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Ga segregation in MnSb epitaxial growth on GaAs (100) and (111) *B* substrates

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We have found Ga segregation during the epitaxial growth of MnSb thin films on GaAs substrates by core-level photoemission spectroscopy using synchrotron radiation. MnSb films were grown on (100) and (111)B GaAs substrates by molecular beam epitaxy (MBE). While the As 3*d* peak intensities decrease gradually, the Ga 3*d* photoemission peak intensities remain unchanged even with increasing MnSb overlayer thickness. These results suggest that the Ga atoms are segregated at the surface during growth. The thickness of the segregated Ga layers is estimated to be 0.5 monolayer (ML) for MnSb on GaAs(100) and 1.0 ML for MnSb on GaAs(111)*B*. It is considered that the segregated Ga layer acts as a surfactant and decreases the surface energy and contributes to the layer-by-layer growth. The thickness of the surfactant layer is thought to have an influence on the surface morphology of the MnSb film.

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I. INTRODUCTION

A heterojunction of semiconductors and ferromagnetic metals has attracted much interest as a new type of magnetoelectronic device utilizing not only the charge but also the spin of carriers.[1] Recently, epitaxial Mn-based magnetic compounds such as MnSb,[2,3] MnAs,[4–8] MnGa,[9] and MnAl (Ref. 10) have been successfully grown on semiconductor substrates using molecular beam epitaxy (MBE). In the epitaxial growth of such ferromagnetic metals on semiconductors, we are confronted with the difficulty that there is a strong reactivity at their interface. In order to apply these films to realistic devices, the control of these surfaces and interfaces is very important.[11] The practical demands for the hybrid structure of ferromagnetic metals on semiconductors are a very abrupt interface, which is very important for spin injection, and a flat surface. In this article, we present the surface and interface structures and the initial growth mechanism of MnSb thin films on GaAs substrates studied by core-level photoemission spectroscopy using synchrotron radiation. The heterostructure of epitaxial ferromagnetic metals on semiconductors is very important for new applications such as integrated spintronic devices, as well as for the study of low dimensional magnetism because it can be used as the model material. MnSb has attracted much attention because of its large magneto-optic effect, a large magnetic anisotropy in the direction of the easy magnetization axis and the large magnetoresistance.[3,12,13] The bulk MnSb has a stable ferromagnetic phase below the Curie temperature (578 K) and the structure is a NiAs type (space group $P63/mmc$) with lattice constants of $a = 0.4128$ nm and $c = 0.5789$ nm.[14,15] Recently Akinaga *et al.* observed a very large magnetoresistance, so called ‘‘magnetoresistive switch,’’ in MnSb dots on *S*-passivated GaAs(100) substrates.[16–18] To clarify the origin of the magnetoresistive switch effect, investigation for the initial growth mechanism of MnSb on GaAs is indispensable.

II. EXPERIMENTAL

The MBE growth of MnSb films on GaAs (100) and (111)*B* substrates and the *in situ* photoemission spectroscopy measurements were carried out at the BL-1A of the Photon Factory. The epi-ready n^+-GaAs substrate was introduced into the MBE growth chamber and the clean surface was obtained by annealing in an As atmosphere. The base pressure of the MBE growth chamber was 8×10^{-10} Torr. After cleaning the GaAs substrate, we exposed the substrate only to an Sb beam to obtain the Sb-terminated surface. After obtaining the Sb-terminated surface, the substrate is exposed to both Mn and Sb beams. The temperature of the substrate during MBE growth was set to 300 °C. The Sb/Mn flux ratio was set to 2. The growth rate was 0.3 Å/s estimated from the transmission electron microscopy (TEM) images after the growth. For both samples, we grew MnSb films and measured the photoemission spectra for each thickness without breaking the ultrahigh vacuum. The surface structure of the samples during the MBE growth was measured by reflection high-energy electron diffraction (RHEED). We investigated the chemical states of Ga 3*d*, Sb 4*d*, and As 3*d* core levels by synchrotron radiation photoemission spectroscopy. Photoelectrons were measured by a high-resolution hemispherical angle-integrated electron analyzer (Scienta SES200). The photon energy was set at 118.9 eV and the take-off angle of photoelectrons was set normal to the sample surface. The structure of the MnSb films and the interfaces were observed by *ex situ* cross-sectional TEM. Epitaxial orientations were checked by *ex situ* x-ray diffraction (XRD).

III. RESULTS AND DISCUSSION

A. Surface and interface structures of MnSb films on GaAs(100) and (111)B

Figure 1(a) shows a RHEED pattern along the ⟨011⟩ azimuth of the MnSb film grown on the GaAs(100) substrate

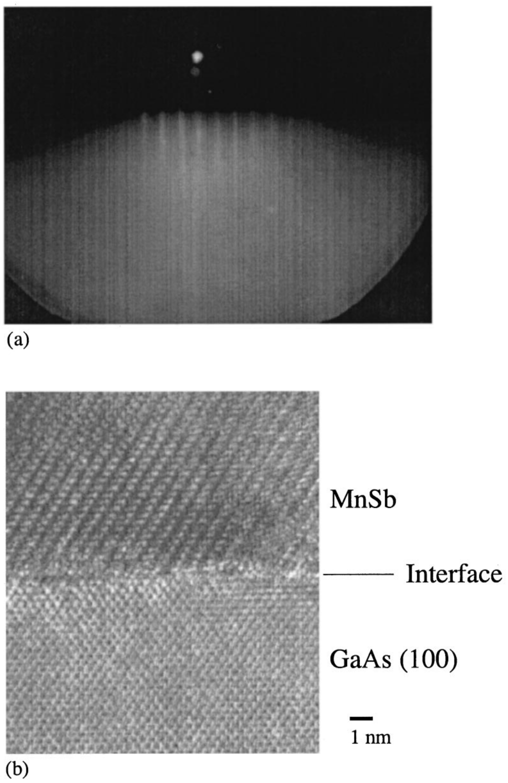

FIG. 1. (a) RHEED patterns of MnSb/GaAs(001) along the ⟨011⟩ azimuth during the MBE growth of the MnSb film when the thickness of the MnSb film is 4 ML. Clear (1×1) streaks are observed. (b) A cross-sectional TEM image of the interface region of MnSb/GaAs(001) taken along ⟨011⟩ with the thickness of 300 Å. The MnSb region, GaAs region, and the interface are illustrated in the figure. A scale for the TEM image is also shown in the figure. The image shows the abrupt interface between the MnSb film and the GaAs(100) substrate.

during the MBE growth. The thickness of the film is 4 ML. This RHEED pattern shows (1×1) streaks, and the in-plane spacing of the MnSb film in the GaAs [011] direction as estimated by the streak spacing in the RHEED pattern is similar to that of bulk ferromagnetic MnSb[1101]. The background intensity of the RHEED pattern is rather high indicating a considerable surface roughness exists in the MnSb(1101) surface on GaAs(100). In the XRD pattern, we observed GaAs(002) and (004) reflection as well as MnSb(1101) and (2202) peaks, confirming the epitaxial orientation of MnSb(1101) on GaAs(100). Figure 1(b) shows a cross-sectional TEM image of an interface region taken from MnSb/GaAs(100) along ⟨011⟩ with the thickness of the MnSb film of 300 Å. This TEM image shows clear lattice fringes from both MnSb and GaAs regions, and that the interface between the MnSb film and the GaAs(100) substrate is very sharp indicating the formation of abrupt interface between the MnSb film and the GaAs(100) substrate. The *c* axis of MnSb observed in the TEM image is tilted about 70° from the GaAs ⟨100⟩ direction which is consistent with the crystal orientation of MnSb(1101) on GaAs(100). It should be noted that although the MnSb surface roughness observed by RHEED is considerably large, the interface structure observed by TEM is very abrupt and dislocations are relaxed near the interface. XRD and cross-sectional TEM results show that the MnSb epitaxial films on GaAs(100) substrate are of high quality.

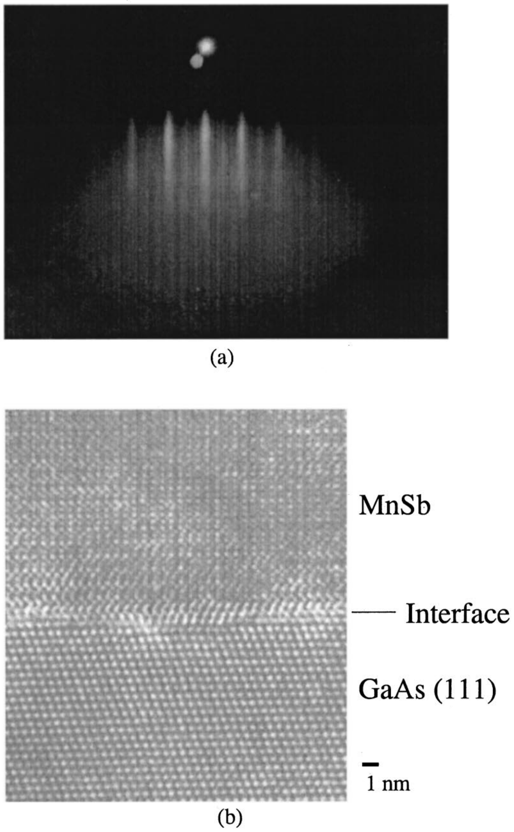

FIG. 2. (a) RHEED patterns of MnSb/GaAs(111)*B* along the ⟨112⟩ azimuth during the growth of the MnSb film when the thickness of the MnSb film is 4 ML. Clear (2×2) streaks are observed. (b) A cross-sectional TEM image along ⟨112⟩ of the interface region of MnSb/GaAs(111)*B* taken with the thickness of 300 Å. The MnSb region, GaAs region, and the interface are illustrated in the figure. The scale for the TEM image is also shown in the figure. The image indicates the abrupt interface between the MnSb film and the GaAs(111)*B* substrate.

Figure 2(a) shows a RHEED pattern along the ⟨112⟩ azimuth of the MnSb film grown on the GaAs(111)*B* substrate during the MBE growth. The thickness of the film is 4 ML. This RHEED pattern shows (2×2) streaks, and the in-plane spacing of the MnSb film in the GaAs [112] direction as estimated by the streak spacing in the RHEED pattern is similar to that of bulk ferromagnetic MnSb[0001] as in the case of MnSb on GaAs(100). Compared with the RHEED patterns of MnSb on GaAs(100), the background intensity of the RHEED patterns of MnSb on GaAs(111)*B* is considerably low, which indicates the smooth surface is obtained in MnSb(0001) on GaAs(111)*B*. In the XRD pattern, we observed GaAs(111), GaAs(222), and GaAs(333) reflection with MnSb(0002), and MnSb(0004) peaks, confirming the epitaxial orientations of MnSb(0001) on GaAs(111)*B*. Figure 2(b) shows a cross-sectional TEM image of an interface region taken from MnSb/GaAs(111)*B* along ⟨112⟩ with the thickness of the MnSb film of 300 Å. In this TEM image, clear lattice fringes are observed from both MnSb and GaAs regions, and the interface between the MnSb film and the GaAs(111)*B* substrate is very sharp indicating the formation of abrupt interface between the MnSb film and the GaAs(111)*B* substrate, with the *c* axis of MnSb perpendicular to the substrate which is consistent with the crystal orientation of MnSb (0001) on GaAs (111)*B*. At the interface dislocations are completely relaxed. RHEED, XRD, and cross-sectional TEM results again show that the MnSb film on GaAs (111)*B* is of very high quality. It is concluded that MnSb films on both GaAs(001) and (111)*B* substrates are epitaxially grown and the very abrupt crystallographic interface structure is observed, however the surface morphology of the film shows the dependence on the crystallographic orientation of the substrate surface.

B. Core-level photoemission results of MnSb films on GaAs (100)

To investigate the chemical structure in MnSb films on GaAs substrates, we have performed synchrotron radiation photoemission measurement. In order to perform a quantitative analysis, we fit photoemission spectra by using Voigt function, that is a convolution of a Lorentzian that represents the natural line shape and a Gaussian that represents broadening contribution from both instrumental factors (limited experimental resolution from the monochromator and electron analyzer) and sample related factors (phonon broadening and disorders in the sample which gives rise to contributions with slightly different binding energies), with an integrated background. Photoemission spectra of Ga 3*d*, As 3*d*, and Sb 4*d* core levels from MnSb/GaAs(100) with their fitted components for various MnSb overlayer thickness are presented in Figs. 3(a), 3(b), and 3(c), respectively.

In the case of Ga 3*d* core-level spectra, the spin-orbit separation for $J=5/2$ and 3/2 components is fixed at 0.45 eV and Lorentzian width is fixed at 0.18 eV, where both of the parameters are consistent with other studies.[19–21] The branching ratio is set at 0.67, which is almost the same as the theoretical value. In Fig. 3(a) Ga 3*d* photoemission spectra are shown with the fitting results for various MnSb overlayer thickness. Photoemission spectrum of Ga 3*d* on GaAs(100) clean surface shows two surface components (one with higher and another with lower binding energy) and one components from a bulk GaAs. In the Sb-terminated surface, one surface component with a bulk component is observed. This component corresponds to a Ga-Sb chemical bonding state. The chemical shift of this Ga-Sb component from the bulk component is -0.37 eV. The origin of this negative chemical shift in Ga-Sb bonding state is considered as due to a weaker bonding in Ga-Sb compared to Ga-As which is originated from smaller electron negativity of Sb. After the MnSb overlayer growth, one more additional component at lower binding energy is observed. This additional component corresponds to a Ga-Mn chemical bonding state because a bulk MnGa is metallic and a core hole of Ga in Ga-Mn bonding are considered to be more screened as compared with a core hole in Ga-Sb bonding. The chemical shift of the Ga-Mn component from the bulk component is estimated to -0.81 eV. Above the thickness of MnSb overlayer of 23 Å, the intensity of Ga 3*d* peak remains unchanged indicating the Ga segregation to the surface of the film. This behavior of Ga 3*d* peak intensity is discussed later. With increasing the MnSb overlayer thickness, an enhancement of Ga-Sb component is observed in the first stage. However, while the overlayer thickness increases from 23 to 72 Å, a Ga-Mn component drastically increases and in the thick MnSb film (237 Å), the intensities of both Ga-Sb and Ga-Mn are almost equal.

In the case of As 3*d* spectra, the spin-orbit separation, Lorentzian width, and the branching ratio are set to be 0.68, 0.14, and 0.66 eV, respectively. These values are consistent with other studies on the photoemission spectra of GaAs.[19–21] As 3*d* photoemission spectra are shown with the fitting results for various MnSb overlayer thickness in Fig. 3(b). As 3*d* photoemission spectrum of a clean (100) surface shows two surface components (located at a higher and a lower binding energy) with a bulk As-Ga component. In the Sb-terminated surface, the similar features are observed. After the MnSb overlayer growth, one additional component at the lower binding energy is observed. This additional component shows the chemical shift (from the bulk component) of about -0.52 eV and corresponds to an As-Mn chemical bonding state because a bulk MnAs shows metallic characteristics and the core-hole screening is quite large compared to that in semiconducting GaAs. The intensity of the As 3*d* peak decreases gradually with increasing the thickness of MnSb film and the As 3*d* peak could not be observed at the thickness of 273 Å. The behavior of the As 3*d* peak intensity is also discussed later.

In the case of Sb 4*d*, the fitting procedure is rather complicated because the Sb peaks from Sb-Mn bonding state show the asymmetric line-shapes due to the metallic characteristics in MnSb and the Sb 4*d* peaks also show the branching ratio anisotropy. The spin-orbit separation and Lorenzian width are set to be 1.24 and 0.18 eV, respectively, and are consistent with other photoemission studies.[21,22] The branching ratio is set at 0.7 for all the component. For a component from Sb-Mn chemical bonding state, we use Doniach-Sunjic asymmetric line shapes.[23] For Sb-Mn components the singularity is set at 0.16 and for the other components the singularity is set at zero. Sb 4*d* photoemission spectra are shown with the fitting results for various MnSb overlayer thickness in Fig. 3(c). Two components were found in the photoemission spectra of Sb-terminated GaAs(100). The component at the lower binding energy corresponds to a Sb-Ga chemical bonding state and the components at the higher binding energy corresponds to a Sb-As chemical bonding state. The chemical shift of Sb-As component from the Sb-Ga component is 0.79 eV. With increasing the MnSb overlayer thickness, asymmetric peaks are observed in the photoemission spectra. This additional component corresponds to a Sb-Mn chemical bonding from MnSb overlayers with the chemical shift from the Sb-Ga of -0.55 eV. At the first stage the

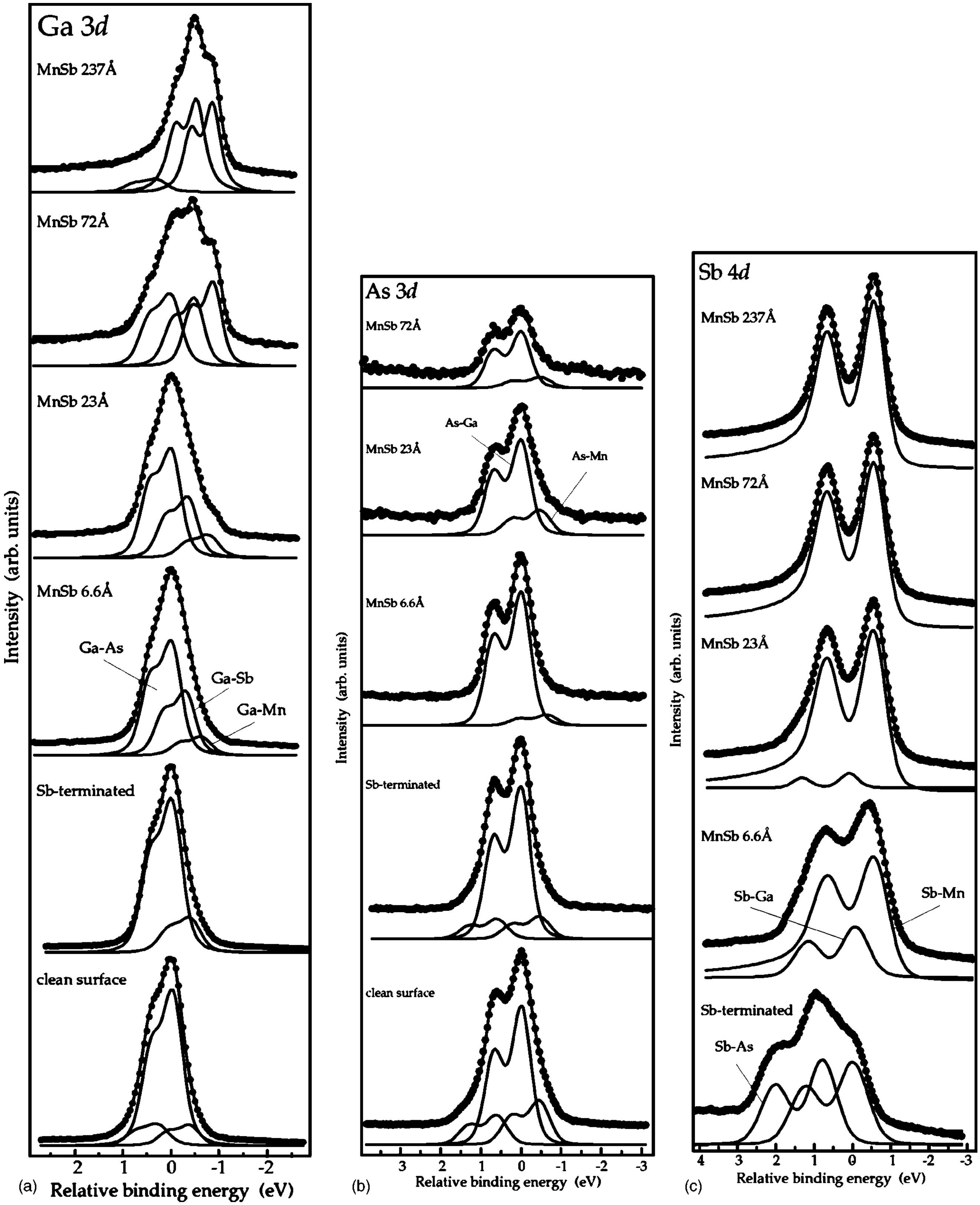

FIG. 3. Photoemission spectra of MnSb/GaAs(100) taken at $h\nu = 118.9$ eV for various MnSb thicknesses (as indicated) from (a) Ga 3d, (b) As 3d, and (c) Sb 4d core levels. The raw photoemission data are shown by solid circles and the deconvoluted components are shown by solid lines below the raw data. Furthermore, fitted reslults obtained from the sum of these deconvoluted components and an integrated background are shown by solid lines at the same level as the raw photoemission data. The assignments of each deconvoluted component are also presented and are discussed in the text.

Sb-Ga bonding state remains in the MnSb overlayers, while above 72 Å the Sb-Ga bonding state could not be observed and Sb 4*d* peaks contains only single component of the Sb-Mn bonding state.

C. Core-level photoemission results of MnSb films on GaAs (111)B

Similar behavior is also observed in the epitaxial growth of MnSb on GaAs(111)*B* substrates. Figures 4(a), 4(b), and 4(c) show the photoemission spectra of Ga 3*d*, As 3*d*, and Sb 4*d* core levels from MnSb/GaAs(111)*B* with their fitted components for various MnSb overlayer thickness, respectively. In the fitting procedures for Ga 3*d*, As 3*d*, and Sb 4*d*, the same parameters of spin-orbit splitting, branching ratio, and Lorentzian width are used, namely, 0.45, 0.67, and 0.18 eV for Ga 3*d*, 0.68, 0.66, and 0.14 eV for As 3*d*, and 1.24, 0.70, and 0.18 eV for Sb 4*d*, respectively. However for the Sb-Mn component of Sb 4*d*, we used the singularity of 0.115 which is slightly smaller than that is used for MnSb/GaAs(100).

Figure 4(a) shows Ga 3*d* photoemission spectra with the fitted components for various MnSb overlayer thickness. Photoemission spectrum of GaAs(111)*B* clean surface shows one surface components at the lower binding energy and one components from a bulk GaAs. In the Sb-terminated surface, the similar features are observed, however, a component at the lower binding energy in the spectra is thought to correspond to the Ga-Sb chemical bonding state, since the chemical shift of this Ga-Sb component from the bulk component is -0.44 eV which is similar to the chemical shift of Ga-Sb observed in MnSb/GaAs(100). After the MnSb overlayer growth, the Ga-Sb component is rapidly enhanced. Above the MnSb overlayer thickness of 25 Å, one more additional component at the lower binding energy is observed, corresponding to the Ga-Mn chemical bonding state. The chemical shift of the Ga-Mn component from the bulk component is estimated to -0.94 eV. Above the thickness of MnSb overlayer of 9.3 Å, the intensity of Ga 3*d* peak remains unchanged indicating the Ga segregation to the surface of the film. This behavior of Ga 3*d* peak intensity is also discussed later. Different from the growth of MnSb on GaAs(100), the main contribution of the Ga component in MnSb on GaAs(111)*B* is the Ga-Sb chemical bonding state. In the thick MnSb films (75 and 240 Å), the Ga-Sb bonding state is dominant in the Ga 3*d* spectra.

In the case of As 3*d*, the situation is very similar to that observed in MnSb/GaAs(100). As 3*d* photoemission spectra are shown with the fitting results for various MnSb overlayer thickness in Fig. 4(b). The As 3*d* photoemission spectrum of a clean (111)*B* surface also shows two surface components (located at a higher and a lower binding energy) with a bulk As-Ga component. In the Sb-terminated surface, the similar features are observed. After the MnSb overlayer growth, one additional component also appears at the lower binding energy. This additional component shows the chemical shift of about -0.47 eV from the bulk component and corresponds to the As-Mn chemical bonding state. The intensity of As 3*d* peak decreases gradually with increasing the thickness of MnSb film and the As 3*d* peak could not be observed at the thickness of 75 Å. The behavior of As 3*d* intensity is also discussed later.

The behavior of Sb 4*d* is also very similar to that in MnSb/GaAs(100). Sb 4*d* photoemission spectra are shown with the fitting results for various MnSb overlayer thickness in Fig. 4(c). Two components were also found in the photoemission spectra of Sb terminated GaAs(111)*B*. The component at the lower binding energy corresponds to a Sb-Ga chemical bonding state and the components at the higher binding energy corresponds to a Sb-As chemical bonding state with the chemical shift of Sb-As component from the Sb-Ga component of 0.43 eV. The smaller chemical shift may be originated from the different surface structures in different GaAs substrates. With increasing the MnSb overlayer thickness, asymmetric peaks are observed in the photoemission spectra. This additional component corresponds to the Sb-Mn chemical bonding state with the chemical shift from the Sb-Ga of -0.50 eV. At the first stage the Sb-Ga bonding state remains in the MnSb overlayers, while above 75 Å the Sb-Ga bonding state could not be observed and Sb 4*d* peaks contain only single component of the Sb-Mn bonding state.

D. Ga segregation in MnSb epitaxial growth on GaAs (100) and (111)*B*

Considering the above photoemission results, we discuss the Ga-segregation in MnSb epitaxial growth on both GaAs(100) and (111)*B* substrates. To clarify the Ga-segregation behavior, we must discriminate between Ga-segregation at the MnSb surface and Ga interdiffusion into the MnSb film. To eliminate the possibility of Ga-interdiffusion into the MnSb film, we plotted the normalized absolute Ga 3*d* and As 3*d* photoemission intensities as a function of MnSb overlayer thickness on the GaAs(100) substrate [Fig. 5(a)] and the GaAs(111)*B* substrate [Fig. 5(b)]. To determine the absolute photoemission intensity, the sum of areas in all photoemission components was normalized by background intensity. The absolute intensity in each overlayer thickness is normalized by that for the Sb-terminated surface. In the case of MnSb growth on GaAs(100) which is shown in Fig. 5(a), As 3*d* decreases gradually, whereas Ga 3*d* remains constant above 23 Å. The decrease of the As 3*d* intensity as a function of overlayer thickness is relatively slow, indicating a little As interdiffusion into MnSb occurs at the initial growth until the overlayer thickness up to 72 Å. The thickness of the segregated Ga layer estimated by the absolute photoemission peak intensity ratio of Ga 3*d* and Sb 4*d* and the escape depth of photoelectrons is about 0.5 ML. In the case of MnSb growth on GaAs(111)*B* which is shown in Fig. 5(b), As 3*d* rapidly decreases, whereas Ga 3*d* remains constant above 9.3 Å. The rapid decrease of As 3*d* intensity indicates a less As interdiffusion into MnSb films in the initial growth of MnSb on GaAs(111)*B*. The absolute intensity of Ga 3*d* in MnSb/GaAs(111)*B* is about twice of that in MnSb/GaAs(111)*B* and the thickness of the segre-

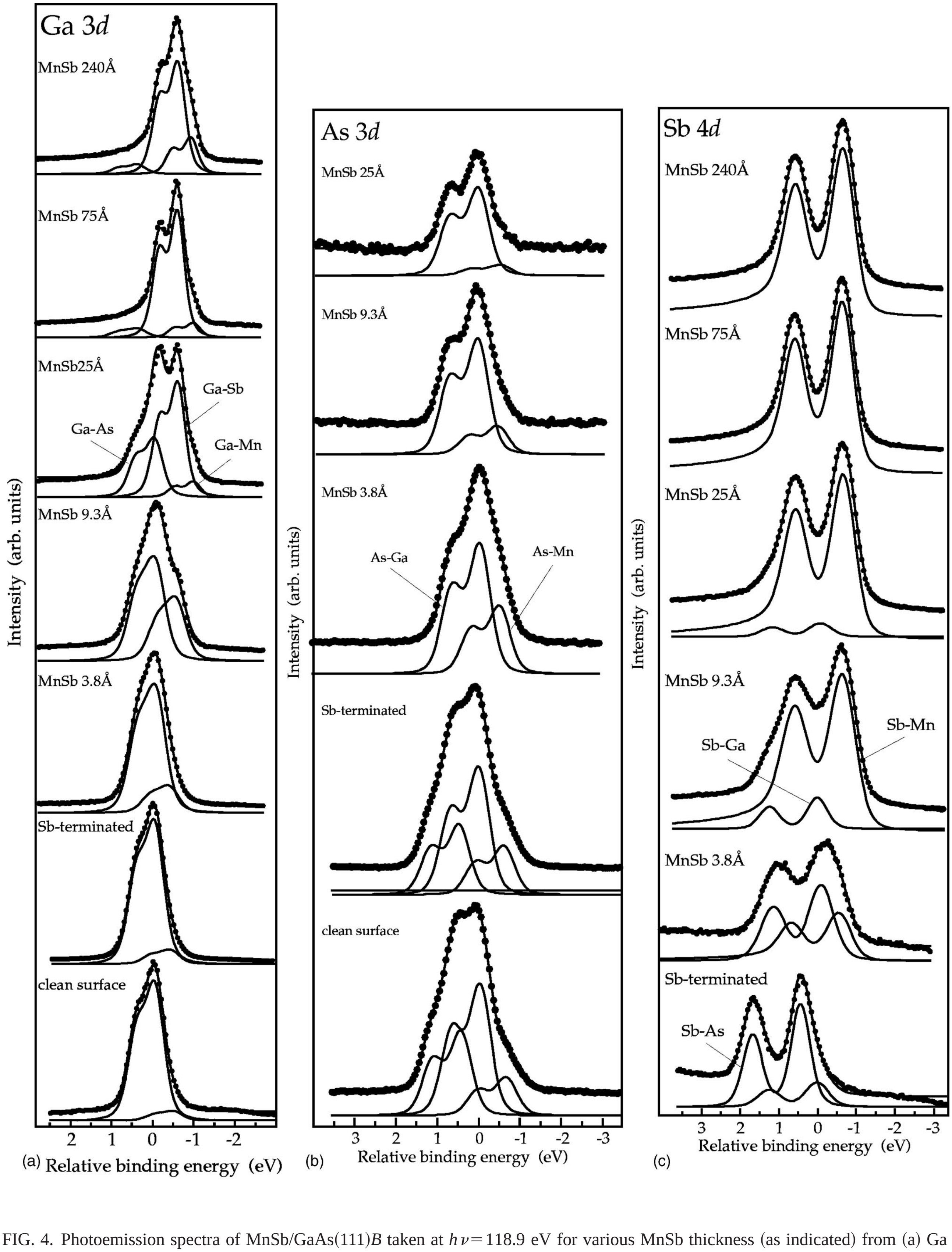

FIG. 4. Photoemission spectra of MnSb/GaAs(111)*B* taken at $h\nu = 118.9$ eV for various MnSb thickness (as indicated) from (a) Ga 3*d*, (b) As 3*d*, and (c) Sb 4*d* core levels. The raw photoemission data are shown by solid circles and the deconvoluted components are shown by solid lines below the raw data. Furthermore, fitted reslults obtained from the sum of these deconvoluted components and an integrated background are shown by solid lines at the same level as the raw photoemission data. The assignments of each deconvoluted component are also presented and are discussed in the text.

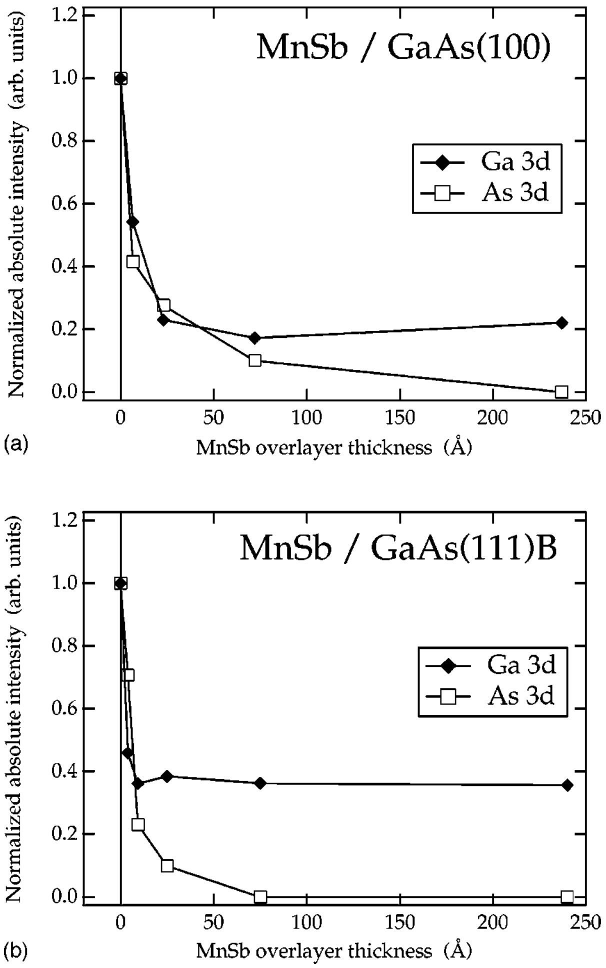

FIG. 5. Normalized absolute Ga 3d and As 3d photoemission intensities as a function of MnSb overlayer thickness on (a) the GaAs(100) substrate and (b) the GaAs(111)B substrate. The intensity of Ga 3d is shown in filled diamonds and the intensity of As 3d is shown in open squares. The MnSb overlayer thickness of zero indicates the Sb-terminated surface and the absolute intensity in each thickness is normalized by that of the Sb-terminated surface.

gated Ga layer is about 1.0 ML. These results strongly suggest that the Ga segregation occurs at the surface. The thickness of the segregated Ga layer depends on the crystallographic orientation of the substrate surface. In the case of MnSb(0001)/GaAs(111)B, a top surface layer of MnSb(0001) surface is a Sb layer and just 1 ML of segregated Ga layer is bonded to the Sb layer, which is confirmed by Ga 3d photoemission in Fig. 4(a). A hexagonal lattice of MnSb is fitted for GaAs(111)B, which has sixfold symmetry. Whereas in the case of MnSb(1101)/GaAs(100), both Mn and Sb atoms are existed on the top surface layer, and segregated Ga layer is bonded to both Mn and Sb as observed by Ga 3d photoemission in Fig. 3(a). The surface structures of MnSb surfaces, however, could not be revealed yet and detailed structure of segregated Ga layer is not understood. The surface of MnSb film on GaAs(111)B observed by RHEED is very smooth compared to that on GaAs(100), furthermore in the TEM observation, the dislocations are relaxed near the interface in both films. As the reason for the difference in surface roughness, it is suggested that the segregated Ga layer acts as a surfactant and decrease the surface energy and contribute to the layer-by-layer growth. The thickness of the surfactant layer is thought to have an influence on the surface morphology of the MnSb film.

IV. CONCLUSION

In conclusion, we have investigated the initial growth mechanisms and found the Ga segregation during the epitaxial growth of MnSb thin films on GaAs (100) and (111)B substrates by quantitative analysis of Ga, As, and Sb core levels. The Ga 3d photoemission peak intensity remains unchanged with increasing MnSb overlayer thickness. However the As 3d peak intensity decreases gradually. These results suggest that the Ga atoms are segregated at the surface during the growth. The thickness of the Ga segregation layers is estimated to 0.5 monolayer (ML) for MnSb on GaAs(100) and 1.0 ML for MnSb on GaAs(111)B. The thickness of the segregated Ga layer is thought to have an influence on the surface morphology of the MnSb film.

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