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InGaAsP *EX-SITU* CHARACTERIZATION

A. Roshko and K.A. Bertness

Optoelectronics Division

National Institute of Standards and Technology, Boulder, Colorado, USA.

Abstract

An interlaboratory comparison has been undertaken of X-ray and photoluminescence measurements of InGaAsP on InP. Six 1 cm^2 specimens were measured, with nominal photoluminescence wavelengths of 1.1, 1.3 and $1.5 \mu\text{m}$ (2 of each). Preliminary maps of the specimens by X-ray and photoluminescence showed relatively large degrees of nonuniformity. This specimen nonuniformity appears to have dominated the variation in the X-ray measurements, making the results more sensitive to beam positioning and peak identification than to other instrument and/or measurement differences. The photoluminescence uniformity was sufficient to allow the variation between measurements by different instruments to be assessed. Offsets between measurements from different instruments were observed, and potential causes, such as pump wavelength, wavelength calibration, sample temperature, pump power density, and peak identification were identified.

I. Background

High accuracy characterization of InGaAsP is critical for the application of this material in lasers, detectors, and other telecommunications components. However, evidence suggests there are large variations in composition assessment between laboratories. In fact, several major optoelectronics manufacturers have identified lack of standardized assessment procedures as a major impediment to productivity. This paper reports on a study of the *ex-situ* characterization of InGaAsP materials for optoelectronics, being conducted with the goal of developing reliable measurement and data analysis methods.

II. Experimental

In this study X-ray diffraction (XRD) and photoluminescence (PL) of InGaAsP films are being examined. A single set of samples is being measured sequentially by industry, government and university laboratories to allow comparison of measurements. The specimens are 1 cm^2 pieces of uncapped, $1 \mu\text{m}$ thick InGaAsP layers deposited by MOCVD on slightly misoriented (001) InP substrates. There are six samples with three nominal photoluminescence wavelengths: 1.1, 1.3 and $1.5 \mu\text{m}$.

Initially two-inch wafers were grown for the study and their uniformity was determined from maps of the XRD peak separation and peak PL wavelength. A large degree of nonuniformity was found in the wafers. To minimize the impact of variations in the samples themselves, specimens of 1 cm^2 were cleaved from the

most uniform region of each two-inch wafer and participants were asked to make their measurements as close to the center of each specimen as possible.

The samples have been sent to eight laboratories for measurement. The extent of variations between measurements from different laboratories is being analyzed and means of minimizing it sought. An important aspect of the study is to distinguish variations intrinsic to the materials from those caused by the measurement systems or techniques. Measurement variables, such as temperature, spot size, beam power, sample alignment and resolution, and data analysis techniques, are being assessed with the goal of developing standardized procedures.

III. Results and Discussion

1) X-ray Diffraction

To date, measurements have been made with eight different XRD instruments. A fairly wide range of conditions has been used, including differences in the X-ray line, monochromator type, spot size, step size, count time and optimization method. The variation in separation between the substrate and epilayer peaks, measured for the six samples, is shown in Figure 1. This figure is a plot of the difference between the individual and average measurements for each sample.

As can be seen in Figure 1, instruments 1, 3, and 8 each appear to have a systematic offset. However, correlations of the data with the operating parameters listed above (X-ray line, monochromator, etc.) have not been found. Instead the difference between measurements appears to depend more on the specimen

measured, with some specimens showing much larger variations than others. In addition, although the data are not reported here in chronological order, no systematic change in the samples with time has been found.

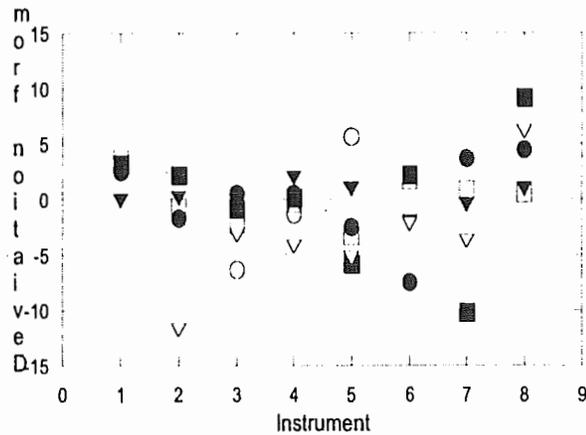


Fig. 1. Deviation of the XRD peak separation, measured using eight different instruments, from the average of the eight measurements for six samples: \square sample 1, \square sample 2, \square sample 3, \circ sample 4, \triangle sample 5, and \bullet sample 6. (The data point from instrument 1 for sample 6 is 24.7 and is out range for the graph.)

The standard deviation, the maximum variation between reported values, and the maximum variation converted to percent strain for each specimen are reported in Table I, along with the maximum variation measured by mapping each 1 cm² piece. As can be seen from the table, there is relatively good agreement among the X-ray data. For most of the specimens the maximum variation is 12 arcsec or less, and for all but one sample it is less than the variation measured over the 1 cm² specimen by the original mapping.

Table I. XRD Peak Separation Statistics (arcseconds)

| specimen | center std dev | center max var | % strain ¹ | map max var |
|----------|-------------------|-------------------|-----------------------|----------------|
| 1 | 5.9 | 19 | 0.0152 | 150 |
| 2 | 2.2 | 7 | 0.0055 | 9 |
| 3 | 3.9 | 12 | 0.0094 | 10 |
| 4 | 5.1 | 12 | 0.0094 | 80 |
| 5 | 1.3 | 4 | 0.0031 | 50 |
| 6 | 11.1 | 36.5 | 0.0287 | 51 |

¹ Calculated using: % strain = $7.86 \times 10^{-4} \times$ (max diff).

For half of the samples (2, 3 and 6) there was disagreement among the measurements as to whether the peak separation was positive or negative. This is a result of the substrate peak not being clearly identifiable. Because the InGaAsP layers are 1 μ m thick their peak intensity is similar to that of the substrate peak. In addition, in some samples the substrate and epitaxial layer peaks are closely spaced and it can be difficult to distinguish them. This results in uncertainty as to

whether the strain in the films is tensile or compressive. 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. As can be seen from the table, the measurements for two of these samples (2 and 3) are in good agreement regarding the peak separation; it is only the sign of the separation that is in question.

It is anticipated that identification of the substrate peak will be less of a problem for device structures than for these test specimens in which the epitaxial layer is very thick. If it were an issue in a device wafer, however, the indeterminate nature of the sign of the separation could lead to major difficulties. For example, if the sign is incorrectly identified for one or more spots on a wafer, this error can create the appearance of nonuniformities in the wafer that are not actually present. This can be especially problematic for automated mapping systems where the instrument determines the peak positions independent of input from adjacent data points, or where the map density is too low for adjacent data points to provide guidance.

For two of the specimens, 4 and 6, it was reported that there were more than two peaks in the X-ray spectra and that their overlap made the measurements difficult. For sample 4 there were four reports of a peak separated from the substrate peak by ~ 410 arcsec. These are the data plotted in Figure 1 and evaluated in Table I. The agreement between these four measurements is fairly good; the standard deviation is ~ 5 arcsec and the maximum deviation is 12 arcsec. For sample 6, although it was reported that there were several peaks, only one peak separation was reported for each instrument, and the deviation in these is much larger than that of the other measurements. These variations are the result of multiple phases in the materials. We have tried to minimize the problem of inhomogeneity in this study by using relatively small specimens with the maximum possible uniformity. But inhomogeneity remains a very real issue for the industry.

The other sample for which uniformity is a major problem is specimen 1. Although the measurements for this specimen have the second largest variation (19 arcsec), the difference is small compared with the variation measured in a map of the 1 cm² piece (150 arcsec). For samples such as 1, 4, 5 and 6, where large variations across the specimen were found by mapping, the ability of the instrument and/or operator to position the measurement beam in the center of the sample can have a large effect on the results. Although part of this can be compensated for by the fairly large spot sizes used for XRD (typically from 1 to 10 mm²), it is likely that in some cases (such as for specimen 1) the extremely large sample variation still dominates the measurement scatter. In addition, the existence of more than one epilayer peak in several of these samples complicates the XRD analysis. It seems likely, therefore, that the variation (7 arcsec) between measurements on the most uniform sample (sample 2),

which had only two peaks in the XRD patterns, is indicative of the true variation between measurements on different instruments.

2) Photoluminescence

One might expect problems due to sample uniformity to be similar or worse for PL measurements, since the spot diameter for PL is typically much smaller (≤ 0.5 mm) than that for XRD. However, mapping of the 1 cm^2 specimens revealed relatively small variations in the peak wavelengths, as shown in the last column of Table II. Also given in the table are the standard deviation and maximum variation between reported peak wavelengths from PL measurements made on each specimen with eight different systems.

Table II. PL Peak Wavelength Statistics (nm)

| specimen | center std dev | center max var | map max var |
|----------|-------------------|-------------------|----------------|
| 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 | 5.8 | 16.7 | 6 |
| 2* | 3.6 | 8.9 | |
| 1* | 4.5 | 12.7 | |
| 6* | 2.3 | 6.3 | |

Entries with * recalculated as described in text.

Unlike the XRD measurements, the variation in the PL measurements at the center of each specimen is larger than the variation found by mapping the 1 cm^2 piece, for all of the samples. This suggests that the variations are indicative of true differences between the measurements, rather than the result of sample nonuniformity. The deviations of the peak wavelengths from their average, determined for the six samples, are shown in Figure 2.

As can be seen in the figure, there appear to be offsets in the data taken with some of the instruments. The results for all samples from instrument 7 are lower than the average, while those from instrument 6 are all higher than average. Likewise, the results from instrument 8 are mostly lower and the results from instrument 2 are mostly higher than average. These systematic variations may indicate a calibration difference between the instruments. All but two of the instruments, were reported to have been wavelength calibrated within three weeks of the measurement. Other potential sources of offset include: the sample temperature during measurement, sample heating by the pump beam, and the fitting of the PL data to obtain the peak wavelength.

Participants were asked to report several of these parameters for the study, so it was possible to look for relationships with the PL peak data. No definitive correlations 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 affect each of these variables has on the measured PL peak position.

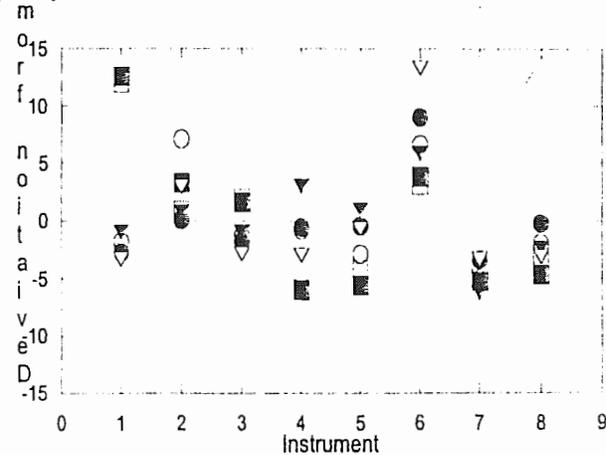


Fig. 2. Deviation of the PL peak wavelength measured using eight different instruments, from the average of those eight measurements for six samples: \blacktriangle sample 1, \square sample 2, \square sample 3, \circ sample 4, \triangle sample 5, and \circ sample 6.

It is also interesting to note that instrument 1 has much higher than average values for specimens 1 and 2 but not for the other samples. The measurements on this instrument were made with a pump wavelength ($1.060 \mu\text{m}$) close to the nominal luminescence wavelength ($1.1 \mu\text{m}$) of specimens 1 and 2. In these cases, since the pump energy is close to the bandgap energy, there will be less absorption of the pump beam resulting in a lower carrier density and a lower Fermi level. This in turn will lead to a lower energy luminescence peak due to lack of filling of higher energy levels. The standard and maximum deviations for specimens 1 and 2, if the data from instrument 1 are excluded, are given in the lower part of Table II, indicated with asterisks. These values are similar to the variations found for the other specimens.

Specimen 6 also has one measurement that is much higher than the average. The standard and maximum deviations for this sample, if the results from instrument 6 are ignored, are also given in the lower part of Table II, marked with an asterisk. In this case, however, the reason for the difference in the instrument 6 measurement is not obvious. As mentioned earlier, instrument 6 appears to have an offset relative to the other instruments. However as measured with instrument 6, the relative PL maximum for specimen 6 is much higher than the relative maximum for specimen 5, which has nominally the same emission wavelength. This indicates that the offset is not solely responsible for the high value. Specimen 6 was also reported to have multiple XRD peaks. It is possible, therefore, that the

offset of the peak maximum of specimen 6 measured with instrument 6 results from a combination of the instrument offset and sample nonuniformity.

Even if these exceptionally different data points are eliminated from the analysis, the remaining data still has a larger variation than the variation found by mapping each 1 cm² piece (see Table II). The goal of this study is to identify which of the measurement variables are responsible for this variation. Similar to the XRD measurements, the PL measurements were made under a wide range of operating conditions. Parameters that varied include the pump wavelength, spot size, power density, and wavelength resolution. In addition, several different methods were used to find the peak maximum. Since the PL peaks are asymmetric and, therefore, more difficult to fit than the symmetric X-ray peaks, this may also contribute to the differences in the PL data. As mentioned earlier, independent measurements in a controlled environment at NIST are underway to assess the affect each of these variables has on the PL peak position. Identifying the critical PL parameters to control is especially important since PL is the industry tool of choice for qualifying wafers.

IV. Summary

Measurements from several different instruments, of InGaAsP layers, have shown interesting trends. X-ray measurements are in fairly good agreement, with less variation found between measurements from different

instruments than in maps of 1 cm² specimens. The major difficulties identified for X-ray measurements are associated with sample inhomogeneity and distinguishing the substrate peak from the epilayer peak(s). In contrast, most of the variations among the PL measurements can be attributed to either instrument and/or technique differences. There are several parameters in the PL systems that could contribute to the variation, including wavelength calibration, sample temperature, pump power density, and identification of the peak maximum. Systems with possible offsets, due in one case to the pump wavelength, were identified. Further study is underway to clarify these issues and to establish standardized assessment procedures.

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