Growth of polysilicon and silicide films for MOS-VLSI application

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Abstract. Doped and undoped polycrystalline silicon films were grown by APCVD and thermal evaporation techniques. The effect of growth and annealing conditions on the crystalline nature of the films and their properties were studied by electrical, optical, x-ray diffraction and SEM techniques. Metal silicides such as TiSi_2 and PtSi_2 were prepared by co-evaporation technique over polysilicon layers to study their suitability in microelectronic applications. Some of the properties of polysilicon and silicides are discussed.

Keywords. Metal-oxide semiconductor; atmospheric pressure chemical vapour deposition; very large scale integration; polycrystalline silicon films

1. Introduction

Polycrystalline silicon films have many applications in microelectronics technology. These films are being used to simplify the fabrication technology and to obtain improved parameters of semiconductor devices.

Undoped polycrystalline films are used in planar high voltage diodes, transistors, dielectrically isolated circuits and in integrated circuits made with silicon on an insulated substrate (Clarke and Zoroglu 1970; Davidson and Lee 1969; Kuhn and Rhee 1973). Doped polycrystalline films have been used as interconnect lines, as gate electrodes in silicon gate mos integrated circuits (Faggin and Klein 1970), and as high value resistors in static memory circuits. Other applications include distributed RC line filters, lossy dielectric in SIPOS devices, low cost solar cells, a dopant source in bipolar and in self-aligned contact metal-oxide semiconductor (mos) technology (Fripp and Sterner 1970).

Use of polycrystalline silicon films is therefore of special importance in the preparation and characterization of these films (Kamins 1975; Mai *et al* 1971; Richter and Ley 1981; Feldman *et al* 1978; Anderson 1973).

A major limitation of polycrystalline silicon films as a gate material for very large scale integration (VLSI) applications is their limited conductivity which restricts their usefulness as an interconnection level (Crowder and Zirinsky 1979). Metal silicides such as TiSi_2 , PtSi_2 were prepared to study their suitability in microelectronic applications.

This paper examines some of the properties of polysilicon and silicides, grown by atmospheric chemical vapour deposition and by thermal/electron beam evaporation techniques.

2. Experimental

2.1 Atmospheric pressure chemical vapour deposition (APCVD) technique

Polycrystalline silicon films were deposited from $\text{SiH}_4\text{-N}_2$ mixtures at temperatures ranging 600–750°C in a horizontal resistance heated reactor shown in figure 1. These films were grown on polished fused quartz substrates and SiO_2 deposited silicon wafers. However, the best films were deposited at 650°C with a silane-to-nitrogen ratio of $1:1.5 \times 10^2$. The deposition rate was kept at about 500 Å/min.

2.2 Evaporation technique

High purity (5 N) silicon powder was evaporated using either a beryllium oxide crucible heated by tantalum helical filament surrounded by tantalum heat shields or an electron beam gun, on polished quartz substrates and SiO_2 deposited silicon wafers at a working pressure of about 2×10^{-5} torr. The substrates were heated by a radiation heater made of tantalum sheet positioned on top of the substrate holder. Films were deposited at different substrate temperatures ranging from 600°C to 900°C and annealed at the deposition temperature for about 1 hr. This enabled reduction of the internal stresses developed during deposition and ensured better film adhesion on the quartz substrates. The rate of deposition was of the order of 1000 Å/min.

2.3 Formation of silicides

Several potential fabrication techniques were investigated including sputtering, chemical vapour deposition and coevaporation of elements with a multihearth electron beam

Figure 1. Schematic layout of polysilicon deposition system.

evaporation unit. Direct reaction between silicon and the refractory metal was not employed because of the strong dependence of this reaction upon the interface between the metal and silicon (e.g. native oxides) (Kumar 1976; Locker and Capico 1973).

The following techniques were used: coevaporation from the elements on fused polished quartz substrates, and evaporation of titanium metal on single crystalline silicon wafers having sheet resistance of the order of 60 ohms/ $\square$. Using an electron beam gun, titanium was evaporated at a pressure of about 10^{-6} torr at 350°C. The vacuum chamber was flushed a number of times with argon to remove traces of oxygen which are detrimental to silicide formation (Murarka *et al* 1980). Samples were annealed at temperatures ranging from 600 to 750°C in a vacuum coating unit at 10^{-6} torr for about 30 min.

3. Results and discussion

3.1 Effect of annealing temperature

When annealed at higher temperatures the grains grow in size. The effect of annealing on the grain size was investigated between 700 and 1100°C for APCVD polysilicon films grown at 650°C. Heat treatment was performed in a standard resistance heated furnace in an inert atmosphere of argon gas for 30 minutes. The effect of annealing temperature upon grain size is shown in figure 2. No noticeable increase was observed in grain size with annealing between 700°C and 1000°C. Above 1000°C grain size increases from 0.2 μm to 1.2 μm . SEM micrographs of poly Si film grown at 650°C followed by annealing at 1050°C are shown in figure 3. These results agree with published data (Wada and Nishimatsu 1978; Mei *et al* 1981).

3.2 Effect of deposition temperature

X-ray diffraction patterns of polysilicon films deposited at different substrate temperatures are shown in figure 4. Using Sherrer formula the grain size of these films

Figure 2. Annealing temperature vs mean grain size of polysilicon films grown at 650°C.

Figure 3. SEM micrograph of polysilicon films (a) deposited at 650°C. (b) annealed at 1050°C for 30 min.

Figure 4. X-ray diffraction pattern of polysilicon films, deposited at different temperatures.

can be calculated from x-ray diffraction pattern. The x-ray diffraction patterns of the films deposited at different substrate temperatures are almost identical; however, an increase in crystallinity with increasing substrate temperature is observed. With increasing substrate temperatures, the width of diffraction peaks decreases and the intensity ratio of (220) to (111) peak increases due to the change in the preferential orientation of the crystallites. This has also been observed by Veprek *et al* (1981). The grain size calculated using Scherrer formula for films deposited from 700°C to 900°C varies from 100 Å to 200 Å. The films grown below 600°C, however, show an amorphous nature.

3.3 Effect of doping

A mixture of high purity silicon with 1% aluminium was evaporated at 10^{-5} torr pressure on mirror polished quartz substrates kept at 500°C followed by annealing (for 30 min) at the deposition temperature. The following conclusions have been drawn:

- (i) Grains are uniformly distributed and their size increases as a result of aluminium doping.

- (ii) An undoped poly Si film deposited below 600°C shows amorphous nature. On aluminium doping films grown at 500°C show polycrystalline nature.
- (iii) The resistivity of the films decreases as a result of aluminium doping and sheet resistance of the order of 150 ohm/□ has been obtained.

The Arrhenius plots of doped and undoped polysilicon films are shown in figures 5 and 6. For the undoped films the calculated activation energy is 0.55 eV which is half the band gap of single crystal silicon. For doped films the activation energy decreases and its value is 0.19 eV which has also been observed earlier (Richter and Ley 1981).

3.4 Optical absorption

Figure 7 shows a typical dependence of the absorption coefficient on photon energy in the range of 1 to 3 eV for thermally evaporated Si samples deposited at 900°C and 700°C respectively. The APCVD film grown at 650°C is also shown in figure 7 as also the data of Dash and Newman (1955) for single crystal silicon. It can be seen that the absorption coefficient of a polycrystalline Si film is high in the photon energy range of 1 to 3 eV and exceeds that of a single crystal silicon by an order of magnitude and decreases as the substrate temperature increases. With increasing deposition temperature the crystalline size increases as has been seen from SEM and x-ray studies.

In figure 8 the value of $(\alpha h\nu)^{1/2}$ vs $h\nu$ is plotted in the limited range of 0.5–2.5 eV. As expected in the case of indirect transitions there is linear dependence with a threshold at 1.1 eV. This value is in agreement with the indirect band gap of single crystal silicon at room temperature. There is a tail of optical absorption extending to about 0.5 eV. This suggests that at low energies the absorption is associated with some rather well-defined

Figure 5. Arrhenius plot for undoped polysilicon films.

Figure 6. Arrhenius plot for doped polysilicon films.

states in the gap which might be due to (paired) dangling bonds at the surface of the crystallites or due to other defects.

3.5 Silicide characteristics

Sheet resistance of titanium silicide as measured by the four-point probe is of the order of 5 to 7 ohm/ $\square$. Sheet resistance vs annealing temperature is shown in figure 9. Decrease in sheet resistance at higher temperature may be due to either an intermetallic formation or recrystallization of film or both. The lowest resistance obtained is due to complete transformation of metal into its silicide. The thickness of the film is measured by creating a step (by etching the film in buffered hydrofluoric acid solutions) and measuring the step height using the tally step. Silicide thickness of the order of 2000 Å to 6000 Å is obtained by changing the deposition time. The titanium: silicon ratio in the silicide is 33:67 as observed by EDAX measurements.

4. Conclusions

Polycrystalline silicon films grown by CVD and vacuum evaporation techniques were characterized using x-ray diffraction, optical absorption, electrical and SEM techniques. It was found that the doped and the undoped Si films meet all the requirements for gate material and load resistors in MOS devices. Initial experiments on the fabrication and characterization of silicides were carried out.

Figure 7. Absorption coefficient vs photon energy for polysilicon films deposited at different temperatures.

Figure 8. $h\nu$ vs $(\alpha h\nu)^{1/2}$ for polysilicon films deposited at 900°C by vacuum evaporation.

Figure 9. Sheet resistance vs annealing temperature of TiSi_2 films grown by coevaporation of Ti and Si at 300°C .

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