

# Spatial mapping of ordered and disordered domains of GaInP by near-field scanning optical microscopy and scanning capacitance microscopy

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Imaging of topography, locally induced photoluminescence and Fermi-level pinning in adjacent ordered and disordered domains on a cleaved GaInP sample is performed using a near-field scanning optical microscope and scanning capacitance microscope at room temperature in air. Highly localized photoluminescence spectra obtained by the near-field scanning optical microscope on these domains show spectral peaks at 680 nm (ordered) and 648 nm (disordered) GaInP. The near-field scanning optical microscope and scanning capacitance microscope data confirm previously published data, indicating that the electronic surface structure of ordered GaInP is significantly different from that of disordered GaInP. Both approaches indicate that the Fermi-level at the surface of ordered GaInP is pinned, while the Fermi-level at the surface of disordered GaInP is not pinned. The size, structure, and position of the ordered and disordered domains observed by the near-field scanning optical microscope and scanning capacitance microscope agree with those obtained by cathodoluminescence and Kelvin probe force microscopy. © 1996 American Vacuum Society.

In the past few years, atomic ordering has been observed for a wide variety of III-V semiconductor alloys.<sup>1</sup> The ordering in GaInP affects the optical and electrical properties of the material and is therefore interesting and important for optoelectronic devices. It is found that under certain growth conditions, Ga<sub>x</sub>In<sub>1-x</sub>P lattice-matched to GaAs spontaneously orders on a group III sublattice with gallium and indium atoms preferentially occupying alternate {111} planes.<sup>2</sup> The ordering is identified by the appearance of superspots in transmission electron diffraction (TED) patterns. Recently, atomic force microscopy<sup>3,4</sup> (AFM) and Kelvin probe force microscopy<sup>5</sup> (KPFM) have been used to study the surface morphology and surface electronic properties of GaInP. The ordering of GaInP reduces the energy band gap, as observed by cathodoluminescence<sup>6</sup> (CL) and photoluminescence<sup>7</sup> (PL). The measurement of band gap by PL is macroscopic, while the measurement of CL provides approximately 1 μm spatial resolution. Recently, partially ordered GaInP is studied by a low temperature NSOM with a spatial resolution of 270 nm.<sup>8</sup> In this work, we report the results of direct imaging of PL from adjacent ordered and disordered domains in GaInP by NSOM (Ref. 9) at room temperature and correlate these measurements with scanning capacitance microscopy<sup>10,11</sup> (SCM) with a resolution of 150 nm.

Figure 1 shows a schematic of the NSOM used in this work. The NSOM consists of a shear force imaging (topography) system<sup>12</sup> and a near-field optical imaging system.<sup>9</sup> The shear force imaging system is used to control the distance between the tip and sample. The tip is vibrated at its resonance frequency (15–30 kHz) with an amplitude of about 10–20 nm. As the tip approaches the sample surface,

the oscillation of the tip is damped because of the tip-sample interaction via shear force. Keeping a constant force results in a constant tip-sample distance. At the heart of the near-field imaging system is the tip, which is a tapered single mode fiber made opaque everywhere except at its very end by deposition of a layer of aluminum (100 nm).<sup>9</sup> The output of an Ar<sup>+</sup> laser (488 nm) is launched into the fiber. This light exits through the 150 nm fiber aperture exciting a small region of the sample and inducing PL from this region. The PL signal is collected by a 0.25 NA objective lens from the side. The pump light (488 nm) is filtered out by a notch filter. The PL signal is then sent to a monochromator and detected by an avalanche photodiode (APD) with a dark count of 8 counts per second.

Figure 2 shows a combination AFM and SCM.<sup>10,11</sup> A metal-coated silicon tip is brought to the surface of the sample. A contact mode AFM is used to position and scan the tip over the surface of the sample. A high sensitivity capacitance sensor (operating at 915 MHz) is connected to the tip to measure the capacitance between the tip and the sample. The tip is grounded by an inductor (at low frequencies) and a 10–100 kHz ac bias voltage is applied to the sample. The tip-sample capacitance change associated with the ac bias voltage is measured by the capacitance sensor. When the tip is scanned across the sample, a topography and a capacitance change image are obtained simultaneously.

NSOM and SCM measurements have been performed on a GaInP sample (AO-98). The ordering in this sample is controlled by substrate misorientation. The GaInP layer is grown by organometallic vapor phase epitaxy (OMVPE) on a grooved GaAs (001) substrate misoriented by 9° toward [110]. The period of the groove is 40 μm, and the depth is 4 μm. The grooves run along the [110] direction. The growth

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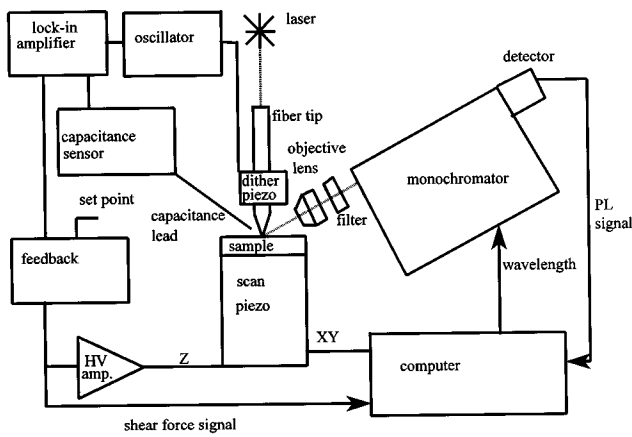


FIG. 1. Schematic diagram of the near-field scanning optical microscope.

temperature is 670°C and growth rate is 2 μm/hr. The thickness of the GaInP epilayer is approximately 5 μm. The sample is cleaved perpendicular to the surface and grooves. CL measurements indicate the existence of a highly ordered domain of several micrometer length and approximately 2 μm thickness at the bottom of the grooves close to the GaAs interface, and a highly disordered GaInP region (3 μm×2 μm) above the ordered region.<sup>13</sup> In Fig. 3, a sketch of the sample cross section is shown together with the ordered and disordered regions which have been identified by CL.<sup>13</sup>

NSOM PL spectra are obtained by locating the fiber probe on the cross sectional surface of sample AO-98. A pump beam (488 nm) is sent through the fiber providing approximately 1 nanowatt at the surface. The collected PL intensity is then measured as a function of wavelength with the spectrometer. The measured PL spectra are shown in Fig. 4. The peak wavelengths are 648 nm (for the disordered region) and 680 nm (for the ordered region) respectively. Both peaks are well separated. By fixing the monochromator bandwidth (10 nm) at 648 nm (to select PL from the disordered regions) or 680 nm (to select PL from ordered regions), a map of the disordered and ordered PL intensity is obtained. The results

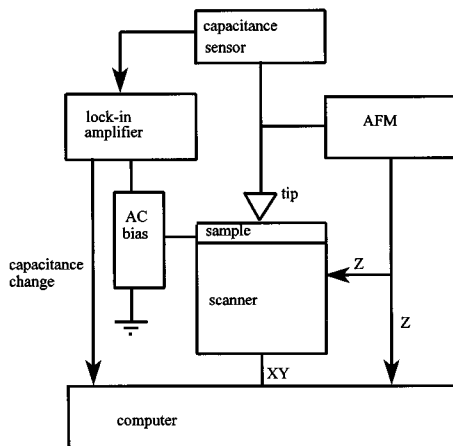


FIG. 2. Block diagram of the scanning capacitance microscope.

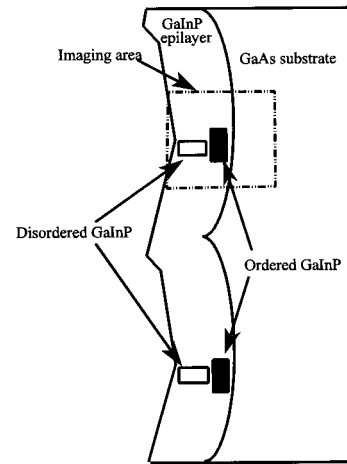


FIG. 3. Schematic drawing of the groove structure of sample AO-98 (in cross section).

are shown in Fig. 5(a) and 5(b), respectively. The scan area is 9.5 μm by 12.0 μm and the full width at half maximum (FWHM) of the smallest feature in the NSOM images indicates a spatial resolution of 150 nm. The corresponding topographic images are also shown in Fig. 5(c) and 5(d), respectively. These topographic images show the same area except for a slight shift of 0.4 μm. The maximum height variation in the topographic image is less than 100 nm. The black regions in Fig. 5(a) and 5(b) indicate that no photoluminescence is detected from a large part of the GaInP epilayer or from the GaAs substrate. The small white region in Fig. 5(a) shows the photoluminescence signal from the disordered region, which corresponds to the highly disordered domain shown in Fig. 3. It is close to the sample surface (the growth surface). The size of the domain is approximately 2

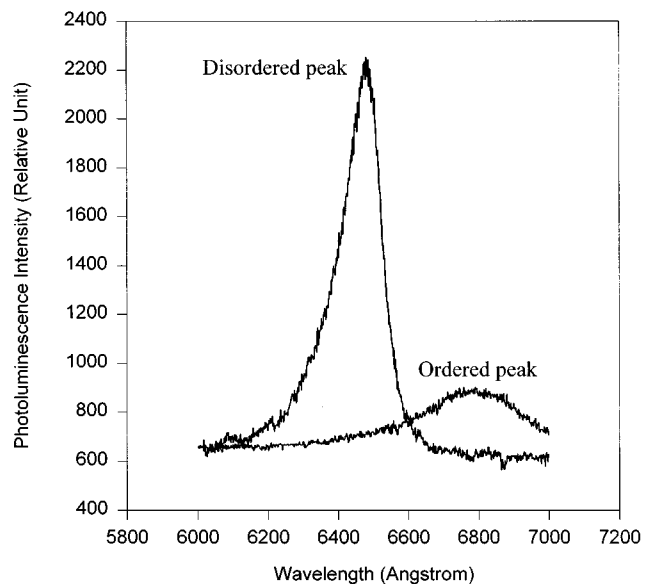


FIG. 4. Near-field photoluminescence spectra of ordered and disordered GaInP.

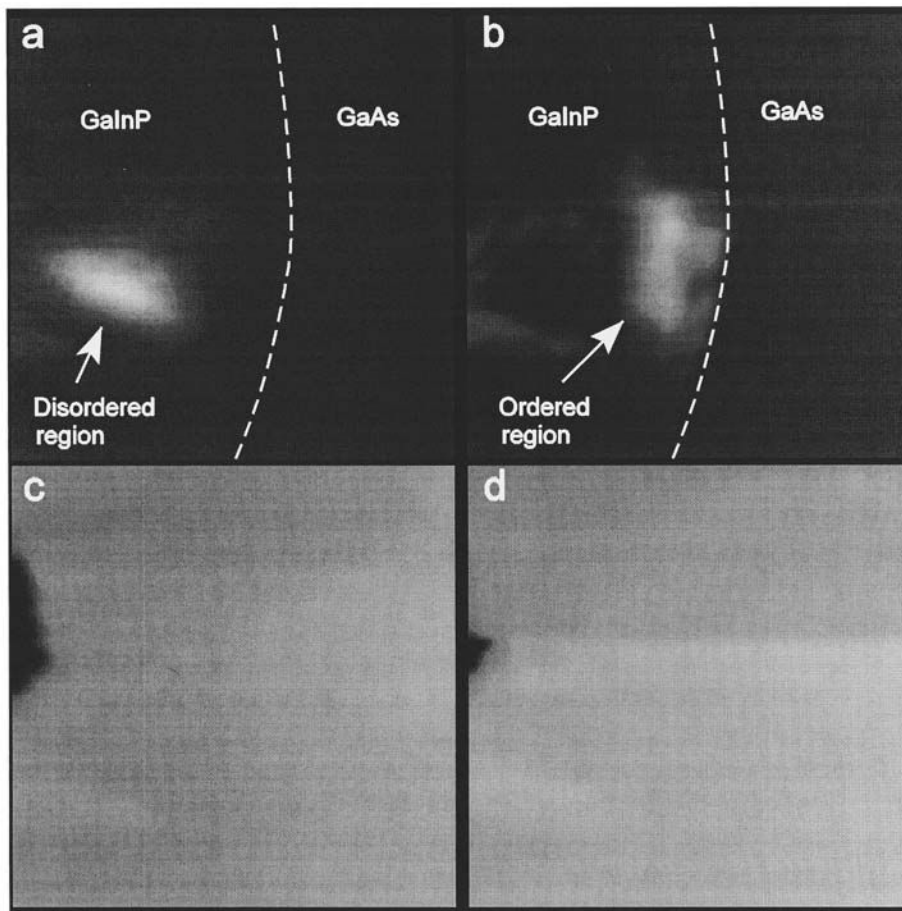


FIG. 5. (a) and (c) Near-field photoluminescence intensity map ( $\lambda=648$  nm, 10 nm bandwidth) of cross sectioned GaInP/GaAs sample (AO-98) and simultaneously obtained topography ( $9.5 \mu\text{m} \times 12.0 \mu\text{m}$ ). (b) and (d) Near-field photoluminescence intensity map ( $\lambda=690$  nm, 10 nm bandwidth) of AO-98 and simultaneously obtained topography ( $9.5 \mu\text{m} \times 12.0 \mu\text{m}$ ). All four images have been taken in the same sample area with the same tip.

$\mu\text{m}$  wide and  $3 \mu\text{m}$  long. The small white region in Fig. 5(b) represents the highly ordered domain, which is between the highly disordered domain and the GaAs substrate. The size of this domain is about  $2 \mu\text{m}$  wide and a few micrometers long. The positions of these two regions are near the lateral center of the groove, which can be seen by comparison to the topographical images [Fig. 5(c) and 5(d)]. There also appears to be a halo of ordered material around the disordered region as seen in Fig. 5(b). Seven different places of sample (AO-98) have been measured by the NSOM, and it is found that the size and shape of these domains varies slightly from groove to groove. The NSOM results are in very good agreement with that of CL (Ref. 13) and KPFM (Ref. 5). A large part of the GaInP epilayer shown in Fig. 5(a) and 5(b) emits no photoluminescence. This region (outside the ordered and disordered domains identified in Fig. 3) is not well characterized by other methods and requires further study.

Figure 6 shows two NSOM photoluminescence line cuts, approximately perpendicular to the GaInP/GaAs interface, through the center of both disordered and ordered domains shown Fig. 5(a) and Fig. 5(b). The PL intensity in the ordered domain is smaller than that of the disordered domain, as can also be seen in Fig. 4. Our previous results, using the

KPFM,<sup>5</sup> have shown that the surface of ordered GaInP is electrically pinned, indicating a large surface state density. This is consistent with the fact that the PL intensity is reduced in the highly ordered region, since it is expected that a large surface state density should decrease PL yield. Note that the PL intensity drops relatively sharply at the GaAs/GaInP interface in Fig. 6, because the carriers quickly diffuse into the GaAs substrate.

The SCM is also used to image the sample (AO-98) in cross section. A capacitance change image and a topographic

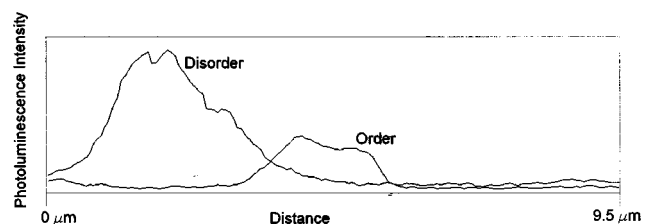


FIG. 6. Line cuts, approximately perpendicular to GaInP/GaAs interface, through the center of both ordered and disordered regions shown in Fig. 5(a) and 5(b).

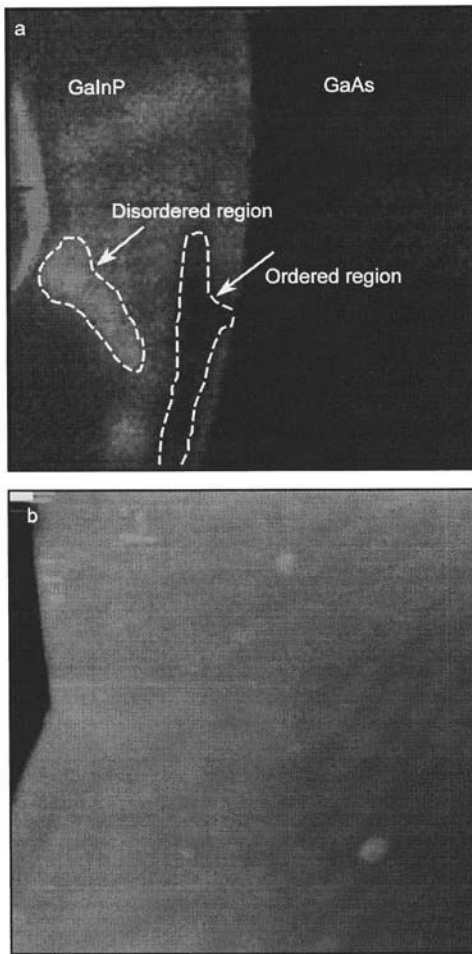


FIG. 7. (a) and (b) simultaneously obtained capacitance change image and topographic image ( $12\ \mu\text{m} \times 12\ \mu\text{m}$ ) of cross sectioned AO-98 sample. This image is not of the same groove as that of Fig. 5.

image obtained simultaneously by the SCM are shown in Fig. 7(a) and 7(b). The image size is  $12\ \mu\text{m} \times 12\ \mu\text{m}$ . The applied ac bias voltage between the tip and a sample is 2.5 Volts peak at 89.9 kHz. The capacitance change is measured by a lock-in amplifier at that frequency. In Fig. 7(a), the black color represents a negligibly small capacitance change signal. Because the GaAs surface is electrically pinned, there is a negligible amount of capacitance change with voltage. Just to the left of the GaAs substrate and at the bottom of the GaAs groove, there is an approximately  $2\ \mu\text{m}$  wide and several  $\mu\text{m}$  long region (black color) which corresponds to the highly ordered GaInP. The capacitance change signal of the ordered domain is the same as that of GaAs, indicating that the ordered domain is also electrically pinned. To the left of the highly ordered region, there is a region approximately a  $2\ \mu\text{m}$  wide and  $3\ \mu\text{m}$  long (gray color) which corresponds to

the highly disordered region identified by NSOM. This region has the largest capacitance change. Compared with the PL images by NSOM, the size, shape, and position of these two domains are very similar. We believe that the SCM and NSOM data strongly confirm our previous observations that surface Fermi-level pinning is directly related to ordering in GaInP.

Since the atomic structure of the ordered and disordered GaInP is different in the bulk, it is not unreasonable to assume that ordering may also modify the electronic structure at the surface [in this case the cleaved (110) plane]. Our observation that the Fermi-level of ordered GaInP domains is pinned at the surface, while highly disordered GaInP domains are unpinned, supports the idea that ordering causes an increase in the density of surface states. We are not aware of the existence of any detailed model of the electronic structure of GaInP (ordered or disordered) at the surface.

The SCM image also shows several other clear variations in the GaInP layer which are not seen by NSOM and KPFM. We believe that the contrast seen by SCM may be directly related to the degree of order in the GaInP epilayer. Further study is required to substantiate this claim.

In conclusion, NSOM and SCM have been used to study GaInP in which ordering was controlled by substrate misorientation. The measurements have been made in cross section and at room temperature. The results clearly show that NSOM and SCM are capable of distinguishing between ordered and disordered GaInP. A previous result (KPFM) and these results (NSOM and SCM) all indicate that the Fermi-level in ordered GaInP is pinned at the surface in contrast to that of disordered GaInP.

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