TEM of Aluminium-Induced Crystallisation of A morphous Silicon

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INRODUCTION

The solid phase crystallisation of amorphous silicon (a-Si) to obtain polycrystalline silicon for various applications, such as thin film transistors (TFTs), solar cells, image sensors, etc., has received a significant amount of attention. Various metals, for example, Cu, Ag, Ni, Au and Al, have been used to convert a-Si into thin poly-Si films. Aluminium-induced crystallisation (AIC) and doping of amorphous silicon has attracted researchers attention because of its ability to produce poly-Si with good crystallographic and electrical properties at low temperatures [1-3]. Although AIC has received attention in the past and is presently investigated the mechanisms involved in the conversion are still not very clear. Transmission electron microscopy [4] has an advantage over other techniques because one can see the microstructure of the material to have direct insight into the grain growth and also one can identify the initiation of the crystallisation by closely monitoring the electron diffraction pattern. In the present work specimen preparation for the TEM study of a-Si has been carried out in such a way that the films along with the specimen supporting grids can be annealed at temperatures close to 400°C. From TEM investigations it has been shown that AIC of PECVD grown amorphous silicon (a-Si:H) can be achieved at temperatures as low as 150°C which is a very significant achievement because this can be used to fabricate devices at a very low temperature resulting in low cost and low processing temperatures. The AIC temperature of sputtered amorphous silicon however was found to be higher (240°C) than that of PECVD grown amorphous silicon [5].

MATERIALS AND METHODS

PECVD Growth of Amorphous Silicon

Carbon coated nickel grids were used in our studies and were found to be quite satisfactory up to annealing temperatures as high as 400°C. Intrinsic a-Si:H films of nearly 50nm thickness were deposited on these grids using an ultra - high vacuum plasma enhanced chemical vapour deposition (UHV - PECVD) system. The deposition chamber was evacuated to a base pressure of 4.8 x 10⁻⁶ Torr and the well temperature was kept at 250°C. Pure silane (SiH₄) was used as the source gas and its flow rate was kept at 20 sccm. The SiH₄ pressure in the deposition system using a 4 inch dia pure Si disc as the target material. The grids were mounted on a steel jig which was loaded into the sputtering chamber using a computer controlled automatic substrate loading arm. The chamber was load locked using a gate valve after positioning the samples in the sputtering chamber. The chamber was then evacuated to a base pressure of 6.7x10⁻⁸ Torr using a turbomolecular
pump backed by a high speed rotary vacuum pump. The well temperature was maintained at 250°C during the whole sputtering process. High purity Argon gas was introduced into the deposition chamber and its flow rate was maintained at 25 sccm. The chamber pressure was maintained at 0.005 Torr during the sputtering process. A continuous RF power of 150 watt was maintained for the whole deposition period. The sputtering was carried out for a period of 30 minutes and under these conditions approximately 50 nm thick sputtered a-Si film was deposited on the grids. The system was precalibrated and a deposition rate of 1.3 -1.5 nm/min was obtained on Si and glass substrates under similar conditions. After the deposition the RF power was switched off and the carbon-coated nickel grids were then taken out from the sputtering chamber and kept in the specimen transfer chamber under vacuum to cool down and finally removed from the deposition system. The grids coated with sputtered a-Si were then transferred immediately to a vacuum evaporation system where an aluminium (Al) film of approximately 50 nm was deposited over a-Si film. The thickness of both the sputtered a-Si and Al were intentionally made thin to enable the viewing of the microstructure of the samples using plan view TEM and selected area diffraction patterns in order to study the crystallisation behaviour.

Vacuum Annealing of Al/a-Si Film

The grids containing a-Si deposited by PECVD as well as sputtering and having Al films on them were annealed under vacuum (2 mTorr) at different temperatures ranging between 140-250°C for 30 minutes duration. A separate grid was used for each annealing temperature and time. For every annealing experiment, a fresh grid was used so that no grid saw more than one anneal temperature. The Al film was chemically etched from surface of the silicon film on the grid using a 0.855 M KOH solution.

Microstructural Characterisation of the Films

The carbon coated nickel grids after ex-situ annealing at different temperatures were mounted on a J EOL 100CX TEM specimen holder and the microstructure of the films was observed in plan view mode. The SAD pattern was also recorded for each specimen in order to observe the crystallisation behaviour. Some grids were coated with a-Si only but not Al in order to observe the microstructure and electron diffraction pattern (EDP) of the bare film for comparison purpose.

RESULTS & DISCUSSION

Figure 1a & b show the microstructure and EDP of a PECVD grown amorphous silicon (a-Si:H) film annealed at 140°C for 20 minutes. The Al film was etched off before the TEM analysis. It can be observed that there are no large nucleation sites in the film and the film is amorphous. Figure 2a shows the microstructure of a film annealed at 150°C for 30 minutes and the EDP of the same film is shown in Fig 2b. It shows that the a-Si:H is completely crystallised into randomly-oriented, polycrystalline silicon, and the 111, 220, and 311 silicon rings are seen very prominently. Grains as large as 0.2 - 0.3µm can be seen in the micrograph and the distribution appears quite uniform. Figure 2c shows a micrograph of the same area taken at a lower magnification and reveals a network of crystals formed during AIC. The growth looks dendritic in nature. Similar features were observed by Liao and Lee [6] in their scanning electron micrographs for thicker films and annealed for much longer duration. Konno and Sinclair [7] had also shown that Al grains act as a solid phase medium for the nucleation and growth of c-Si. However, we observe in this work that the diffraction pattern, following the removal of the aluminum, contains no rings, halos, or spots except for those attributable to Si. Thus, there are no other phases at A/Si present in the crystallised silicon. We had also carried out the SEM and EDS investigations of Al induced crystallised a-Si/Al thin film structure grown on crystalline silicon substrates and found that a very uniform interface structure can be obtained by this crystallisation process [8].

The microstructure of as grown sputtered a-Si (SP-a-Si) shows that the growth of sputtered film was very smooth and there were no visible cracks or voids in them. No change in the microstructure of the film and the EDP was observed in the films annealed at temperatures ranging between 140 to 210°C. Figure 3a shows the microstructure of a Al/a-Si film annealed at 210°C for 30 minutes. It may be noticed that the Al film has large grains and a smooth a-Si film is also seen inside these grains. A selected area EDP of the same area as shown in Fig 3b reveals a prominent Si (111) halo. A ring corresponding to Al planes is also present. This clearly indicates that sputtered a-Si has not yet crystallised. Figure 4a shows the microstructure of a Al/SP-a-Si film annealed at 240°C for 30 minutes. It may be pointed out here that the unreacted Al film from this specimen was etched in a dilute KOH solution and the microstructure was recorded without Al film on top of the crystallised a-Si film. One can observe polycrystalline silicon sub micron grain size, although some of the grains are slightly larger in size. These grains have further grain boundaries in them. The selected area EDP of the same film as shown in Fig 4b depicts the (111), (220), (311) and other Si rings very clearly. Some of the sharp rings of Al are also visible which may be due to inadequate etching of unreacted Al film.

These results show that sputtered a-Si can be crystallised under vacuum at temperature as low as 240°C and for annealing time as short as 30 minutes. This temperature is higher than the crystallisation temperature demonstrated by us for PECVD grown a-Si films which contain hydrogen. This clearly shows that in PECVD grown a-Si the hydrogen present in a-Si plays a significant role in lowering the temperature of crystallisation during Al assisted crystallisation.

CONCLUSIONS

It has been shown that TEM is an excellent tool to study the crystallisation behaviour of amorphous silicon films as the electron diffraction pattern reveals the crystallisation process and grain size can be measured from the microstructure. Using TEM, conversion of PECVD grown a-Si into polycrystalline silicon by Al assisted crystallisation has been demonstrated at temperatures as low as 150°C and that of sputtered amorphous silicon at 240°C. This may open up the possibility of fabrication of silicon semiconductor devices at low temperature and hence at lower cost and on low melting point substrates. This work also shows that the AIC temperatures are higher for sputtered a-Si than the one which we achieved...
during the aluminum assisted crystallisation of PECVD grown a-Si indicating an important role of hydrogen in the process of crystallisation.

REFERENCES