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Midgap states observed by nonlinear optical spectroscopy of metal:GaAs junctions

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Abstract

We present detailed second-order nonlinear optical spectroscopic studies of the interface electronic structure in metal:GaAs thin films and interfaces. The interface spectra from Au:GaAs, Ga-rich n-type systems exhibit two resonance features in the near infrared at 0.715 eV and 0.731 eV. A single resonance feature at 0.715 eV is observed in Au:GaAs, As-rich n-type interfaces. Similar single resonance features at 0.715 eV were observed in As:GaAs n-type samples. No resonances however were observed in oxide:GaAs and metal:GaAs p-type systems. All of the spectral features are sharp one-photon resonances. They provide compelling evidence for the existence and symmetry of atomic displacement-induced defect states just below the buried interface.

1. Introduction

The physics of buried solid/solid interfaces is of interest from both fundamental and technological viewpoints. Although new methods have been developed and used with limited success to reveal specific features about buried interfaces [1], a basic experimental problem still remains: traditional optical spectroscopies lack interface specificity and traditional surface diagnostics have a limited penetration depth. Second-order nonlinear optical spectroscopy possesses long penetration depths characteristic of most

optical methods and intrinsic interface specificity characteristic of second-order optical processes. These nonlinear optical techniques have been used recently to probe buried interfacial features associated with new bonding [2], band profiles [3], and strain [4]. The experiments have helped us to understand better the microscopic role played by interfacial excitations in affecting nonlinear optical phenomena, and their elucidation has accelerated the development of second-order nonlinear optical spectroscopy as a probe of these systems.

In this paper second-order nonlinear optical spectroscopy (second-harmonic generation (SHG) and sum-frequency generation (SFG)) was used to probe the electronic structures of different GaAs based interfaces, 80 Å Au epitaxial films on n-type and

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p-type GaAs(001) surfaces. We have also studied 80 Å As amorphous films on n-type and p-type GaAs(001) surfaces. In all cases the interfaces were either As-rich or Ga-rich as a result of different GaAs substrate growth conditions. Native oxide GaAs(001) samples were also studied in both n- and p-type. In Au:GaAs n-type systems, we have observed two resonance features at 0.715 eV and 0.731 eV for the Ga-rich interface, and one resonance feature at 0.715 eV for the As-rich interface. Similar single resonance features at 0.715 eV were observed in the As:GaAs n-type samples, but were not present in oxide:GaAs and metal:GaAs p-type systems. All resonance features were confirmed to be one-photon resonances. After consideration of various three-step optical processes, we suggest that the transitions originate from the midgap states. We further suggest that the interfacial resonance features are brought about by atomic displacement-induced defect states near the junction.

There are a number of experimental observations consistent with the existence of midgap states in this system, however there are very few direct spectroscopic measurements of interface energy levels. Some spectroscopic evidence for interface states has been derived from cathodoluminescence (CLS) in Au:GaAs(100) systems [5], inverse photoemission (IPS) in Ti:GaAs(110) systems [6], and ultraviolet-photoemission spectra (UPS) in Au:GaAs(110) systems [7]. These spectra however, are very broad, and the measurements are not intrinsically sensitive to the buried interface. In contrast to the CLS, IPS and UPS measurements [5–7], *sharp* resonant features were observed in our SHG and SFG experiments. The intrinsic interface sensitivity of the second-order nonlinear optical spectroscopies enable us to suppress spectral contributions from the adjoining bulk media, resulting in more specific assignments of the spectral features, and a narrowing of the features by comparison to those observed with competing spectroscopies.

The remainder of the paper is organized as follows. We first describe the experimental set-up and our samples in Section 2. In Section 3, we present measurements of the SHG and SFG spectra from different GaAs based interfaces. Then the results of measurements are discussed in Section 4. In this section we critically examine the evidence and sug-

gest that atomic displacement-induced defect states are responsible for these resonances. A brief conclusion is presented in Section 5.

2. Experiment

A schematic of the experimental apparatus is shown in Fig. 1. A 10 Hz *Q*-switched Nd:YAG pumped tunable optical parametric oscillator (OPO) or dye-laser was used as the fundamental SH generating light source for the SHG measurements. The samples were irradiated at an incidence angle of 75°. The incident light pulses had a temporal duration of 10 ns, and a fluence of ~ 1 mJ/cm². The fundamental (SH) photon energy range was from 0.68–0.78 eV (1.36–1.56 eV) (using the OPO), and from 1.29–1.40 eV (2.58–2.80 eV) (using the dye-laser). The reflected SH power was measured as a function of output light wavelength. Unfortunately continuous scanning from the visible to infrared wavelength range (0.8–1.36 eV) was impossible with our light sources. Our KTP based OPO cut off when photon energies exceeded 0.85 eV. Additionally, as the fundamental OPO photon energy was scanned towards the mid-infrared range (< 0.68 eV), we encountered several problems that made our spectroscopic measurements more difficult. For example, the very low

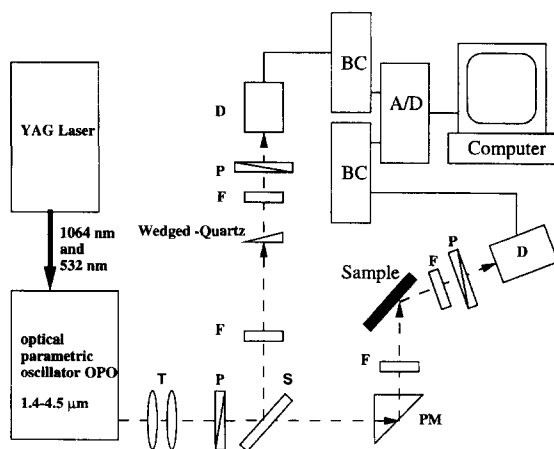


Fig. 1. Schematic diagram of experimental setup. F, spectral filter; P, polarizer; PM, prism; S, beam splitter; T, telescope; D, photomultiplier tube and monochromator; BC, boxcar averager; A/D, analog to digital converter.

quantum efficiency of photo-multiplier tubes limited our sensitivity to SH photons in near infrared (i.e. < 1.36 eV); also the fundamental probe light was strongly absorbed by water molecules in air for energy below ~ 0.65 eV. SFG spectra were obtained by irradiating the samples with the Nd:YAG beam at 1064 nm, and the tunable infrared beam at the same incidence angle and with the same polarization. To compensate for intensity fluctuations of the input beam, SH or SF signals were simultaneously produced and measured in a parallel (reference) optical path which contained a wedged quartz plate [3]. The maximum SHG or SFG output of the reference at each frequency was obtained by translating a wedged quartz plate along a direction perpendicular to the laser beam propagation vector. Sample intensities were normalized using this reference SHG or SFG signal.

GaAs(001) has a zinc-blende crystal structure and carries a single nonzero bulk second-order susceptibility $\chi_{yxz}^{(2)}$, whose contribution to the output SH radiation is highly anisotropic. Au bulk has an isotropic structure. Therefore it does not contribute SH radiation in the dipole approximation [8]. The interface signals from the metal:GaAs(001) samples were separated from the bulk signals by proper choice of sample orientation and light polarization [3]. By setting $\phi = 0$ and employing the p-in/p-out polarization configuration, where ϕ is the angle between the [100] direction and the plane of incidence, we were able to suppress the bulk signal, $\chi_{xyz}^{(2)}$, by $\geq 10^4$, thereby greatly enhancing our sensitivity to interface features. Results obtained in the p-in/p-out configuration with $\phi = 0$ will hereafter be referred to as interface signals.

Our GaAs(001) samples were doped ($\sim 1 \times 10^{16}$ cm^{-3}) with Si (n-type) and Be (p-type). They were grown on an undoped GaAs substrate by molecular-beam epitaxy (MBE) at temperatures around 580°C, and with background chamber pressures of $\sim 3 \times 10^{-10}$ Torr. It is well known that GaAs(001) exhibits a wide variety of surface reconstructions. These reconstructions are often related to the relative Ga to As ratio at GaAs surface [9]. RHEED measurements were performed on all samples to determine the surface reconstructions before growth of the Au epitaxial film. GaAs(001) samples were either As-rich (2×4 RHEED surface reconstruction) or Ga-rich

(4×2 surface reconstruction) [10]. The ratio of As:Ga was 75%:25% (25%:75%) for the 2×4 reconstruction (4×2 surface reconstruction). An 80 Å epitaxial Au film was then grown on the GaAs(001) surface with a surface temperature of $\sim 400^\circ\text{C}$. We confirmed the Au film was epitaxial by RHEED. We also grew amorphous, 80 Å As metal films on GaAs. When such a sample is exposed to air, a fraction of the As film is converted into an oxide, but Auger spectroscopy confirmed that oxygen did not penetrate as far as the As:GaAs interface. Finally we studied GaAs(001) n- and p-type samples without metal films. In the air, a ~ 50 Å oxide formed on the sample surface.

3. Results

Fig. 2 exhibits the full range of SHG spectra measurements from Au:GaAs n-type samples with different GaAs surface reconstructions (As-rich 2×4 and Ga-rich 4×2). The full wavelength range used both the OPO and the dye-laser. There are no sharp resonance features within energies probed by the dye-laser. The increase in signal at higher energy occurs as the fundamental photon energy increases above the direct band-gap of GaAs. However, we see that the interface spectra possess a single sharp peak at one-photon energy 0.715 eV for the As-rich Au:GaAs n-type sample and two closely spaced peaks for the Ga-rich Au:GaAs n-type sample. Comparison measurements of interface SHG and SFG spectra from Au:GaAs n-type samples using OPO with different GaAs surface reconstructions (As-rich 2×4 and Ga-rich 4×2) are exhibited in Fig. 3. The solid lines in the spectra from the Ga-rich Au:GaAs n-type sample represent a best fit to the data using two Lorentzian lineshape functions. According to the fitting, the SH (SF) peak positions are 0.708 ± 0.014 eV (0.713 ± 0.014 eV) and 0.731 ± 0.014 eV (0.730 ± 0.014 eV) respectively. We believe that the first peak in the SHG (SFG) spectra at ~ 0.708 eV (0.713 eV), has the same origin as the single sharp peak in the As-rich Au:GaAs n-type system, while the latter peak is a new feature resulting from the different interface preparation. By comparison to SFG spectra, all resonance features were confirmed to be one-photon resonances. SHG and SFG experi-

ments were also performed on As:GaAs(001) n-type samples within the same photon energy range. A similar single resonance feature around 0.715 eV was observed (see Fig. 4(a)), and was confirmed to be a one photon resonance. Interestingly in contrast to previous spectroscopic experiments [5–7], all of the observed resonances are sharp, suggesting a narrow distribution of states at or just below the buried interface.

In Au:GaAs p-type and oxide:GaAs systems, the interface second-order nonlinear optical spectroscopies have also been performed within the same spectral region. No resonance features were observed in either system (see Fig. 4(b) and (c)). These results are surprising at first glance, since one would expect that the energy levels at the interface will not be changed by variations in bulk doping and overlayer oxidation [11]. However, the carrier occupation density in these states is modified, since the Fermi level can shift with respect to the midgap levels. In the p-type metal:GaAs system these midgap states will become empty, as the Fermi level shifts towards the valence band maximum [5]. The effects in the oxide:GaAs system are less clearly explained. However the oxide is rarely well characterized, and it is possible that the midgap levels were shifted out of our probe range.

In general the second order susceptibility for SHG, $\chi_{ijk}^{(2)}$, can be written as [8]

$$\chi_{ijk}^{(2)} = N \frac{e^3}{\hbar^2} \sum_{n,n'} \langle g|r_i|n\rangle \langle n|r_j|n'\rangle \langle n'|r_k|g\rangle \rho_{gg} \times \left[(2\omega - \omega_{ng} + i\Gamma_{ng})^{-1} (\omega - \omega_{n'g} + i\Gamma_{n'g})^{-1} + (2\omega + \omega_{ng} + i\Gamma_{ng})^{-1} (\omega + \omega_{n'g} + i\Gamma_{n'g})^{-1} + (\omega + \omega_{ng} + i\Gamma_{ng})^{-1} (\omega - \omega_{n'g} + i\Gamma_{n'g})^{-1} \right]. \quad (1)$$

Here $|g\rangle$ represents the ground state, and $|n\rangle$ and $|n'\rangle$ are two excited states of the medium. ρ_{gg} is the occupied electron density of the ground states. Resonances can be viewed to arise through particular terms in this sum over states. In our case, there are two probable excitation processes that can be invoked to explain the one photon resonance. In one case the three-step process starts from the top of valence band. Here the state $|g\rangle$ is at the valence band edge. In the other case the process starts from the interface midgap state. Here the state $|g\rangle$ is an interface midgap state. Our results suggest that the one photon resonances on metal:GaAs n-type samples are due to

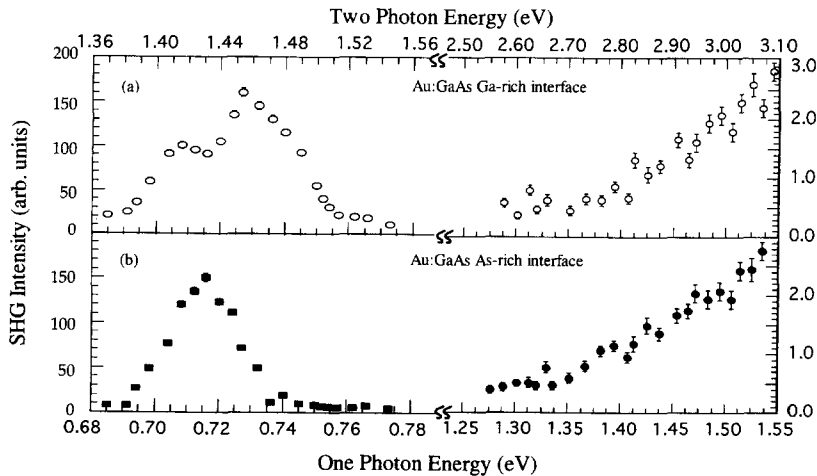


Fig. 2. Interface SHG from Au:GaAs n-type samples. The left (right) SHG intensity axis corresponds to SHG spectra from 0.68–0.78 eV (1.29–1.40 eV). Note, the horizontal axis is not continuous.

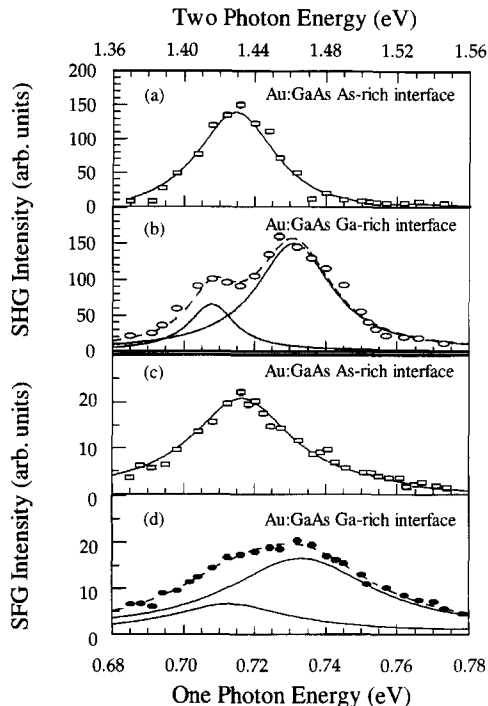


Fig. 3. Interface SHG and SFG spectra from Au:GaAs n-type samples. (a) SHG spectrum from As-rich Au:GaAs n-type sample. The solid line is a guide for the eye. (b) SHG spectrum from Ga-rich Au:GaAs n-type sample. The two solid lines are fit using two Lorentzian line shape functions, and the dashed line is the combination of the above two lines. The peak positions are estimated to be 0.708 ± 0.014 eV and 0.731 ± 0.014 eV respectively. (c) SFG spectrum from the As-rich interface sample. The solid line is a guide for the eye. (d) SFG spectrum Ga-rich Au:GaAs n-type sample. The two solid lines are fit using two Lorentzian line shape functions, and the dashed line is the combination of the above two lines. The peak positions are estimated to be 0.713 ± 0.014 eV and 0.730 ± 0.014 eV respectively.

the resonant transition from the occupied midgap states to the conduction band minimum. This follows from consideration of observations in the p-type systems. In p-type systems the resonance process originating from the midgap states would be very weak as a result of their low occupation density. Indeed no resonance was observed in these systems (see Fig. 4).

Additional measurements were performed to rule out several other possible origins for these features. Bulk SHG $\chi_{xyz}^{(2)}$ spectra measurements were performed in the p-in/s-out polarization configuration at $\phi = 0$. Using this configuration we maximized our

sensitivity to the bulk nonlinearity [3]. Fig. 4(d) reveals no resonance features in the bulk signals.

The interface signals in principle contain contributions from the front metal surface, and higher-order bulk nonlinearities. To examine the contributions from the Au surface, we measured SFG and SHG spectra from a thick Au-epitaxial film ($\sim 5 \mu\text{m}$) and found that SFG and SHG signals were below our noise level (see Fig. 4(a)). Also, since the bulk nonlinear response of the Au epitaxial layer on GaAs(001) is expected to contribute in the same way in both n- and p-type GaAs systems, the fact that no resonance features were observed in Au:GaAs p-type systems effectively rules out possible contributions

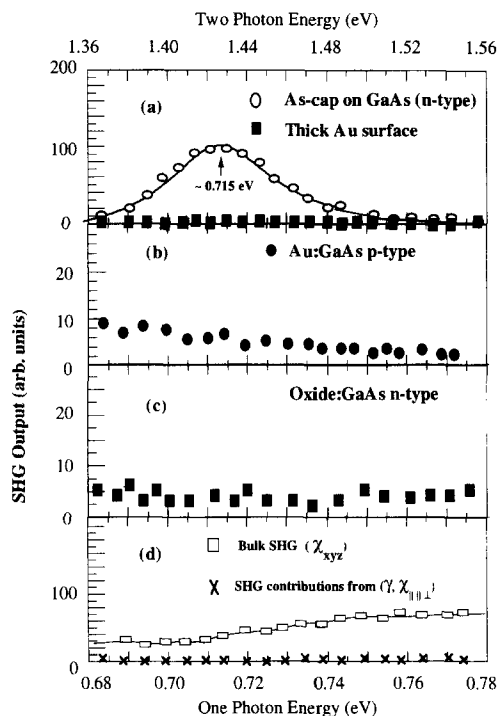


Fig. 4. (a) Interface SHG spectra from the As capped GaAs n-type sample. The solid line is a guide for the eye. The SHG from thick Au surface was at our noise level. (b) Interface SHG spectra from Au:GaAs p-type system; no resonance features were observed. (c) Interface SHG spectra from native oxide GaAs surface; no resonance features were observed. (d) Bulk SHG spectra from the Au:GaAs n-type sample; the solid line is a guide for the eye. Both As-rich and Ga-rich interface samples have the same bulk spectra. No resonance features have been observed from the bulk SHG studies. The SHG contribution from the linear combinations of γ and $\chi_{||\perp}^2$ was at least two orders of magnitude smaller than the interface signal in both n- and p-type systems.

from bulk epitaxial Au films. The possibility of higher-order GaAs bulk contributions were also examined in the s-in/s-out and s-in/p-out polarization configurations. The bulk anisotropic contribution (ξ) was below our noise level and the signal resulting from linear combinations of γ and $\chi_{\parallel\perp}^{(2)}$ was at least two orders of magnitude smaller than the interface signal [3] (see Fig. 4(d)). In addition, the higher-order bulk terms contribute in the same way to metal:GaAs and oxide:GaAs systems, and no resonances in oxide:GaAs systems were observed. In total, these findings rule out higher-order bulk contributions as sources of the resonance features. The 0.715 and 0.731 eV resonance features most likely originate from the buried interface.

4. Discussion

Our second-order nonlinear optical spectral measurements of As-rich Au:GaAs and As:GaAs n-type samples possess a similar resonance at 0.715 eV, suggesting that the common interface state may be related to As atoms. On the other hand, the interface state at ~ 0.731 eV was only found at the Ga-rich interface in Au:GaAs n-type systems. This suggests that this latter interface state is related to the presence of excessive Ga atoms at the interface.

These observations can be understood within a simple theoretical framework. A gradient in the chemical potential for As or Ga atoms can induce the migration of these atoms from the interface into the GaAs substrate. As a result of relative concentrations of As and Ga atoms during growth, we might expect the formation of high concentrations As atom displacement-induced defect states near the As-rich interface [11]. In this case the As atom sits in a Ga atom site in the interfacial region. Such As atom displacements are the first step in the production of several primary defect states in GaAs [11–13]. Two defect-associated, strongly bound energy levels with s-like and p-like symmetries respectively, are predicted to lie near the middle of the band gap (see Fig. 5(c) and (d)) [14]. We note that the calculated states are generally more complicated than the simple atomic displacement described above; for example they may involve coupling of the antisite to a nearby vacancy [15]. Since vacancy concentrations

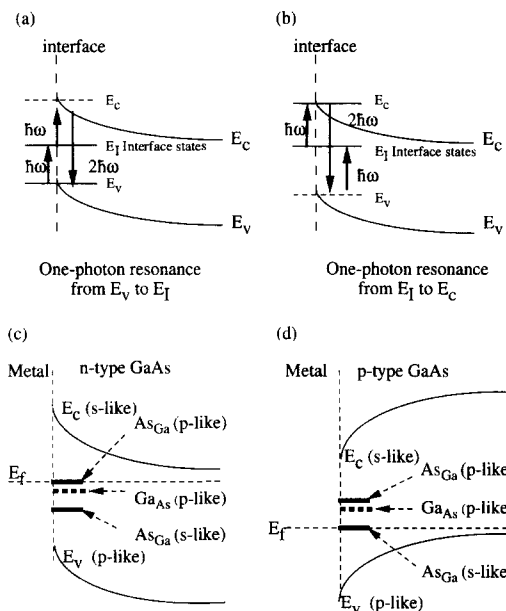


Fig. 5. Schematic of two most probable three-step optical processes giving rise to the one photon resonance. (a) Time-ordered process starts in valence band, and is represented by $E_v \xrightarrow{\hbar\omega} E_1 \xrightarrow{\hbar\omega} E_c \xrightarrow{2\hbar\omega} E_v$. (b) Time-ordered process starts from interface states, and is represented by $E_1 \xrightarrow{\hbar\omega} E_c \xrightarrow{2\hbar\omega} E_v \xrightarrow{\hbar\omega} E_1$. (c) Schematic representation of As (As_{Ga}) and Ga (Ga_{As}) displacement defect state energies and symmetries in n-type GaAs:metal interfaces. (d) Same as (c) except in p-type GaAs:metal interface. E_c (E_v) denote conduction (valence) band energy minima (maxima), E_f denotes the Fermi-level in these systems, and E_1 denotes interface state energy.

may be higher near the interface, it is also plausible that a higher density of these types of defect states may exist near interfaces. We note however, that on very general grounds one would expect the As anti-site midgap state overlap strongly with bulk |s>-like and |p>-like states [15].

Similarly a high concentration of Ga displacement-induced defect states are expected for the Ga-rich interface. In this case Ga atoms sit in As atom sites. A single strongly bound Ga displacement defect state has been theoretically predicted to have an p-like symmetry and an energy level near the middle of band gap [14]. Again on general theoretical grounds one would expect this state to be pulled from p-like bulk states below the valence band edge.

Our observations can be explained quite neatly if

we assume that the theoretical assignment of defect state relative energies and symmetries is correct [14], and that Ga-rich interfaces still contain some As defect states near the interface. In this case the n-type GaAs systems will experience Fermi-level pinning by the p-like defect states, while the p-type GaAs system will experience Fermi-level pinning by the s-like defect states (see Fig. 5). (Note, in the former case it is not essential that the p-like states pin the Fermi level, only that their energy is equal to or less than the energies of states that pin the Fermi level.) The observed resonances are derived by three-step processes that progress from occupied p-like defect states, to the s-like conduction band, to the p-like valence band, and then back to the original defect state. The last step is allowed as a result of weak strain- or field-induced interfacial state symmetry breaking. In general the strain-induced symmetry breaking is strong, but is limited to a few monolayers near the interface [4]; on the other hand, the depletion field-induced symmetry breaking may occur through the depletion length range (i.e. $\sim 0.1 \mu\text{m}$ in our samples). In principle both effects could contribute to the nonlinear optical processes. The model explains the observation of two resonance peaks in the Ga-rich interface, and it predicts no transitions in p-type GaAs systems since p-like states will be unoccupied. Finally the model predicts new resonances in spectral regions not studied here, as a result of processes coupled to the s-like defect states.

By comparison to broadened interface states observed in photoemission at very low Au coverages on GaAs surfaces [7], the very sharp observed spectra suggest that overlayer metal atoms displace the As or Ga, and that the detected atomic displacement defects occur in bulk GaAs just far enough from the interface so that their interaction with the metal overlayer free electron states is weak. Since the As displacement is among the most common defects in GaAs, the resonance state at 0.715 eV was still observed in the Ga-rich interfaces, albeit more weakly [12]. Signal sizes in our experiment suggest that the interface susceptibility $\chi^{(2)} \leq 10^{-16}$ esu. Model-dependent estimates of defect number densities may be derived using this nonlinearity. In the case of strain-induced effects, we might expect $\chi^{(2)} \sim N\alpha$, here N is the surface defects density within a few monolayers of the junction and α is single defect second-

order susceptibility. If we assume $\alpha \sim 10^{-29}$ esu, then $N \sim 10^{13} \text{ cm}^{-2}$. In the case of depletion field-induced effects, there are several hundred layers within the depletion region that can contribute to the signal. We may estimate the bulk antisite defect density N within this depletion region using simple models; it is of order $N \sim 10^{18} \text{ cm}^{-3}$. In reality both effects coexist and may affect our second order nonlinear processes. Qualitatively our spectra also suggest that the ratio of the two resonance peaks (0.715 eV, 0.731 eV) can be related to the ratio of As and Ga displacements defect at the interface. For example, if we assume the single defect has the same order susceptibility coefficient, the relative peak heights ratio $I_{\text{AsGa}}/I_{\text{GaAs}} \sim N_{\text{AsGa}}^2/N_{\text{GaAs}}^2$. This may prove to be a useful methodology for in situ chemical analysis during growth.

5. Conclusion

We have used second-order nonlinear optical spectroscopies (SHG and SFG) to probe the interface states at buried metal:GaAs junctions. Sharp resonance interface spectra arise from transitions between the midgap states and the conduction band minimum. Two interface resonance states at ~ 0.715 eV and ~ 0.731 eV were observed and are associated respectively, with As and Ga atomic displacements just below the buried interface.

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