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Evidence for amorphous multilayered silicon obtained by deposition under modulated pressure of hydrogen

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A new method of fabrication of layered structure of silicon is presented. The silicon was evaporated continuously under a modulated pressure of hydrogen. A contrast between dense and less dense silicon layers is evidenced by low-angle x-ray scattering and cross-sectional electron microscopy.

INTRODUCTION

During the past five years, amorphous semiconductor superlattices have been fabricated to make quantum wells and to improve optical and electrical properties of these materials. Semiconductor superlattice structures ($\text{GaAs}/\text{GaAsAl}^1$ or Si/SiGe^2) have been obtained in alternating layers of materials by molecular beam epitaxy. On the other hand, amorphous semiconductor multilayers have been obtained by glow discharges in which the composition of the reactive gas was periodically changed during the deposition process. By this method $a\text{-Si:H}/a\text{-Ge:H}$ and $a\text{-SiN:H}/a\text{-SiC:H}$ multilayers have been fabricated.³ Recently, we presented⁴ a technique of reactive evaporation to obtain hydrogenated silicon and hydrogenated silicon tin alloys. In this paper, we show that $a\text{-Si:H}_x/a\text{-Si:H}_y$ amorphous multilayers can be obtained with the same technique by evaporating silicon under a modulated pressure of hydrogen. To the best of our knowledge, this method is new and could be used to obtain a new class of multilayers.

EXPERIMENTAL TECHNIQUE AND RESULTS

Silicon was evaporated from an electron gun crucible. The deposition rate was measured and controlled by a quartz monitoring system. The quartz controller was previously calibrated by optical measurement of the thickness of stan-

dard materials. The deposition rate was the equivalent of 1 Å/s of pure silicon of bulk density. The substrate was a float glass strip. It was kept at liquid-nitrogen temperature during the evaporation process.

Hydrogen was introduced in the evaporation chamber through a 1-mm-diam tube heated at 2000 °C in order to decompose molecular hydrogen and to send a maximum of atomic hydrogen to the samples. The molecular hydrogen pressure in the evaporation chamber was measured with an ionization gauge and automatically controlled by using a RVG040 Balzers valve control unit. It was possible to make the pressure vary from 2×10^{-6} to 5×10^{-5} Torr in a few seconds (Fig. 1). For the sample characterized in this paper, the sequence of hydrogen pressure was 2×10^{-6} Torr during 30 s, then 5×10^{-5} Torr during 30 s, etc. The total number of layers was 100; the theoretical thickness of each layer was 30 Å. The samples were removed at room temperature to achieve their characterization.

Small angle x-ray scattering was performed with an incident wavelength of 1.789 Å ($K\alpha$ Co). The $\theta/2\theta$ scan is pictured in Fig. 2. At very low angle, we see the total reflexion plateau; at higher angle, the diffraction pattern exhibits two peaks and thus gives evidence of the layered structure of the sample. The intensity of the first peak is rather important for a multilayered system from which a small scattering contrast is expected. The width of this peak is reasonably nar-

FIG. 1. Hydrogen pressure cycle in the evaporation chamber during the silicon deposition.

FIG. 2. Low-angle x-ray scattering from a 65-Å wavelength-modulated Si/Si:H sample ($\lambda = 1.789$ Å).

row. The wavelength of the modulation (i.e., the width of the bilayers), as calculated from the position of the peaks, is 65 Å, which is significantly larger than the modulation expected from a deposition in high vacuum, 60 Å.

By using microcleavage technique,⁵ we collected some fragments of the sample on an electron microscope grid. The observation of the sample in cross section visually confirms the modulation. A sequence of dark and light stripes is observed very clearly in Fig. 3. A rough measurement of the distance between the stripes confirms the modulation wavelength deduced from low-angle x-ray scattering.

Then arises the question of the contrast between a sparsely hydrogenated layer and a layer deposited under a higher hydrogen pressure. The x-ray scattering from hydrogen is very weak, and a modulation of hydrogen concentration in the material would lead to an insignificant signal and is likely not the origin of the x-ray scattering peaks or of the striped pattern of the micrographs.

More likely, the contrast originates in the modulation of the density of silicon during the deposition process. The density of silicon would be large when evaporated under low pressure of hydrogen and small when the silicon is evaporated under high pressure of hydrogen.

FIG. 3. Cross-sectional observation of the sample, the x-ray scattering of which is pictured in Fig. 2.

CONCLUSION

In conclusion, we showed that the evaporation of silicon under a modulated pressure of hydrogen leads to a modulated structure. At this stage, we can only say that we have observed a modulation of the density of silicon. The question of the profile of hydrogen concentration in the sample is not resolved. During the evaporation process, the less dense layers are certainly very rich in hydrogen. It is not quite certain to what extent this condition is maintained at room temperature or after subsequent annealing. Accurate x-ray and neutron low-angle scattering have now to be performed in these samples. The comparison of the data collected from these two techniques should allow one to ascertain any possible difference between the hydrogen concentration and the silicon density profiles at room temperature or after annealing at higher temperature. This technique may provide a useful approach to the fabrication of modulated structure.

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