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REVIEW

Potentialities and Basic Principles of Controlling the Plastic Relaxation of GeSi/Si and Ge/Si Films with Stepwise Variation in the Composition

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Abstract—GeSi/Si heterostructures consisting of a plastically relaxed layer that includes various fractions of Ge and which is grown on Si (001) span the values of the lattice parameter from equal to that in silicon to equal to that in germanium. The corresponding substrates are conventionally referred to as artificial. A number of methods exist for growing high-quality GeSi layers with as large as 100% of Ge on Si (001) substrates through an intermediate GeSi layer with a varying composition. However, it is desirable in a number of cases to have ultrathin (<1 μm) GeSi and Ge layers directly on the Si (001) substrate for practical applications. The results of new methods such as the use of a buffer Si layer grown at a comparatively low temperature (300–400°C) in plastic relaxation of the GeSi/Si(001) heterostructures and also the use of surfactants (antimony and hydrogen) are analyzed. The examples of artificial introduction of centers for origination of misfit dislocations as an alternative to their introduction from the rough surface are considered. It can be concluded that, in order to expand the range of potentialities of growing perfect plastically relaxed GeSi (001) films, it is necessary to (i) make it possible to form in a controlled manner the centers for origination of the misfit dislocations and (ii) retard or completely suppress the transition of the growth mechanism from two- to three-dimensional in order to prevent the formation of additional misfit dislocations from the surface of the stressed film and, correspondingly, additional threading dislocations.

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1. INTRODUCTION

In order to fabricate electronic devices using semiconductor materials whose growth technology is well developed, it is desirable to have a set of substrates that are based on Si and ensure the growth of heterostructures with various lattice parameters. Heterostructures consisting of a plastically relaxed Ge_xSi_{1-x} layer with various values of x span the entire range of lattice parameters, from those characteristic of Si to those characteristic of Ge. Such substrates are usually referred to as artificial substrates. In recent years, the technology of obtainment of these substrates and Ge_xSi_{1-x} layers finds more and more effective uses in optoelectronics, microelectronics, and nanoelectronics. Outstanding examples of device applications of SiGe technology have been demonstrated by the IBM company as far back as in 2003. This company developed integrated digital circuits with a response speed as high as 40–56 Gb/s [1]. At present, the technology of epitaxy of the Ge_xSi_{1-x} alloys by IBM is becoming a key procedure in fabrication of integrated circuits with the densest integration, lowest power consumption, and highest speed of response. Operating frequencies in excess of 60 GHz are attained for detectors and emitters [2]. IBM in collaboration with Georgia Tech developed a GeSi processor that operated at a frequency of 350 GHz at room temperature. The new device can operate at 500 GHz when properly cooled, while the modern processors used in personal computers have maximum frequencies of about 4 GHz. The use of germanium in silicon integrated circuits makes it possible to realize the extended-silicon effect and reduce the leakage currents, thus attaining a higher efficiency. According to the opinion of specialists, the highest frequency that can be attained using the SiGe chips is 1 THz. It is quite possible that such processors will be used in supercomputers [3]. Scientists at the Massachusetts Institute of Technology under the guidance of Fitzgerald developed the technology for formation of virtual GeSi substrates on silicon [4–6]. On these substrates, GaAs layers were grown and the light-emitting diode structures were practically realized; these structures were integrated with high-rate-response digital circuits for optical transmission and processing of signals [7]. Von Koenel supervised the development of a new technology of growth of GeSi/Si films, the so-called low-energy plasma-enhanced chemical vapor deposition (LEPECVD) [8]. Artificial substrates obtained using the low-energy plasma were used (in collaboration with a German Company RWE Space Solar Power

GmbH) for the growth of GaAs films; as a result, operational single-stage elements for solar cells were obtained [9]. The same team of researchers as in [8] fabricated the heterojunction-based GeSi photodetectors that operate at a wavelength of 1.55 μm [10]. The above-given examples of device applications of silicon-germanium (SiGe) technology open new opportunities in the evolution of component base for the devices of optoelectronics, microelectronics, and nanoelectronics.

The above methods of growth of high-quality Ge_xSi_{1-x} layers with $x \longrightarrow 1$ are based on the use of GeSi intermediate buffer layers with variable composition [4–10]. The thickness of this layer is approximately 10 μm, which is not always suitable for practical applications. In addition, chemical polishing of the surface of a GeSi buffer layer is necessary both at the initial stage of its growth ($x = 0.5$) [5] and in the course of subsequent technological operations [7]. However, in some cases, it is desirable to have ultrathin (<1 μm) Ge_xSi_{1-x} and Ge layers directly on the Si (001) substrate.

High-quality Ge_xSi_{1-x}/Si(001) films with steplike variation in the composition and a density of threading dislocations no higher than 10^6 cm^{-2} and x as large as ≈0.15 can be grown using various methods [11]. The problem of plastic relaxation of GeSi/Si films with a large fraction of Ge and differing stepwise in the lattice parameter is still topical and in the stage of discussions and a search for new methods. In our previous reviews [11, 12], we systematized and generalized the level of understanding of basic physical mechanisms of formation of plastically relaxed heterostructures in a Ge_xSi_{1-x}/Si system. The aim of this review is to continue the analysis of the results of studies published in the years elapsed since the previous reviews were published; we include the discussion of problems and the results of direct growth of germanium films on silicon substrates.

As is well known, plastic relaxation (i.e., the formation of misfit dislocations (MDs)) in stressed GeSi/Si(001) films grown by the molecular-beam epitaxy (and also by other methods) in modern setups sets in at a thickness that exceeds by tenfold and more the critical thickness of the pseudomorphic film as determined by the Matthews–Blakeslee method [13]. This is related to the absence of the centers of origination for MDs, in particular, to the absence of threading dislocations. Nevertheless, in the case of attaining a large thickness, MDs appear in an amount sufficient for accommodation of mismatch in the lattice parameters of joined materials (f) and for ensuring the equilibrium residual elastic deformation in the films. At present, the most widely accepted mechanisms of formation of MDs are the following.

The most popular mechanism of generation of MDs in heterosystems of the Ge_xSi_{1-x}/Si type is origination of dislocation half-loops from the surface of growing film with subsequent extension of these loops to a heteroboundary. This mechanism was suggested as far back as 1975 by Matthews [14] and was then regularly discussed in reviews devoted to this subject (see, for example, [15, 16]). However, in the end of 1980s, it was shown in a number of studies that origination of the above half-loops on the smooth surface of the growing Ge_xSi_{1-x} film at $x \le 0.5$ is improbable as a result of a large value of the energy barrier, which amounts to tens and hundreds of electronvolts [17, 18]. The main attention of researchers was attracted to the detection and analysis of internal centers as sources of MDs; these centers can be represented by various defects [17, 19–21]. Also, multiplication of previously generated dislocations [22–24] should be considered.

The energy prohibition of the surface origination of dislocation can be relieved if the surface is roughened. The surface of a stressed layer can be roughened in the course of growth as a result of the effect of the elastic-relaxation mechanism. The driving force for formation of the roughened surface of a stressed film is a decrease in the free energy of the system due to a decrease in the energy of elastic strains in the region of the peaks in the three-dimensional (3D) profile [25], while the mechanism of realization of this formation is the surface migration of adatoms. The theoretical basics of this mechanism, referred to as the ATG mechanism [26], were laid in publications by Asaro and Tiller [27] and Grinfel'd [28]. The effect of roughening of the surface obtained at present a wide-spread practical application in the heteroepitaxy of highly stressed structures, i.e., the Ge nanoislands on Si and InAs nanoislands on GaAs (see for example, the reviews [29, 30]). The barrier for formation of MDs is lowered at the roughened surface as a result of a geometric increase in stresses at nonuniformities of the profile. Apparently, Jesson et al. [31] were the first to attract the attention to the fact that the morphologically unstable surface can be a source of MDs. It is important that, as a result of appearance of stress's concentrators at the surface's areas with abrupt dips, an activationless origination of dislocations can take place. The onset of introduction of MDs is controlled by the transition of the surface of the growing stressed film from the planar feature corresponding to the two-dimensional (2D or layer-by-layer) growth to the roughened (three-dimensional, 3D) state. It was also noted [31] that the known effect of surfactants on smoothening of the surface of growing film can also affect the behavior of the plastic relaxation. Tersoff and LeGoues [32] compared the calculated values of the energy of activation for the MD origination at the internal and surface-located stress concentrators. The activation energy for the MD origination at internal stress concentrators is proportional to $1/f$, while roughening of the surface and the related process of origination of MDs are proportional to $1/f^4$. Correspondingly, the origination of dislocations at internal structural defects and multiplication of dislocations are dominant at low mismatch levels (lower than 1% according to calculations). At values of f larger than 1%, origination of dislocations at the surface-located stress concentrators

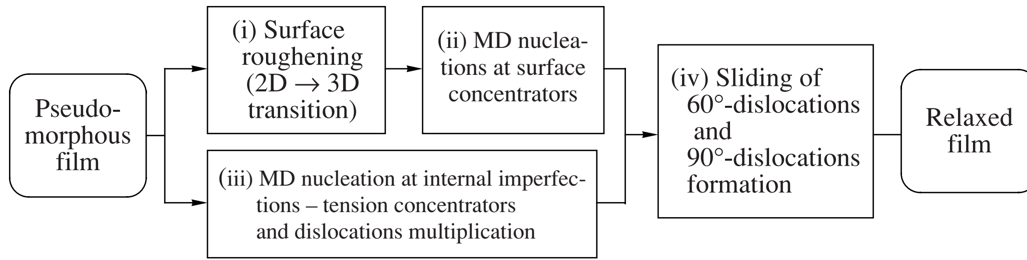

Fig. 1. Schematic representation of the interaction between limiting mechanisms of MD origination and their involvement in plastic relaxation of initially stressed film.

becomes dominant. Since the process of surface roughening is thermally activated, an increase in the growth temperature for the film should be conducive to the appearance of the first MDs at smaller values of the thickness. This conclusion is consistent with many experimental results concerned with determination of the critical thickness for the MD introduction [15]: the higher the growth temperature for the GeSi films, the smaller the effective critical thickness.

The extent of plastic relaxation of an initially stressed film is controlled by the total MD length that increases in the course of relaxation owing to a slide of inclined MD segments that come to the surface and are referred to as threading dislocations (TDs), and also owing to an increase in the number of MDs. Therefore, the processes limiting the development of plastic relaxation are the origination of MDs, their lengthening, and interaction of MDs if their density is high.

On the basis of the above statements, these limiting mechanisms can be illustrated in the form of schematic representation shown in Fig. 1, where the MD origination and the TD glide that ensure an increase in the MD length and, correspondingly, an increase in the extent of plastic relaxation are included in series. The processes of the MD origination at the internal or surface-located sources are competing and are included in parallel. The following part of this paper is concerned with the problem of controlling the factors (i)–(iv) in Fig. 1.

2. THE USE OF SPECIFIC FEATURES OF LOW-TEMPERATURE EPITAXY OF Si AND GeSi

The plastically relaxed Ge_xSi_{1-x}/Si(001) heterostructures that had a stepwise variation in the Ge fraction in excess of 0.15 and were grown at a temperature of no lower than 550°C (we refer to these heterostructures as classical) have a TD density at a level of 10^8–10^9 cm^{-2} [18, 33–36] (Fig. 2). In the past decade, a number of publications have appeared [37–44] in which it was shown that the Ge_xSi_{1-x}/Si(001) films with values of x as large as 0.3 relax to the final TD density of 10^5–10^6 cm^{-2}, if a buffer Si layer with the thickness of 50–200 nm is preliminarily grown at temperatures of 350–400°C (the so-called low-temperature Si, LT Si). The main data reported in the above publications are also represented in Fig. 2. The area enclosed by the dash–dot line in Fig. 2 indicates that the range of the TD density in the above heterostructues is lower by three to four orders of magnitude than this density in the films with the same composition but grown without an LT-Si film. In Fig. 3, we show a typical image of the transverse section of this heterostructure; this image was obtained by the method of transmission electron microscopy (TEM). The TDs are not observed in the GeSi layer. The observed dislocation half-loops penetrating into the substrate indicate [45] that the Frank–Read mechanism of multiplication of dislocations is in

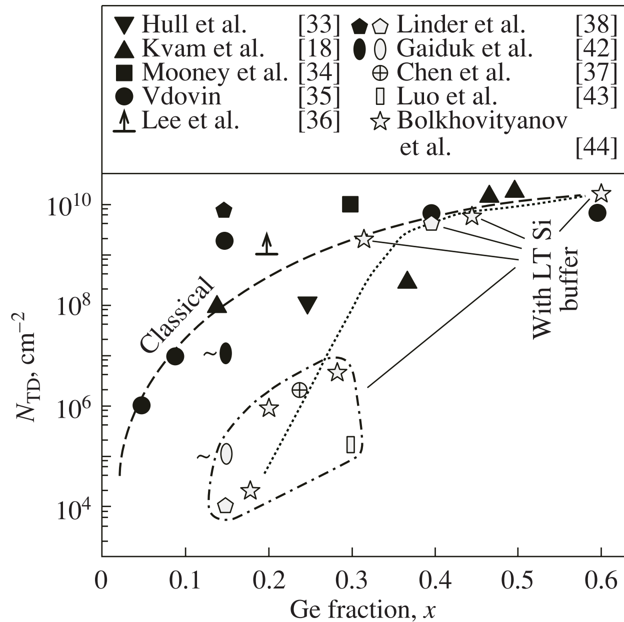

Fig. 2. The density of threading dislocations (TDs) in plastically relaxed GeSi layers with stepwise variation in composition in relation to Ge fraction according to the data of various researchers. Filled symbols represent the growth of the films without LT Si at temperatures of 550°C and higher (the "classical" variant). Empty symbols represent the TD density in the layers grown using the buffer LT-Si layer. The dashed and dotted lines are plotted for visualization of general tendency to variation in the quantities. The dash-dotted line indicates the contour of the data on the growth of GeSi layers with a reduced density of TDs.

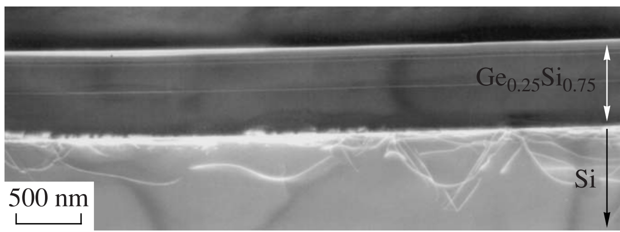

Fig. 3. A TEM image of transverse cut of the $Ge_{0.25}Si_{0.75}$/Si(001) sample formed using a buffer Si layer grown at a comparatively low temperature (350°C). Bright bands correspond to built-in layers of stressed Si with a thickness of 15–20 nm.

effect. It is believed [45] that this mechanism sets in necessarily if the TD density is not high enough to ensure the plastic relaxation. In this case, it is necessary to attain a certain film thickness in order for this mechanism to become operative [46]. It was assumed [45] that introduction of dislocations into the substrate, which accompanies the effect of this mechanism, can be considered to be an indirect indication that there is a low density of structural defects in the bulk of the relaxed film.

An analysis of published data shows that the "low-temperature" buffer Si (and GeSi) layers affect differently the process of plastic relaxation of the GeSi film in relation to the temperature of their growth. Thus, Luo et al. [43] showed that, as the thickness of the buffer LT-Si layer is increased, (the growth temperature $T \approx$ 400°C), the degree of plastic relaxation is reduced markedly, while the half-width of the film's peak in the X-ray rocking curve is decreased under the conditions where all other factors are equal, which implies the passivation of internal centers of origination of dislocations and a decrease in the TD density. It was shown in [47–49] that, in addition to a decrease in the initial density of threading dislocations in the Ge_xSi_{1-x}/Si(001) heterostructures with $x \leq 0.3$ (the LT-Si growth temperature $T \approx 350$–400°C), an increase in the glide velocity is observed in comparison with the velocity in classical heterosystems grown at a temperature of 550°C without buffer layers. Such an increase in the TD glide velocity represents one of the causes of a decrease in their final density in the plastically relaxed Ge_xSi_{1-x}/Si(001) heterostructures with $x \leq 0.3$ (for details, see Section 8). At the same time, the Si and GeSi buffer layers grown at temperatures below 200°C affect inversely the process of plastic relaxation of the main GeSi film. For example, it is established [50, 51] that the Si and GeSi buffer layers grown at a temperature around 150°C bring about the 100% plastic relaxation of the main GeSi film and a high TD density (see Section 7 for details).

As can be seen from Fig. 2, the density of threading dislocations increases again as the fraction of Ge becomes larger than 0.3 (despite the use of LT Si) and attains large values equal to 10^8–10^9 cm^{-2} [47, 48]. In our opinion, the main cause of the limited beneficial effect of the "low-temperature" buffer Si layer is the fact that the contribution of surface imperfections as sources of the MD origination increases with increasing Ge fraction and becomes dominant [32].

3. ORIGINATION OF DISLOCATIONS AT SURFACE SOURCES

The surface of a stressed film was always assumed to be the main source of MDs [31]; however, direct experimental confirmation of this assumption has not been obtained so far. Electron microscopy studies of transverse cuts of plastically relaxed heterostructures showed that only quite complex defects located at the depressions at the rough surface of the InGaAs/GaAs films [52] and also of the GeSi/Si films annealed at 800°C were observed [53]. The dislocation half-loops originating in the vicinity of the surface were first detected in our study concerned with initial stages of plastic relaxation in the GeSi films with a Ge fraction close to 0.3 [54].

A significant variation in the diffraction pattern of fast electrons (reflection high-energy electron diffraction, RHEED) was observed as the film thickness became as large as 40–50 nm, which indicated that there was a transition from the two-dimensional to three-dimensional mechanisms of growth (2D–3D). This transition is illustrated in Fig. 4. The diffraction pattern observed at the initial growth stage in the form of extended reflections (Fig. 4a) is transformed: point reflections appear in this pattern (Fig. 4b). This observation indicates that a three-dimensional profile appears. Additional bands positioned at an angle (the so-called chevron-like reflections) indicate that vicinal planes of the (115) or (117) types appear on the growth surface.

The appeared three-dimensional surface profile can be conducive to origination of dislocations [55]. A decrease in the growth temperature prevents the formation of a 3D profile; however, as will be clear from what follows, origination of misfit dislocations at the surface inhomogeneities is found to be possible even at 350°C.

In Fig. 5, we show a fragment of the image of a transverse cut of a $Ge_{0.32}Si_{0.68}$/Si(001) heterostructure and the corresponding scheme of the configuration of dislocation lines. The image shows the supposed location of the surface origination of dislocations, a darkened spot *1* at the film surface; this spot is located symmetrically relative to the side branches of the dislocation half-loop *2* that extends from the surface to the film bulk but does not yet reach the interface. One can also see the half-loop that already formed the segment of a misfit dislocation *3a* and a threading dislocation *4a*. Misfit dislocation *3b* and its branch *4b* that reaches the surface are located in the glide (111) surface inclined to another side and, apparently, belong to a dislocation family formed by another origination center.

A more detailed analysis of the TEM images of this sample was reported in the publication by Bolkhovity-

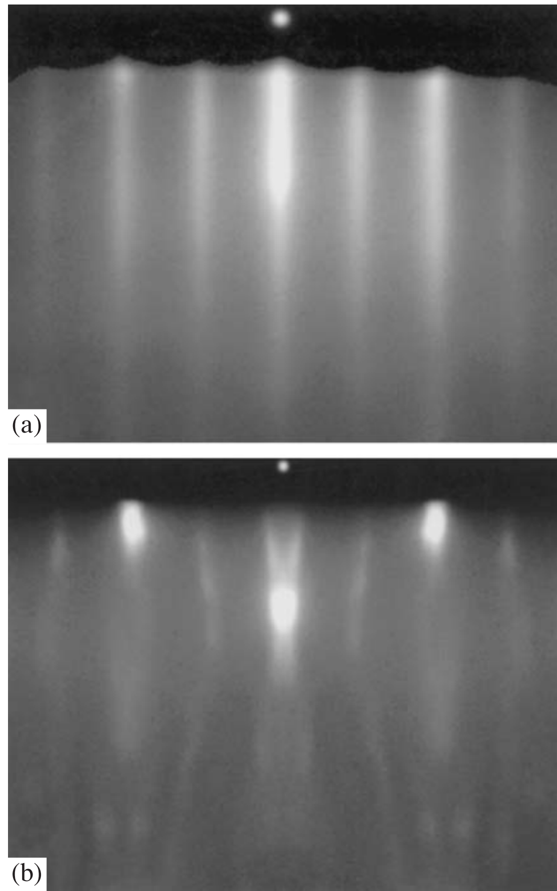

Fig. 4. Patterns of diffraction of high-energy electrons (a) from the smooth surface of the $Ge_{0.3}Si_{0.7}$/Si(001) film with the thickness of 30 nm and (b) from a thicker film with the same composition. The data were taken from [47].

anov et al. [54]. It was managed to determine experimentally for the first time the initial stage of the MD formation owing to the following factors [54]. First, a comparatively low growth temperature (equal to 300°C) for the $Ge_{0.32}Si_{0.68}$/Si(001) film made it possible to obtain this film in the pseudomorphic state at a thickness of 200 nm and then study the initial stages of its relaxation. Second, the relatively low temperature of the postgrowth annealing (350°C) at which the glide velocity of dislocation half-loops was no higher than 10^{-6} cm/s [49]. Third, there was a high density of the centers of MD origination ($\sim 10^7$ cm^{-2}) as a result of a high level of stresses due to a large fraction of Ge in the film. Under such conditions, some dislocation half-loops originated in the near-surface region of the film had no time to reach the boundary with the substrate during annealing and were visualized using the transverse TEM cuts.

The density of the surface centers (sources of the misfit dislocations in this sample) was, according to the TEM data, $(2–3) \times 10^7$ cm^{-2}, while the TD density was 1.1×10^8 cm^{-2} after 10 min of annealing in the argon

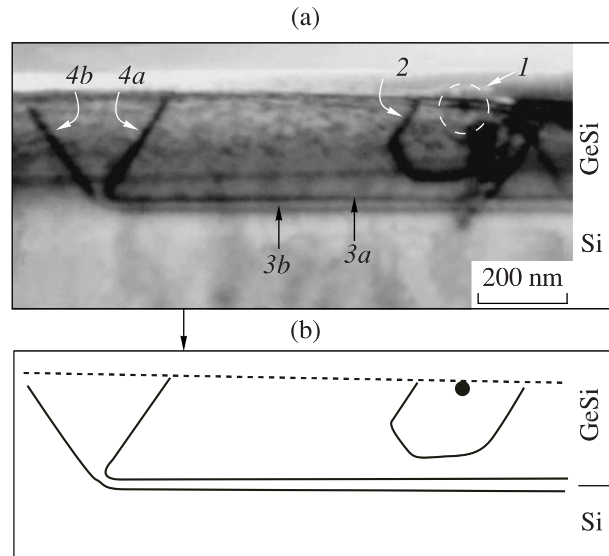

Fig. 5. (a) The bright-field two-beam TEM image of cross section of the $Ge_{0.32}Si_{0.68}$/Si(001) sample after its annealing for 10 min at 350°C; arrow *1* indicates the presumed site of the MD origination and arrow *2* indicates the dislocation half-loop; arrows *3a* and *3b* indicate the misfit dislocations and arrows *4a* and *4b* indicate the threading dislocations. The Ge/Si(001) boundary is inclined by 10° about [110]. (b) The schematic representation of the sites of location of dislocations. The data are taken from [59].

atmosphere. The TD density exceeds the density of the centers for origination of MDs by several times owing to the fact that these centers are of nonexpendable type. For example, it can be seen in Fig. 5 that source *1* is the center of origination of at least two dislocation half-loops. Another example of formation of several closely spaced MDs is shown in Fig. 6. Here, in the fragment (Fig. 6a), the numbers *1–4* indicate the threading segments of the MD dislocations that have the same inclination and glide in the same {111} family of planes. These facts indicate indirectly that there is a single source common for all MDs under consideration; this source is of nonexpendable nature. In the expanded

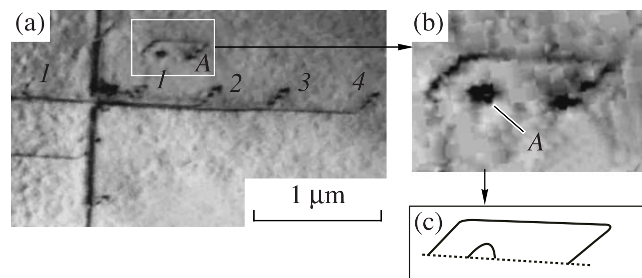

Fig. 6. (a) A planar TEM image of a $Ge_{0.32}Si_{0.68}$/Si(001) heterosystem, (b) an enlarged fragment of this image, and (c) a schematic representation of the location of dislocations. Arrow *A* indicates the dislocation source; direction of the electron beam coincides with the [001] axis.

The density of threading dislocations (TDs) and the degree of relaxation of stresses in the $Ge_{0.32}Si_{0.68}$ films annealed at a temperature of 350°C in the atmosphere of Ar and H_2

Annealing duration, min	Annealing atmosphere	TD density, cm^{-2}	Degree of relaxation
30	Ar	$(4–8) \times 10^8$*	9**
30	H_2	10^8*	2*

Note: * indicates that the data were obtained using TEM, and ** indicates data obtained using X-ray diffraction.

fragment of Fig. 6 (Fig. 6b), arrow *A* indicates the dark region that features a diffraction contrast and is located along the line connecting two threading segments of a larger dislocation half-loop. This contrast can appear in the image as a representation of either a small stress concentrator or a small dislocation loop (see the schematic representation in Fig. 6c). This example represents an additional confirmation of the operation of the surface MD sources. In that case, a single dislocation half-loop that reached the heteroboundary and formed an MD segment can represent such a source.

4. PASSIVATION OF THE ORIGINATION OF MISFIT DISLOCATIONS AT THE SURFACE IMPERFECTIONS. SURFACTANTS

The possibility of using surfactants as tools for affecting the process of plastic relaxation has been noted in as far back as 1993 [31]. Under the conditions where the transition from the two-dimensional to three-dimensional mechanisms of the growth of a stressed film brings about a massive origination of MDs at the arising roughness of the surface, passivation of this transition delays the onset of intense plastic relaxation of this film. As is well known, surfactants (antimony and atomic hydrogen) can smoothen the surface [56–58]. It was found when studying the $Ge_{0.32}Si_{0.68}$/Si(001) heterostructures annealed in the atmosphere of various gases [54] that there is a significant decrease in the degree of plastic relaxation and also in the density of threading dislocations in the sample annealed in the hydrogen atmosphere as compared to the sample annealed in the atmosphere of argon with the other conditions being the same (see table). A decrease in the degree of plastic relaxation of stresses and TD density as a result of annealing in the hydrogen atmosphere indicates that there is a decrease in both the rate of origination of MDs and the rate of plastic relaxation.

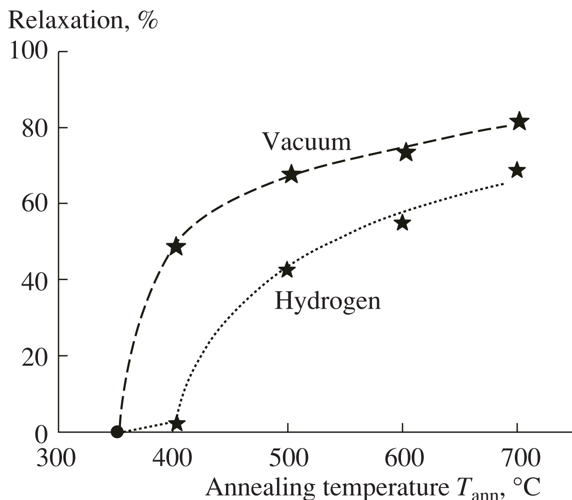

Fig. 7. An increase in the extent of plastic relaxation of the $Ge_{0.28}Si_{0.72}$/Si(001) film as the annealing temperature T_{ann} is increased; the annealing was performed for 1 h in vacuum (the upper curve) and in the hydrogen atmosphere (the lower curve). The film thickness is 200 nm. The lines were plotted in order to facilitate comparison.

Even a larger difference was observed after annealing of a similar heterostructure in vacuum and in hydrogen atmosphere [59]. In Fig. 7, we show the dependences of the degree of plastic relaxation in the samples of this heterostructure on the annealing temperature. The most pronounced difference is observed at the initial stages of plastic relaxation of initially pseudomorphic films. It can be seen that the degree of relaxation in hydrogen atmosphere at $T_{ann} \approx 400$°C is lower by a factor of 20 than in the case of annealing in vacuum. As a result of a further annealing at higher temperatures, this difference becomes smaller; nevertheless, the degree of relaxation of the films annealed in vacuum is found to be always higher.

Taking into account the fact that the hydrogen diffusion into the film bulk is insignificant at a temperature $T_{ann} \approx 400$°C, the results reported indicate the following. First, these results confirm the conclusion that the MDs originate from surface sources. Second, formation of such sources is substantially retarded in the case of annealing in hydrogen; i.e., the surface of the stressed film is passivated by hydrogen and the surface profile is not roughened. Despite the small coefficient of dissociative adsorption of H_2 on Si (001) at temperatures of 400–500°C [60], one may assume that atomic hydrogen is present on the film surface if the sample is annealed in hydrogen under atmospheric pressure. Vasek et al. [61] found that small values of coverage with hydrogen increase the activation energy for the motion of Si adatoms on the Si (001) surface. LaPierre et al. [62] assumed that atomic hydrogen reduces the diffusion length for surface migration of adatoms. In accordance with these notions, the presence of atomic hydrogen on the surface of growing GeSi film widens the range of conditions under which the mechanism of growth of the stressed GeSi/Si(001) film is two-dimensional (layer-by-layer) [63]. Thus, one may conclude on the basis of the above results that we can control the process of plastic relaxation of stressed films by controlling the profile of growth surface of these films.

Specific features of plastic relaxation of GeSi/Si films in the presence of Sb as a surfactant were studied

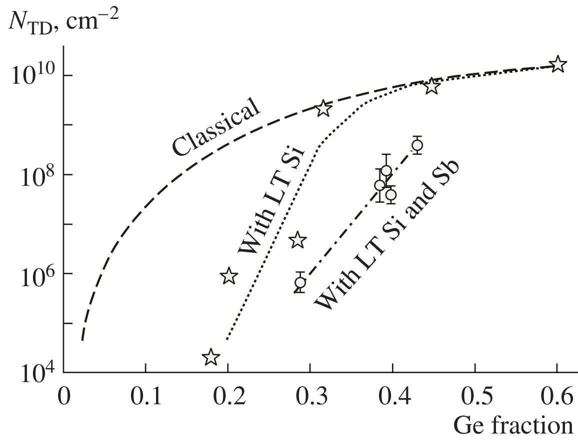

Fig. 8. The density of threading dislocations in relation to composition of a relaxed GeSi/Si(001) film grown with the involvement of the Sb surfactant. For the sake of comparison, the dashed and dotted lines and also a part of experimental points are taken from Fig. 2.

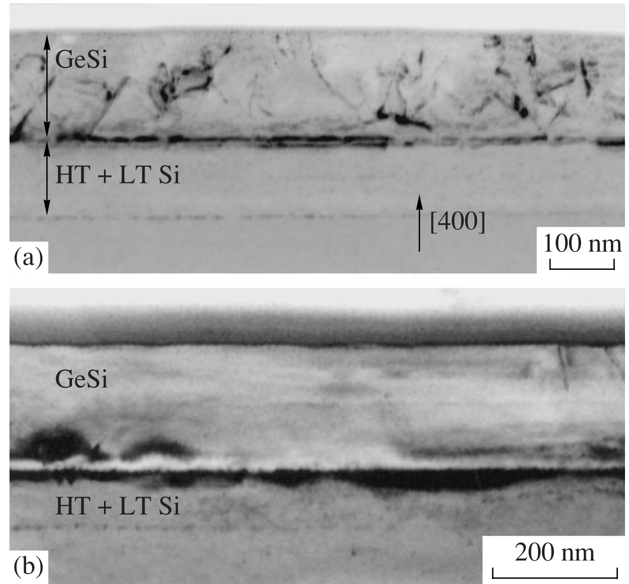

Fig. 9. A TEM image of cross sections of Ge_xSi_{1-x}/Si(001) films grown (a) without the use of surfactants ($x = 0.45$) and (b) using Sb as a surfactant ($x = 0.43$). The data are taken from [65].

by us recently [64, 65]. In Fig. 8, we show the values of the TD density in relaxed GeSi/Si(001):Sb films in relation to composition of these films. For the sake of comparison and determination of a trend, some of the data were taken from Fig. 2. Introduction of the Sb surfactant (Fig. 8, the dash–dotted line) that smoothens the film surface at the stage of pseudomorphic growth and reduces the TD density in the plastically relaxed heterostructure by more than an order of magnitude. In Fig. 9, we show the images of the transverse cuts of the samples with GeSi films with almost identical composition and grown without (Fig. 9, sample (a)) and with (Fig. 9, sample (b)) the surfactant. It follows from comparison of transverse cuts that sample (b) has a lower density of TDs by an order of magnitude. Apparently, if the fraction Ge in the GeSi films is larger than 0.3, the use of Sb as a surfactant retards the formation of the rough surface of the stressed film and delays the onset of the MD formation. Nevertheless, despite a decrease in the TD density, the TD number, for the composition of the GeSi films with $x \approx 0.4$, remains too large for these films to be required for practical applications.

At the same time, the $Ge_{0.29}Si_{0.71}$ film (Fig. 10, sample (b)) with the Ge fraction equal to 0.29 grown using a single Sb monolayer (LT Si + 1 ML of Sb) features not only a fairly low TD density (which could be attained even without the Sb involvement) but also a very low degree of surface roughness. In Fig. 10b, we show the image of the surface of this film; this image was obtained using the atomic-force microscopy (AFM). For the sake of comparison, we show in Fig. 10a an image of the surface of the $Ge_{0.28}Si_{0.72}$ film with similar thickness but grown without a surfactant. Both patterns of the surface shown in Fig. 10 demonstrate a profile in the form of prolonged straight lines caused by the presence of extended MDs in the film; however, the GeSi film grown with the use of Sb features an rms roughness of the surface of 0.85 nm, which is one of the smallest for GeSi/Si(001) films with a composition close to that with a Ge fraction of about 0.3. In Fig. 10c, we show (on the same scale) the surface profiles along the lines *A*–*A* and *B*–*B* on the corresponding AFM images. It can be seen that the roughness of the surface of sample (b) is much smaller.

A decrease in the roughness of the surface of a plastically relaxed heterostructure in the presence of Sb can be attributed to both a decrease in the multiplicity of the effect of the MD surface sources in the presence of the surfactant and a decrease in the lateral mobility of adatoms in these conditions. Thus, the use of a surfactanct in the course of plastic relaxation of a stressed heterostructure is a useful procedure that makes it possible to delay the onset of the introduction of MDs that originate at the surface sources and decrease the density of these dislocations. Simultaneously, the roughness of the surface in the relaxed heterostructure is reduced.

5. ORIGINATION OF MISFIT DISLOCATIONS AT INTERNAL SOURCES

Various stress concentrators related to residual contaminations of the substrate surface or to crystal-lattice defects of the grown layers can be considered as belonging to the MD sources [66]. Contemporary methods of cleaning the silicon-substrate surface prior to growth make it possible to reduce appreciably the concentration of residual contaminations [67] and remove spatially the stressed film from these imperfections using the growth of buffer Si layers.

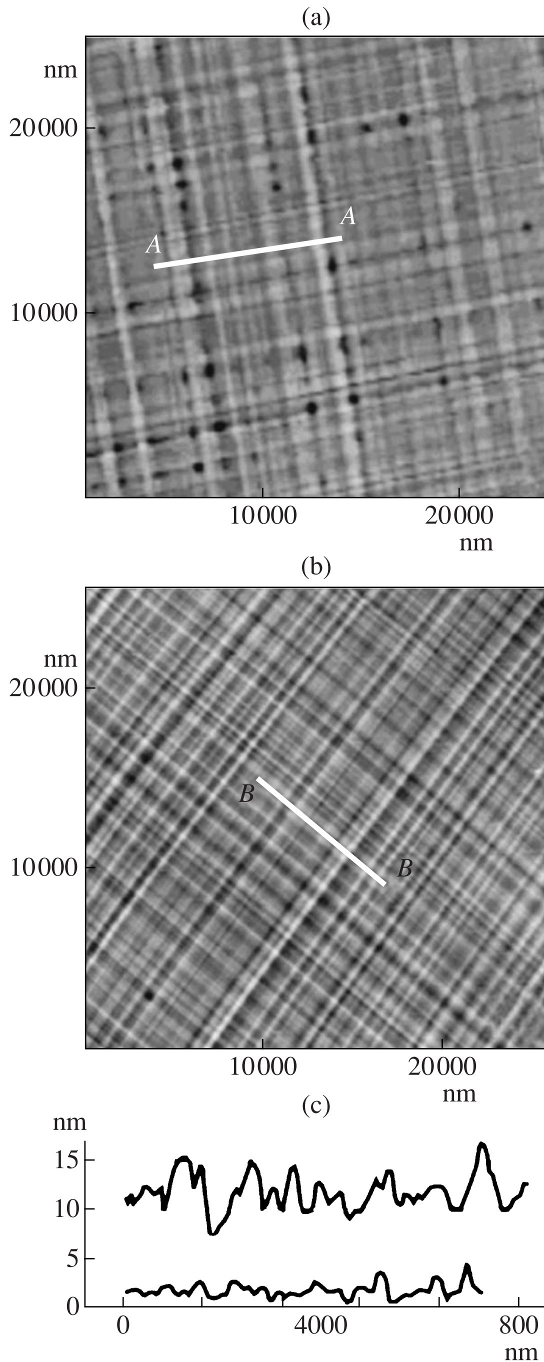

Fig. 10. (a, b) AFM images of the surface of the GeSi/LT-Si(001) films with a Ge fraction of (a) 0.28 and (b) 0.29. The film (b) was grown using Sb. The rms roughness amounts to 2.2 nm for film (a) and 0.85 nm for film (b). (c) The profiles of variation in the thickness over the sections *A–A* and *B–B* chosen arbitrarily in images (a) and (b). The data are taken from [64].

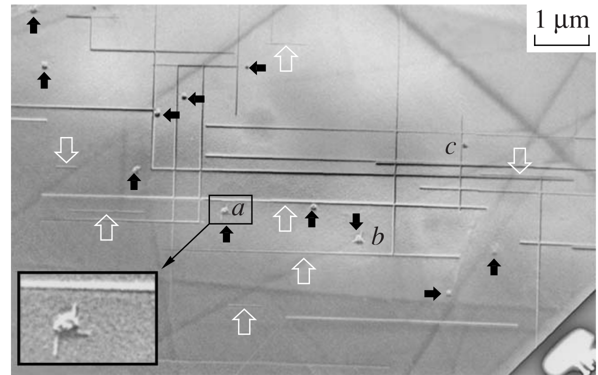

Fig. 11. A TEM image of a longitudinal section of the $Ge_{0.42}Si_{0.58}$/Si(001) film with a thickness of 20 nm grown using the LT Si layer grown at 350°C and approximately one monolayer of Sb at 450°C. The observed density of threading dislocations is 6×10^7 cm^{-2}. The data are taken from [65].

In Fig. 11, we show an electron microscopy image of the $Ge_{0.42}Si_{0.58}$ film at the initial stage of plastic relaxation. According to the data of X-ray diffraction, the degree of relaxation of this film amounts to 0.7%. Mainly short MD lines are observed in the image; these lines are characteristic of initial stages of the plastic relaxation. In addition, local regions with a dark–bright diffraction contrast can be seen; this contrast arises typically at the concentrators of stresses in the bulk of the thin film under study (denoted by black arrows). In our opinion, these concentrators can be related to residual carbide particles at the substrate surface. As can be seen from Fig. 11, dislocations can be visualized only in the vicinity of three of all the stress concentrators visible in the image; these dislocations can subsequently form MDs (centers *a*, *b*, *c*). The mechanism of formation of MDs at these concentrators was described in detail in our previous publication [68]. We attract attention to the fact that all dislocation lines fitted completely in the Fig. 11 range (these lines are indicated by bright arrows) are not related to any visible structural defects. We may assume that, despite the use of Sb as a surfactant, these misfit dislocations were generated by some surface sources. Thus, in this case, we have both the surface and internal centers of dislocations' origination. In this case, origination of MDs at surface imperfections is found to be dominant compared to the MD generation at internal stress concentrators. The TD density in this film even at the very first stage of the plastic relaxation was close to 10^8 cm^{-2} (which can also be deduced from Fig. 8). It follows that, in order to grow the relaxed films with a low TD density, it is necessary to attain a comparatively small (~10^5 cm^{-2}) number of controllably formed centers of the MD origination; these centers, first, should have a low activation energy for origination of dislocations and, second, should be nondispesable, i.e.,should generate more than one dislocation.

Internal stress concentrators as the sources of MD origination can be formed artificially either using special conditions of growth [50, 51] or by the method of ion bombardment [69–72]. In the recent publication by Cai et al. [72], the data are reported on the artificial

stimulation of plastic relaxation of previously grown GeSi/Si(001) films by forming the MD-origination centers in the near-surface region using ion bombardment with He^+ ions with subsequent annealing. In order to form the MD-origination centers prior to annealing of the films, He^+ ions were implanted into the heteroboundary region. In Fig. 12, we show the data [72] on the degree of the plastic relaxation for various values of the thickness of the GeSi/Si(001) films annealed at 850°C. The GeSi films free of artificially introduced MD sources (the lower curve) do not relax, as the thickness of these films is smaller than the ~300 nm that exceeds by more than a factor of 20 the critical thickness h_c of the pseudomorphic film. As this thickness is surpassed by 50 nm, the degree of relaxation of stresses is ~50%. However, the same films irradiated with He^+ ions prior to annealing start to relax actively even at values of thickness in excess of h_c by no more than a factor of three. These results indicate that there is a competition between the MD origination at the surface and at internal sources. The fact that the pseudomorphic state of the films is observed at values of the thickness as large as exceeding h_c by a factor of 10 and more indicates that there are no MD origination centers (both in the bulk and at the surface) necessary for plastic relaxation. As a certain thickness is attained, the 2D–3D transition occurs and the surface is roughened according to the ATG mechanism. Avalanche-like generation of MDs at the surface-located sources sets in. This assumption is confirmed by a relatively narrow range of an increase in the thickness (~50 nm) within which the extent of relaxation varies from 0 to 50% (Fig. 12). The same films into which the MD sources were introduced by ion bombardment start to relax during annealing at much smaller values of the thickness at which the surface is not yet roughened. Under these conditions, there are no prerequisites for the formation of the surface-located dislocation sources; therefore, intense plastic relaxation is accomplished by dislocations generated by internal sources. Choosing properly the radiation dose and penetration depth of He^+ ions, Cai at al. [72] managed to attain practically complete relaxation of stresses in the $Ge_{0.2}Si_{0.8}$ films with the TD density at a level of 10^7 cm^{-2}. Thus, one can achieve a decrease in the TD density in plastically relaxed Ge_xSi_{1-x} films by controlled introduction of the MD-generation centers, without the use of LT Si.

6. MULTIPLICATION OF MISFIT DISLOCATIONS

Beanland [73] called attention to two stages of plastic relaxation of the stressed layer. The first stage takes place at small thicknesses of the layer and is caused by primary MD sources. The second stage develops on the basis of secondary sources related to different variants of dislocations' multiplication. The best known inexhaustible sources of this kind are (i) the source based on the mechanism suggested by Hagen and Strunk [22] and (ii) the Frank–Read sources [74]. Mechanism (i) is

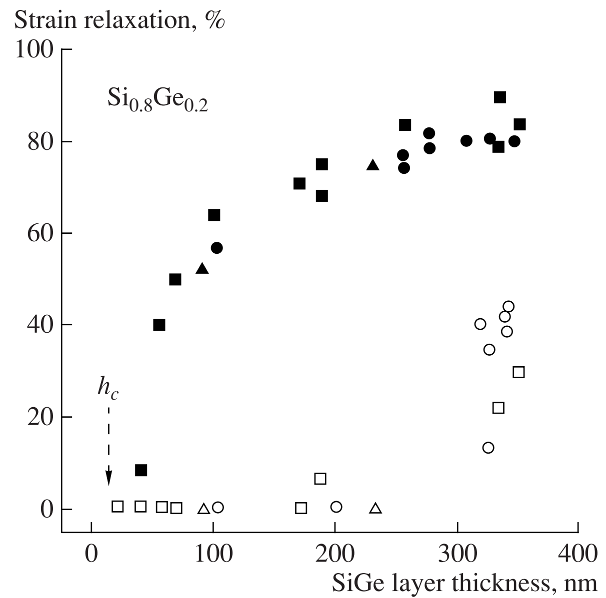

Fig. 12. The extent of plastic relaxation in relation to the thickness of GeSi/Si(001) films grown and then annealed at 850°C. Open symbols are related to the films immediately after growth and closed symbols refer to the films after formation of centers of MD origination using ion bombardment. Three types of points correspond to three different installations. The data are taken from [72] with the permission of the authors.

supposed to be operative in thin films since it is necessary that the branches of split MD reach the surface. It is necessaty for the onset of operation of the Frank–Read mechanism that the film thickness exceeds appreciably the critical thickness [46]; it is this mechanism that is most appropriate for explanation of the appearance of the second stage of plastic relaxation, which was noted in the earlier publication by Beanland [75].

Formation of a new dislocation loop according to the Frank–Read mechanism is related to appearance of pinning of TDs within the stressed film. LeGoues et al. [23, 76, 77] studied the specific source in stressed GeSi/Si(001) films of gradient composition; this source was referred to as the modified Frank–Read (MFR) source that operates at crossing glide planes (111). A characteristic feature of this mechanism is the appearance of groups of closely spaced misfit dislocations with the Burgers vector of the same sign. As a result, we have deep penetration of dislocations into the substrate as can be seen, for example, in Fig. 3. Therefore, observation of this pattern in the image of the transverse cut of the film is considered as the sign that the MFR mechanism is operative [45].

However, as can be seen from the results reported above, the second wave of plastic relaxation for a film with steplike composition can be most of all caused by a massive origination of MDs at the film surface that becomes rough when the film thickness exceeds a certain value. As we noted above, we found that the sur-

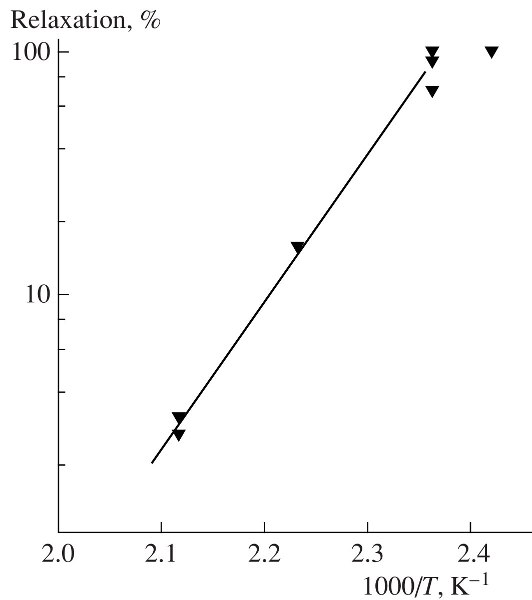

Fig. 13. Dependence of the extent of plastic relaxation in the $Ge_{0.26}Si_{0.74}$ on the temperature of growth of the "low-temperature" buffer layer. The data were taken from [51] with the permission of the authors.

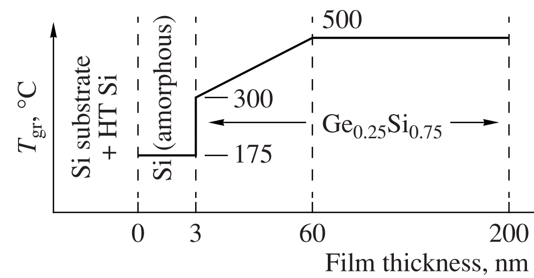

Fig. 14. Schematic representation of conditions of growth of a GeSi/Si heterostructure according to the data reported by Rahman et al. [78]. T_{gr} denotes the growth temperature.

face sources are inexhaustible and give rise to a family of misfit dislocations with the Burgers vectors of the same sign. We may assume that, under the conditions where origination of misfit dislocations from the surface is dominant, it is the multiplicativity of the surface sources that is responsible for pushing dislocations into the substrate.

7. POTENTIALITIES OF CONTROLLED GENERATION OF MISFIT DISLOCATIONS IN THE COURSE OF EPITAXIAL GROWTH

The method suggested by Cai et al. [72] and aimed at formation of the MD-origination centers by ion bombardment can be used only after preliminary growth of a GeSi film that should be in the pseudomorphic state. This circumstance limits the use of the method under consideration to the films whose plastic relaxation is necessary in the course of growth. In addition, the use of this method requires extra instrumentation for ion implantation, while, at the same time, it is desirable to have the opportunity to affect the process of plastic relaxation directly in the epitaxial-growth systems. Apparently, one could use the method of "rapid" plastic relaxation suggested in [50, 51] for artificial formation of MD-formation centers. The authors [50, 51] introduced thin Si and GeSi layers prior to growth of the main GeSi film grown under standard conditions; the growth temperature of the Si and GeSi layers was in the range of 150–200°C. According to the assumptions, a large amount of the MD-origination centers are contained in these layers. In Fig. 13 taken from [51], we show the variation in the degree of relaxation in the $Ge_{0.26}Si_{0.74}$ layer that had a thickness of about 100 nm and was grown at 550°C, in relation to the temperature of growth of the initial part of the film that consisted of Si and GeSi. It can be seen that the extent of plastic relaxation increases from several percent to 100% as the growth temperature for the first stage is reduced from 200 to 150°C. Consequently, such a buffer layer grown at ultralow (relatively) temperatures close to those of amorphization represents a built-in source of MDs. As can be seen from the analysis of the electron microscopy image of the transverse cut [50], the TD density in these relaxed compositions exceeds 10^9 cm^{-2}. We may also assume that the number of the centers for MD origination in a buffer layer grown at relatively low temperatures has the same order of magnitude. However, as can be seen from Fig. 13, as the temperature of deposition of the initial layer is increased to 200°C, plastic relaxation of the main part of the stressed film grown at 550°C amounts to no more than 3%, which signifies a decrease in the number of MD-origination centers as the growth temperature of the buffer layer is increased.

The run of the dependence shown in Fig. 13 makes it possible to assume that, by varying the growth temperature of the "low-temperature" layer along with its thickness, one can control the density of the MD-originaton centers. On the basis of this approach, Rahman et al. [78] grew 200-nm-thick GeSi films using a two-stage variant; in the first stage, a thin (1–3 nm) Si layer deposited at a temperature of 175°C was used. According to observations, this layer was amorphous. The most successful variant of further growth suggested in [78] can be traced in the scheme shown in Fig. 14. After depositing an ultrathin amorphous (UTA) Si buffer with the thickness of 1–3nm, the substrate temperature was raised to 300°C and the growth of the GeSi film was started. The growth temperature was increased gradually to 500°C when the film thickness did not exceed 60 nm. This part of the layer is an analogue of the layer grown at relatively low temperatures in the well-known methods of growth of high-quality GeSi layers with the LT-Si (LT-GeSi) buffer [37–44], with a corresponding effect of the entire composition on the structural quality as is generally accepted.

The extent of relaxation of a GeSi layer with a UTA-Si buffer was greater than in the absence of this buffer, which confirms the effect of this buffer on the process of plastic relaxation (due to earlier origination of MDs, according to the above assumptions). Direct experimental data on the TD density in the systems under consideration are not available. Rahman et al. [78] reported the results of studying the morphology of the surface of these films using the AFM method; according to these results, two orthogonal systems of extended crossing steps (cross hatches) are observed, which can indicate that there is a regular MD network in the heteroboundary with the TD density of 10^5–10^7 cm^{-2}.

Apparently, the suggested method for controlling the onset and rate of plastic relaxation of the films should be considered to be a promising technique for growing high-quality plastically relaxed GeSi/Si(001) films with a Ge fraction of approximately 0.3 and a thickness no larger than 500 nm.

8. ANNIHILATION AND SLIDING VELOCITY OF THREADING DISLOCATIONS

As mentioned above, the use of LT Si (grown at 350–400°C) brings about a decrease in the TD density in the relaxed GeSi/Si(001) layers if the Ge fraction is no larger than 0.3. These results stimulated studies of specific features of these layers grown at relatively low temperatures. Positron-annihilation spectroscopy was used [79, 80] to show that, in the case of "low-temperature" (200–400°C) epitaxy of silicon and its alloys with germanium, the grown layers contain defects of the vacancy type with a concentration of 10^{17}–10^{18} cm^{-3}. At temperatures of 350–400°C, these defects agglomerate into clusters with the largest size [80]. It is logical to assume that it is these defects that represent the cause of a decrease in the TD density in these layers.

Along with practical advantage of this approach, gaining insight into specific features of physical processes caused by introduction of the buffer Si or GeSi layers grown at a relatively low temperature should be conducive to a deeper insight into the process of the GeSi/Si(001) plastic relaxation in general. We witnessed recently rather exotic assumptions intended for explanation of this phenomenon, such as "LT Si acts as a pliable substrate" [81], "the LT-Si layer blocks TDs" [37], and "point defects capture the TDs" [39]. However, more realistic explanations are the following. In the initial stage of plastic relaxation, the vacancy-type point defects contained in LT Si grown at 350–400°C passivate the bulk MD-origination centers, in particular, those related to imperfections at the Si surface, by reducing their density or activity. This conclusion is confirmed by experimental data reported in [43] and was discussed in [47, 48]. Further on, two scenarios can take place in the course of plastic relaxation: (i) increased TD annihilation caused by the increased concentration of point defects and (ii) acceleration of plastic relaxation at a lowered TD density as a result of an increase

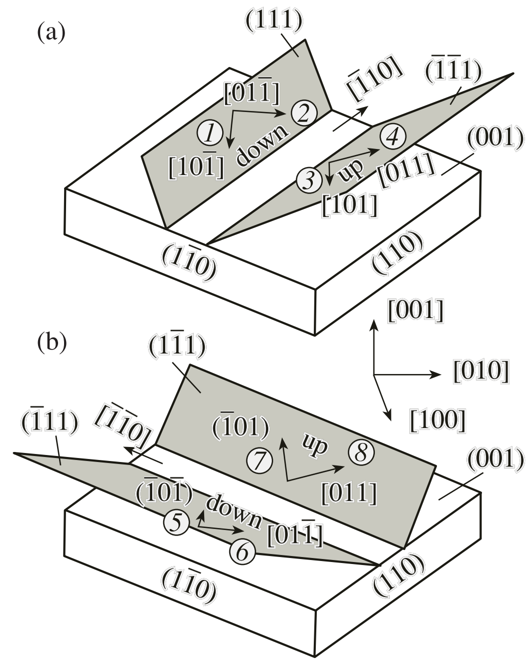

Fig. 15. The main systems of gliding for 60° dislocations involved in plastic relaxation of stressed GeSi films on Si (001).

in the glide velocity of TDs. We now consider these possibilities in more detail.

It is established with assurance that plastic relaxation of germanium–silicon heterostructures proceeds according to the mechanism of glide of 60° dislocations in inclined (to the (001) surface) {111} planes in the ⟨110⟩ directions (the glide systems $(a/2)\langle 110\rangle\{111\}$). In Fig. 15, we show the scheme of geometric distribution of these systems. For the sake of simplification, the pairs of mutually orthogonal glide planes {111} are represented separately in Figs. 15a and 15b. Under annihilation of TDs, we mean realization of one of the dislocation reactions according to which either the TDs with parallel propagation and with antiparallel Burgers vectors of the types $(a/2)[101]$ and $(a/2)[\bar{1}0\bar{1}]$ annihilate each other or a single TD with a Burgers vector of the type $(a/2)[1\bar{1}0]$ remains after an encounter of two TDs with the Burgers vectors $(a/2)[0\bar{1}\bar{1}]$ and $(a/2)[101]$ [82]. If TDs glide towards each other over not the same {111} plane but over the closely spaced parallel planes, the realization of the above reactions requires the climb of TDs that propagate close to each other. Therefore, we introduce some effective radius of dislocations' interaction. It is clear that, as the TD density is lowered, the average distance between TDs increases, the probability of their encounter decreases, and the annihilation efficiency becomes lower. For example, Hull et al. [83] showed that a decrease in the TD density due to annihilation as a result of the effect of stressed superlattices became inefficient at a TD den-

sity lower than 10^8 cm^{-2}. Theoretical calculations [82] based on the consideration of annihilation reactions between dislocations showed that the TD density decreases as the film thickness h is increased (this decrease follows the $1/h$ law) and is consistent with experimentally observed dependences in the range of TD densities 10^8–10^9 cm^{-2}. At the same time, at lower TD densities, these dependences become less steep, i.e., the dependence of the TD density on the increasing thickness becomes less heavy. Consequently, if the TD density attains a value of ~10^8 cm^{-2} at the stage of plastic relaxation of the GeSi/Si(001), the film thickness should be increased by more than 100 times, which is unacceptable in the majority of cases.

In the presence of a high concentration of point defects, the TD annihilation rate can increase owing to an increase in the interaction radius of TDs that propagate in the glide planes close to each other [84]. Typically, the stressed layers are grown at a temperature at which the dislocation climb can be disregarded. A high concentration of point defects formed as a result of either irradiation or growth of a "low-temperature" buffer layer should, in general, increase the probability of inclusion of this mechanism into the process of annihilation of threading dislocations that are located in the glide planes close to each other. As is well known, the concentration of the vacancy-type point defects is at a level of 10^{18} cm^{-3} in the buffer LT-Si or LT-GeSi layers grown in the growth-temperature range of 300–400°C [80]. Apparently, this defect concentration cannot be sufficient for appreciable activation of the TD-annihilation mechanisms and a decrease in the TD density to a level of 10^5–10^6 cm^{-2}. The proof for the annihilation-mechanism operation could be obtained by an experimentally observed decrease in the TD density to 10^5–10^6 cm^{-2} in the course of isothermal annealing of a plastically relaxing sample with large lateral sizes (the latter is necessary in order to exclude the artifacts related to a decrease in the TD density due to the fact that TDs reach the side faces of the sample subjected to annealing). However, we failed to find such data in available publications. In contrast, there are data that indicate that the TD density increases when the systems under consideration are annealed. For example, Bolkhovityanov et al. [47] reported data on the variation in the TD density in the GeSi/Si(001) films grown at the same temperatures as those corresponding to the growth of the LT-Si layer (i.e., 300–400°C). The TD density just increased with the plastic-relaxation extent, which indicates that there is additional generation of MDs in the course of annealing. This fact casts doubt on the mechanism that implies a decrease in the TD density due to annihilation of inclined MD segments in a "cloud" of point defects [84], at least in LT-Si heterostructures grown at temperatures of 300–400°C.

Therefore, we consider another factor that can induce a decrease in the TD density. The rate of plastic relaxation of a stressed film depends on the average rate of the MD lengthening, i.e., on the TD glide rate. If this rate according to some causes can increase with other factors being the same (temperature, the lattice mismatch between the film and substrate), then a decrease in the stresses in the film should occur faster; for complete plastic relaxation of the film, a smaller amount of TDs is required, which is conceptually one of the main goals in the formation of artificial substrates based on GeSi/Si(001). The assumption that one of the factors conducive to a decrease in the TD density in GeSi films grown on LT Si can be an increase in the TD glide velocity was first made by Bolkhovityanov et al. in 2004 [47, 48] and was later confirmed experimentally [49].

According to classical concepts [85], the dislocation velocity can be written as

$$V_d = V_0 \tau_{\text{eff}}^m \exp(-E_V/kT), \quad (1)$$

where V_0 is a constant, E_V is activation energy for glide motion of a dislocation, and τ_{eff} is the effective cleavage stress that represents the driving force for the TD propagation in stressed films with the thickness h. This effective stress is proportional to the strain ε (at the initial stage of relaxation, we have $\varepsilon \propto f$) and increases as the film thickness h is increased as soon as the critical thickness (according to Matthews et al. [13]) is exceeded (a more detailed consideration can be found in the review by Fitzgerald [15]).

In Fig. 16, we show the TD glide velocities determined in the course of annealing of the Ge_xSi_{1-x} films (with $x \approx 0.3$) grown at relatively low temperatures [49]. We show both the averaged and calculated (based on measurements of the lengths of unit MDs) TD glide velocities in comparison with well-known published data referred to by us as classical. Solid straight lines are plotted on the basis of calculations using expression (1) and taking into account the parameters reported by Tuppen et al. [86] for $x = 0.3$ and the stressed-film thickness of 50 and 200 nm. Points *4* and *5* represent experimental data [87] for GeSi films with a Ge fraction close to 0.3. It can be seen that experimental data are in satisfactory agreement with theoretical dependence. Points *1–3* represent our data obtained using GeSi films (with a Ge fraction close to 0.3) grown at relatively low temperatures. The velocities were determined using both the length of MDs detected in the TEM images of longitudinal cuts of the samples after their rapid annealing and the variation in the extent of plastic relaxation taking into account the TD density determined also from the TEM images [49]. It can be seen that the TD glide-velocity values, both the average and that calculated from the length of separate MDs in the films that included a "low-temperature" buffer Si layer and were grown by us at 300–350°C, are larger than the classical data.

The TD-glide velocity calculated using the length of separate misfit dislocations in heterostructure A (symbol *1* in Fig. 16) exceeds (by an order of magnitude) the calculated expected velocity of a unit dislocation as cal-

culated using expression (1). The average calculated dislocation velocity V_d for the same heterostructure A was found to be, as expected, smaller by several times (symbols *2* in Fig. 16). Nevertheless, this velocity is also higher than the calculated expected velocity of a unit dislocation. The values of the average dislocation velocity V_d for heterostructure B annealed at 400 and 500°C also exceeded the calculated expected velocity of a unit dislocation.

The mechanism bringing about an increase in the TD glide velocity in the above films can consist in the following. It is well known [87] that dislocations glide because of the formation of double kinks at this dislocation. This double kink expands over the dislocation line and brings about a transition of the dislocation line to a neighboring energy valley. Hull et al. [87] showed that, under conditions of molecular epitaxy of thin GeSi films with limited TD length, only a single double kink is formed within the entire dislocation line at each instant of time, so that the glide velocity of the dislocation becomes dependent on the frequency of their formation. If we assume that, as a result of the appearance of vacancy clusters, two or more double kinks are formed simultaneously in some part of the film at the dislocation line intersecting these clusters, this circumstance should be conducive to enhancement of TDs' glide.

Thus, in the region of Ge fractions $x < 0.3$ for the Ge_xSi_{1-x}/LT-Si/Si(001) structures, the following factors can be considered as the main causes of a decrease in the TD density in relaxed GeSi layers: the density of the MD origination centers is lower than in the "classical" case and the glide velocity of threading branches of these dislocations is higher than the corresponding velocity in classical heterosystems grown at a temperature of 550°C without buffer layers.

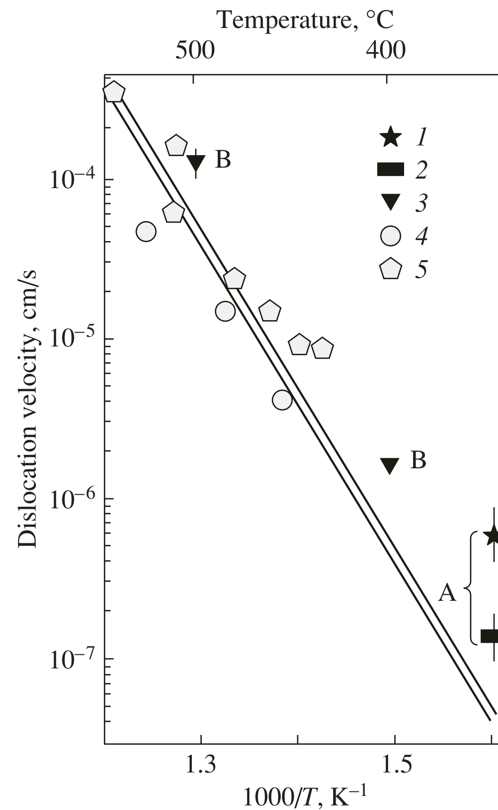

Fig. 16. Temperature dependences of TDs' glide velocities. Theoretical dependences were calculated for stressed $Ge_{0.3}Si_{0.7}$ films with thicknesses of 200 nm (the upper solid line) and 50 nm (the lower solid line). Symbols (*1–3*) represent our data. Data *1* and *2* represent the velocity of a unit dislocation and average velocity determined for sample A, i.e., the $Ge_{0.32}Si_{0.68}$/LT-Si/Si(001). Data *3* represent the average velocities determined for sample B (i.e., the $Ge_{0.28}Si_{0.72}$/LT-Si/Si(001) structure) with a thickness of 200 nm. See [49] for details for data *1–3*. Data *4* and *5* are taken from [87]: $x = 0.3$ and $h = 35$ nm for (*4*) and $x = 0.33$ and $h = 45$ nm for (*5*).

9. FORMATION OF EDGE MISFIT DISLOCATIONS

As follows from the classical energy-balance model suggested by Matthews [14, 15], the purely edge dislocations are more advantageous compared with 60° dislocations. If the equilibrium linear density of edge dislocations is defined as f/b (b is the length of the Burgers vector), the density of 60° dislocations should be twice as high ($2f/b$) for the (001) heterointerface. However, the edge MDs are not gliding in materials with the diamond (sphalerite) crystal lattice, so that introduction of these dislocations into the (001) heterointerface is impossible by the mechanism of glide over the {111} planes. At the same time, according to a large amount of experimental data, this type of MD is practically always present at the film–substrate interface. In addition, the fraction of these MDs in the total number of MDs increases if the value of f increases and if the degree of relaxation of heteroepitaxial stresses becomes higher [88]. As an example, Fig. 17 shows the TEM images obtained by us for an MD network in the $Ge_{0.29}Si_{0.71}$/Si(001) heterosystem annealed at 800°C.

Two dark-field images of the same area (Figs. 17a, 17b) obtained under conditions of two-wave diffraction from mutually perpendicular set of planes {220} illustrate the complete quenching of purely edge Lomer MDs if the condition $\mathbf{g} \times \mathbf{b} = 0$ is satisfied, where $\mathbf{g} \parallel \langle 220 \rangle$. At the degree of plastic relaxation of approximately 60%, the fraction of edge dislocations was ~50%.

The issue of formation of edge MDs in a large number in the course of plastic relaxation of stressed heterosystems remains under discussion until now. One of the models accounting for the appearance of the edge MDs at the (001) heteroboundary (the Lomer dislocations) is based on the fact that, at large values of f (like in a GeSi system), the stressed film transforms into an island film during the initial stages of growth. In this case, a large fraction of edge MDs (as large as 100%) is accounted for by introduction of dislocations from the islands' edges. However, this conclusion contradicts the fact

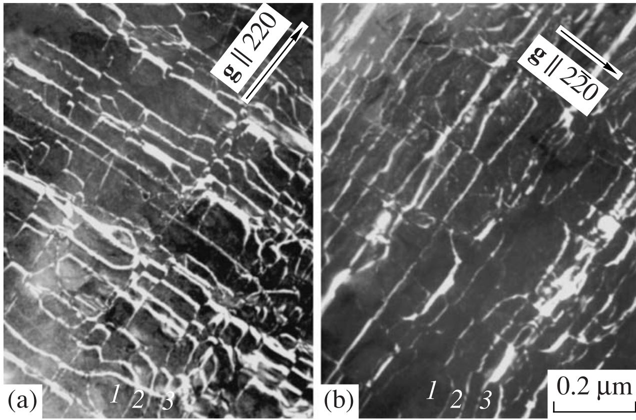

Fig. 17. Planar TEM images of misfit dislocations in the $Ge_{0.29}Si_{0.71}$/Si(001) system grown at T = 400°C and annealed for 1 h at T_{ann} = 800°C. The film's thickness was 100 nm. The images were obtained under conditions of a "weak beam" at (a) **g** || ⟨220⟩ and (b) **g** || ⟨2$\bar{2}$0⟩ . Numbers indicate the reference dislocations.

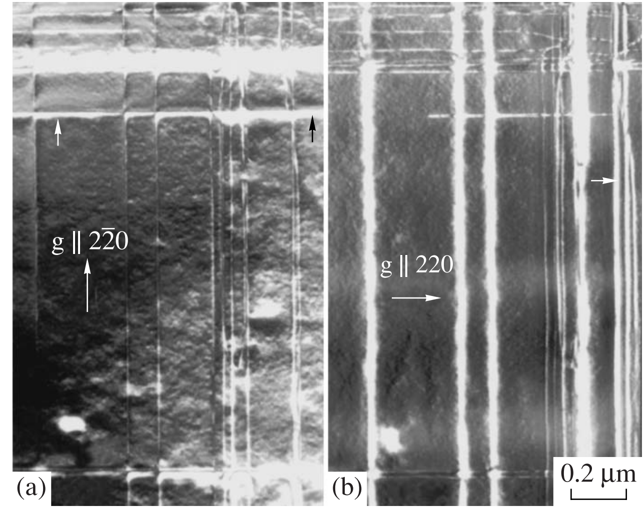

Fig. 18. The dark-field planar TEM images of misfit dislocations; the images were obtained under conditions of a "weak beam" in order to reveal the Lomer dislocations. A $Ge_{0.33}Si_{0.67}$ film with a thickness of 100 nm was grown on Si (001) at T = 400°C. The images were obtained at (a) **g** || ⟨2$\bar{2}$0⟩ and (b) **g** || ⟨220⟩.

that only 60° dislocations with the Burgers vectors inclined to the heterointerface are observed in the bulk of these films. As is well known, these dislocations represent threading segments of MDs. It is worth noting that the same dislocation should have an identical Burgers vector over its entire length. In addition, the purely edge MDs are observed also at much smaller values of f (see Fig. 17) at which the growth of the film at all stages proceeds according to the layer-by-layer mechanism without formation of 3D islands.

Another mechanism of formation of Lomer dislocations in heterosystems with a large value of f was first observed in a study of interfaces in an InAs–GaAs (001) system (f = 7%) [89, 90], was then discovered independently by Eaglesham et al. [17], and was later considered in detail by Vdovin [91]. The essence of this mechanism consists in the following. In the case of glide of two complementary 60° dislocations over inclined mutually intersecting planes {111}, these dislocations interact via the classical reaction known from the theory of dislocations:

$$\frac{a}{2}[101] + \frac{a}{2}[01\bar{1}] = \frac{a}{2}[110]. \quad (2)$$

It can be seen from Fig. 15 that these reactions can involve systems (1) and (4), (2) and (3), (5) and (8), and (6) and (7) (see designations in Fig. 15), i.e., the dislocations that reside in the {111} planes deflected in different directions from the ⟨110⟩ axis. In this case, if the glide planes intersect each other not precisely at the heteroboundary, the closely lying 60° MDs or their separate branches should either be elevated to the film or descend to the substrate, to the line of intersection of the inclined glide planes. These configurations were observed experimentally by Vdovin [91]. However, in the majority of cases, the observed edge dislocations are located exactly at the film–substrate interface, which suggests that there are other mechanisms for their formation that do not imply an accidental encounter of two complementary 60° MDs. Kvam et al. [18] suggest, as an alternative, origination of a complementary 60° dislocation loop at the surface of a thin layer under the effect of the stress field of a 60° MD already existing at the interface. Gosling [92] showed theoretically that origination of the above loop in the region of an already existing 60° MD (with subsequent transformation into a 90° MD) features a much lower energy barrier than in the case of origination of a single dislocation loop in the absence of an MD. The situation illustrated in Fig. 18 can correspond to one of the above mechanisms. Here, long edge MDs (indicated by arrows) are observed even at the initial stage of plastic relaxation (of about 10%) in a GeSi/Si(001) film grown at a temperature of ~450°C.

It is mentioned in available publications that the fraction of edge MDs in relaxing films increases as the initial degree of mismatch is increased, i.e., as the Ge fraction is increased [88]. Apparently, mechanisms of introduction of MDs change appreciably as f is increased. In a GeSi system, the limiting and most important case is the epitaxial growth of "pure" Ge on Si (001) substrates. We now consider this case in more detail.

10. PROBLEMS OF EPITAXY AT A LARGE MISMATCH BETWEEN THE LATTICE PARAMETERS OF JOINED MATERIALS

The mismatch of lattice parameters f amounts to ~4% for a Ge–Si heterosystem. In addition, there is

a large mismatch between linear coefficients of thermal expansion: 5.92×10^{-6} and 2.3×10^{-6} K^{-1} for Ge and Si, respectively. The problem of accommodation of crystal lattices for this pair of materials is mainly solved by using Ge_xSi_{1-x} buffer layers in which the value of x varies from zero at the heteroboundary to unity at the surface of the buffer layer [5]. In addition, the thickness of the buffer layer should be approximately 10 μm in order to ensure that the TD density in this layer is lower than 10^{-6} cm^{-2}. At such large thicknesses, which are often unacceptable for practical applications, a fairly developed profile arises at the surface of buffer layer (and then at the surface of the Ge epitaxial layer grown on the buffer), which requires the finish polishing of the surface [93]. Therefore, researchers recently return again to the growth of Ge films directly on Si substrates. In this case, the problems of high density of threading dislocations and difference between the thermal-expansion coefficients for the film and Si substrate become very important.

The methods used to reduce the TD density in the heterosystems under consideration can be divided into two groups. These are

(i) "Off-line" methods, i.e., control of threading dislocations after heteroepitaxy; and

(ii) "On-line" methods, i.e., control of threading dislocations in the course of heteroepitaxy.

The main postepitaxial method consists in the use of cyclic heat treatments of the grown relaxed heterostructure with an upper temperature limit of 900°C [94, 95]. Efficiency of thermocycling is caused by the fact that the Ge films become alternatively compressed or extended in a wide range of variations in temperature; as a result, TDs move in one or the opposite direction. The TDs actively annihilate when they encounter each other in various combinations. In addition, at high temperatures (900°C), the climb of dislocations is appreciably accelerated, which also facilitates their annihilation. High temperatures of cyclic annealing result in diffusion-related broadening of the Ge/Si interface by lowering the magnitude of the stresses' jump caused by the difference in the thermal-expansion coefficients for joined materials. However, the upper limit for heating the heterostructures is close to the melting temperature of Ge (937°C), which is unacceptable in the majority of cases for growth of compounds with preliminarily formed p–n junctions.

Prior to considering the second group of methods used to reduce the TD density, we state the causes of the TDs' high density in heterosystems with a large value of f. It is well known that either the Volmer–Weber growth mechanism or the Stranski–Krastanov mechanism is realized in the case of heteroepitaxy of films with a large value of f. Formation of 3D epitaxial islands is characteristic of both of these mechanisms. In the case of the Volmer–Weber mechanism, islands are formed immediately on the surface of the substrate, while the islands are formed on the surface of the thin continuous wetting layer in the case of the Stranski–Krastanov mechansm. It is noteworthy that, depending on the type of the material and conditions of epitaxy, one can observe the formation of both coherent stressed islands without defects and relaxed islands with MDs at the interface. At present, this specific feature of growth of the films with a large value of f finds wide practical application in nanotechnology for formation of high-density arrays of islands with nanometer sizes, so-called quantum dots (see, for example, the reviews [29, 30]). At the same time, formation of islands at the initial stage of heteroepitaxy is an unwanted phenomenon in the growth of continuous and comparatively thick layers. The causes of this effect consist of the following.

In the initial stages of islands' formation, plastic relaxation (formation of MDs) can set in before the first continuous layer is formed. In this case, the MD length is limited by lateral sizes of islands at the instant of their coalescence; TDs are bound to be formed at the coalescence boundaries. Since the sizes of islands at coalescence do not typically exceed several tens of nanometers, the TD density is found to be very high ($>10^{10}$ cm^{-2}). As the film thickness is increased, a fraction of these dislocations annihilates; however, this is a very small fraction (see Section 8). We studied in detail the effect of origination of TDs in the course of coalescence of islands previously [96–98]; we then formulated for the first time one of the necessary conditions for obtainment of heteroepitaxial films with a low TD density. This condition consists in the following: the critical thickness of the pseudomorphic film should be larger than the thickness of the first continuous layer. This condition is necessary but not sufficient. For example, if the Stranski–Krastanov mechanism is in effect, relaxation of stresses sets in after formation of the continuous (wetting) layer due to the formation of elastically relaxing islands (the ATG mechanism). However, these islands also represent the effective centers for origination of MDs (see Section 3). All the aforesaid is directly related to a Ge–Si system.

It is well known [26] that growth of Ge on Si sets in by the Stranski–Krastanov mechanism, in which case the first four monolayers of the Ge film are continuous (the wetting layer); if this thickness is exceeded, Ge islands with a density of 10^{10}–10^{11} cm^{-2} are formed. As a result of such surface inhomogeneity, appearance of high-density stress concentrators [32] gives rise to large density ($\sim 10^{10}$ cm^{-2}) defects penetrating through the Ge film [99].The "on-line" methods are exactly directed at elimination of the above cause. We now consider some of these methods.

In order to kinetically retard the formation of islands on the wetting layer, some researchers performed the heteroepitaxy of Ge on Si at lower temperatures in the initial stage of growth. The growth began at the temperatures of 330 [100], 350 [94], and 370°C [101]. It was found that the Ge layers grown at relatively low temper-

atures featured again a tendency towards formation of islands and high-rate introduction of defects. For example, Halbwax et al. [100] showed that films grown at relatively low temperatures are unstable at thicknesses smaller than 27 nm. Notwithstanding the almost complete relaxation observed at the thickness of 7.5 nm, a much larger thickness is needed in order for the film to grow still according to the 2D mechanism as the growth temperature is increased to 600°C.

Another possibility for suppressing the island growth of Ge consists in the introduction of surface-active impurities (surfactants) in order to stabilize the 2D layer-by-layer mechanism of growth. Indeed, if the surfactants as arsenic, antimony, or atomic hydrogen are used in the growth of Ge on the Si (001) substrates, the 2D growth of the films continues at a thickness larger than four monolayers [102–106]. However, this circumstance cannot prevent at a later time the roughening of the growth surface of the Ge film; this roughening is caused by the effect of the ATG mechanism, i.e., by the appearance of elastic relaxation via the formation of 3D surface profile. As can be seen from Fig. 8, the density of threading dislocations in relaxed GeSi film exceeds 10^8 cm^{-2} even at a Ge fraction larger than 0.4 in spite of the presence of Sb. Consequently, the 2D–3D transition still represents the main cause of the high density of the centers for the origination of dislocations, although this occurs at a somewhat larger fraction of Ge than in the absence of Sb. It may be assumed that the pseudomorphic Ge film can also become rougher (notwithstanding the presence of a surfactant) as the film thickness is increased, which, as a result, will give rise at a later time to a high density of TDs and other defects. Indeed, according to Horn von Hoegen et al. [107], the Ge film remains continuous if Sb is used as a surfactant and the growth temperature is ~500°C; however, the roughness of the film's surface increases and defects with a high density are generated at an average coverage amounting to ~12 monolayers.

Based on the assumption that there is competition between the sources of MD origination (see Section 5), we can assume that the use of either the Ge layer grown at a relatively low temperature or a surfactant in the course of Ge growth on Si (001) had not made it possible to find the conditions under which the sources of low-density MDs would manifest themselves and compete with high-density surface sources.

Positive results were obtained in the case of heteroepitaxy of the Ge:Sb films on the Si (111) substrates (see, for example, publication [108] and references therein). It was shown previously [109, 110] that, in the case under consideration, an ordered network of misfit dislocations is formed at the interface; these dislocations are of the edge type, i.e., are the most favorable energetically. The mechanism of formation of such a dislocation network as suggested in [109, 110] is based on the fact that mobile dislocations originated at the rough surface of a still thin film (eight or more Ge monolayers [111, 112]) glide over inclined {111} planes to the interface that also represents a glide plane for these dislocations. This circumstance makes the interaction of dislocations easier; as a result, an ordered network of edge misfit dislocations is formed at the interface, while the threading segments are supposedly annihilated effectively. As a result, the TD density is no higher than 10^8 cm^{-2} [111].

As is evident from the analysis of mechanisms of plastic relaxation in a Ge/Si(111) system, a necessary step in the sequence intended to reduce the TD density is the formation of an ordered MD network of the edge type at the initial stage of plastic relaxation. Apparently, in a Ge/Si(111) system, this MD-network formation is favored by the fact the energy of formation of a step on the {111} plane is higher than that on the {001} plane [113], which makes roughening of the surface difficult and is conducive to formation of extended islands with flat tops. Another factor is represented by the possibility of a glide of dislocations over the interface, which facilitates annihilation of threading segments. The {001} surface does not feature these properties; attempts to grow thin (~1 μm) high-quality Ge films on the substrates with this (most widely used in semiconductor technology) orientation failed until 2005, in spite of the use of surfactants [107, 114, 115].

In studies by Wietler et al. [116] it was shown that one can grow high-quality Ge films with thicknesses no larger than 1 μm on the Si (001) substrates. The ordered and dense network of misfit dislocations was observed at the interface Figs. 19a, 19b). According to Fig. 19c, the half-width of the X-ray rocking curve for the Ge film was at a level of 100″, which is an unprecedentedly small value for Ge films with a thickness no larger than 1 μm grown directly on Si (001).

The main changes effected in this study in comparison with previous studies were the following: (i) an increase in the growth temperature for Ge film on Si (001) to 700°C and (ii) an uninterrupted flux of Sb during the entire growth time. The latter circumstance is necessary since, at the above temperature, antimony is actively desorbed from the surface. The continuous Sb flux was exactly necessary to preserve the effect of antimony as an element that passivates the 2D–3D transition. We may assume that in this case, due to increased film-growth temperature, the mechanisms of origination of complementary dislocations and formation of an ordered network of edge misfit dislocations became the main factors that control the process of plastic relaxation at its earliest stages, while the spontaneous introduction of dislocations as a result of roughening of the surface was retarded owing to the presence of the surfactant Sb impurity.

Another method for growing thin high-quality Ge films on the Si (001) substrates is known. Bauer et al. [117] used the CVD method with involvement of deuterium-stabilized tin hydrides, which made it possible to grow GeSn films with a Sn content of several per-

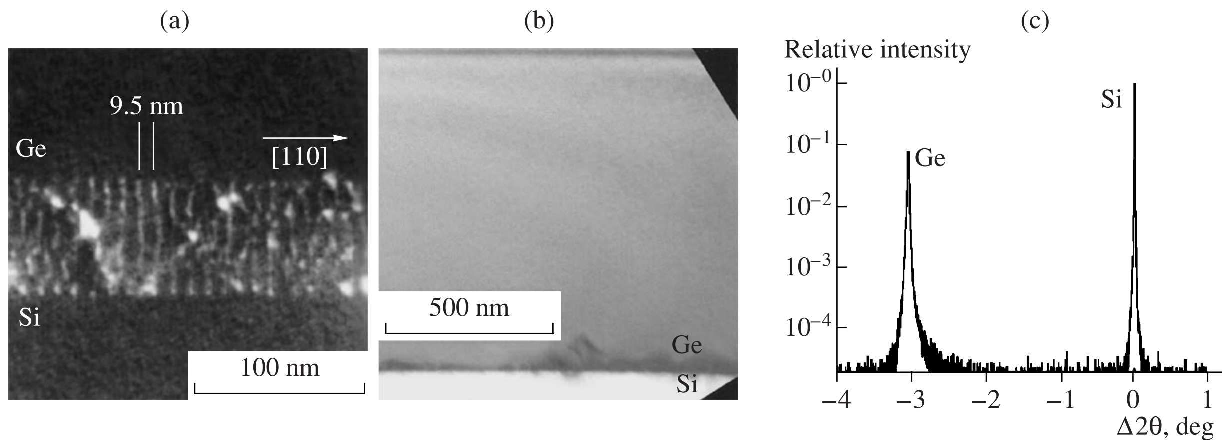

Fig. 19. (a) The dark-field TEM image obtained under conditions of a “weak beam” for the Ge/Si(001) heteroboundary inclined by 25° around the [110] axis. (b) The bright-field TEM image of a cross section of 1-μm-thick Ge film grown on Si (001). (c) The X-ray rocking curves in reference to the (004) planes for a 1-μm-thick Ge film grown on Si (001). The data were taken from [116] with the permission of the authors.

cent. It was argued [117–119] that, even at a film thickness smaller than 100 nm, the films were relaxed with the network of the Lomer edge dislocations at the interface. Using this method, Ge films were grown in the $Ge/Sn_{0.02}Ge_{0.98}/Si$ structure. It is argued that the Ge films had a low concentration of defects (~10^{-6} cm^{-2}), while the half-width of the X-ray rocking curves in the (004) reflection amounted to 250″.

11. CONCLUSIONS

The problem of growth of high-quality relaxed Ge_xSi_{1-x} films on Si (001) substrates remains topical and is still in the stage of the search for methods that can reduce the density of structural defects (mainly, threading dislocations) in the layers with a comparatively small thickness (<1 μm) with a minimum roughness of the growth surface.

The use of the Si buffer layer grown at a comparatively low temperature (300–400°C) (LT Si) and deposited onto the Si substrate immediately before the GeSi alloy growth makes it possible to solve this problem for alloys with Ge content no larger than 30% and grow relaxed $Ge_{0.3}Si_{0.7}$ layers with a thickness smaller than 1 μm and a low density of threading dislocations (10^5–10^6 cm^{-2}). The main causes of this reduction in the TD density are a decrease in the number of centers and in the MD generation rate and also an increase in the glide velocity of threading dislocations compared to this velocity in classical heterosystems grown at a temperature of 550°C without buffer layers. However, at $x > 0.3$, a decrease in the growth temperature for the buffer and main layers does not provide the desired effect; the density of threading dislocations remains at a level of 10^8–10^9 cm^{-2}. The main cause of this phenomenon is the transition from the layer-by-layer mechanism to the 3D mechanism of growth of the stressed film (the 2D–3D transition), at which the number of centers of the MD generation is increased.

On the basis of analysis of the above experimental data, we may conclude that the following is necessary to extend the potential of growth of high-quality plastically relaxed GeSi/Si(001) films: one should have a possibility of (i) controlled formation of MD-origination centers and (ii) retardation or complete suppression of the 2D–3D transition in order to restrain origination of additional MDs from the surface of the stressed film and, correspondingly, additional TDs. Origination of MDs from the surface occurs at the stress concentrators caused by the existence of 3D profile. The use of a surfactant (antimony) makes it possible to retard the transition to a 3D surface profile and, as a result, restrain the introduction of MDs and additionally improve the structural properties of the Ge_xSi_{1-x}/Si(001) films with $x \approx 0.3$. Nevertheless, the related improvement of structural properties of GeSi/Si(001) films with a Ge fraction larger than 0.3 is insufficient since the surface profile is still formed according to the ATG mechanism simultaneously over the entire area of the structure, while the density of the MD-origination centers correlates with the density of pseudoislands; the latter density is at a level of 10^8–10^{10} cm^{-2}. Therefore, it is unlikely that one can reduce in a controlled way the rate of introduction of MDs from the surface to any appreciable extent. At the same time, the controllable introduction of MDs by origination of them in the bulk of the film seems to be more promising. The possibility of affecting the glide velocity of TDs in the course of plastic relaxation of the GeSi/Si films, i.e., an increase in this velocity, is, in our opinion, one of the promising methods for reducing the TD density; however, this effect is inadequately studied so far.

Attempts to grow Ge films with low dislocation densities and a thickness on the order of a micrometer on

Si (001) substrates without using postgrowth thermocycling mostly failed until recently. Recent publications by Wietler et al. [116] and Roucka et al. [118, 119] showed that, if some conditions are satisfied, one can grow high-quality Ge films on Si (001) substrates. Plastic relaxation of these films is accompanied by formation of an ordered network of edge MDs without formation of a large number of threading defects if the thickness the films is small. The search for an extended range of these conditions and the detailing of relaxation mechanisms represent the most topical problem at present.

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