東北大学機関リポジトリ
TOUR
Tohoku University Repository

Ion Energy, Ion Flux, and Ion Mass Effects on Low-Temperature Silicon Epitaxy Using Low-Energy Ion Bombardment Process

著者	大見　忠弘
journal or publication title	Journal of Applied Physics
volume	79
number	5
page range	2347-2351
year	1996
URL	http://hdl.handle.net/10097/48044

doi: 10.1063/1.361161

Ion energy, ion flux, and ion mass effects on low-temperature silicon epitaxy using low-energy ion bombardment process

Wataru Shindo[a)]
Department of Electronic Engineering, Faculty of Engineering, Tohoku University, Aza-Aoba, Aramaki, Aoba-ku, Sendai 980-77, Japan, and Laboratory for Electronic Intelligent Systems, Research Institute of Electrical Communication, Tohoku University, 2-1-1 Katahira, Aoba-ku, Sendai 980-77, Japan

Tadahiro Ohmi
Department of Electronic Engineering, Faculty of Engineering, Tohoku University, Aza-Aoba, Aramaki, Aoba-ku, Sendai 980-77, Japan

(Received 11 September 1995; accepted for publication 20 November 1995)

In low-temperature (300–350 °C) silicon epitaxy employing low-energy inert-gas ion bombardment on a growing film surface, the effects of ion bombardment energy and ion flux as well as that of ion species on the crystallinity of a grown silicon film have been experimentally investigated. It is shown that the energy dose determined by the product of ion energy and ion flux is a main factor for epitaxy that compensates for the reduction in the substrate temperature. Large-mass, large-radius ion bombardment using Xe has been demonstrated to be more effective in promoting epitaxy at low substrate temperatures than Ar ion bombardment. Thus, low-energy, high-flux, large-mass ion bombardment is the direction to pursue for further reducing the processing temperature while preserving high crystallinity of grown films. *© 1996 American Institute of Physics.* [S0021-8979(96)03405-3]

I. INTRODUCTION

Deep-submicrometer ultralarge scale integrated (ULSI) fabrication requires total low-temperature processing. Reduction in silicon epitaxial growth temperature is, in particular, indispensable not only to avoid dopant redistribution by solid-phase diffusion and/or by autodoping effect, but also to achieve advanced device structures using metals.[1] For a high quality crystal growth to occur, however, a certain amount of energy must be provided for surface adatoms to migrate and find appropriate lattice sites. Such energy is supplied by substrate heating as thermal energy in conventional high temperature processing. Therefore, conventional silicon epitaxy process, such as thermal chemical vapor deposition (CVD), have been carried out at temperatures as high as 1000 °C or higher.

Low-energy (<100 eV) ion beam irradiation during film deposition is an effective method used to modify the grown film properties of various materials.[2–9] We have already established device-grade silicon epitaxy at temperatures as low as ~300 °C[10–12] using a low-kinetic-energy ion bombardment process. The idea of this process is to use the kinetic energy of bombarding ions incident on the growing film surface for crystal growth. The concurrent ion bombardment activates the topmost surface layer and enhances the surface migration of deposited silicon atoms, thus promoting epitaxy at such a low substrate temperature. Thus, the major factors dominating the film quality are ion bombardment energy, ion flux density, and substrate temperature, which all determine the degree of surface activation during the crystal growth. However, it is anticipated that the ion bombardment would cause damages in the film. Therefore, the influence of ion energy and ion flux as well as that of ion species on the process must be well understood. The purpose of this article is to present the effects of ion energy, ion flux, and ion species on the crystal growth at various substrate temperatures. We found that the energy dose determined by the product of ion energy and ion flux dominates the grown film quality, and large-mass, large-radius ion bombardment encourages a lowering of the epitaxial growth temperature.

II. EXPERIMENT

Figure 1 shows the rf-dc coupled mode bias sputtering system[10] used in the present work for silicon film growth. A 100 MHz rf power supply was employed to generate a high density plasma under a gas pressure of 10 mTorr. Two dc power supplies are connected to the target and to the wafer holder via low pas filters that separately control their dc potentials.

The dc potential applied to the target determines the sputtering rate, i.e., the film growth rate, while that applied to the wafer holder determines the magnitude of ion kinetic energy for bombarding the film surface. Since the excitation frequency of 100 MHz is much higher than the ion frequency (typically 2–3 MHz), the ions cannot respond to the rf component of the sheath field and are accelerated only by the dc voltage component appearing across the sheath, which is primarily determined by the external dc source. Therefore, the ion bombardment energy ϵ_i, defined as the kinetic energy of individual bombarding ions incident on the substrate surface, was determined by the potential difference between the time-averaged plasma potential V_p and the externally applied substrate dc voltage V_s. The time-averaged plasma potential V_p was evaluated using advanced single probe measurement.[13] Thus, ion energy was controlled accurately by substrate dc bias V_s. On the other hand, the rf power input controls the plasma density, i.e., the ion flux provided to the wafer. Ion

[a)]Electronic mail: shindo@sse.ecei.tohoku.ac.jp

Downloaded¬11¬Mar¬2010¬to¬130.34.135.83.¬Redistribution¬subject¬to¬AIP¬license¬or¬copyright;¬see¬http://jap.aip.org/jap/copyright.jsp

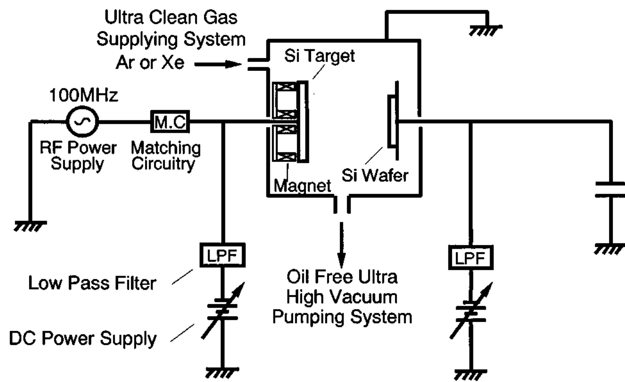

FIG. 1. Schematic of a rf-dc coupled mode bias sputtering system.

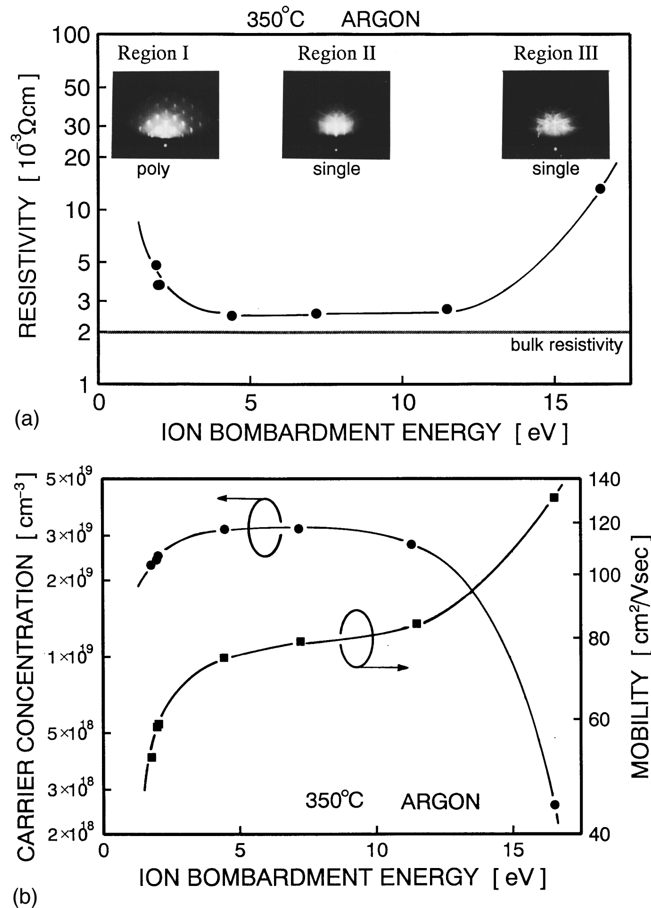

FIG. 2. Effect of the ion energy using argon ion bombardment at a substrate temperature of 350 °C. (a) The resistivity of a grown silicon film and typical RED patterns. (b) The carrier concentration and the carrier mobility.

flux density was determined from the measurements of the saturation ion current on a negatively biased substrate. Thus, by adjusting the target bias, the wafer bias, and the rf power, the key parameters in film growth such as the film growth rate, the ion bombardment energy, and the ion flux density can be selected to fit any desired combinations. It is quite essential to manage the plasma process with internal plasma parameters such as ion energy and ion flux, and not with external subparameters such as rf power and gas pressure. Thus, the principal deposition variables studied were ion bombardment energy, ion flux, and substrate temperature during deposition, while maintaining constant deposition rate (10 nm/min).

The base pressure of the sputtering chamber was 2×10^{-10} Torr and ultraclean argon or xenon gas was used. (The remaining moisture and oxygen levels were less than 1 ppb.) After performing *in situ* substrate surface cleaning[10,14] by low-energy ion bombardment, epitaxial layers were grown on *p*-type (100) silicon substrates under concurrent Ar or Xe ion bombardment, where the sputtering target was phosphorus-doped *n*-type silicon with an impurity concentration of $3-4\times10^{19}$ cm^{-3}. The *p-n* junctions formed at the epilayer/substrate interface can electrically separate the deposited layer from the substrate, making electrical measurement of epitaxial layers possible. The substrate temperature was monitored by a thermocouple attached to the back side of the wafer and was kept constant during the deposition by adjusting the substrate heating power.

Resistivity of a grown silicon film was measured by four-point probe measurements. Carrier concentration and carrier mobility were evaluated by Hall measurement. Film crystallinity was investigated by reflection electron diffraction (RED) analysis.

III. RESULTS AND DISCUSSION

The resistivity of a grown silicon film as a function of the Ar-ion bombardment energy is shown in Fig. 2(a). The substrate temperature and the ion flux were 350 °C and 1.3×10^{16} $cm^{-2}\,s^{-1}$, respectively. Typical RED patterns of grown silicon films are also presented in the figure. Film resistivity exhibits three regions with the increase in the ion energy. When the ion bombardment energy is lower than ~4 eV (i.e., region I), the films grown are poor-quality single crystal or polycrystal, and the film resistivity increases as the ion energy decreases. In region II, clear Kikuchi lines are recognized in the RED pattern, indicating that grown layers are high-crystallinity single crystal. Film resistivity becomes minimum and is very close to the bulk resistivity. In the case of region III, however, the resistivity increases with the increase in the ion energy. Figure 2(b) indicates the reason for the existence of the minimum resistivity. Figure 2(b) shows the carrier concentration and the carrier mobility as a function of the ion bombardment energy. In region I, the mobility becomes smaller as the ion bombardment energy decreases. This indicates that the lack of total energy supply to the growing film surface results in poor film quality. In region II, maximum carrier concentration is at the same level of the target impurity concentration, i.e., the dopant impurities in the sputtering target are fully incorporated into the grown film and electrically activated as well. On the other hand, when the ion bombardment energy is higher than ~13 eV, the carrier concentration steeply decreases, i.e., the carrier activity decreases with the increase in the ion bombardment energy. Therefore, the carrier mobility increases because of the decrease in the ionized impurity scattering. If the ion energy is further increased, grown films become defective due to the damage caused by ion bombardment.

Figure 3 shows the film resistivity as a function of the

Downloaded¬11¬Mar¬2010¬to¬130.34.135.83.¬Redistribution¬subject¬to¬AIP¬license¬or¬copyright;¬see¬http://jap.aip.org/jap/copyright.jsp

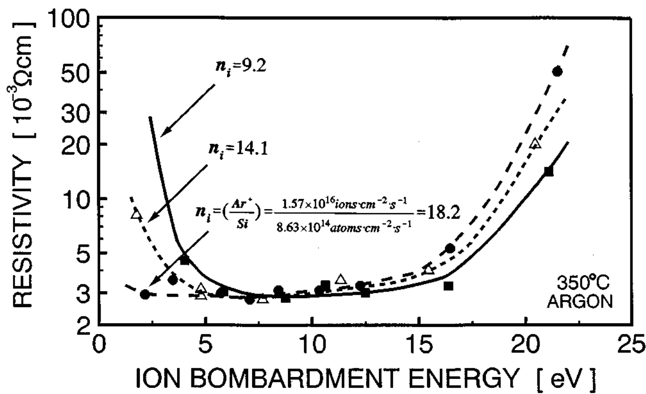

FIG. 3. The film resistivity as a function of the ion kinetic energy for three different values of the normalized Ar ion flux: n_i=9.2, n_i=14.1, n_i=18.2. Substrate temperature was kept at 350 °C during the deposition.

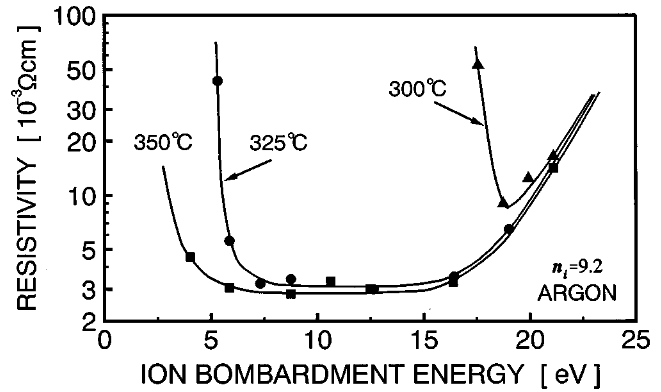

FIG. 5. The resistivity of silicon films grown at 350, 325, and 300 °C as a function of the ion kinetic energy (n_i=9.2).

ion bombardment energy under three different values of the normalized ion flux n_i, where normalized ion flux is defined as the ratio of ion flux to Si flux. In other words, n_i represents the number of bombarding Ar ions per single deposited Si atom. In the case that n_i=9.2, low-resistivity films are formed only when the ion energy is higher than 5 eV. Increasing n_i up to 18.2, low-resistivity films are realized even when ion bombardment energy is lower than 3 eV. By increasing the ion flux, high-quality films can be grown even when bombardment energy of individual ions is very low. On the other hand, however, the increase in the resistivity due to the dopant deactivation occurs when the ion energy exceeds 13 eV for all n_i, although the increasing rate is affected by ion flux.

Figure 4 summarizes the results of epitaxy at 350 °C using Ar ion bombardment. The vertical axis represents the normalized ion flux n_i, while the horizontal axis represents the ion bombardment energy ϵ_i. The quality of the Si crystal can be classified into four categories, which are indicated in the figure as regions I–IV. The region boundaries are determined by using a number of experimental results, which are also shown in the figure by symbols (○, ▲, ■). The boundary line separating region I from other regions represents the constant value of $\epsilon_i \times n_i$(≈40 eV). When the combination of ϵ_i and n_i is in region I, the carrier mobility becomes smaller and the film crystallinity degrades with the decrease in the ion energy, which was confirmed by reflection electron diffraction analysis [see Fig. 2(a)]. Within regions II and III, grown layers are single crystal. While carriers are fully activated in region II, the carrier concentration in epitaxially grown silicon film decreases drastically as the bombardment energy increases in region III. In region IV, the crystal structure of a grown film changes from defective single crystal to polycrystal and then to amorphous as the ion bombardment energy increases. Boundary energy between regions III and IV is not clearly determined yet, but it is approximately ϵ_i=25 eV.

Region I indicates that the lack of total energy supply to the growing film surface results in a poor film quality. Since $\epsilon_i \times n_i$ represents the total energy dose that is supplied to each deposited silicon atom from bombarding ions, the boundary for region I ($\epsilon_i \times n_i$=40 eV) represents the minimum energy dose for the epitaxial growth to occur at 350 °C using argon ion irradiation. Therefore, sufficient energy to grow single crystal films is provided in all regions II, III, and IV. However, epitaxial films with fully activated carriers can be obtained only in region II. It can be interpreted that ions having kinetic energies of about 13–25 eV (region III) do not induce damage in silicon but break the silicon-phosphorus bonds at the growing film surface, resulting in incomplete dopant activation. In region IV (ϵ_i>25 eV), ion bombardment energy is too excessive, resulting in the damage generation in a grown silicon layer.

The resistivity of silicon films grown at 350, 325, and 300 °C as a function of the ion kinetic energy is shown in Fig. 5, where the normalized ion flux n_i is 9.2. While dopant deactivation (positive slope—region III) is not affected by the deposition temperature, the negative slope region (crystallinity degradation—region I) shifts to higher ion energy as the substrate temperature decreases. In particular, low resistivity film growth is not achieved at a temperature of 300 °C.

Figure 6 shows the deposition temperature dependence of the region boundary. Only the region I boundary shifts toward higher energy dose as the substrate temperature de-

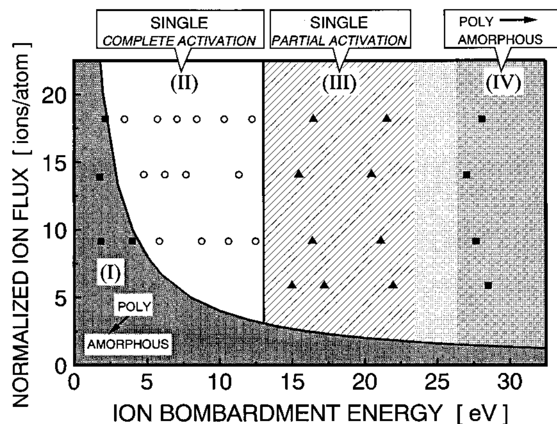

FIG. 4. The dependence of silicon film property on the combination of ion kinetic energy and normalized ion flux. (○) single crystal—complete carrier activation; (▲) single crystal—incomplete carrier activation; (■) defective single crystal, polycrystal, or amorphous.

Downloaded¬11¬Mar¬2010¬to¬130.34.135.83.¬Redistribution¬subject¬to¬AIP¬license¬or¬copyright;¬see¬http://jap.aip.org/jap/copyright.jsp

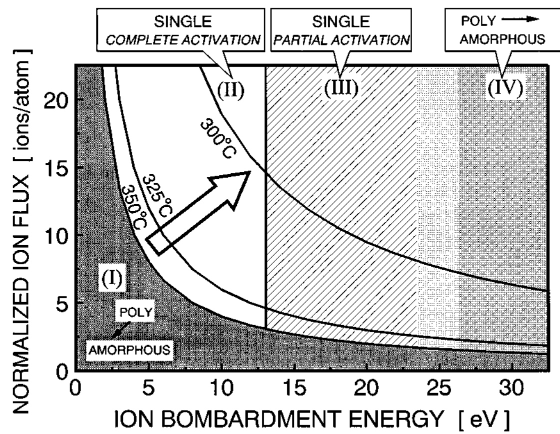

FIG. 6. The dependence of the region boundary on the deposition temperature (300–350 °C) using argon ion bombardment.

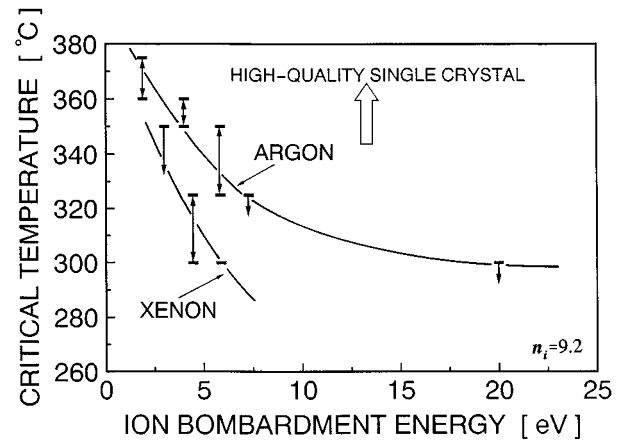

FIG. 8. The minimum substrate temperature for obtaining high-quality single crystal film as a function of the ion bombardment energy using Ar or Xe ion bombardment (n_i=9.2).

creases ($\epsilon_i \times n_i$=40, 60, and 190 eV for 350, 325, and 300 °C, respectively). It has been experimentally shown that the deficit in the substrate temperature can be compensated by increasing the energy dose. More specifically, higher energy dose is necessary to promote epitaxy at lower substrate temperature. Thus, increasing $\epsilon_i \times n_i$ is the primary requirement for obtaining good epitaxy at very low substrate temperatures. However, dopant deactivation occurs when ion energy is higher than about 13 eV, regardless of the substrate temperature. Thus, increasing the ion flux density while keeping the ion bombardment energy low is the proper method to further reduce the processing temperature.

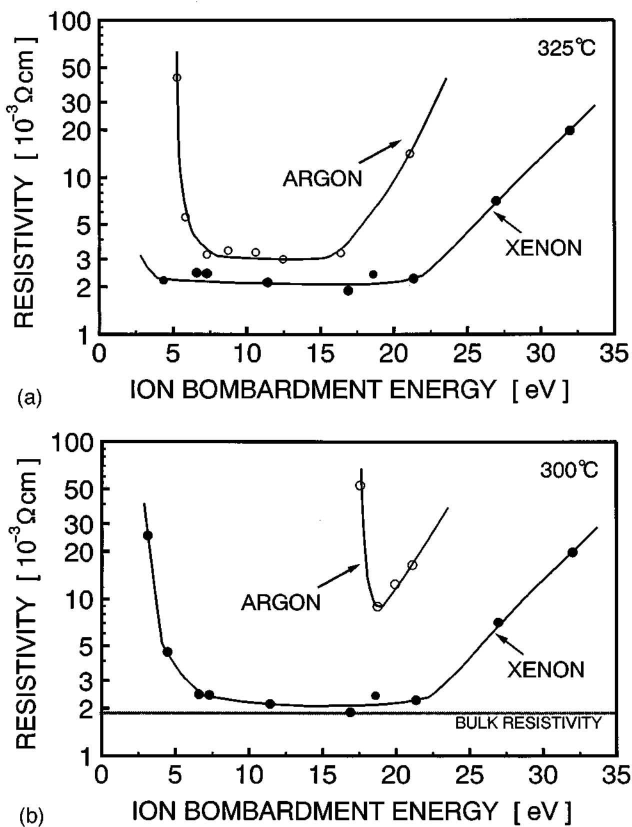

FIG. 7. The resistivity of a silicon layer grown at (a) 325 and (b) 300 °C as a function of the ion bombardment energy. The normalized ion flux of both Xe and Ar is 9.2.

The film resistivity as a function of the ion energy using Ar and Xe ion irradiation is shown in Fig. 7(a). Substrate temperature was maintained at 325 °C. In the case of Ar, the low-resistivity layers are grown only from 7 to 15 eV of the ion bombardment energy. In the case of Xe, however, high-quality low-resistivity films are obtained in a much wider region, i.e., from 4 to 20 eV. The advantage of using Xe as bombarding species becomes more evident by reducing the substrate temperature to 300 °C. Figure 7(b) shows the results of experiments conducted by using either Ar or Xe at the substrate temperature of 300 °C. While argon ion bombardment does not produce low-resistivity film growth because of the reduction in the substrate temperature, excellent low-resistivity films are realized in a very wide range of the ion bombardment energy by using xenon, even under the same ion flux density. It is obvious that the incidence of Xe ions effectively activates the growing film surface. This result indicates that the region boundaries in Figs. 4 and 6 are not absolute lines but strongly depend on the bombarding ion species.

The minimum deposition temperature for obtaining high-quality single crystal film as a function of the ion bombardment energy is shown in Fig. 8. Much lower ion energy is sufficient for achieving high-quality film growth in the case of xenon than in the case of argon under the same flux density: n_i=9.2. In other words, Xe^+ bombardment is very effective in enhancing the crystal growth at low temperatures. The difference between Xe and Ar increases with the reduction in the deposition temperature. This result clearly proves the advantage of using large-mass, large-radius ions for lowering the processing temperature of silicon epitaxy.

Table I shows the atomic masses[15] and the atomic radii[16] of argon, xenon, and lattice silicon. There are two possible reasons for the advantage of xenon compared to argon. The first point is that xenon has a larger mass than argon. Assuming a simple center-of-mass elastic collision of two isolated

Downloaded¬11¬Mar¬2010¬to¬130.34.135.83.¬Redistribution¬subject¬to¬AIP¬license¬or¬copyright;¬see¬http://jap.aip.org/jap/copyright.jsp

TABLE I. The atomic masses and the atomic radii of argon, xenon, and lattice silicon (Refs. 15 and 16).

	Atomic mass	Atomic radius (Å)
Ar	39.948	1.88[a]
Xe	131.30	2.17[a]
Si	28.086	1.17[b]

[a]Standard atomic radius in inert gas configuration.
[b]Atomic radius when in tetrahedral covalent bonds.

particles, the energy transfer efficiency η from impinging ion to Si atom is given by

$$\eta=\frac{4\mu}{(\mu+1)^2}, \tag{1}$$

where μ is the mass ratio of bombarding ion (M_{ion}) to Si atom (M_{Si}), viz.,

$$\mu=\frac{M_{\mathrm{ion}}}{M_{\mathrm{Si}}}. \tag{2}$$

The mass ratio of Ar/Si is only 1.4, but that of Xe/Si is 4.7. While 97% of the bombarding energy is transferred from the Ar ion to the Si atom in a single collision event, only 58% of the bombarding energy is transferred from Xe to Si. This model is too simple to describe the complicated process occurring between the impinging inert-gas ion and the surface of the growing silicon film. However, we can say with some certainty that large-mass ion transfers less energy with each collision. Therefore, it is assumed that less defects are generated in the case of large-mass ions. The second point is atomic radius. Large-radius ions have high probability of hitting Si adatoms on a growing film surface. Furthermore, the penetration probability of incident xenon ions into the silicon lattice might be smaller than that of argon ions. Thus, it is presumed that xenon ion transfers less energy with each collision, but such energy is focused effectively on the near-surface region. The point is that large-mass, large-radius ion bombardment might equivalently achieve very low-energy, high-flux ion bombardment. Therefore, the use of large-mass, large-radius ions in the low-energy ion bombardment process is the proper way to reduce epitaxial silicon growth temperature.

IV. CONCLUSION

The relationship between the Xe or Ar ion bombardment condition and properties of epitaxial silicon films grown at very low substrate temperatures is investigated. It is shown that the energy dose that is determined by the product of ion energy and ion flux can compensate for reduction in the substrate temperature, and large-mass, large-radius ion (Xe^+) bombardment is quite effective in promoting epitaxy. Thus, low-energy, high-flux, large-mass ion bombardment is the direction to pursue for further lowering of the processing temperature.

ACKNOWLEDGMENT

The majority of this work was carried out in the Superclean Room of the Laboratory for Electronic Intelligent Systems, Research Institute of Electrical Communication, Tohoku University, Japan.

[1] T. Ohmi, Jpn. J. Appl. Phys. **33**, 6747 (1994).
[2] J. L. Vossen, J. Vac. Sci. Technol. **8**, S12 (1971).
[3] J. W. Pattern and E. D. McClanahan, J. Appl. Phys. **43**, 4811 (1972).
[4] N. Savvides and B. Window, J. Vac. Sci. Technol. A **4**, 504 (1986).
[5] J. E. Yehoda, B. Yang, K. Vedam, and R. Messier, J. Vac. Sci. Technol. A **6**, 1631 (1988).
[6] G. K. Wehner, R. M. Warner, Jr., P. D. Wang, and Y. H. Kim, J. Appl. Phys. **64**, 6754 (1988).
[7] T. Ohmi, H. Kuwabara, S. Saitoh, and T. Shibata, J. Electrochem. Soc. **137**, 1008 (1990).
[8] T. Ohmi, T. Saito, M. Otsuki, T. Shibata, and T. Nitta, J. Electrochem. Soc. **138**, 1089 (1991).
[9] Y. Kawai, N. Konishi, J. Watanabe, and T. Ohmi, Appl. Phys. Lett. **64**, 2223 (1994).
[10] T. Ohmi, T. Ichikawa, H. Iwabuchi, and T. Shibata, J. Appl. Phys. **66**, 4756 (1989).
[11] T. Ohmi, K. Hashimoto, M. Morita, and T. Shibata, J. Appl. Phys. **69**, 2062 (1991).
[12] W. Shindo, M. Hirayama, and T. Ohmi, Jpn. J. Appl. Phys. **34**, 800 (1995).
[13] M. Hirayama and T. Ohmi, in *Extended Abstracts of the 1994 International Conference on Solid State Device and Materials* (Japan Society of Applied Physics, Yokohama, 1994), p. 697.
[14] T. Ohmi, T. Ichikawa, T. Shibata, K. Matsudo, and H. Iwabuchi, Appl. Phys. Lett. **53**, 45 (1988).
[15] *American Institute of Physics Handbook*, 3rd ed., edited by D. E. Gray (McGraw-Hill, New York, 1972), pp. 7–6.
[16] C. Kittel, *Introduction to Solid State Physics*, 6th ed. (Wiley, New York, 1986), p. 76.

Downloaded¬11¬Mar¬2010¬to¬130.34.135.83.¬Redistribution¬subject¬to¬AIP¬license¬or¬copyright;¬see¬http://jap.aip.org/jap/copyright.jsp