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The influence of target composition and thermal treatment on sputtered Al thin films on Si and SiO_2 substrates

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ABSTRACT

Al thin films deposited by DC magnetron sputtering from two different target compositions Al–1%Si and Al–1%Si–0.5%Cu on n-type Si (100) and on SiO_2 substrates were investigated. Surface morphology was studied as a function of deposition temperature and thermal annealing of deposited Al thin films by optical microscopy, SEM and AFM analyses. Hillock formation in the Al layer was found to be strongly dependent on the deposition temperature in the range of 373–573 K and less on the annealing temperature in the range of 573–773 K. Hillock size and density were significantly increased when Al was sputtered on SiO_2 substrate compared to Si substrates. Al grain size was increased when sputtered from Al–Si target composition compared to Al–Si–Cu and was not influenced significantly by the annealing process. Deposition of Al films from Al–Si–Cu target composition resulted in lower hillock density and orthogonally packed fine grain structure when deposited on (100) Si substrate. Strong (111) texture of Al films on SiO_2 substrate for both target composition and (101) texture when deposited on Si substrate were determined by EBSD method.

1. Introduction

Sputter deposition of metal thin films is widely used in the fabrication of various microelectronic and microelectromechanical devices. Al is a common metal in microelectronics for interconnections and ohmic contacts to the external periphery of devices and adheres well to silicon and dielectric layers. To suppress Al/Si interdiffusion, which can cause spiking, 1% of Si is commonly added to pure Al. In addition to that, copper is added to avoid electromigration phenomenon when conducting high current densities.

Often, roughness of such thin films is increased by the appearance of hillocks. Hillocks are considered as local out-of-surface protrusions of polycrystalline Al thin films. It has been established that hillocks grow in response to the relaxation processes of compressive stress in Al film which occurs mainly due to thermal mismatch of deposited Al film and substrate in particular when films are subdued to the annealing heat treatment [1–5]. The relative softness of the aluminum easily results in plastic deformation of thin films [1]. Hillock growth is also observed in the deposition process of amorphous materials such as thick plasma enhanced chemical vapor deposited (PECVD) amorphous silicon and can be successfully suppressed by tailoring the residual stress [6].

During the annealing treatment step, the processes such as grain growth and dislocation motion cause additional favorable conditions for hillock growth. The resulting Al grain size also affects the $1/f$ noise in RF applications [7]. Hillocks usually result in a film that is milky and exhibits reduced optical reflectivity. Hillocks can also cause short circuits between the conducting layers which are separated by thin dielectric layer when their height is in the order of dielectric layer thickness in multilevel metallization schemes. Capping layers deposited on the top of Al layer were found to suppress the hillock growth [8], however in many cases this approach can not be realized due to process restrictions.

To provide more accurate information on hillock formation and their suppression, our investigation was focused on thermal conditions that potentially influence the hillock growth and can be applied during or after the metallization process step. The first thermal process investigated was the preheat step of the substrates in vacuum chamber just prior to Al sputtering sequence, defining the deposition temperature, and the second thermal process was the annealing step after the Al film was deposited on Si or SiO_2 substrate. A comparative study between two target compositions Al–Si and Al–Si–Cu has been carried out to reveal the influence and to determine optimal conditions for preparing the fine grained Al films with low hillock density.

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Texture is one of the basic microstructural properties of polycrystalline thin films and, as pointed out by some authors [9–12], it is also related to the growth of hillocks, heat treatment effects and residual stress in thin films. Very extensive study was performed by Martin et al. [9], providing the strong correlation between the Al film texture and hillock growth on TEOS silicon oxide and Ti-W substrates. While strong (1 1 1) texture was determined in the surrounding film, the analyses of individual hillocks revealed randomly orientated grains. In the work of Birkholz [11], thin Ti adhesive layers, deposited at high ion flux were investigated and significant interrelation between residual stress and preferred orientation was presented. On the basis of Al crystallinity study by X-ray diffraction and shifts in 2θ peak values, Quintana et al. [12] determined that thinner films are more stressed. They showed that by increasing the film thickness the stress is diminishing while the degree of crystallinity is increased. In the work of Park and Kim [13], the texture of Al/Ti films on SiO_2 substrate was investigated with respect to the deposition parameters. It was shown that Al (1 1 1) texture is increased by decreasing the Ar pressure and increasing the RF bias power. Due to significance of this microstructural property and to revealing the influence of different Al film composition and substrate type used in our case on the film texture, electron back scatter diffraction (EBSD) analyses were also performed.

2. Experimental methods

The experimental part of our work was carried out on low resistivity (20–25 Ohm cm), n-type, single side mechanically polished float zone (FZ) silicon wafers with < 100 > crystal orientation. Si wafers were thermally oxidized to grow 620 nm thick SiO_2. This was followed by the standard photolithographic step which defined the $150 \times 150\ \mu m^2$ thermal oxide squares to be left on the surrounding Si substrate.

By implementing this approach, each Si wafer contained both types of underlying substrates, i.e. bare Si and SiO_2 islands which were subsequently covered by sputtering the Al thin film in a single deposition run. Another advantage of this approach is that for microstructural analyses both types of substrate were included on each analyzing specimen ($5 \times 5\ mm^2$).

Al thin films were sputtered on substrates by DC magnetron sputtering process using MRC603-I sputtering equipment. MRC-603-I is a side sputtering deposition system which employs horizontal transfer of sputtered material onto substrates. This type of sputtering approach reduces defect density by preventing dislodged particles to incorporate into the film as they fall to the chamber floor. A substrate pallet is a vertically oriented plate where samples are attached and traverse the stationary target with a controlled scan speed, determining the thickness of sputtered film at chosen DC power and Ar gas pressure parameters. The substrate pallet is positioned at a distance of 45 mm from the target. Single or multiple passes can be used for obtaining the final desired thickness.

Prior to the sputtering process the Si wafers were immersed for 15 s in 5% HF solution (to remove the native oxide), cleaned with deionized water and dried with nitrogen in order to establish equal surface conditions for each sputtering sequence.

Al target composition of 5 N Al–1%Si and Al–1%Si–0.5%Cu were utilized for the sputtering of Al layers. The vacuum system was evacuated by CT-8 cryo pump backed with two stage mechanical pump. Base vacuum of the system was maintained below 2.6×10^{-5} Pa prior to each deposition process. The sputtering process was performed with 7.2 kW DC power and Ar working gas partial pressure of 1.3 Pa. Short pre-sputtering step, i.e. target cleaning, is performed prior to each deposition followed by *in situ* substrate preheat step just prior to sputtering cycle. The pallet with wafers was scanned in front of the target at the speed of 20 cm/min, corresponding to deposition rate of 7.5 nm/s. Two consecutive passes were carried out for each Al deposition. The thickness of Al, as determined by measuring the step profile with surface profiler, was $0.8 \pm 0.05\ \mu m$ and kept equal for all samples. Annealing step was performed in a quartz tube under 5% H_2:95% N_2 forming gas protective atmosphere at temperatures between 573 and 773 K. A series of isochronal annealings (30 min) were performed with heating and cooling rate of 10 K/min. In order to determine the influence of individual process parameters on surface morphology of Al layers and in particular hillocks and grain size, analyses were performed by optical microscopy, Jeol scanning electron microscopy (SEM) and atomic force microscopy (AFM) instrument (Solver PRO, NT-MDT). To investigate Al thin film texture the EBSD method was used. The specimens were analyzed by a JEOL JSM 6500F field-emission scanning electron microscope using attached EBSD camera HKL Nordlys II and Channel 5 software.

3. Results and discussion

3.1. Deposition and annealing temperature influence on the Al grain and hillock formation

One of the indicators of residual mechanical stress in sputtered Al films are hillocks which form mostly by stress relief mechanisms in the growing layer and are further developed by additional heat treatments [1]. Hillocks originate from deposition process (growth hillocks) and from the annealing process (annealing hillocks). During the deposition process, heating from the heat flux originating from the sputtering target and/or from the nucleation process results in built-in intrinsic stress in deposited layer which develops as the film grows. In turn, this can result in hillock growth or void formation, depending on process parameters such as sputter power or working gas pressure [6,14]. In situ preheat step of the substrates just before sputtering starts is applied in this study mainly to facilitate desorption of the residual contaminants from the substrate and to define the deposition temperature, thus providing additional increase of the surface mobility of sputtered material during nucleation phase on the substrate surface. Preheat temperature corresponds to the deposition temperature when the target cooling is sufficient and ion energy transfer to the substrate is moderate.

The first set of experiments was performed by sputtering Al–Si layers on Si substrate at three different deposition temperatures and subsequently annealed isothermally at 673 K, which is the most common temperature applied in our microelectronic annealing processes where Al metallization is used. In Fig. 1, sequence of SEM micrographs is presented, showing the influence of three different deposition temperatures on grain size and hillock distribution. For these analyses, formations larger than 0.5 μm were taken into account as hillocks. Hillock density was estimated and compared by counting the number of hillocks from the SEM micrographs shown below. The hillocks were clearly identified by distinctive light intensity and size with respect to the surrounding grains.

Hillocks were found to appear already on samples without the preheat step (deposition at RT), however, their growth was suppressed by increasing deposition temperature to 373 and 473 K. For the deposition temperature of 573 K, both, Al hillock size and density were observed to significantly escalate.

Further, the Al grain microstructure was analyzed with respect to the deposition temperature. Magnified view in Fig. 2 demonstrates that the polycrystalline grain size is increasing by the in-