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Interlaboratory comparison of InGaAsP ex situ characterization ☆

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Abstract

The accuracy of ex situ characterization of InGaAsP materials for optoelectronics has been assessed by circulating a single specimen set among different laboratories. Six InGaAsP thin film specimens, with nominal photoluminescence (PL) peak wavelengths of 1.1, 1.3 and 1.5 μm, were measured with X-ray diffraction and PL. X-ray measurement reproducibility appears to be dominated by specimen nonuniformity. The distributions of the X-ray rocking-curve peak separations measured by the different laboratories had standard deviations from 1 to 11 arcsec, depending on the specimen, while the lateral variations across specimens were between 9 and 150 arcsec. In contrast, the variation among PL measurements was larger than the variation within individual samples. Consistent relative offsets between instruments were observed, but these had no apparent correlation with factors such as pump wavelength, wavelength calibration, sample temperature, pump power density, and peak identification. Analysis of the raw PL data with identical methods revealed that the variations are intrinsic to the data, not artifacts of the methods used to extract a characteristic energy from the PL spectra.

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1. Introduction

InGaAsP forms the basis for lasers, detectors, and other telecommunications components. High accuracy characterization of the material composition and strain is critical for these applications. Evidence suggests, however, that there are large variations in measurements of these parameters by different laboratories even in a relatively well-characterized material such as AlGaAs [1]. In fact, several major optoelectronics manufacturers have identified lack of standardized assessment procedures as an impediment to productivity [2]. To address this, an interlaboratory comparison of ex situ measurements of InGaAsP was undertaken with the goal of identifying reliable, reproducible methods for measurement and data analysis.

2. Experimental procedure

The measurement methods studied were X-ray diffraction (XRD) and photoluminescence (PL).

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Six different specimens were measured. The specimens were 1 cm^2 pieces of uncapped InGaAsP layers 1 μm thick. The layers were deposited by organometallic vapor phase epitaxy (OMVPE) on slightly misoriented, (0 0 1) InP substrates. Of the six samples, there were two each having the nominal PL peak wavelengths of 1.1, 1.3 and 1.5 μm.

The InGaAsP layers were grown on 51 mm (2 in) wafers, and the lateral nonuniformity as determined from maps of X-ray rocking curve peak separation and peak PL wavelength was found to be significant. To minimize the impact of these variations on the study, specimens of 1 cm^2 were cleaved from the most uniform region of each wafer. Fig. 1 shows a contour plot of the XRD peak separation over the central $2 \times 2\,cm^2$ region of one of the wafers. The difference between the contour lines is 10 arcsec and the maximum variation over the area is 130 arcsec. The outlined square in the figure shows the region from which the specimen used for the study was cleaved. The maximum variation over this piece is 50 arcsec. Because of the specimen nonuniformity, a single set of samples was used for the study and measured sequentially by different laboratories.

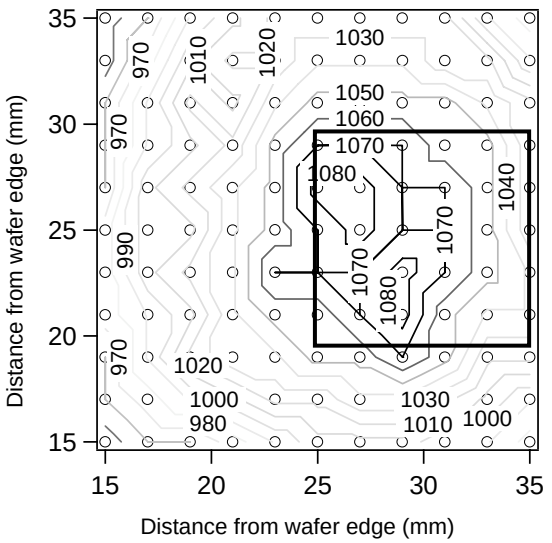

Fig. 1. Contour plot showing the variation in XRD peak separation, in nm, across the center $2 \times 2\,cm^2$ region of one of the wafers. The outlined square is the region from which the specimen for the study was cleaved.

Participants were asked to make their measurements as close as possible to the center of each specimen.

Participants were also asked to report the conditions under which each measurement was made. Variables such as temperature, spot size, beam power, wavelength, resolution, and data analysis technique were all tracked in an effort to identify which have the greatest influence on the measurement results. The samples were measured with eight different XRD instruments and eight different PL systems. The XRD systems were all commercially obtained and represented more than one manufacturer and more than six model types. The PL systems were commercially obtained except for one system and represented more than one manufacturer and more than four model types. No measurements by NIST were included in the comparison.

3. Results and discussion

The results of the XRD and PL measurements, made with different instruments, are shown in Fig. 2(a) and (b), plotted as the difference between the individual and average measurements for each sample. The XRD data plotted are the measured separation between the substrate and epilayer peaks; the PL data are the peak wavelength identified. Both sets of data show fairly large variations. For the PL data there appear to be systematic offsets between individual instruments. For the XRD data, however, the differences between measurements appear to depend more on the specimen measured, with some specimens showing much larger variations than others.

The statistics of the XRD measurements are given in Table 1. The standard deviation and maximum deviation of the measurements at the center of each specimen, by different instruments, are given in columns 2 and 3. The maximum lateral variation measured by mapping each 1 cm^2 piece is given in column 4. As can be seen from the table, and as described earlier, the lateral variation measured over each specimen is quite large. In fact, it is much larger than the maximum variation between measurements by different instruments on

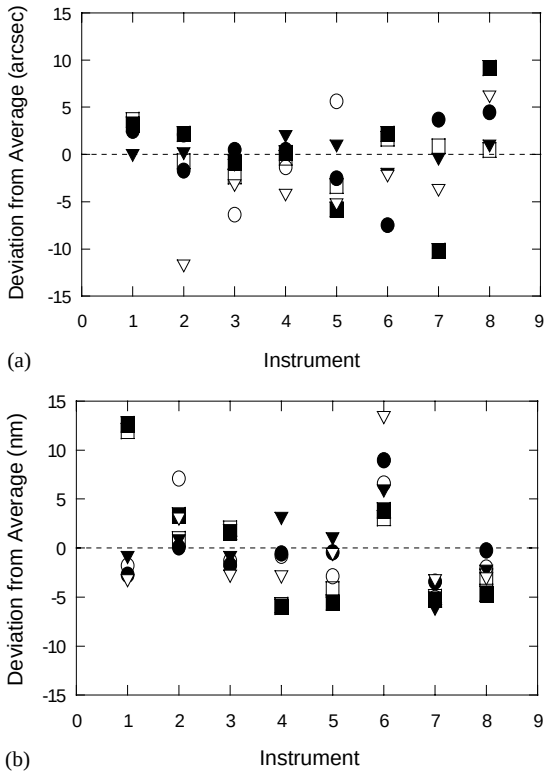

Fig. 2. Deviation from the average of: (a) the XRD peak separation; and (b) the PL peak wavelength, each measured using eight different instruments, for six samples: (■) sample 1, (□) sample 2, (●) sample 3, (○) sample 4, (▼) sample 5, and (▽) sample 6 (for XRD the point from instrument 1 for sample 6 is 24.7 and is out range for the graph).

Table 1
XRD peak separation statistics (arcsec)

Specimen	Instrument standard deviation[a]	Instrument maximum deviation[a]	Maximum lateral variation[b]
1	5.9	19	150
2	2.2	7	9
3	3.9	12	10
4	5.1	12	80
5	1.3	4	50
6	11.1	36.5	51

[a] Variation over eight instruments, at the center of the specimen.
[b] Measured with one instrument over the entire specimen.

most of the specimens. This indicates that the variation between measurements made with different instruments probably results from differences in the position of the X-ray beam on the specimen, rather than from true differences in the measurements. Contributing to this was the fact that positioning the beam on a specific spot on a small sample is difficult in many XRD systems; most participants in the study did not have experience with this.

For half of the samples (2, 3 and 6) there was disagreement among the XRD measurements as to whether the peak separation was positive or negative, i.e., the substrate peak was not clearly identifiable. Because the InGaAsP layers are 1 μm thick their peak XRD intensity is similar to that of the substrate peak. In addition, in some samples the substrate and epitaxial layer peaks are closely spaced and can be difficult to distinguish. Identification of the substrate and epilayer peaks should not be a problem for device structures, where the epilayers are thinner. For samples where there was disagreement about whether the peak separation was positive or negative, the absolute values of the separation were used to obtain the average peak separation and standard deviation. It was also reported that for two of the specimens (4 and 6) there were more than two peaks in the X-ray spectra and that their overlap made the measurements difficult. From this study it seems likely that the variation between measurements on the most uniform sample (2), which had only two peaks in the XRD spectra, is indicative of the true variation between measurements on different instruments (7 arcsec).

One might expect problems due to sample nonuniformity to be similar or worse for PL measurements since the spot diameter for PL is typically much smaller ($\leqslant 0.5$ mm) than that for XRD. However, mapping of the 1 cm^2 specimens revealed that the variation in the PL measurements from different instruments, at the center of each specimen, is larger than the variation found by mapping, for all of the samples (columns 3 and 4 of Table 2). This suggests that the variations result from true differences in the instruments or conditions used, rather than from sample nonuniformity.

As can be seen in Fig. 2(b), there appear to be wavelength offsets in the data taken with different instruments. For example, the results for all samples from instrument 7 are lower than the

average, while those from instrument 6 are all higher than average. These systematic variations may indicate a calibration difference between the instruments. All but two of the instruments (1 and 6) were reported to have been wavelength calibrated within three weeks of the measurement.

Table 2
PL peak wavelength statistics (nm)

Specimen	Instrument standard deviation[a]	Instrument maximum deviation[a]	Maximum lateral variation[b]
1	6.6	23.0	3
2	5.9	17.8	2
3	3.8	12.4	2
4	4.4	12.2	2
5	3.6	12.1	4
6	6.6	16.7	6

[a] Variation over eight instruments, at the center of the specimen.
[b] Measured with one instrument over the entire specimen.

Other potential sources of offset were also examined. The variation in peak wavelength measured for the different samples was examined as a function of the sample temperature during measurement, the incident beam power density, and the incident beam wavelength, using values reported by the participants. Plots of these variables are shown in Fig. 3 for sample 4, which has a typical variation (see Table 2). No systematic variation with temperature was observed. The data do indicate a shift to shorter wavelengths (higher energy) with increasing power density, although data from other samples is not as neatly clustered as for sample 4. But this is opposite to what would

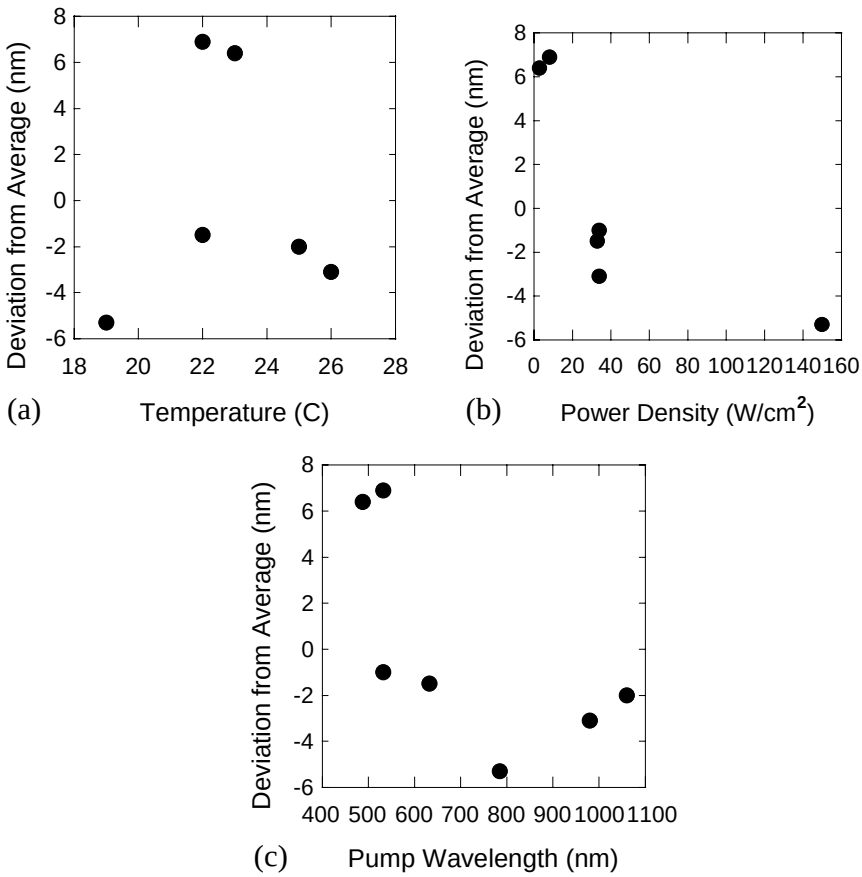

Fig. 3. The variation in the PL peak wavelength with different measurement parameters for specimen 4: (a) sample temperature, (b) incident power density, and (c) incident wavelength.

be expected if the power density shifts the PL spectra by increasing the sample temperature. The band gap of InGaAsP decreases with increasing temperature [3], and it has been reported that the PL peak position of InGaAsP shows the same temperature dependence as the band gap of InGaAsP [4]. Thus, the PL peak would be expected to shift to longer wavelengths with increasing temperature. The apparent peak shift with power density in Fig. 3(b) is opposite to what has been measured for AlGaAs samples under a controlled environment [5]. Fig. 3(c) indicates that the peak may shift to longer wavelength when a shorter pump wavelength is used. This trend, however, was not observed for most of the samples, and is not considered significant. It should also be noted that, although the data are not reported here in chronological order, no systematic change in the samples with time has been found.

Identification of the PL peak position can also contribute to the difference between reported values. To check this, the raw data taken on different instruments was analyzed by two different methods at NIST, using a fourth-order polynomial and an asymmetric bell curve [5]. The results of these fits along with the peak positions reported by the participants are shown for specimen 2 in Fig. 4. For instrument 1 PL peak wavelengths much higher than average were reported for specimens 1 and 2, but not for the other samples. As Fig. 4 shows, these are due to the peak identification method used. Measurements of these specimens by instrument 1 were made with a 20 nm step size, and the peak position was reported as the data point with the highest intensity. Using a peak fitting method brings these data into good agreement with the other results. The reason for the difference between the participant and NIST values for the data from instrument 8 has not been identified. Except for instruments 1 and 8, however, the small differences between peak wavelengths found by the different fit methods indicate that peak identification is not a major source of variation.

Although the PL measurements were made under a wide range of operating conditions, no definitive correlations with measurement offsets were found. However, since the sample temperature, spot size, and beam power were measured with different tools and differing degrees of precision for each instrument, this does not necessarily mean correlations do not exist. Independent measurements in a controlled environment are underway at NIST to assess the effect each of these variables has on the measured PL peak position. Identifying the critical PL parameters to control is especially important since PL is the industrial tool of choice for qualifying wafers.

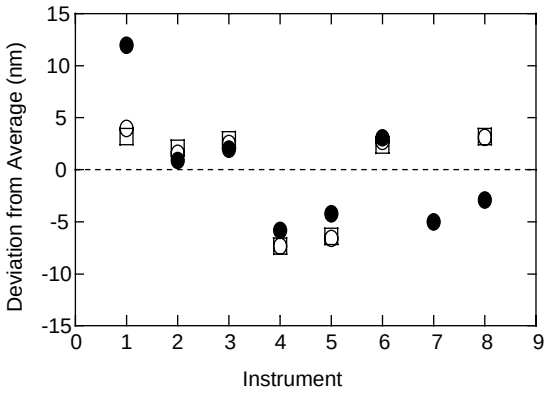

Fig. 4. The PL peak wavelength for specimen 2: reported by participants (●), from a fourth order polynomial fit (○), and from an asymmetric bell curve fit (□).

4. Summary

An interlaboratory comparison of ex situ characterization of 1 μm thick InGaAsP films by XRD and PL has been conducted. Lateral nonuniformity in the specimens appears to have dominated the variation in the XRD measurements, causing the results to be more sensitive to beam positioning and peak identification than to differences in other instrument and/or measurement conditions. Interestingly, although for XRD the variation across each 1 cm^2 specimen is larger than the maximum difference between measurements from different instruments, the PL variation across each sample was smaller than the variation between measurements and allowed differences between different measurement systems to be identified. Definitive correlations with wavelength calibration, sample temperature, pump power

density, incident wavelength, and peak identification method were sought but not found. Further study is underway to clarify these issues and to establish standardized assessment procedures.

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