C for 30 min. The agglomeration of Cu is also obtained and the process is similar. Different from the 90 nmCu/20 nmTi/Si thin film, the agglomerated Cu islands in the surface of 50 nmCu/20 nmTi/Si thin film annealed after 700°C are almost isolated and only a few islands are still connected.

The formation of complex patterns and shapes such as dendritic patterns can be found in processes under nonequilibrium conditions such as aggregation. Figure 6 shows the process of dendritic growth for the 90 nmCu/20 nmTi/Si thin film annealed at temperature from 600°C to 800°C. When

FIGURE 4: AFM topographic images of 90 nmCu/20 nmTi/Si thin film after annealing at various temperatures: (a) 500°C, (b) 600°C, (c) 700°C, and (d) 800°C.

the annealing temperature is more than 600°C, the distribution of Cu_3Ti particles is regular as a straight line at local position as shown in Figure 2(b). Taken as a whole, these line structures can form a particular pattern such as tree branch. With the annealing temperature increasing, the density of the Cu_3Ti particles increases. When annealing temperature increases up to 800°C, large number of bright intermetallic compounds Cu_3Ti particles distribute in the surface to form a complex dendritic patterns. These symmetrically branched dendritic patterns are composed of large clusters with an open ramified structure and grow in a certain line orientation. In the processes of Cu/Ti/Si thin film annealed at the temperature from 500°C to 800°C, the random fractal is transformed to the dendritic growth.

The dendritic growth may be a combined action as a result of the interplay between microscopic interfacial dynamics

such as surface tension, surface kinetics and anisotropy, and external macroscopic forces [28, 29]. In general, the transition from random fractal to dendritic growth is regarded as a manifestation of the predominance of anisotropy over random noise in the growth process [20]. Microscale molecular symmetries manifested that the growth anisotropy comes from the anisotropy of the crystallite itself. The fractal growth of metal thin film is restricted to densely hexagonal surface such as the $\{1\ 1\ 1\}$ plane of Fcc metal [20] and (0001) plane of Hcp metal [30]. Therefore, anisotropic polygonal plates covered by $\{1\ 1\ 1\}$ facets are preferentially produced. Furthermore, surface energies associated with different crystallographic planes are usually different. A general sequence is $\gamma\{1\ 1\ 0\} > \gamma\{1\ 0\ 0\} > \gamma\{1\ 1\ 1\}$ [31], and the surface free energy of a FCC crystal is also in this order. According to Fan et al. [29], Sun et al. [32], and Umar and Oyama [33],

FIGURE 5: Scanning electron micrograph on the surfaces of 50 nmCu/20 nmTi/Si thin film after annealing at 700°C.

FIGURE 6: The process of dendritic growth to the 90 nmCu/20 nmTi/Si thin film annealed at temperature from 600°C to 800°C, (a) 600°C, (b) 700°C, and (c) 800°C.

FIGURE 7: The dendritic patterns of 50 nmCu/20 nmTi/Si thin film annealing after 800°C.

the $\{1\ 1\ 1\}$ plane of Cu may possess the lowest surface energy. This stabilizes the Cu plates with the $\{1\ 1\ 1\}$ plane as the basal plane for the dendritic growth.

Heat absorption of a system can be given as $Q = cm\Delta t$, where c is specific heat capacity, m is the mass related to the thickness, and Δt is the change of temperature. In the 70 nmCu/20 nmTi/Si film system reported in our previous research [27], the thickness of Si substrate (0.4 mm) is bigger than the film thickness for several orders of magnitude, so the substrate will play a major role in the endothermic process. For the same absorption heat, when the substrate thickness increases, the change of temperature becomes smaller. This is the reason why the dendritic growth occurred at 700°C for thin film system with substrate thickness of 0.4 mm but at 800°C for both film systems with that of 0.6 mm.

It can be found from Cu/Ti binary phase diagram that the Ti atoms can not diffuse into Cu to form solid solution and there is not any intermetallic compounds to generate after annealing below the annealing temperature of 500°C. If the annealing temperature increases up to 600°C, Ti can solid soluble in Cu and the new intermetallic compounds Cu_3Ti engendered in the surface of Cu film by the solid-phase reaction. Because of the anisotropic of the Cu film surface, the distribution of Cu_3Ti particles appears regular. With the temperature increasing the diffusion of Ti atoms intensified and the content of Ti solid soluble in Cu increase, the product (Cu_3Ti) after annealing grows in number or quantity and the distribution density in the surface increased. Furthermore, the film thickness also influences the density of dendritic patterns by changing the number of Cu_3Ti particles. Figure 7 shows that the dendritic patterns of 50 nmCu/20 nmTi/Si thin film annealing after 800°C. It shows the dendritic patterns are obvious, but the density is lower than the 90 nmCu/20 nmTi/Si thin film.

Multifractal analysis can be used to characterize the morphology evolution from random fractal to dendritic growth. The multifractal analysis theory has been studied [21–26, 34]. The topographic images were imported into computer and converted into black-white images [34]. The

FIGURE 8: Multifractal spectra $f(\alpha) - \alpha$ of 90 nmCu/20 nmTi/Si annealed at temperature from 500°C to 800°C.

images were stored in the computer as two-dimensional arrays in 256 grey levels. Each pixel is related to certain grey value. And then the images were divided into many boxes of size $n \times n$ ($\varepsilon = n/N$, $N = 256$, $\varepsilon < 1$) with the certain gray value. Box-counting method is used in multifractal analysis. To calculate the multifractal spectrum, we use the following definition of measure: $P_{ij} = n_{ij} / \sum n_{ij}$, where P_{ij} is the gray value distribution probability in the box (i, j) and n_{ij} is the summation of the gray values of the pixels in the box (i, j) . A singularity spectrum $f(\alpha)$ varying as the singularity of the subset of probabilities α is nominally used in analysis.

The multifractal spectra $f(\alpha) - \alpha$ of 90 nmCu/20 nmTi/Si after annealing are shown in Figure 8. It can be seen that the shapes of the multifractal spectra $f(\alpha) - \alpha$ are different and are all mainly hook-like to the left. The parameters of the multifractal spectra are also summarized in Table 1. Since $\varepsilon \leq 1$, $\alpha_{\min}$ indicates the maximum gray value distribution probability ($P_{\max} \sim \varepsilon^{\alpha_{\min}}$) and $\alpha_{\max}$ the minimum one ($P_{\min} \sim \varepsilon^{\alpha_{\max}}$) [21–26]. Thus, width $\Delta\alpha$ ($\Delta\alpha = \alpha_{\max} - \alpha_{\min}$) can be used to describe the range of the probabilities. If the $\Delta\alpha$ value is larger, the probability distribution is wider, and the difference between the most uniform and the most nonuniform gray value distribution probability is more. The $\Delta\alpha$ value is also plotted in Figure 9 for purpose of clarity. With the annealing temperature being higher, the $\Delta\alpha$ values increase from 0.3646 at 500°C to 0.9379 at 800°C. This means that nonuniformity of gray value distribution probability of 90 nmCu/20 nmTi/Si thin film increases and the surface will be rougher with the annealing temperature increasing. Since $f(\alpha_{\min})$ indicates the number of the boxes having the same minimum gray value distribution probability while $f(\alpha_{\max})$ denotes the maximum one, $\Delta f (= f(\alpha_{\min}) - f(\alpha_{\max}))$ illustrates the ratio of the number of the maximum probability and that of the minimum one. The Δf values of the thin films after annealing are all more than zero, so there is more possibility

FIGURE 9: Relationship between $\Delta\alpha$ and Δf and annealing temperature.

for the gray value distribution to occur at the maximum than at minimum sites. The value of Δf increases with the annealing temperature from 500°C (0.4386) to 700°C (1.4195) but reduces from 700°C to 800°C (0.5995) (Figure 9). This means that the probability for the gray value distribution occurring at the maximum location increases from 500°C to 700°C but reduces from 700°C to 800°C.

4. Conclusion

The 90 nmCu/20 nmTi/Si and 50 nmCu/20 nmTi/Si bilayer thin films were fabricated using DC magnetron sputtering technique. After fabrication, the films were annealed by RTA at the temperature from 500°C to 800°C. The surface morphology of the thin film had a procedure of agglomeration, compact and void-free surface, the formation of intergrain voids, the formation of connected agglomerated islands, and finally completely isolated islands. With the annealing temperature increasing, the film growth transforms from random fractal to dendritic growth evidently. The substrate thickness has effects on the occurrence of dendritic growth. Until annealed at 800°C, the dendritic patterns with symmetrical branches were generated in the surfaces of thin film systems with substrate thickness of 0.6 mm. The film thickness influences the agglomeration and dendritic growth. If the film becomes thinner, agglomeration occurred easier and the density of dendritic patterns is smaller. The shapes of the multifractal spectra $f(\alpha) - \alpha$ for 90 nmCu/20 nmTi/Si after annealing are all mainly hook-like to the left. The width $\Delta\alpha$ increased with the annealing temperature rising up. It indicates that the surface was rougher when the annealing temperature increased. The Δf values were more than zero and increased with the annealing temperature rising from 500°C to 700°C but reduced from 700°C to 800°C.

Conflict of Interests

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

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