Growth of lattice-matched GaInAsP grown on vicinal GaAs(001) substrates within the miscibility gap for solar cells

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Abstract

The growth of quaternary $\text{Ga}_{0.68}\text{In}_{0.32}\text{As}_{0.35}\text{P}_{0.65}$ by metal-organic vapor phase epitaxy is very sensitive to growth conditions because the composition is within a miscibility gap. In this investigation, we fabricated 1 μm -thick lattice-matched GaInAsP films grown on GaAs(001) for application to solar cells. The growth temperature and substrate miscut are varied in order to characterize the effect of the surface diffusion of adatoms on the material quality of alloys. Transmission electron microscopy and two-dimensional *in-situ* multi-beam optical stress determine that growth temperatures of 650 °C and below enhance the formation of the CuPt_B atomic ordering and suppress material decomposition, which is found to occur at the growth surface. The root-mean-square (RMS) roughness is reduced

from 33.6 nm for 750 °C to 1.62 nm for 650 °C, determined by atomic force microscopy. Initial investigations show that the RMS roughness can be further reduced using increased miscut angle, and substrates miscut toward (111)A, leading to an RMS roughness of 0.56 nm for the sample grown at 600 °C on GaAs miscut 6° toward (111)A. Using these conditions, we fabricate an inverted hetero-junction 1.62-eV $\text{Ga}_{0.68}\text{In}_{0.32}\text{As}_{0.35}\text{P}_{0.65}$ solar cell without an anti-reflection coating with a short-circuit current density, open-circuit voltage, fill factor, and efficiency of 12.23 mA/cm², 1.12 V, 86.18%, and 11.80%, respectively.

Keywords

A3. Metalorganic vapor phase epitaxy, B1. Alloys, B2. Semiconducting quaternary alloys, B3. Solar cells

1. Introduction

Quaternary $\text{Ga}_{1-x}\text{In}_x\text{As}_{1-y}\text{P}_y$ alloys are of significant interest to researchers for the development of optoelectronic devices, such as lasers [1], photodetectors [2], and solar cells [3]. GaInAsP lattice-matched to InP [4,5] is well studied in the infrared spectral range, in which the bandgap can be tuned between 0.74 eV ($\text{Ga}_{0.47}\text{In}_{0.53}\text{As}$) and 1.35 eV (InP). On the other hand, GaInAsP lattice-matched to GaAs [6,7] can offer an alternative to well-established AlGaAs systems, because it covers the bandgap range between 1.42 eV (GaAs) and 1.9 eV ($\text{Ga}_{0.49}\text{In}_{0.51}\text{P}$) while maintaining lattice-matching to GaAs. Thus, this material could provide aluminum (Al)-free active layers in optoelectronic devices with fewer deep-level defects [8,9]. In solar-cell applications, cells with a 1.7-eV bandgap are in especially high demand for III-V/Si dual-junction solar cells [10] as well as for five- or more junction solar cells with conversion efficiency (η) greater than 50% [11].

GaInAsP single-junction solar cells grown by gas-source molecular beam epitaxy (MBE) [12] and metalorganic vapor phase epitaxy (MOVPE) [13,14] and with bandgaps of 1.5 eV and 1.7 eV, have previously been created. Because the miscibility gap exists in a wide composition range of GaInAsP [15], GaInAsP alloys are often susceptible to compositional fluctuations owing to spinodal-like decomposition [5,6,16-18]. In addition, atomic ordering is well known to occur for ternary GaInP, where CuPt-type ordering on the group III sublattice occurs on $\{111\}$ B planes [19,20]. Knauer et al. revealed that such atomic ordering also occurs in quaternary GaInAsP grown on GaAs substrates by MOVPE

[21,22], though the ordering could occur on both the group III and group V atomic sublattices. The degree of both atomic ordering and compositional fluctuation strongly depends on growth parameters, such as temperature, growth rate, V/III flux ratio, doping, and substrate miscut. However, detailed studies on the correlation between the CuPt ordering and the phase separation on lattice-matched $\text{Ga}_{1-x}\text{In}_x\text{As}_{1-y}\text{P}_y$ grown on GaAs with $0.5 < y < 0.8$, which is within the predicted immiscibility region for conventional MOVPE growth [15], are scarce. In this paper, we investigate the effects of growth temperature and substrate miscut on $\text{Ga}_{0.68}\text{In}_{0.32}\text{As}_{0.35}\text{P}_{0.65}$ films grown by MOVPE. The aim is to develop high-quality, Al-free, quaternary, wide-gap solar cells under conditions where the growth would be significantly affected by the miscibility gap.

2. Experiments

Growth of GaInAsP was carried out by MOVPE at atmospheric pressure in a custom-built vertical reactor. Phosphine (PH_3), arsine (AsH_3), trimethylgallium, triethylgallium, and trimethylindium were used as III-V precursors, while hydrogen selenide, disilane, diethylzinc, and tetrachloromethane were used as dopant gases.

One challenging aspect of mixed As-P epitaxy in MOVPE is the difference in the adsorption efficiency of As and P at a given temperature; P is much more difficult to adsorb onto the surface than As. This makes it difficult to evaluate the effect of growth temperature on the material quality of GaInAsP films with identical lattice-matched conditions.

Therefore, we firstly investigated the relationship between the P content, y , in the solid and the gas flux ratio $R = f_{\text{PH}_3}/(f_{\text{PH}_3}+f_{\text{AsH}_3})$, where f_X is the flux of chemical species X, in $\text{Ga}_{0.68}\text{In}_{0.32}\text{As}_{1-y}\text{P}_y$ and $\text{GaAs}_{1-y}\text{P}_y$ alloys. We fabricated 1 μm -thick intrinsic thin films on p-GaAs(001) substrates miscut 2° towards (111)B (denoted as 2°B) at various temperatures between 600 $^\circ\text{C}$ and 750 $^\circ\text{C}$. The growth rate and the V/III flux ratio were fixed at 4.5 $\mu\text{m}/\text{h}$ and 331. The composition of the epilayer was determined from symmetric (004) and asymmetric (224) Bragg reflections in high-resolution x -ray diffraction (XRD) using Panalytical X-Pert Pro or Bede D1 diffractometers. We **roughly** assumed that the compositional ratio of group III atoms did not change for the temperature range used, **because the degree of temperature dependence on the adsorption efficiency for group V atoms is much greater than those for group III atoms [23]**. Figure 1 shows a plot of y as a function of R obtained for samples grown at various temperatures. Solid lines are fitting curves in accordance with the Langmuir adsorption relationship [24, 25], which is given by

$$\frac{1}{R} = a + \frac{b}{y} \quad (1)$$

where a and b are constants independent of R and y . A similar trend has been observed for As-P epitaxy using gas-source precursors, such as gas-source MBE [7] and MOVPE [23-26], though the constants significantly depend on the growth method. The fact that y increases as temperature increases at any given R indicates the enhanced adsorption efficiency of P. Because the plot of y as a function of R becomes very steep for large R and for low temperature, we needed to very precisely control R to achieve lattice-matched

$\text{Ga}_{0.68}\text{In}_{0.32}\text{As}_{0.35}\text{P}_{0.65}$ represented as the dashed line in Fig. 1. The values of R used in this range were 0.992 for 600 °C, 0.986 for 650 °C, 0.962 for 700 °C, and 0.933 for 750 °C.

We fabricated 1 μm -thick lattice-matched intrinsic $\text{Ga}_{0.68}\text{In}_{0.32}\text{As}_{0.35}\text{P}_{0.65}$ films grown at temperatures between 600 °C and 750 °C using the above-mentioned R parameters. In addition to the 2°B substrates, p-GaAs(001) substrates miscut 6° towards (111)B (6°B) and 3° or 6° towards (111)A (3°A and 6°A) were used. We confirmed by XRD that the lattice-mismatch ($\Delta a/a$) between the GaInAsP layer and the GaAs substrate was within 0.1% for all samples (data not shown). Real-time two-dimensional (2D) curvature and reflectivity of the wafer were measured *in-situ* using a two-dimensional k-Space Associates multi-beam optical stress (MOS) sensor with the optical axis of the tool normal to the substrate growth surface, described previously [27]. The axes of the 3×3 laser-spot array were set along [110] and [-110] by eye. Fig. 2 shows the laser output power and the 2D curvature obtained in MOS measurements as functions of thickness during growth for the 1 μm -thick GaInAsP grown at (a) 600 °C and (d) 750 °C. Because the laser output power changes with feedback to maintain a constant light intensity at the detector during growth, the plot qualitatively represents the real-time reflectivity at the cumulative thickness. An almost constant output was observed during the growth of the GaInAsP layers for Fig. 2(a) except for Fabry–Perot oscillations associated with the interference of the reflection at the growing surface and that at the hetero-interfaces of epi-layers [28]. An obvious increase in the output power was detected at 0.6 μm for Fig. 2(b) (denoted as arrows), indicating that

the degradation of the surface morphology began at a certain thickness. In addition, the slope of curvature along each direction with respect to the thickness represents the stress applied in each direction of the film [29]. Although a small variation in the curvature was observed in the entire region for Fig. 2(a) due to an imperfect lattice-match condition, the abrupt change in the curvature for Fig. 2(b) was observed at 0.6 μm due to the drastic change in the stress condition as well.

Morphology and structure were examined by Veeco D3100 tapping mode atomic force microscopy (AFM) and transmission electron microscopy (TEM). Orthogonal $\langle 110 \rangle$ cross-section TEM samples were prepared using standard mechanical polishing techniques followed by final thinning to electron transparency via low energy (4 kV with final polishing at 1 kV) Ar^+ ion milling, with the sample stage rotated and cooled by liquid nitrogen to minimize any possible changes in microstructure. The thinned samples were then examined in an FEI Tecnai G²30 SuperTwin high-resolution TEM operated at 300 kV. Photoluminescence (PL) measurements were performed at temperatures between 10 K and 294 K using a silicon charge coupled device array detector. A 442-nm laser with a spot diameter of 250 μm , at an intensity of 1 mW, was used as the excitation light source.

A lattice-matched $\text{Ga}_{0.68}\text{In}_{0.32}\text{As}_{0.35}\text{P}_{0.65}$ inverted heterojunction solar cell was grown at 600 °C on the 6°A substrate as shown in Fig. 3. The inverted solar cell structures, described in more detail later, were processed on the Si supporting substrate as described previously [30]. The cell size was 0.1 cm^2 . No anti-reflective coating was applied. The solar cell was

characterized by external quantum efficiency (EQE) measurements using a custom-built tool under chopped, monochromatic light. The light current–voltage (I – V) characteristic was measured using an adjustable 1-sun simulator under air mass 1.5 direct (AM1.5D) illumination.

3. Results and discussions

3.1 Growth temperature dependence

Phase separation is thermodynamically unfavorable at high temperature, but can also be kinetically limited at low temperature, making growth temperature a key parameter in GaInAsP material quality. The effect of temperature on phase separation was studied by varying the GaInAsP growth temperature between 600 °C and 750 °C. Roughness can correlate with phase separation [18,31], and so was analyzed using AFM and *in-situ* reflectance. TEM was used to investigate the location and magnitude of phase separation, and atomic ordering was investigated using transmission electron diffraction (TED) and 2D wafer curvature [17,32].

Fig. 4 plots the root-mean-square (RMS) roughness measured for the 1 μ m-thick lattice-matched $\text{Ga}_{0.68}\text{In}_{0.32}\text{As}_{0.35}\text{P}_{0.65}$ samples grown on 2°B substrates at between 600 °C and 750 °C by AFM. RMS was determined from 2 μ m $\times$ 2 μ m scans. RMS roughness becomes smaller as the growth temperature decreases and reaches a minimum value of 1.62 nm at 650 °C. On the other hand, a large RMS roughness of 33.6 nm was obtained for the

sample grown at 750°C.

Fig. 5 shows dark field [110] and [-110] cross-sectional TEM images for the sample grown at 600 °C and 750 °C with arrows indicating the direction of the diffraction vectors $g = 2\cdot 20$ and 220 . Alternating bright/dark contrast regions arising from microscopic strains present in the material along directions roughly parallel to the layer surface were observed in both samples. For the sample grown at 600 °C, the periodic strain contrast is more pronounced and on a finer scale along the [110] direction, as seen in the [-110] cross-section image of Fig. 5(b). Such strain contrast features are well known to be associated with a [110] lateral composition modulation due to the spinodal decomposition in the alloy, which have previously been reported in AlInAs [33, 34], AlInP [35], GaInP [36], and GaInAsP alloys [16, 17]. Strong strain contrast was observed in both [110] and [-110] cross-sections for the sample grown at 750 °C possibly indicating an enhanced phase separation at high temperature and kinetic limitation at low temperature. Fig. 5(a) and (b) show that a homogeneous film was obtained except for the lateral composition modulation in the sample grown at 600 °C, while a highly defective region appeared at approximately 0.5 μm from the GaAs buffer layer in the sample grown at 750 °C, Fig.5 (c) and (d).

Table I summarizes the decomposition thickness determined by the drastic change in the reflectivity as shown in Fig. 2(b). The decomposition thickness for 600 °C and 650 °C are not shown, since the abrupt change did not occur during 1 μm -thick GaInAsP growth. Conversely, this phenomenon occurs earlier in the growth for the higher growth

temperature. The decomposition thickness of 0.6 μm obtained by MOS for the 750 $^{\circ}\text{C}$ is in good agreement with TEM results. Thus, the large RMS obtained for high temperature growth was mainly caused by the occurrence of the defective region during the growth. In addition, the combination of MOS and TEM provides information about both the time and thickness during growth that the decomposition occurred, as well as the amount of material affected by the decomposition. The agreement of the decomposition thickness between MOS and TEM suggests that the defective region is related not to a bulk phase separation, but due to severe decomposition at the growth surface, which is consistent with previous literature [37].

Atomic ordering was evaluated using TED measured for a selected area of the GaInAsP layers as shown in the inset of Fig. 5. The large spots represent the fundamental reflections and smaller extra spots and streaks represent the superlattice reflections. Note that there is clearly a single CuPt_B variant present for 600 $^{\circ}\text{C}$, because the TED image of the orthogonal $[-110]$ cross-section does not show any superlattice reflections, and there is only one set of superlattice reflections in the $[110]$ cross-section pattern. In addition, there are no superlattice reflections for the 750 $^{\circ}\text{C}$ sample, indicating no ordering present at high growth temperature. Atomic ordering can also be revealed by anisotropic strain applied in the wafer between the $[110]$ and $[-110]$ directions in MOS measurements [32]. Table I also summarizes the stress along $[110]$ ($\sigma_{[110]}$) and $[-110]$ ($\sigma_{[-110]}$) direction, which is determined at the initial portion of the growth by Stoney's equation [29]. The strain

anisotropy ($\Delta\sigma$) was determined by $\Delta\sigma = \sigma_{[110]} - \sigma_{[-110]}$. The $\Delta\sigma$ for 750 °C is negligible, whereas the largest $\Delta\sigma$ of 90 MPa was obtained for 600 °C. Therefore, the degree of the CuPt_B ordering is significantly enhanced at 600 °C and 650 °C growth temperatures.

These results indicate the decomposition is correlated with the degree of atomic ordering. Konaka et al. [6] and Diéguez et al. [38] argued that the formation of (2×4) surface reconstruction causes CuPt ordering and the CuPt ordering forming at the surface suppresses the generation of phase separation especially at lower temperature in GaInAsP and GaInP alloys. This is consistent with the results reported for the samples used in this study. At higher temperature, the degree of CuPt ordering becomes small due to the disruption of the surface reconstruction due to longer adatom diffusion, resulting in an enhancement of the phase separation [6]. Furthermore, the phase separation leads to the creation of GaP- and InAs- rich regions in GaInAsP alloys, leading to nonuniform stress in the bulk and at the growing surface. The defective region in the high temperature samples could potentially be a result of the relaxation of locally accumulated stress followed by a change in the growth composition.

3.2 Substrate miscut dependence

The above-mentioned results suggest the diffusion length of adatoms as well as the atomic ordering at the growing surface has an important role to control the material quality of GaInAsP alloys. The surface miscut can be related to the diffusion length of adatoms,

since adatoms are preferentially incorporated at kink and step sites on the (2×4) reconstructed surface [39]. Thus, we characterized GaInAsP films grown on the different substrate miscut of 2°B, 6°B, 3°A, and 6°A. In this study, we chose the growth temperature of 600 °C in order to ensure the growth without any decomposition arisen from the growth temperature, because lower temperature prevented the occurrence of decomposition during the growth. However, further work optimizing the temperature is ongoing. Fig. 6 shows AFM images measured for samples grown on each substrate. Note that the island structure associated with pronounced phase separation [18, 31] becomes significantly reduced in size by both an increase in miscut angle and the use of substrates miscut toward (111)A, leading to the improvement of RMS roughness. The smallest RMS roughness of 0.66 nm was obtained for the sample grown on the 6°A substrate. Asai reported that in MOVPE growth, the fastest growth direction was [110], and the slowest one was [-110] [40]. This is because the diffusion length of adatoms at the growth surface is longer for [110] than that for [-110] because of the (2×4) surface reconstruction. The A-type miscut forms steps perpendicular to the [110] direction, resulting in a decrease of surface diffusion along [110]. Furthermore, the increase in the miscut angle generally increases the step density leading to further decrease in the surface diffusion.

Temperature-dependent PL has previously been used to characterize phase separation in GaInAsP [16]. Here, we compare in Fig. 7 the PL spectra measured at various temperatures between 12 K and 294 K for the GaInAsP films grown at 600 °C on (a) 2°B

and (b) 6°A substrates. Single PL emission from the GaInAsP was observed in Fig. 7(b) over the entire temperature range, while the PL emission at around 850 nm is originated from the emission in a GaAs bulk. An odd PL peak appears at 820 nm at 294K in Fig. 7(b) with unknown origins. The emission is much below the EQE cutoff wavelength of 760 nm discussed in section 3.3, and so is likely not originated from the the GaInAsP bulk, but possibly due to a localized state generated at the heterointerface and/or surface. Conversely, anomalous emission behavior was clearly observed in Fig. 7(a). Fig. 5(c) plots the PL peak energy as a function of the temperature for each sample. The solid line shows the fitted curve using Varshni relationship [41]. PL emission in the longer wavelength region around 760 nm was observed up to 150 K, followed by the PL emission in the shorter wavelength region around 740 nm. Note that the PL emission wavelength of 760 nm taken at 294 K in Fig. 7(a) is almost identical with that in Fig. 7(b), indicating the band-to-band transition from the nominal compositional bulk region. Using the expression $E_g = E_{PL} - kT/2$, where k and T are the Boltzmann constant and the temperature, we estimated the bandgap of the alloy to be 1.62 eV at room temperature. The longer wavelength PL emission observed up to 150 K in Fig. 7 (a) may be explained by localized states caused by the compositional fluctuation [16] and/or atomic ordering [42]. In either case, we conclude that the optical properties of the alloys grown on A-type miscut substrates are superior to those grown on B-type miscut substrates because the emission closely follows the expected Varshni relationship.

3.3 Cell performance

To gauge the GaInAsP material quality for solar cells, an inverted n-on-p solar cell was grown and processed using 600 °C growth temperature and on 6°A. The structure was similar to common front-junction inverted GaInP solar cells, described previously [30], except that the GaInP base was replaced with 1.5-um thick Zn-doped ($2 \times 10^{17} \text{ cm}^{-3}$) GaInAsP, and the back surface field was Zn-doped GaInP. Fig. 8 shows the EQE spectrum for the inverted hetero-junction $\text{Ga}_{0.68}\text{In}_{0.32}\text{As}_{0.35}\text{P}_{0.65}$ solar cell. Fringe peaks were observed from 650nm due to the GaInP emitter layer and the gold mirror causing Fabry-Perot cavity. We were able to determine the absorption edge to be 760 nm (1.62 eV) [43]. Because the bandgap calculated from EQE was identical with the bandgap measured from PL at 294 K as shown in Fig. 7(c), phase separation in GaInAsP layers grown on 6°A does not affect significantly the cutoff wavelength of EQE spectrum. However, EQE gradually decreases above 600 nm due to inadequate absorption thickness and short minority-carrier diffusion length. Figure 9 shows the I - V curves measured for the cell under AM1.5D (1 sun). The short-circuit current density (J_{SC}), V_{OC} , fill factor (FF), and efficiency (η) were 12.23 mA/cm^2 , 1.12 V, 86.18%, and 11.80%, respectively. The corresponding bandgap-voltage offset ($W_{\text{oc}} = E_g/q - V_{\text{oc}}$), which is as an indication of material quality for solar cells with different bandgaps [44], was determined to be 0.50 V. The W_{OC} value obtained in this work are lower than those obtained for previously reported GaInAsP cells with different 1.55 eV and 1.70 eV bandgap as shown in the inset of the Fig. 9 [14], which is likely a result of the

growth temperature and substrate miscut optimization. However, we found that the obtained W_{OC} was slightly larger than that of AlGaAs cells reported in Ref. 9. Further reducing both the non-radiative recombination and the phase separation can lower the W_{OC} .

4. Conclusions

We carefully investigated effects of growth temperature and substrate miscut on the material quality of $\text{Ga}_{0.68}\text{In}_{0.32}\text{As}_{0.32}\text{P}_{0.65}$ films grown on GaAs(001) substrates for application to solar cells, since the growth is significantly affected by the miscibility gap in MOVPE. The results suggest that the phase separation was effectively suppressed using low growth temperature and an increase in miscut angle, with substrates miscut toward (111)A preferable over (111)B. Furthermore, we found CuPt_B ordering present in the alloy for the growth at 650 °C and 600 °C. The presence of CuPt_B ordering correlates with the suppression of a surface-driven phase separation, which suggests that the surface reconstruction during growth plays a factor in phase separation. This correlation between the phase separation and the atomic ordering agree with previous works on other GaInAsP alloy compositions. Initial inverted hetero-junction GaInAsP solar cells exhibit a relatively low W_{OC} value of 0.50 V after temperature and substrate optimization, although the growth must be further optimized to improve the cell performance for operation in a multijunction device. We have made progress on the GaInAsP solar cell and it is promising, but AlGaAs is still a little better at this point. This demonstration represents an important step toward

the development of highly efficient, monolithic MJSCs with five or more junctions as well as III-V/Si dual junction cells.

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Figure captions

- Fig. 1 P content, y , in the $\text{Ga}_{0.68}\text{In}_{0.32}\text{As}_{1-y}\text{P}_y$ and $\text{GaAs}_{1-y}\text{P}_y$ thin films grown at 600 °C, 650 °C, 700 °C, and 750 °C as a function of the flux ratio R .
- Fig. 2 The laser output power and the 2D curvature for 1 μm -thick GaInAsP layers grown at (a) 600 °C and (b) 750 °C.
- Fig. 3 Schematic structure of inverted heterojunction GaInAsP solar cells.
- Fig. 4 RMS roughness measured with AFM as a function of the growth temperature for GaInAsP films grown on 2°B.
- Fig. 5 {220} dark field [110] and [-110] cross-sectional TEM images for the GaInAsP grown at 600 °C ((a) and (b)) and 750 °C ((c) and (d)). The inset shows the TED measured for a selected area of the GaInAsP layer.
- Fig. 6 AFM images measured for GaInAsP films grown at 600 °C on (a) 2°B, (b) 6°B, (c) 3°A, and (d) 6°A. Scan area is $2\mu\text{m} \times 2\mu\text{m}$.
- Fig. 7 PL spectra measured at various temperature between 12 K and 294 K for the GaInAsP films grown at 600 °C on (a) 2°B and (b) 6°A. (c) plots the PL peak energy as a function of the temperature for each sample. Solid line shows the fitted curve using Varshni relationship.
- Fig. 8 EQE spectrum measured for the inverted heterojunction GaInAsP solar cell.
- [MO069]

Fig. 9 Light I-V curve measured for the inverted heterojunction GaInAsP solar cell.

[MO069]

Table captions

Table I Summary of the decomposition thickness, the stress ($\sigma_{[110]}$ and $\sigma_{[-110]}$), and the strain anisotropy ($\Delta\sigma$) determined by MOS for GaInAsP grown at 600 °C, 650 °C, 700 °C, and 750 °C. [MN854, MN896, MN875, and MN874]

Table I

Sample ID	Temperature (°C)	Decomposition thickness (μm)	$\sigma_{[110]}$ (MPa)	$\sigma_{[-110]}$ (MPa)	$\Delta\sigma$ (MPa)
MN854	600	-	58.8	-32.8	91.6
MN896	650	-	-4.3	-82.7	78.4
MN875	700	0.78	65.8	43.6	22.2
MN874	750	0.60	26.5	18.0	8.5

Figure 1

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Figure 2

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Figure 3

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Figure 4

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Figure 5

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Figure 6

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Figure 7

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Figure 9

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