Abstract
In the buffer layer-assisted growth method, a condensed inert gas layer of xenon, with low-surface free energy, is used as a buffer to prevent direct interactions of deposited atoms with substrates. Because of an unusually wide applicability, the buffer layer-assisted growth method has provided a unique avenue for creation of nanostructures that are otherwise impossible to grow, and thus offered unprecedented opportunities for fundamental and applied research in nanoscale science and technology. In this article, we review recent progress in the application of the buffer layer-assisted growth method to the fabrication of Ge nanoclusters on Si substrates. In particular, we emphasize the novel configurations of the obtained Ge nanoclusters, which are characterized by the absence of a wetting layer, quasi-zero dimensionality with tunable sizes, and high cluster density in comparison with Ge nanoclusters that are formed with standard Stranski-Krastanov growth methods. The optical emission behaviors are discussed in correlation with the morphological properties.

Keywords
Nanocluster · Buffer layer-assisted growth · BLAG · Ge nanocluster · Photoluminescence · Semiconductor growth

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Introduction
Nanostructure self-assembly is currently a topic of considerable interest at both fundamental and applied levels. Semiconductor nanoclusters, in which electrons are confined in all three dimensions, show novel physical phenomena related to dimensionality, such as an atom-like energy spectrum and quantum size effects. Among self-assembled semiconductor nanostructures, Ge nanoclusters on Si, in particular, have been intensively studied as it could promote the development of Si-based optoelectronics. Although remarkable successes have been achieved [1–3] it remains challenging to create 0D Ge clusters on Si(100) with small, dense, and uniform dot formation. One of the dominant growth mechanisms is the Stranski-Krastanov (SK) growth mode, which leads to the formation of coherently strained, pseudomorphic 3D islands after the growth of a 2D wetting layer. Germanium SK dots grown on Si have lateral dimensions of 10–50 nm and are 1–5 nm in height. Their larger lateral size means that quantum confinement effects are determined by the Ge size in the growth direction. Moreover, SK growth is initiated with the formation of a 2D Ge wetting layer beneath the dots [4–6]. Wetting layer formation is due to the lattice mismatch (4.2%) between Si and Ge, which leads to a layer with built-up strain (layer-by-layer growth) and a relief of strain energy (3D island formation) when the layer grows beyond a critical thickness. This wetting layer is electronically coupled to the Ge islands increasing the structures’ thickness further, thereby reducing quantum confinement effects. It also couples the dots to 2D quantum well-like states, reducing the electronic isolation of the dots from one another, and thus affecting device performance.
To avoid wetting layer formation, an alternate growth technique is required to promote a 3D growth mode. In the phenomenological 3D Vollmer-Weber island growth mode, the interaction between neighboring layer atoms should be stronger than that between substrate and layer atoms. This means that a low-surface free energy of the substrate is generally required for island growth. Thus, a key concept to prevent wetting layer formation is the modification of the surface free energy of a substrate. An inert gas that has normally very low-surface free energy was first proposed as a buffer layer by Weaver’s group [7–9]. This process, in which a buffer layer is applied to interrupt the normal growth process and artificially create a necessary condition for the nanoclusters formation, has developed into a unique buffer layer-assisted growth (BLAG) method. A group at Oak Ridge National Laboratory has successfully employed this method to the growth of a variety of semiconductor and magnetic nanoclusters [10–12]. In this paper, we review the application of BLAG in the fabrication of Ge nanoclusters on Si substrates. The nanoclusters thus produced are characterized by absence of a wetting layer, quasi-0D with tunable sizes, and high-cluster density compared with Ge nanoclusters that are formed with the normal SK growth method. The novel morphology, structure, and photoluminescence (PL) from Ge nanoclusters are discussed.

**Buffer layer-assisted growth method**

The general approach to the fabrication of self-assembled nanoclusters using the BLAG method contains the following steps: (1) Condense and freeze a thick buffer layer of an inert gas and maintain the system at a temperature below the sublimation point of the buffer gas. (2) Deposit atoms of a semiconductor on top of the buffer layer. These atoms will exhibit extremely high mobility on top of the buffer layer (compared with direct deposit without the buffer layer) and will diffuse to form a relatively uniform 3D clusters. (3) Continue the growth of the nanoclusters to the desired size. Each nanoclusters acts as a nucleus for further growth and since each may collect arriving adatoms from approximately the same areas, they will grow to approximately the same size. (4) Raise the substrate temperature above the sublimation temperature of inert gas to remove the buffer layer. This provides a gentle landing of the nanoclusters to the Si substrate.

**Morphology and structure of Ge nanoclusters**

As already mentioned foremost, without the presence of a Xe buffer layer, Ge growth on Si(100) is well known to form SK clusters [13–15] in the absence of a Xe buffer layer. Strain-driven SK growth proceeds in two steps. First, below a critical thickness, a layer-by-layer growth can be maintained with built-in strain. Beyond the critical thickness, a 3D island growth sets in to relieve the strain energy. Figure 2 shows the scanning tunneling microscopy (STM) image of a 2.0 ML Ge layer on Si without a buffer layer. This is below a critical thickness (3.0 ML) of Ge coverage, and a layer-by-layer growth mode is observed, leading to
the formation of a 2D wetting layer [12]. The inset of Fig. 2 shows a thinner Ge layer (~1.2 ML) on Si [16]. At a Ge coverage slightly above the critical thickness, a strain relieving transition leads to 3D island formation [12]. Figure 3a shows a typical SK Ge-island with a base size of 60 nm, which formed on top of the Ge wetting layer with a Ge coverage of 3.2 ML. It is well known that SK Ge islands-shapes display several forms such as hut, pyramid, and dome depending on growth conditions. The background of the STM data also clearly indicates a Ge wetting layer, which is described as a domain $2 \times n$ structure.

Ge nanoclusters grown by the BLAG method show a strikingly different morphology as compared to the SK clusters. Figure 3b shows a STM image of Ge nanoclusters after the deposition of 0.5 ML equivalent of Ge atoms on a 6 ML xenon buffer layer. The image was taken at room temperature after the xenon buffer was removed by sublimation. With such a small amount of deposited Ge, it would not be possible to see any cluster formation in the SK growth regime since 0.5 ML is less than the critical thickness [3, 5, 17]. However, with the BLAG method, the Ge nanoclusters clearly appear with an irregular 3D or dome-like shape as shown in Fig. 3b. A pristine Si surface along with two-step edges can be seen in the background of the STM image in Fig. 4a. The equivalent Ge coverage derived from the nanocluster size and density confirms that all Ge adatoms exist in the form of nanoclusters. It proves that the mediation of the Xe buffer prevents direct interactions of deposited atoms with the substrate and no strained wetting layer formation in-between the nanoclusters. The average width of those nanoclusters is 3 nm, and the average height is 0.6 nm with a narrow size distribution as displayed in Fig. 4b. The nanoclusters are remarkably smaller than SK Ge clusters, which normally have a width of 60–200 nm and a height of 12 nm for pyramid or dome shaped clusters on top of a wetting layer [3]. Even the hut clusters are 20–40 nm laterally and 1–3 nm in height [1, 3]. The cluster density is deduced from Fig. 4a to be about $5 \times 10^{12}$ cm$^{-2}$; i.e., more than three orders of magnitude higher than SK dots with a density of $10^8$–$10^9$ cm$^{-2}$ in dome or pyramid shapes, and two orders of magnitude higher than hut clusters [1, 3]. The dimensions of these quasi-0D clusters provide the
Possibility of a 3D carrier confinement, which is desirable for applications in optoelectronics. The sizes of Ge nanoclusters can be tuned by changing the amount of deposited Ge when using the BLAG method. It has been shown that 4 ML Xe is sufficient to buffer the substrate [18]. With a constant Xe buffer layer thickness of 6 ML, the average nanocluster size is approximately 2.5 nm laterally and 0.4 nm in the growth direction when the Ge coverage is 0.5 ML, while at a Ge coverage of 6 ML, the nanoclusters grow to approximately 7.5 nm wide and 1 nm high (Fig. 5).

The nanocluster density increases slightly in the low coverage range of 0.5–1 ML and then decreases at higher coverage. This is consistent with conventional molecular beam epitaxy growth of the first monolayer where the early stages exhibit nucleation of islands followed by a steady-state regime with constant island density and the later stages exhibit island coalescence. The weak coverage dependence of the nanocluster density in the low coverage regime indicates that the adatoms on the Xe buffer are mobile enough to reach the existing nuclei with very few new nanoclusters being nucleated. At high Ge coverage as shown in Fig. 5c, coalescence of nanoclusters results in a decrease of density compared to the low coverage case.

The morphologies of the nanoclusters can also be modified by varying the Xe buffer-layer thickness. The initial shapes of nanoclusters on the buffer layer are determined by the competition between thermodynamics, which favors equilibrium structures with minimal surface areas, and kinetics that imposes constraints due to low temperature, resulting in a high density of nearly round clusters. As shown by comparing Fig. 6a and b, these nanoclusters enlarge as the Xe buffer thickness increases from 6 ML to 10 ML at the same Ge coverage of 0.5 ML. This phenomenon has been ascribed to desorption-assisted coalescence [18] in which the nanoclusters have more time to move and coalesce prior to contact with the Si substrate when landing from a thicker Xe layer. Moreover,
clusters of nanoclusters develop when the Xe buffer thickness increases from 6 ML to 10 ML with an elongation of some nanoclusters appearing, possibly because of incomplete coalescence.

Aggregation of nanoclusters into chains develops for a buffer-layer thickness of 40 ML as shown in Fig. 6c. A similar behavior was recently reported for Au nanoclusters on graphite grown with much thicker buffer layers [8]. The aggregated cluster structure can be attributed to a competition between the arrival rate of new clusters and the time needed to coalesce. Desorption of thicker buffer layers generates more cluster movement and thus leads to a more pronounced cluster aggregation that outpaces the coalescence process. The local structures at the Ge nanocluster/Si interface have been investigated using transmission electron microscopy (TEM). Figure 7a displays a cross-sectional Z-contrast TEM image of the sample with 6 ML Ge, showing the structure of Ge nanoclusters sandwiched between the amorphous Si cap and the crystalline Si substrate. A higher magnification image in Fig. 7b indicates that the Ge island makes an obtuse contact angle with the Si substrate. No coherent Ge layer between the Ge nanoclusters is observed, confirming the absence of a wetting layer [11]. Moreover, no lattice coherence appears at the Ge nanocluster/Si interface. The observed shape of the amorphous islands is different than the hut clusters with {510} facets normally seen when Ge is deposited on the Si(100) surface [13]. This is explained by the BLAG environment in which Ge adatoms interact and form clusters with essentially no influence from the Si substrate. After increased exposure to the electron beam of the microscope, the Ge clusters crystalline structure start to show up. In Fig. 7c, these Ge islands appear to adopt a pseudomorphic structure with the Si substrate.

Fig. 6 STM images showing coalescence of Ge nanoclusters with increasing Xe buffer-layer thickness. (a) 6 ML Xe buffer, (b) 10 ML Xe buffer, and (c) 40 ML Xe buffer. The quantity of Ge deposited (0.5 ML) is the same in all cases. All images are 100 × 100 nm².

Fig. 7 Cross sectional Z-contrast images of 6 ML Ge deposited on 6 ML Xe buffer layer on Si(001). (a) Ge nanoclusters sandwiched between the Si substrate and an amorphous Si capping layer. (b) Higher magnification image showing a Ge cluster making an obtuse contact angle with the Si substrate. (c) Image of a Ge island after increased exposure to the electron beam of the microscope showing the dot has recrystallized to adopt a pseudomorphic structure with the Si substrate.
This experiment has demonstrated that high-energy electron beam bombardment can induce the recrystallization of Ge nanoclusters to align to Si substrate lattices.

**Photoluminescence of Ge nanoclusters**

PL studies on these nanoclusters have revealed intriguing new features in comparison with SK dots. Figure 8 displays PL spectra from Ge nanoclusters grown with the BLAG method with equivalent Ge deposition of 0.3, 0.6, and 6 ML, where the Xe buffer-layer thickness is 6 ML for each case. The dominant PL bands are centered at 0.806 eV (P1) and 0.873 eV (P2) with a full width at the half maximum (FWHM) of 20 meV [11], which are much sharper as compared to those from SK dots (FWHM of 60–100 meV) [2–5]. However, the energies of P1 and P2 are independent of cluster size. Since the clusters are much smaller than SK dots, and should therefore, exhibit much stronger quantum confinement, the absence of size effects on the PL peak energy suggests that the recombination probably is not directly associated with the band structures of the clusters. The emission band at around 1.1 eV is the phonon-assisted recombination of the free-exciton in the Si substrate [2, 4, 19, 20]. The PL bands at around 0.94 and 1.00 eV, which appear on the 6 ML Ge sample and also on other high Ge coverage samples, correspond to those from the excitonic non-phonon and transverse-optical-phonon-assisted transitions of 2D Ge on Si [21, 22]. This implies the possibility that 2D Ge starts to form when Ge coverage becomes higher. However, TEM analysis shows no evidence of a pseudomorphic Ge layer. Thus, if this layer indicated by the PL spectra is associated with a 2D layer, it not the conventional Ge wetting layer.

The excitation power dependence of the PL from a sample with 0.5 ML Ge deposited with 6 ML Xe as a buffer layer is shown in Fig. 9. In contrast to the PL behavior from SK dots, the peak energies of both P1 and P2 do not shift with increasing excitation power from 1 to 5 W/cm² [11]. Coherent strain in SK-grown dots gives them a type-II band alignment, in which the electrons and holes occupy two spatially separate regions [2–5, 19]. Band bending occurs at the interfaces of type-II structures because of the Hartree potential, blueshifting the PL signal as greater excitation power densities increase the carrier density [20–22]. Such a blueshift is not observed in the nanoclusters prepared with the BLAG method, and it is thus probable that these clusters do not possess a type-II band alignment at the Ge/Si interface. This would also be consistent with the fact that negligible strain exists at the Ge nanocluster/Si interface because of the BLAG process. The excitation curves, shown in the inset of Fig. 9, reveal sublinear (~0.50) power exponents for both P1 and P2 bands. Sublinear power exponent with a power exponent of ~0.78 have previously been attributed to a type-II band alignment with a limited density of localized states for excitons [4, 15]. An increase of localization of the radiative centers would lead to a smaller power exponent. A power exponent of ~0.66, which is closer to our value, has been observed from SiGe dots at high excitation power density and attributed to direct competition between Auger and radiative recombination channels in the dots [2, 20, 23]. Coupled with the fact that the peak energy does not blueshift at high excitation power, it is plausible to
attribute our observed sublinear power exponent to a competition of a localized radiative recombination processes against parallel Auger recombination channels. Figure 10 shows the dependence of the PL spectra on measurement temperature for band P1 and P2. The integrated PL intensity and the peak positions as a function of measurement temperature for band P1 and P2 are shown in Fig. 11a and b, respectively. The PL intensity starts to decrease rapidly at 75 K for both bands. The thermal activation energy $E_A$ can be obtained from the Arrhenius plot of the PL intensity. The deduced $E_A$ is 18.6 meV for P1 and 11.2 meV for P2. The obtained $E_A$ would correspond to the energy for the excitons escaping from the radiative centers to non-radiative recombination centers. The low activation energy means a weak confinement and explains a quick PL intensity decrease.

The peak energies of the P1 and P2 bands red-shift with temperature, however, the variations depart significantly from the Varshni relationships of the Ge bandgap and the Si bandgap [24]. This result again confirms that the P1 and P2 transitions are not associated with the Ge band edges or Si band edges, indicating a “bound to bound” nature of the P1 and P2 transitions. These localized luminescence centers

Fig. 10 Temperature dependence of PL spectra from Ge nanoclusters on Si(100) grown with a BLAG method. The nominal Ge thickness is 0.5 ML and Xe buffer thickness is 6 ML.

Fig. 11 The temperature dependence of PL spectra from Ge nanoclusters with 0.5 ML Ge and Xe buffer-layer thickness is 6 ML. (a) Arrhenius plot of PL integrated intensity for band P1 and P2 as a function of measurement temperature. (b) The PL peak shift with temperature of the P1 and P2 bands. The dashed line represents the Varshni relationship of Ge band gap.
likely result from defect centers at the Ge/Si interface or in the clusters since the structure is not pseudomorphic.

Figure 12 compares the spectra obtained for both a single-layer structure and a 3-layer structure. For the 3-layer structure, spacer layers of p-doped Si are grown on top of the nanoclusters at room temperature then the system is cooled again for the BLAG process for each layer. The spectra obtained for both structures have two principal peaks at the same energies. This is remarkable in view of recent results obtained for a 2-layer nanocluster structure of SK grown dots in which the spectrum shows a blueshift and an additional peak resulting from the second layer [5]. This layer dependence stems from the increased stress at the growth front of the second layer that results in increased intermixing of the Si with the Ge, a problem which is clearly not present with the BLAG. The observed spectrum with two principal peaks is not consistent with the published single-layer SK dot spectra that show only one peak. In the conventional SK growth method, the Ge dots possess a highly strained interface with the surrounding silicon resulting in a neighboring confinement structure and a type II band alignment with transitions from electrons trapped in the interface to holes in the germanium dots. The low-temperature deposition of the silicon used in the present work to avoid alloying of the Ge with the Si results in nanoclusters surrounded by amorphous Si and little interface strain.

The PL behaviors show that the Ge nanoclusters fabricated with the BLAG method are not type-II and the radiative recombination is not associated with band edges. To clarify the role of these amorphous Ge nanoclusters in the PL process, a reference sample was fabricated without the mediation of a Xe buffer layer. In this way, clustering was avoided and a uniform Ge amorphous layer of 6 ML equivalent coverage was deposited directly on Si at 10 K, and then capped with an amorphous Si layer at room temperature. An amorphous nature of the deposited Ge layer was confirmed with STM and low energy-electron diffraction analysis. Under the same measurement conditions, only a weak emission band from the Si substrate (1.1 eV) appeared, and P1 and P2 bands were not observed from the uniform Ge amorphous layer or from the amorphous Si cap layer in the reference sample. The exclusive presence of P1 and P2 emission bands in the samples containing the amorphous Ge nanoclusters indicates that these amorphous clusters must play a critical role in the PL process. A possible model of the PL origin is that the excitons are generated in the Ge nanoclusters or in bulk Si and then decay to defect centers located either within the dots or at the dot/Si interface to recombine. The presence of Ge nanoclusters provides high high-energy excitons, higher than the bulk Ge bandgap energy, and a high density of interface states that lead to PL with band energies higher than the bulk Ge bandgap. This model has been extensively used to explain luminescence properties from Si and Ge nanocluster systems [25–29]. We have noticed that our observed P1 and P2 bands are in similar energy positions to the dislocation centers D1 and D2, to which are attributed the emission lines of 0.808 (D1) and 0.875 (D2) meV [20, 30, 31]. A temperature-dependent study of the D1 and D2 emission bands indicated that these bands have an association with Si band edges, showing a Varshni dependence, and thus possess a free-to-bound recombination nature [12]. However, as shown in Fig. 11b, the temperature dependence of the P1 and P2 bands depart significantly from the Varshni relationship, and a bound-to-bound recombination behavior was demonstrated. Thus, the D1 and D2 dislocation centers are probably not the responsible recombination centers for the P1 and P2 bands, and the identification of these defects is not clear.

Summary

A BLAG method has been applied in the growth of Ge nanoclusters on Si. STM images indicate the absence of a Ge wetting layer. These nanoclusters are orders of magnitude smaller and spatially denser than the Ge nanoclusters that are formed with the normal SK growth mode. The nanoclusters sizes are tunable in a range of 2–8 nm by changing the Ge coverage. And the nanocluster morphology changes significantly when varying the Xe buffer thickness. A thicker Xe buffer leads to formation of larger nanoclusters, and at 40 ML of Xe, significant aggregation occurs with the formation of nanocluster Ge chains. Samples with different cluster sizes show strong, sharp PL bands in the near infrared spectral region. PL bands do not shift when tuning cluster sizes. The excitation power and temperature dependences of PL spectra suggest a bound-to-bound nature for the PL transition, which is distinguished from the type-II band alignment structures of SK dots. Although the PL is probably associated with defect centers, the Ge nanoclusters play a role in the PL process. High-resolution microscopy indicates that these Ge nanoclusters crystallize and adopt a pseudomorphic structure with the Si substrate under increased electron beam irradiation.
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