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Novel materials processing and applications by gas cluster ion beams

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Abstract. Gas cluster ion beams are found to offer a number of new and important opportunities for processing of materials. Ultrashallow ion implantation by cluster ion beams has been demonstrated experimentally and confirmed by molecular dynamics simulations. Very high-rate sputtering, with sputtering yields of one or two orders of magnitude greater than those produced by monomer ion beams, has also been studied in detail. Surfaces sputtered by cluster ion beams become smoother when physical sputtering is provided by nonreactive gas species, but chemical sputtering by reactive gases does not produce the same reduction in roughness effect. The smoothing effects produced by cluster ions cannot be produced by monomer ion beams. Unique bombarding characteristics of cluster ion beams have been applied to the formation of source/drain shallow junctions for 40 nm p-MOSFETs, to high-yield etching and surface smoothing of Si, YBCO, diamond, and SiC substrates, and to the production of electronic and optical devices. A low-temperature thin-film formation technique by cluster ion-assisted deposition has also been developed for high-quality oxide films. This paper reviews recent equipment development and discusses several new applications.

PACS. 79.20.Rf Atomic, molecular, and ion beam impact and interactions with surfaces – 36.40.-c Atomic and molecular clusters

1 Introduction

The gas cluster ion beam processes being developed at Kyoto University are based on a new concept of ion and solid surface interactions [1–4]. The clusters may consist of hundreds or thousands of atoms. Gas cluster ion beam processes are distinctly different from those of the traditional ion beam processing. Multicollisions during the impact of accelerated cluster ions upon substrate surfaces produce fundamentally different effects. They offer capabilities for producing very low- energy implantation, extremely high-yield sputtering, unique surface smoothing, low-damage surface cleaning, and low-temperature thin-film formation. Cluster ion beam processing represents a versatile technology that can be applied to satisfy critical requirements associated with many advanced materials.

The research and development of gas cluster ion beam processing toward industrial applications has been supported by JST (Japan Science and Technology Corporation, organized under the Agency of Science and Technology) since 1989. The work is being conducted by Kyoto University Ion Beam Engineering Experimental Laboratory in collaboration with several industrial partners [5]. Topics include: (i) shallow ion implantation (Fujitsu Research Laboratory Ltd. for ULSI applications) [6,7]; (ii) atomic scale surface smoothing and low-damage processes for metals [3], dielectrics, superconductors [8] and diamond film surfaces [9] (using Mitsubishi Materials Co. for diamond surface finishing, for SOR and X-ray lithography, Adachi-Shin Ind. Co., Ltd. for nonspherical plastic lens mold surface smoothing, and Japan Pillar Corp. Ltd., for SiC surface smoothing for SOR mirrors); (iii) very high-yield sputtering and etching processes [2]; and (iv) high-quality thin multilayer film coatings for reliable and durable optical filters [10] (Adchi-Shin Ind. Co., Ltd.). Equipment development and thin-film formation technique development are also being done under a consortium project supported by MITI. This paper reviews recent equipment development and several new applications.

2 Cluster ion beam equipment

Figure 1 shows a schematic diagram of the general gas cluster ion beam apparatus for implantation and sputtering. The cluster beam is formed by the supersonic expansion of gases (at approximately 5 bar) through a small nozzle into a high vacuum. Collimated neutral cluster beams are ionized by electron bombardment. Ionized cluster beams are extracted, and particular sizes of cluster ions can be selected by a mass filter. Cluster size can be selected by a static lens system consisting of several electrostatic field plates with small apertures. This electrostatic field fil-

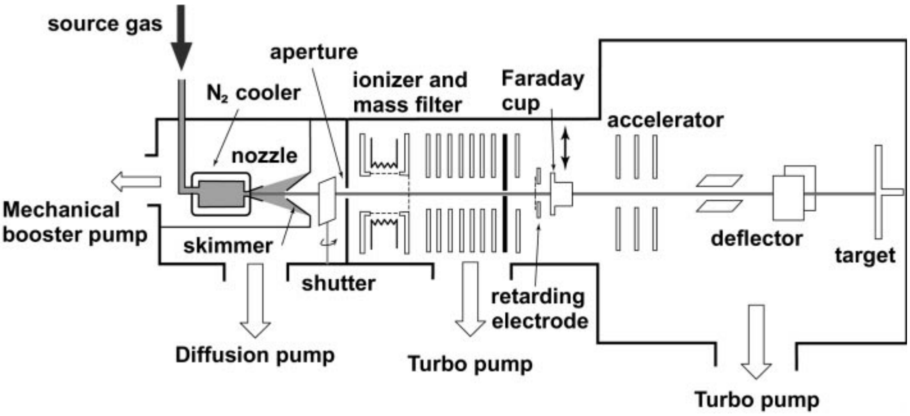


Fig. 1. Schematic diagram of 30 keV gas cluster ion beam equipment.

ter can be employed for processes in which a wide cluster size distribution is acceptable, for example in sputtering apparatus. Some applications require more narrow cluster size distributions. For example, at Kyoto University an EXB-type mass filter is used with a 200 keV cluster ion implantation system and the mass-filtered cluster ions are then electrostatically accelerated towards the target [2].

In Fig. 2, the schematic of a multibeam gas cluster ion beam-assisted deposition system is shown. This multibeam apparatus is equipped with two electron beam evaporators and one gas cluster ion source. The cluster source chamber is evacuated by an 880 l/s turbomolecular pump, and the deposition chamber is evacuated by a 20-inch diffusion pump (17 500 l/s) to pressures below 5×10^{-7} Torr. The deposition chamber can be maintained at high vacuum level during the cluster ion source operation. This equipment employs PC control [11].

Suitably intense cluster beams from gases such as Ar, N_2, O_2, CO_2, N_2O, SF_6, etc. have been generated by supersonic expansion of these gases through a nozzle with throat diameter of 0.1 mm [2]. A TOF mass spectrum of small Ar cluster ions ($2 < n < 30$) at a source pressure $P_0 = 4500$ Torr (6 atms) is shown in Fig. 3a. The spectrum shows many peaks at intervals of 40 atomic mass units, i.e., at intervals corresponding to integral numbers of Ar atoms. Distribution of TOF (time-of-flight) mass spectra for large cluster-sizes at various source pressures (760 to 3800 Torr) are shown in Fig. 3b. Both the beam intensity and the mean cluster size increase with the source gas pressure. At a source pressure of 3800 Torr, the mean cluster size is about 1000 atoms/cluster. With the utilization of a suitable method, such as expansion of a pure gas or gas mixture with cooling or heating of the nozzle system, intense cluster beams can be formed from a wide variety of gases. Details of the spectrum are discussed in another paper [12]. A gas mixture method, for example, Ar and O_2, has been found to increase the cluster beam intensity by up to two orders of magnitude [13]. This characteristic of cluster beam formation by gas mixture expansion will have useful applications to materials processing.

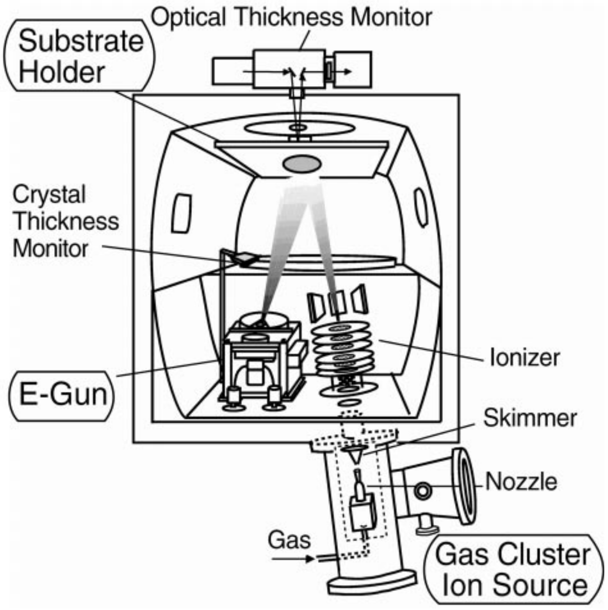


Fig. 2. Schematic diagram of gas cluster ion beam-assisted deposition equipment.

3 Cluster ion implantation

For semiconductor device applications, especially for CMOS fabrication, extensive effort has been placed on the development of very low-energy ion implantation equipment. It is clear that during the next decade, very low-energy ions will become essential for ultrashallow junction formation. However, a major problem for low-energy ion beam equipment is that the maximum current density that can extracted from a plasma ion source is related to the extraction voltage, and is limited by the Child–Langmuir law. In conventional shallow junction implant processes, required implant energies for monomer ions, especially light ions such as B and BF_2, are being reduced to levels of a few hundreds eV.

Clusters can help alleviate this problem, as we will see below. Consider a monomer of mass M_1 and a cluster consisting of N atoms of mass M_1 (mass M_N). For the same

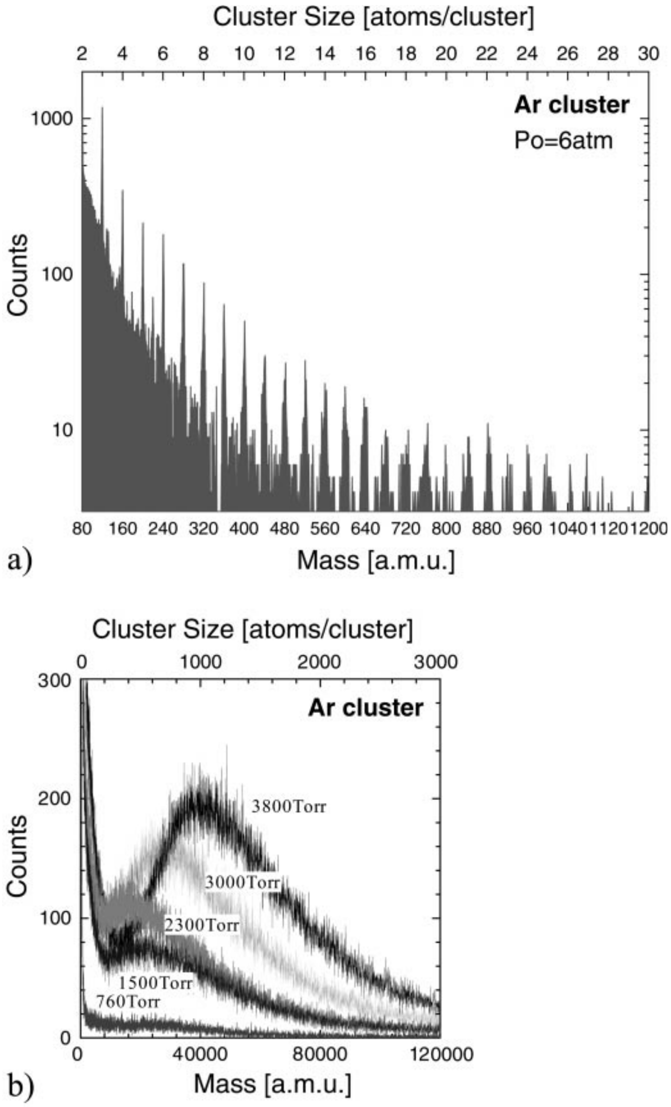


Fig. 3. TOF mass spectra of Ar cluster ions for (a, upper panel) small Ar cluster ions ($2 < n < 30$, $P_0 = 4500$ Torr) and (b, lower panel) large Ar cluster ions at various source pressures ($P_0 = 760$–3800 Torr)

penetration depth of constituent particles, the cluster may be extracted at an energy which is higher by the ratio of masses:

$$E_N = \frac{M_N}{M_1} E_1 = N E_1 \ .$$

Considering both the higher extraction energy and the higher mass of the clusters, the maximum ion current density of the clusters that can be drawn from the ion source is then

$$J_N = \frac{M_N}{M_1} J_1 = N J_1 \ .$$

Further, the flux density (or dose rate) of dopant atoms is larger than the electrical ion beam current indicates, since each of the clusters contains N dopant atoms:

$$\text{dose} - \text{rate} \propto N J_N = N^2 J_1 \ .$$

The supply of dopant to the target for a cluster of N particles can thus be increased by a factor of N^2 relative to that of a monomer of the same species.

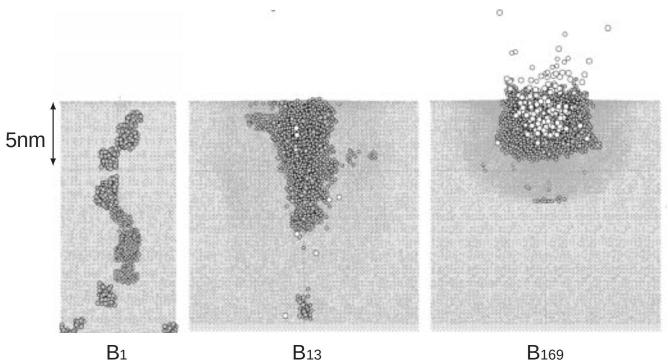


Fig. 4. Molecular dynamics simulation of monomer and small and large boron cluster impact on crystalline Si, after an elapsed time of 385 fs at the same acceleration energy of 7 keV.

Shallow junction formation in Si promises to be one of the most immediate applications of cluster beam implantation. Toward the understanding of cluster ion implantation characteristics, molecular dynamics simulations have been performed for boron cluster ion implantation into Si(001) surfaces. Figure 4 shows the results of 7 keV boron clusters with 1, 13, and 169 atoms at 385 fs after impact upon a Si surface [14]. The implantation characteristics of the small and large clusters are very different. The small cluster with 13 atoms collapses on impact with the surface, and the damage formation within the substrate surface exhibits damage features different from those caused by the monomer implantation. In the 169-atom cluster case, part of the cluster holds together as a unit on impact while isolated atoms break off and form cascades. The penetration is shallower for these cascades, due to the lower energy per atom. In this case, the cluster remains essentially intact during impact, and a shell of damage forms around the cluster as it stops. There is still a possibility of channeling, as the acceptance angle for axial and planar channeling increases as atom energy decreases for small clusters, but the channeling observed in the simulations decreases with increasing cluster size, as hoped.

The first clusters that have been demonstrated for shallow junction formation were decaborane $B_{10}H_{14}$ ions [15, 16]. In those experiments, evaporated decaborane was used as a source material for the clusters. Typical SIMS dopant profiles of Si implanted by $B_{10}H_{14}$ at 5 keV, 3 keV, and 2 keV before and after annealing at 900 °C and 1000 °C are shown in Fig. 5. Transient enhanced diffusion (TED) and thermal diffusion (TD) are considered to be the most important issues in dealing with shallow junction formation in traditional ion implantation technology [17]. TED is fully suppressed by the $B_{10}H_{14}$ implantation; however, TD still occurred after 1000 °C annealing. The results suggest that cluster ion

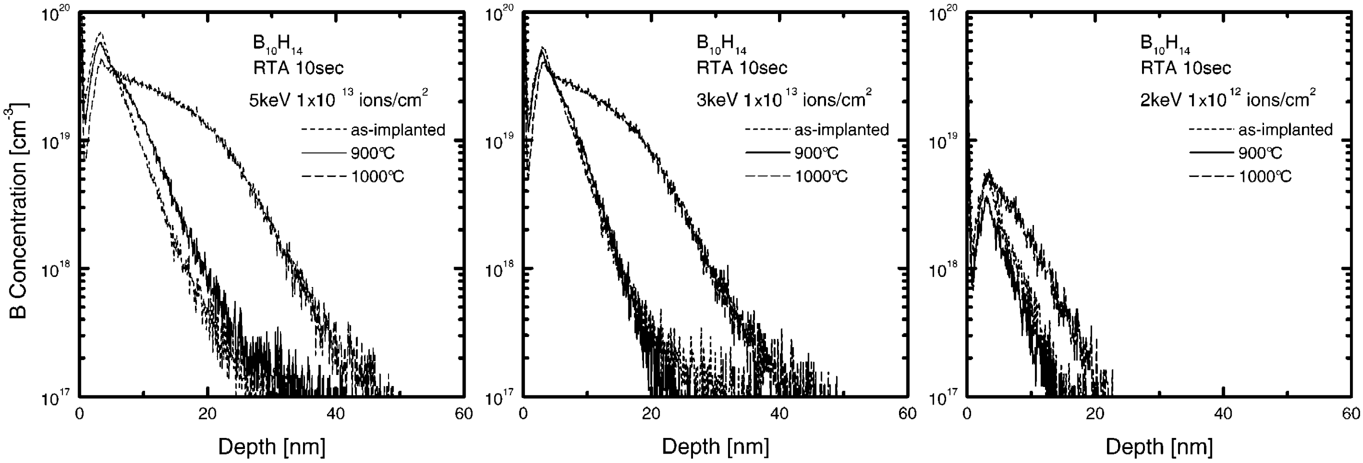


Fig. 5. SIMS analysis of implanted B concentration after implantation of $B_{10}H_{14}$ at 5 keV, 3 keV, and 2 keV, before and after 900 and 1000 °C annealing for 10 s by RTA (rapid thermal annealing).

implantation can be a solution for shallow implantation. Moreover, for obtaining the same boron dose in the substrate, the required dose of $B_{10}H_{14}$, for example, is ten times lower than that necessary for traditional B implantation.

40 nm p-MOSFETs have been fabricated to demonstrate $B_{10}H_{14}$ cluster implantation for shallow source/drain formation [7]. $B_{10}H_{14}$ ion implantation for p-type source/drain (S/D) junctions was performed at an acceleration energy of 30 keV to a dose of 1×10^{13} ions/cm^2 and was followed by annealing at 1000 °C for 10 s. A junction depth of 20 nm was achieved. For S/D extensions, $B_{10}H_{14}$ ion implantation at 2 keV was carried out to a dose of 1×10^{12} ions/cm^2, followed by annealing at 900 °C for 10 sec. A 7-nm ultrashallow junction without TED or TD was achieved. The highest resulting drive current 0.40 mA/μm (@I_{off} of 1 nA/μm and $V_{\text{d}} = -1.8$ V) shows 15% improvement as compared with published data [18,19]. A low S/D series resistance R_{sd} of 760 Ω m was achieved, even when a high sheet resistance (> 20 kΩ/sq) was used for the extension regions (due to the diminished extension length). The smallest p-MOSFET to date, with a L_{eff} of 38 nm, has been demonstrated. This device has the smallest dimension and the highest device performance among many developmental devices. Figure 6 shows an SEM image of the device with a poly-Si gate length of 40 nm after sidewall removal.

3.1 Cluster ion beam sputtering

Ion-enhanced dry etching techniques are widely used for a variety of device fabrication operations because of their high spatial resolution. Recent requirements for etching have become difficult to achieve because of smaller dimensional limitations. Ion bombardment during the etching processes creates considerable long-range damage on the substrate surfaces [20]. In plasma etching, charge accumulation on the substrate surfaces during exposure to the plasma causes damage not only in thin insulating materials but also in delicate devices buried under the exposed surfaces. Elimination of this problem is especially important for novel advanced device fabrication [21,22]. The use of low-energy ion beams with essentially no electrical charge can solve this problem. Moreover, processes are required which can provide high-rate etching even at very low bombardment energy and with low surface damage. Cluster ion beams provide possible solutions for these apparently contradictory requirements.

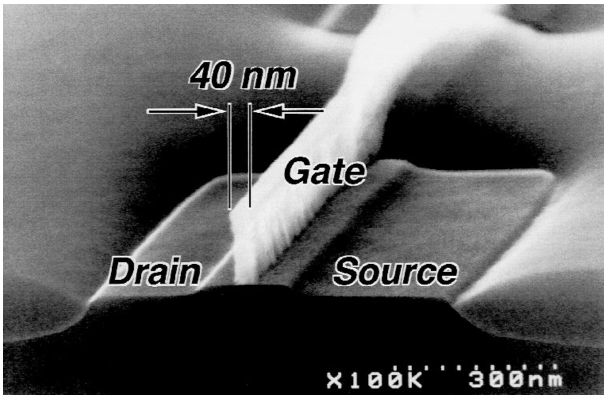
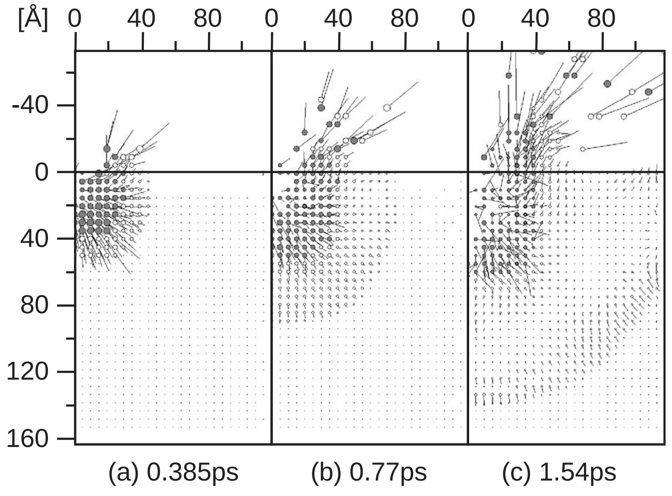


Fig. 6. SEM image of p-MOSFET having a 40 nm gate after sidewall removal.

Very high sputtering yields on metal, semiconductor and insulator surfaces due to bombardment with cluster ions have been experimentally observed [2,3,23], and this effect has been studied by computer simulation [14,25–27]. The sputtering yield Y from Au surfaces due to Ar_n ($n = 80 - 200$) cluster bombardment at energies of 8–20 keV fits a power dependence $Y \sim n^{2.4}$ on the cluster size n; this is in good agreement with experiment [28]. The power exponent of this expression, 2.4, is close to the value of 2 obtained in a thermal spike model for monomer ions [29].

Figure 7 shows the kinetic energy and momentum profiles of an Si substrate irradiated by Ar_{688}, with an acceleration energy of about 55 keV (80 eV/atom) [14]. Figures 7a–c correspond to 0.385 ps, 0.77 ps, and 1.54 ps after



(a) 0.385ps (b) 0.77ps (c) 1.54ps

Fig. 7. Energy (circle) and momentum (line) profile of Ar_{688}, with an acceleration energy of 55 keV impacting on an Si(001) surface.

the impact, respectively. The radius of the circle indicates the mean kinetic energy in a 0.5 nm square. The open circles correspond to substrate atoms, and the filled circles represent cluster atoms. The lines indicate the mean magnitude and the direction of momentum. Even with such a high acceleration energy impact, both the kinetic energy and momentum of cluster atoms are transported to surface atoms in an isotropic direction, and crater-like damage is formed. In the crater formation process, some atoms on the edge of the crater leave the surface in a direction lateral to the surface; this agrees with experimental observations [23]. In Fig. 7c, 10 sputtered atoms are indicated, but this number of sputtered atoms is much lower than the actual experimental value. The reported sputtering yield of Si atoms by Ar cluster ions with an acceleration energy of 20 keV and mean size of 3000 is 25 atoms/ion. It is thought that the atoms on the edge of the crater leave the surface in the subsequent several picoseconds, because each atom in this region still has almost the same momentum and enough kinetic energy (above 2 eV) to leave the surface.

Cluster ion beam sputtering is characterized not only by high etching rates but also by surface smoothing effects from a lateral sputtering process. However, these characteristics depend on the type of surface interaction process; for example, physical sputtering or chemical sputtering. Physical sputtering, e.g., bombardment of SiC by Ar clusters at normal incidence, causes smoothing of the bombarded surface. Chemical sputtering, e.g., bombardment due to SF_6 cluster beams, does not produce the same effect. In Fig. 8, examples are shown of AFM images of surface smoothing on SiC substrates by an Ar_{3000} and $(SF_6)_{2000}$ cluster ion beams.

Surface smoothing and high-rate sputtering by cluster ion beams have been demonstrated for semiconductors, metals and insulators. For example, the original surface of a Cu film deposited on an Si substrate contained large hills with an average diameter of 200 nm, and the average surface roughness was $R_a = 5.8$ nm. After Ar cluster ion bombardment to a dose of 8×10^{15} ions/cm^2 at an energy of 20 keV, the surface was smoothed to $R_a = 1.3$ nm. Similar results have been obtained for a YBCO film (from original roughness $R_a = 7.9$ nm to sputtered surface $R_a = 0.5$ nm by Ar cluster 20 keV at 2×10^{16} ions/cm^2) [8], an SiC film (from $R_a = 14.5$ nm to $R_a = 0.6$ nm by Ar 20 keV at 1×10^{16} ions/cm^2), and a CVD diamond film (from $R_a = 41.3$ nm to 8.2 nm at 20 keV 1×10^{17} ions/cm^2). These measurements were made with an AFM at 1 μm scan size. The potential application of YBCO is for SQUID devices, CVD diamond membrane surface finishing is for SOR and X-ray lithography, metal-plated surface smoothing is for nonspherical plastic lens-mold surface smoothing, and SiC surface smoothing is for SOR mirrors [9].

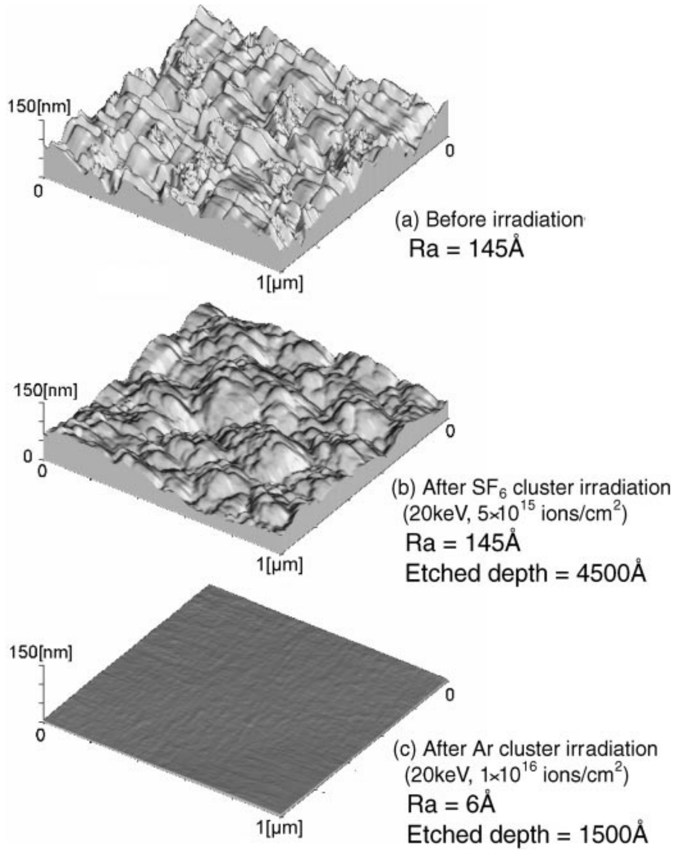


Fig. 8. AFM images of CVD SiC surfaces before and after Ar_{3000} and $(SF_6)_{2000}$ cluster ion beam bombardments at 20 keV with a dose of 1×10^{16} ions/cm^2.

4 Cluster ion beam-assisted thin-film deposition

It is claimed that direct ion beam deposition and ion-assisted deposition are possible candidates for high-quality thin-film deposition processes. However, they have not yet been applied for either delicate semiconductor device fabrication or optical coatings, because of the possible attendant damages. For direct ion beam deposi-

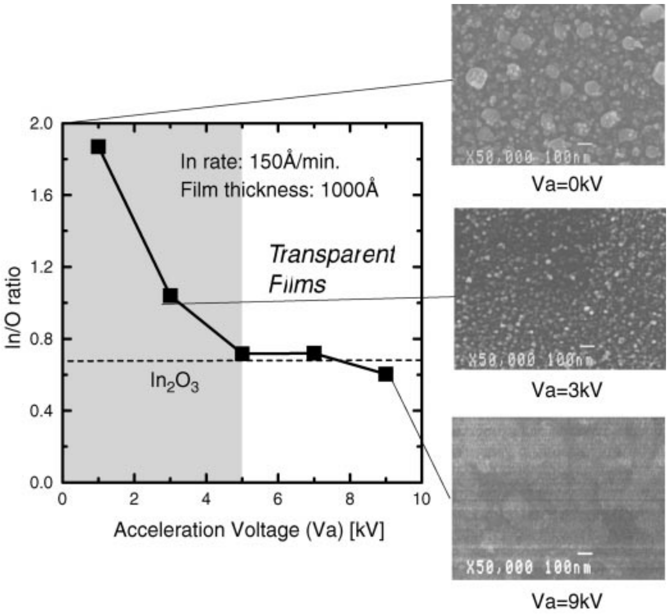


Fig. 9. Dependence of film stoichiometry on acceleration voltage of O_2 cluster ions; micrographs show the surface morphology.

tion, beams of much lower energy and higher current are needed. Generally, ion beams are extracted from a source at high voltage, transported to a substrate chamber, and then decelerated just before their impact upon the substrate surface. It is realized that even this method is not completely adequate for obtaining sufficient deposition rates for actual industrial applications. Energy distributions of ion beams generated by the ion sources have restricted low-energy limits. In ion-assisted deposition methods, beam energies lower than 100 eV are very difficult to obtain. The need exists for very low-energy ion beam systems that can be used to properly control nucleation and growth processes during the initial stages of film deposition.

Gas cluster ion beam-assisted deposition has been applied for the formation of multilayers of TiO_2 and SiO_2. Indium–tin–oxide (ITO) films have been formed by oxygen gas cluster bombardment during Ti, Si, or In deposition [5, 30]. It is believed that films superior to those obtained by conventional ion and excited beam deposition methods can be produced. Very smooth surfaces and high packing density films are expected. For ITO film formation, In and Sn are evaporated during irradiation by O_2 cluster ions [31]. Figure 9 shows the dependence of film stoichiometry on acceleration voltage of O_2 cluster ions, with micrographs indicating the surface morphology. Stoichiometry of InO_x films were measured by Rutherford backscattering (RBS). The results indicate that acceleration voltages higher than 5 keV are needed to enhance the chemical reactions of the oxide. Indium oxide films formed by irradiation with the O_2 cluster ion beam at energies larger than 7 keV at 250 °C of substrate temperature showed a high visible transparency of over 80%, and low resistivity smaller than $1 \times 10^{-5}\ \Omega\,$cm. The results clearly indicate that the kinetic energy of the cluster is effective in enhancing oxidation of the surface without introduction of radiation damage, in spite of the high acceleration voltages.

5 Conclusions

The present status of ion beam processing and of the demands and prospects for future ion beam processing have been reviewed. Processes allowing smaller lateral and vertical dimensions and better reliability are urgently required. Nonlinear and nonequilibrium processes are now gaining more recognition in the ion beam process area. Cluster ion beam processing is presented as an advanced approach which will contribute to further progress this field.

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