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Low Temperature Epitaxial Film Formation by Reactive Ion Beam Deposition

Hiroshi Yamada and Yasuhiro Torii

NTT Atsugi Electrical Communication Laboratories,

3-1 Morinosato Wakamiya, Atsugi-shi, 243-01, Japan

A new low temperature epitaxial film formation technique is proposed, in which reactive ionized species with controlled energy produced by an electron cyclotron resonance (ECR)-type microwave ion source are used as film formation materials. The effectiveness of this method has been demonstrated by growing silicon films on silicon and sapphire substrates by using silane as a material gas. Epitaxial silicon films on Si(111) and sapphire($1\bar{1}02$) substrates were successfully grown at temperatures above 400 °C and 600 °C respectively, by controlling the ion energy to satisfy the epitaxial growth condition.

1. Introduction

Low temperature film formation techniques are very important in the precise fabrication of devices, such as higher integrated VLSI's. Among various techniques for lowering film growth temperature, methods using reactive species, such as ions or excited species, have been expected to achieve this goal[1-6].

A new epitaxial film formation technique is proposed in this study, in which ionized species, produced by an electron cyclotron resonance (ECR)-type microwave ion source with reactive gases, are used as film formation materials. The ionized species beam with controlled energy is irradiated onto a substrate in a growth chamber in high vacuum. This technique (Reactive Ion Beam Deposition: RIBD[7]) opens up possibilities for lowering film growth temperature, since the ionized species impinging on the substrate surface possess high chemical reactivity and surface mobility. It also allows control of film quality, such as crystalline perfection, morphodrom and constitutional homogeneity, by adjustment of ion beam energy.

The effectiveness of this method has been demonstrated by growing epitaxial silicon films on silicon and sapphire substrates using SiH_4 as a material gas. In the latter case, called an SOS structure, the effects of ion energy on this heteroepitaxial growth have been carefully investigated; control of ion energy causes an appropriate mismatched interface layer to develop between the grown film and the substrate; this layer could strongly affect heteroepitaxial film quality.

2. Experiments

The RIBD system shown in Fig. 1 comprises three main regions: The ECR-type ion source, the growth chamber and a substrate exchange chamber.

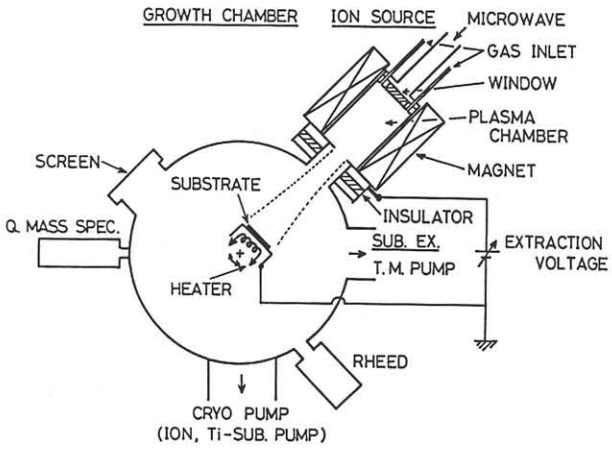


Fig. 1 Schematic diagram of the RIBD system.

This ECR-type ion source is an improvement over a similar type ion source developed by S. Matsuo et al.[4]; it allows operation in a high vacuum (less than about 10^{-5} Torr) and control of the ionized species energy in the lower region of 0-1500 eV. Material gases are supplied to a plasma chamber through a flow controller and decomposed into plasma under ECR conditions. Ionized species energy is controlled by adjustment of voltage between the plasma chamber and a substrate holder. A substrate is set on the holder and heated from the back by a filament. The growth chamber is held in high vacuum, i.e., primary base pressure is less than $3x10^{-8}$ Torr and ordinary operating pressure is less than $2x10^{-5}$ Torr, using helium refrigeration-type cryogenic and ion sputtering vacuum pumps. The substrate exchange chamber is evacuated with a turbo molecular pump.

Films have been grown on Si(111) (10-500 ohm·cm) and sapphire($1\bar{1}02$) substrates. Pretreatment was performed by a similar procedure of "Peroxide Cleaning[8]" using an H_2O_2-H_2SO_4 solution. Final cleaning for these Si substrates was performed in the growth chamber by sputter or thermal cleaning. To lower pretreatment temperature, sputter cleaning was carried out by adjusting ion energy to 500-800 eV at the same temperature as that of the film growth, except in the experiment in homoepitaxial growth maintaining a primary Si(111)-7x7 surface structure.

Film formations were performed at 100-850 °C in the substrate temperature and 0-600 eV in the ion energy, and 4 SCCM SiH_4 gas flow and 100-200 W in microwave power were supplied to the ion source. Films of about 500-6000 Å in thickness were grown. Crystal structures and surface morphology were evaluated by reflection high energy electron diffraction (RHEED) and scanning electron microscopy (SEM), respectively.

3. Results and Discussions

The possibility of decreasing growth temperature for epitaxial films and the effects of ion energy on film growth were investigated using the Si substrates pretreated with the sputter cleaning. Homoepitaxial silicon films were successfully grown at temperatures above 400 °C, by controlling ion energy, as shown in Fig. 2.

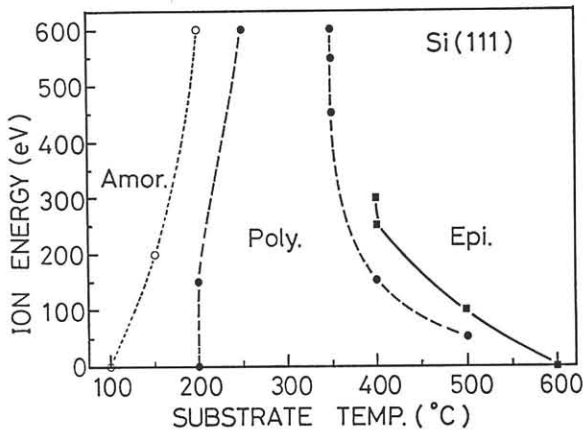
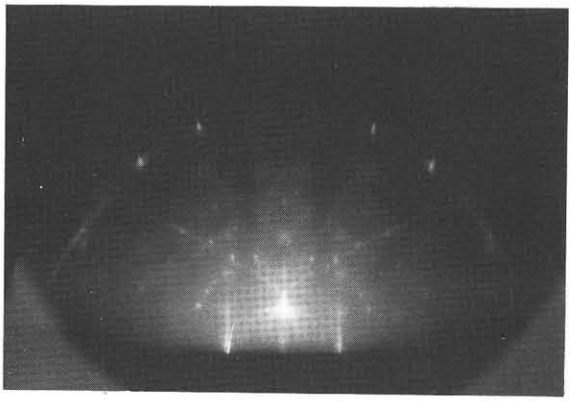


Fig. 2 Relations between the growth temperature and the ion energy, which may be shifted to about -50 eV from the actual ion energy.
Epi, Poly and Amor are abbreviations for epitaxial, polycrystalline and amorphous film growth, respectively.

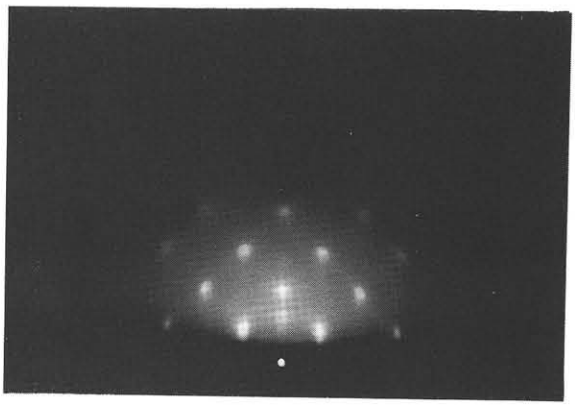
This shows the relationship between growth temperature and supplied voltage, which may be shifted to about -50 eV from the actual ion energy. Moreover, polycrystalline silicon films could be grown at temperatures above 200 °C and 250 °C on silicon and glass substrates (fused quartz and PYREX 7740) respectively.

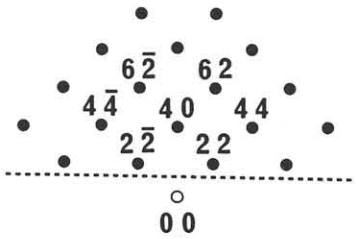
To better the film quality, homoepitaxial growth, maintaining a primary Si(111)-7x7 surface structure prepared by thermal cleaning at a temperature above 800 °C in high vacuum less than $3x10^{-8}$ Torr, was investigated. Epitaxial films with this structure were successfully grown at temperatures above 700 °C, as shown in Fig. 3. RHEED patterns grown at 600-650 °C exhibited very streaks and a trace of the 7x7 surface structure.



(Si <112> incidence)

Fig. 3 RHEED pattern of the homoepitaxial film on Si(111) grown at 700 °C and 100 eV.





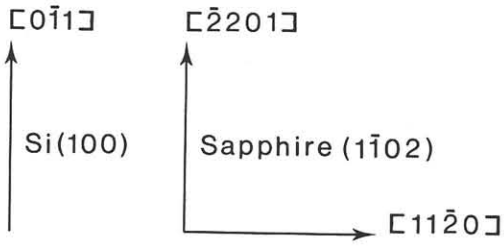
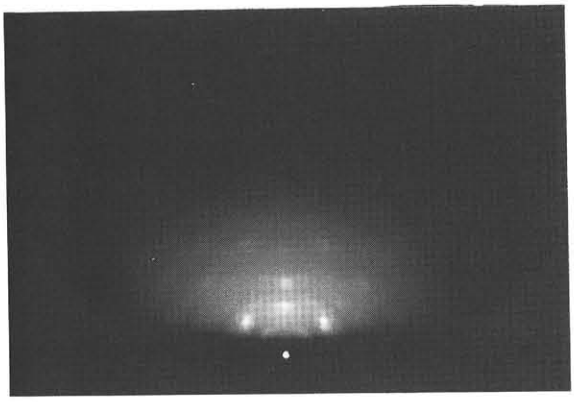


Fig. 4 RHEED pattern of the heteroepitaxial silicon film on sapphire(1$\bar{1}$02) grown at 600 °C and 300 eV, and the schematic reciprocal-space diagram of this pattern. The relationship of film growth orientation between the grown film and the substrate is also shown. (Si <110> incidence)

Heteroepitaxial silicon films on sapphire substrates (SOS) were successfully grown at temperatures above 600 °C. These growth temperatures have been relatively low compared with those for molecular beam epitaxy (MBE)[9,10].

The RHEED pattern of the film grown at 600 °C, shown in Fig.4, exhibits sharp diffraction spots, although twin structures are slightly visible. The relationship of film growth orientation between the grown film and the substrate was Si(100) || sapphire(1$\bar{1}$02) and Si [0$\bar{1}$1] || sapphire [$\bar{2}$201]. At this temperature, ion energy of more than about 300 eV was needed in the initial stage of film growth. As an example, the RHEED pattern of the SOS film grown at this temperature and at about 0 eV is shown in Fig. 5. In this figure, both diffuse diffraction spots, corresponding to the heteroepitaxial film growth and a relatively strong halo pattern are observed. Increasing ion energy sharpened these spots and extinguished the halo. Thus, at about 300 eV, the obvious heteroepitaxial growth pattern, as shown previously, was obtained. On the other hand, by increasing the gas flow, e.g., 8 SCCM, these diffraction spots also became clear. This change in film growth may be caused by increasing the growth rate, which is an important factor of the film growth.

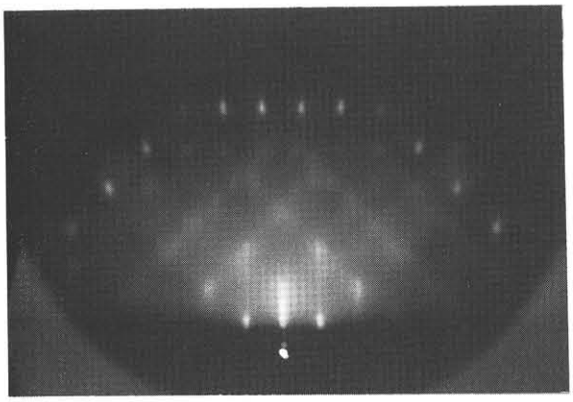
Silicon films on the same substrates grown at temperatures of 500-550 °C displayed imperfect heteroepitaxial growth. Their RHEED patterns exhibited dim diffraction spot patterns at ion energy of 0-400 eV.



(Si <110> incidence)

Fig. 5 RHEED pattern of the silicon film on sapphire($1\bar{1}02$) grown at 600 °C and about 0 eV.

Crystal structure quality was improved by increasing growth temperature, adjusting ion energy or increasing the growth rate. To better the film quality, SOS films were grown at a higher temperature and growth rate. Figure 6 shows the SOS film grown under the conditions, 700 °C and 8 SCCM SiH_4 gas flow. This RHEED pattern exhibits fine diffraction spots and Kikuchi bands, without twin structures.



(Si <110> incidence)

Fig. 6 RHEED pattern of the heteroepitaxial silicon film on sapphire($1\bar{1}02$) grown under the conditions, 700 °C, 100 eV and 8 SCCM SiH_4 gas flow.

4. Conclusions

The RIBD technique can decrease growth temperature of epitaxial and polycrystalline silicon films in the following ways:

(1) Homoepitaxial silicon films on Si(111) substrates were obtained at temperatures above 400 °C by controlling ion energy. Moreover, polycrystalline silicon films were successfully grown at 200 °C.

(2) Homoepitaxial silicon growth with the Si(111)-7x7 surface structure was performed at temperatures above 700 °C.

(3) Heteroepitaxial silicon films on sapphire ($1\bar{1}02$) substrates (SOS) were successfully obtained at temperatures above 600 °C by controlling ion energy, which played an important role in the initial stage of film growth.

(4) By increasing growth temperature and/or the growth rate, the crystal structure quality of SOS films could have been improved. Increasing the growth rate has been very effective in suppressing the generation of twins.

Acknowledgment

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