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LETTER TO THE EDITOR

An HREM, TEM and SAD study of three-dimensional growth and strain relaxation in (Ge_mSi_n) superlattices

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Abstract. The cross-sectional structure of (Ge_mSi_n) superlattices grown on Si(100) at 603 K is investigated via bright-field (BF) images of the diffraction-contrasted transmission electron microscope (CTEM), selected-area diffraction (SAD) and high-resolution electron microscopy (HREM). The superlattices have 3D island structure from the fifth period upward, while the initial few periods have two-dimensional (2D) growth. The boundaries of the 3D islands are approximately parallel to the (011) plane. Analysis of the experimental results suggests that the 3D island structures in $(Ge_{12}Si_{12})_{50}$ are formed by the relaxation of the misfit strain.

The Ge_mSi_n superlattices on Si(100) are interesting for direct band gap structure investigations and have a background of application in optoelectronic devices [1–3]. It is difficult to grow the superlattices in the form of 2D pseudomorphic epilayers, because 4% lattice mismatch between Si and Ge causes a high-misfit strain energy in the epilayer and can easily induce misfit dislocations. In the theories of van der Merwe and others [4–7], the relaxation of misfit strain in a epilayer depends on the thermodynamic balance between the energy of the homogeneous misfit strain and the energy of misfit dislocations. If the misfit strain energy is larger than the dislocation energy, the strain will relax to induce semicircular dislocation loops on the epilayer surface, which will expand to the interface between the substrate and superlattices to form misfit dislocations. In contrast, Ge grown on Si(100) is an example of Stranski–Krastonov (SK) growth which generally occurs when the initial epilayers follow 2D growth on the substrate until the overlayer misfit strain becomes unfavourable for 2D growth, thus 3D island growth occurs [8–12]. The practical modes of growth of the superlattices in the forms of pseudomorphic epilayers or 3D islands are not clear. The determination of the crystallography of the strained (Ge_mSi_n) superlattices and strain relaxation may provide an adequate explanation.

(Ge_mSi_n) superlattices have been grown on the Si(100) substrate by the molecular beam epitaxy (MBE) technique at a temperature of 603 K in the National Laboratory of Applied Surface Science of Fudan University. Before growing the superlattices, 200 nm Si film was grown on the Si(100) to buffer the defects existing on the substrate. Cross sectional TEM specimens were prepared in [011] orientation, and observed in a

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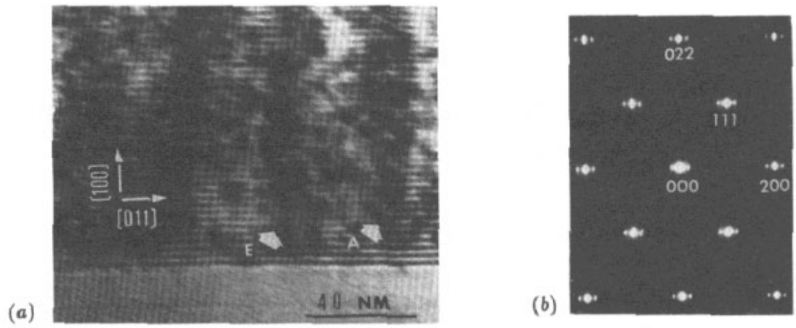


Figure 1. (*a*) BF CTEM micrograph of the superlattices of $(Si_{12}Ge_{12})_{50}$ grown on Si(100) in the [011] projection. (*b*) SAD pattern of the same area as (*a*) in the [011] zone axis.

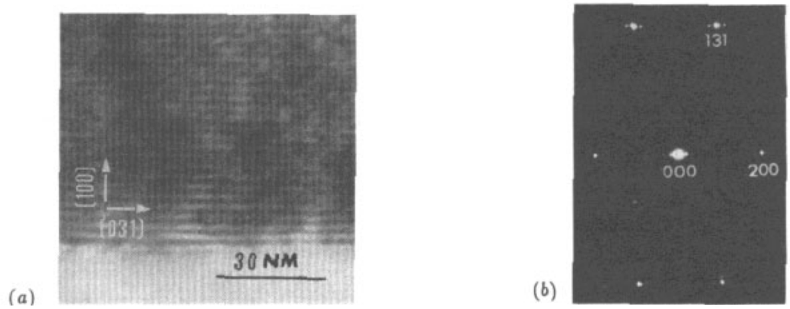


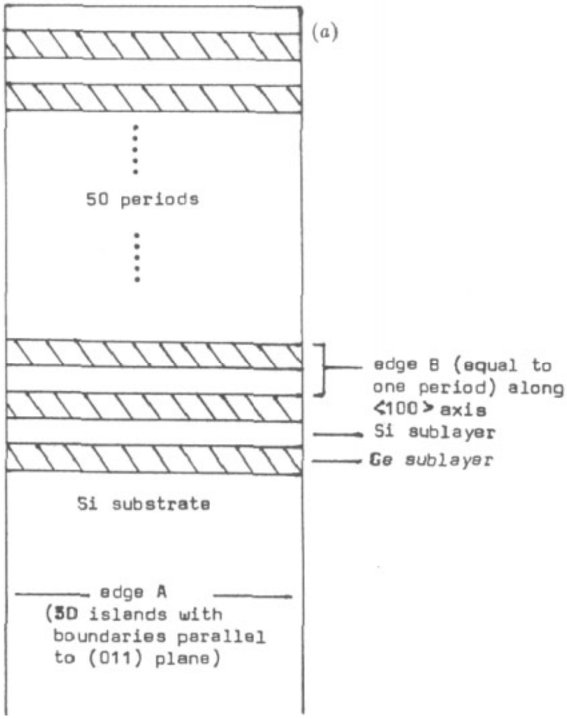
Figure 2. (*a*) BF CTEM micrograph in the [013] projection. (*b*) SAD pattern of the same area as (*a*) in the [013] zone axis.

JEM-4000EX microscope equipped with a 15° goniometer offering a point to point resolution of 0.19 nm.

The BF CTEM micrographs of $(Ge_{12}Si_{12})_{50}$ superlattices taken along [011] or [013] projections (figures 1(*a*) and 2(*a*)) show that the interface between the substrate (buffer layer) and the superlattices has clear images. 3D island structures are observed, and the BF CTEM micrographs of the 3D islands along the [011] projection are clearer than those along the [013] projection. The BF CTEM micrograph of $(Ge_{12}Si_{12})_{50}$ superlattices taken along the [013] projection does not show the boundaries of the 3D islands clearly. Both SAD patterns along the [011] or [013] zone axes (figures 1(*b*) and 2(*b*)) show the following properties: (1) splitting spots along the growth directions of the [100] axis through all spots of the diamond structure, including the undiffracted beam and (2) diffuse streaks along the [011] axis through all spots of the reflections and the additional splitting spots, while the diffuse streaks along the [013] axis in figure 2(*b*) are unsymmetrical, thus indicating that the boundaries of the 3D island are parallel to (011). These additional splitting spots and diffuse streaks are only along certain directions, therefore they must be connected with the structure factors of the superlattices. The SAD patterns can be interpreted by the kinetic diffraction theory with a rectangular parallelepiped crystal model, in which the parallelepiped crystals have edges of A and B as shown in figure 3(*a*). A is about 40 nm which is approximately the width of the bright fringes for the 3D islands along the [011] axis; B is about 3 nm which is the thickness of the period of the superlattices along the growth direction shown in figure 1(a). The diffraction intensity I along the [011] projection has

$$I = F\left[\sin(PIAk_{100})\right]^2\left[\sin(PIBk_{110})\right]^2 / k_{100}^2 k_{110}^2.$$

The simulated diffraction patterns are shown in figure 3(b), and have a half peak width of $1/A$ and diffuse streaks along the [011] direction, at m/A ($m = 1, \ldots, m, \ldots$) which are difficult to separate because A is much larger than the lattice constant of the crystal, as well as a half peak width of $1/B$, and additional splitting peaks at n/B ($n = 1, \ldots, n, \ldots$) along the growth direction. The simulated diffraction patterns agree with figure 2(a) very well.



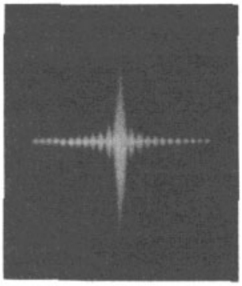


Figure 3. (a) The rectangular parallelepiped crystal model of a 3D island in the superlattices with edges A, B. (b) The simulated diffraction patterns of 3D islands in the superlattices with the rectangular parallelepiped crystal model.

HREM micrographs taken along the [011] zone axis (figure 4) show that the first four periods of the superlattices consist of 2D growth and the lattice images in Si layers and Ge layers are pseudomorphic. The superlattices have 12 monolayers of Ge atoms and 12 monolayers of Si atoms in each of the periods. From the fifth period upward, 3D islands with boundaries approximately parallel to the (011) plane can be observed (the areas blocked out in figure 4). The lattice images of the boundaries, as marked by the arrows, became fuzzy, which means that the atoms in the boundaries are disordered. The boundaries are approximately parallel to the growth direction. the $(Ge_{12}Si_{12})_{50}$ superlattices have a transition of 2D to 3D growth and seem to be the result of the misfit strain relaxing to induce 3D island growth instead of forming misfit dislocations. We have prepared several cross-sectional samples of the $(Ge_{12}Si_{12})_{50}$ superlattices and obtained similar experimental results.

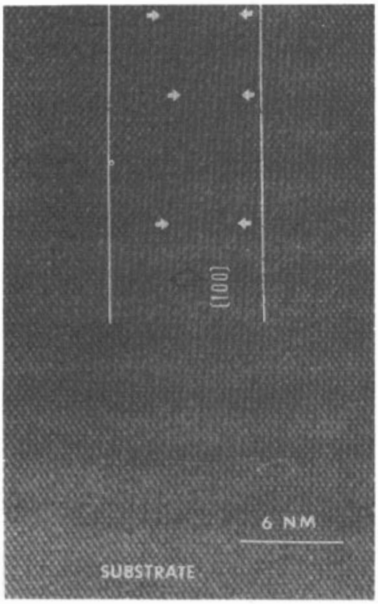


Figure 4. HREM image of 3D islands in the superlattices; the arrows indicate the boundary.

In conclusion, study by TEM, HREM, SAD and the simulated diffraction of 3D islands with a rectangular parallelepiped crystal model show that the crystallographic microstructure of $(Si_{12}Ge_{12})_{50}$ superlattices grown on Si(100) at 603 K is 3D islands from about the fifth period upward with boundaries approximately parallel to the (011) plane. The relaxation of the misfit strain to form 3D island structures is more favourable than that to form misfit dislocations. The 3D islands are believed to be formed by the relaxation of the misfit strain during the growth of superlattices.

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