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Marbled texture of sputtered Al/Si alloy thin film on Si

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ABSTRACT

DC magnetron sputtering is a commonly used technique for the fabrication of silicon based electronic devices, since it provides high deposition rates and uniform large area metallization. However, in addition to the thickness uniformity, coating optical uniformity is a crucial need for semiconductor industrial processes, due to the wide use of optical recognition tools.

In the silicon-based technology, aluminum is one of the most used materials for the metal contact. Both the pre-deposition substrate cleaning and the sputtering conditions determine the quality and the crystalline properties of the final Al deposited film. In this paper is shown that not all the mentioned conditions lead to good quality and uniform Al films. In particular, it is shown that under certain standard process conditions, Al/Si alloy (1% Si) metallization on a [100] Si presents a non-uniform reflectivity, with a marbled texture caused by flakes with milky appearance. This optical inhomogeneity is found to be caused by the coexistence of randomly orient Al/Si crystal, with heteroepitaxial Al/Si crystals, both grown on Si substrate. Based on the microstructural analysis, some strategies to mitigate or suppress this marbled texture of the Al thin film are proposed and discussed.

1. Introduction

Aluminum is a widely used metal both as primary interconnect material in integrated circuits and as forming Ohmic (to p-type silicon region and to heavily-doped n regions) or rectifying contacts (to lightly doped n regions) in discrete silicon devices (e.g. diodes or transistors) [1].

In addition to the properties of the material (namely conductivity, overall processability, good adherence to the silicon dioxide surface, easy to be patterned), which make aluminum ideal for silicon metallization, the variety of Physical (PVD) and Chemical (CVD) Vapor Deposition techniques allow to achieve high productivity and reliability at industrial level.

Among PVD deposition techniques, DC magnetron sputtering is very popular in the semiconductor industry, because it allows the control of the metal layer composition, overcoming oxide formation problems associated with conventional sputtering, the thickness control accuracy and a high deposition rate [2]. Moreover, it allows the direct deposition of Al/Si alloys, which is the most commonly adopted method to inhibit the aluminum silicon interdiffusion at the contacts, causing junction spiking [3].

However, the polycrystalline structure of deposited Al films can influence the electronic properties of electronic devices, through atomic (electro or stress induced) migration along grain boundaries [4–7].

The hetero-epitaxial growth of Al on Si is then required to avoid the inter-grain atomic diffusion. However, Al on Si hetero-epitaxy requires an accurate control of the deposition parameters to get a uniform metal coverage over the entire Si wafer substrate and to ensure high product reliability especially for batch deposition.

During a systematic study aimed to individuate the optimal Al/Si deposition conditions to fabricate Si Schottky diodes, it was find an apparent marbled textured Al/Si film as shown in Fig. 1A. This photograph, taken under standard diffuse illumination conditions of the Al/Si layer deposited by DC magnetron sputtering on a 6″ Si wafer, shows the presence of “milky” flakes, randomly dispersed throughout the wafer surface.

Although there is no evidence of remarkable effects on the electronic properties of the Al/Si contacts, this optical non-uniformity must be reduced not only for an aesthetic issue, since it can affect both the automatic alignment accuracy and critical size obtained during photolithographic processing [8].

To the best of our knowledge, this “marbled texture” has not been previously reported and explained. In this study, the results of different analyses are described and the correlation between the microstructure, surface roughness and crystal orientation with the optical reflectivity of

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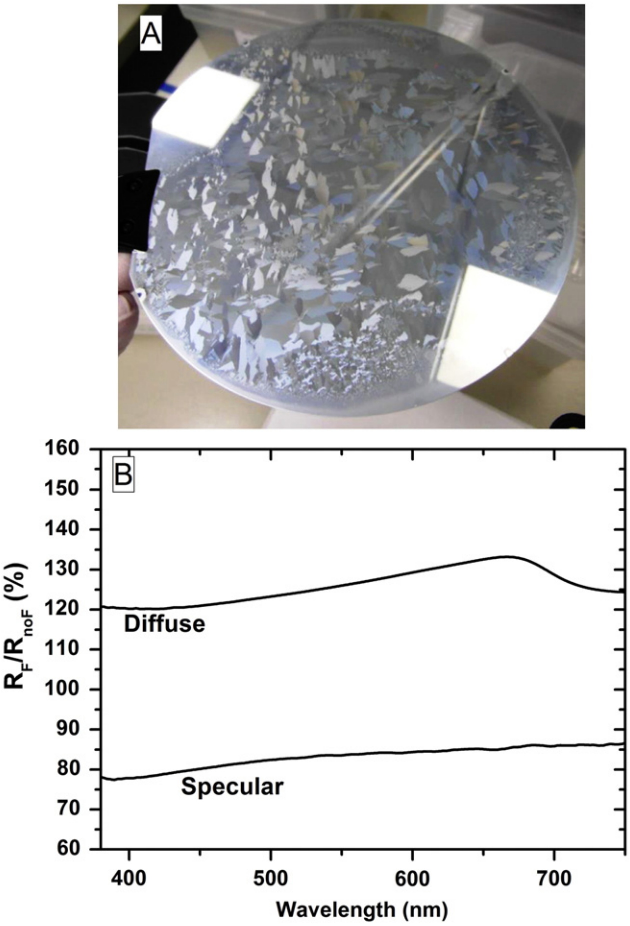

Fig. 1. (A): Photograph of Al/Si film sputtered on a 6″ Si wafer with marbled texture. (B) Specular and Diffuse reflectance spectra in the visible range of the F region, using the noF region as the reference.

the Al/Si alloy thin film are discussed. As an outcome, this study allowed the identification of experimental methods to control or to mitigate the optical non-uniformity of DC magnetron sputtered Al/Si thin films on silicon.

2. Experimental details

The substrates for Al/Si film deposition were 6” Czochralski [100] oriented p-type silicon wafers, with a resistivity of (28 ± 3) ohm·cm.

In order to remove the surface oxide, wafers were dip in aqueous HF ($3.7 \cdot 10^{-4}$ M) solution for 30 s [9]. After rinsing in de-ionized water and drying under nitrogen flow, the surface was H terminated with hydrophobic property; the water contact angles were in the range of 60–70 degree.

Al/Si thin films were then sputtered on the substrates by DC magnetron sputtering process by using a commercial DC sputtering (Varian 3290) system.

In a typical process, each cleaned wafer was loaded into the preheating chamber, and pre-annealed at 623 K in Ar atmosphere. Then the substrate was transferred to the sputtering chamber and placed on a heater plate held at 473 K.

An aluminum silicon alloy sputtering target (Al/Si with 1% Si content), was used to deposit the Al/Si alloy film. The sputtering process was performed at 4.5 kW DC power, the base pressure was $6.7{*}10^{-5}$ Pa, when the Ar was introduced the pressure increase until 0.4 Pa; the O content was <0.2 ppm. The deposition rate was 50 nm/s, and the total Al/Si film thickness about 3 μm, which is typically required for high power device operation.

An Ocean Optics USB4000 spectrometer connected to a R200-7-VIS-NIR probe for specular or to an ISP-REF for diffuse reflectance measurements was used for the optical characterization of the surface.

Morphological analyses of the Al/Si deposited thin film were carried out with a ZEISS Auriga dual beam-field emission scanning electron microscope (Focused Ion Beam (FIB) – Field Emission Scanning Electron Microscopy (FESEM)). An Oxford Energy Dispersive X-ray detector has been used to check the chemical composition.

The interface between the Si substrate and the Al/Si film was analyzed with a FEI Tecnai F20ST Transmission Electron Microscope (TEM) operating at 200 kV. An electron transparent TEM specimen in cross section was prepared from the starting sample, by using FIB, operating at 30 kV. Final polishing was accomplished at 2 kV in order to reduce the ion beam damage on sample.

X-ray diffraction (XRD) analysis was carried out using a PANalytical X'Pert X-ray diffractometer with a Cu x-ray source (Kα $\lambda = 1.54059$ A) in the Bragg-Brentano configuration.

Atomic Force Microscopy (AFM) performed with a Park System XE-100 microscopy operating in non-contact mode; morphological maps were elaborated using the XEI 1.8.0 software.

Finally, the effects of the sputtering temperature on the flake coverage was studied as function of the process temperature and correlated with the film stress measured with a Tencor Flexus Stress Analyzer FLX2320.

3. Results

Fig. 1B shows the specular and diffuse reflectance in the visible range of one region with flakes (denoted in the following as F region), using the surface without flakes (denoted in the following as noF region) as reference. Although the absolute value of the diffuse reflectance vary to a large extent due to the high optical inhomogeneity of the flakes (Fig. 1A), the spectra shown in Fig. 1B are representative of the general optical properties of the flakes, which present a milky aspect because of higher diffuse (and lower specular) reflectance with respect to the noF region.

The correlation between the surface roughness of the F and noF region and their optical properties can be inferred from Fig. 2A, B, where both optical (A) and FESEM (B) micrographs are reported. Actually, surface roughness causes light scattering of the film surfaces, making the reflection less specular in nature [10,11]. The milky aspect of the region on the right side (F) is related to higher hillocks with respect to those visible on the left side (noF), as put in evidence by the AFM map reported in Fig. 2C. The boxplot in Fig. 2D summarizes the main morphological features: the pixel height distribution is wider in F than in noF area, as indexed by the different values of the root mean square (RMS) and of the peak to valley height (Rpv).

The presence of hillocks is commonly attributed to stress relief mechanisms in the growing layer, depending on process parameters (sputter power and working pressure) and film thickness [12,13]. However, to the best of our knowledge, there are no previous reports on the macroscopic and visible to the naked eye morphological nonhomogeneity reported in this study.

In order to investigate the microstructure of these two different regions, a thin lamella straddling the F and noF regions was prepared for TEM analysis by using the conventional FIB cutting method (Fig. 3a).

From Bright-Field TEM image (Fig. 3b) it was possible to observe that the substrate is, as expected, homogeneous without evidence of grain boundaries, indicating that it is a monocrystalline Si material. On the other hand, the noF region of the Al/Si layer consists of several structured crystals (denoted as I—V) arranged in alternating domains of two different widths (narrow with ~0.4 μm and broad with ~1.5 μm). Regarding the F region, denoted as grain VI, there is no evidence of different crystal domains and a single grain is observed.