

Ex-situ Characterization of InGaAsP*

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Abstract An interlaboratory comparison was undertaken to assess differences in X-ray and photoluminescence measurements from different laboratories. Six InGaAsP specimens, with nominal photoluminescence peak wavelengths of 1.1, 1.3 and 1.5 μm , were measured. Lateral nonuniformity in the specimens dominated the variation in the X-ray measurements. In contrast, the variation among photoluminescence measurements was larger than the variation within individual specimens and allowed differences between measurements to be assessed. Offsets between different instruments were identified, however correlations with variables such as pump wavelength, wavelength calibration, sample temperature, pump power density, and peak identification, as reported by the different laboratories, were not observed.

1. Introduction

High accuracy characterization of InGaAsP is critical for the application of this material in lasers, detectors, and other telecommunication components. Evidence suggests, however, that there are large variations in composition determination between laboratories. This has been observed even in the relatively well-characterized AlGaAs system [1]. Lack of standardized assessment procedures has been identified as an impediment to productivity by several major optoelectronics manufacturers [2]. As a first step in addressing this issue, an interlaboratory comparison has been made of *ex-situ* measurements of InGaAsP films.

2. Experimental Procedure

The specimens studied were uncapped, 1 μm thick InGaAsP layers on slightly misoriented, (001) InP substrates. The layers were grown by organometallic vapor phase epitaxy (OMVPE) on 51 mm (2 inch) wafers. The specimens had nominal photoluminescence (PL) peak wavelengths of 1.1 μm (samples 1 and 2), 1.3 μm (samples 3 and 4) and 1.5 μm (samples 5 and 6). Maps of X-ray rocking curve peak separation and PL peak wavelength across each wafer showed substantial lateral variations. To minimize the effect of these variations on the study, specimens of 1 cm^2 were cleaved from the most uniform region of each wafer and participants were asked to make their measurements at the center of each specimen.

The samples were measured with eight different X-ray diffraction (XRD) instruments and eight different PL systems. Participants were asked to report the conditions of their measurements such as temperature, spot size, beam power, wavelength, resolution, and data analysis technique. These were tracked in an effort to identify which have the greatest influence on the measurement results. No measurements by NIST were included in the comparison. More details of the samples and measurements are given in reference 3.

3. Results and Discussion

The statistics of the measurements from eight XRD instruments and eight PL systems are given in Table I. For each specimen the maximum variation in measurements mapped across the 1 cm^2 piece is given, in column 4 for XRD and in column 7 for PL. The other columns give the standard deviation of and the maximum deviation (max - min) between the measurements from the different instruments for each specimen.

It can be seen that, for the XRD measurements, the variation across each sample is larger than the standard deviation between the measurements from different instruments. In all but two cases, the lateral sample variation is significantly larger than the maximum variation between the measurements. This suggests that the differences between the measurements may result from differences in the positioning of the X-ray beam on the sample. Aligning a specific spot on a sample under the beam is difficult in many X-ray systems, and most of the study participants did not have experience with this.

Table I. Measurement Statistics

specimen	XRD peak separation (arcsec)			PI peak wavelength (nm)		
	std dev. of instrument measurements ¹	max dev. of instrument measurements ¹	max lateral variation ²	std dev. of instrument measurements ¹	max dev. of instrument measurements ¹	max lateral variation ²
1	5.9	19	150	6.6	23.0	3
2	2.2	7	9	5.9	17.8	2
3	3.9	12	10	3.8	12.4	2
4	5.1	12	80	4.4	12.2	2
5	1.3	4	50	3.6	12.1	4
6	11.1	36.5	51	6.6	16.7	6

¹ Variation between measurements from eight instruments, at the center of the specimen.

² Measured with one instrument over the entire 1 cm² specimen.

Table I also shows that the variation in the PL measurements, unlike that in the XRD measurements, is larger than the variation across the individual samples. This suggests that the differences result from differences in the instruments and/or conditions used, rather than from sample nonuniformity. Some of the instruments were found to have wavelength offsets; the results for instrument 7 are all lower than average and those for instrument 6 are higher [3]. These systematic differences may indicate a difference in calibration. All but two of the instruments (1 and 6) were calibrated within three weeks of the measurements. Other potential sources of offset include the measurement conditions and the peak identification method used.

The variation of the PL peak wavelength, measured for the different samples, is plotted as a function of the reported measurement temperature in Fig. 1. No systematic variation with temperature is evident. Since the bandgap of InGaAsP decreases with increasing temperature [4], it might be expected that the measurements would shift to longer wavelengths at higher measurement temperatures. There is some indication of this behaviour, especially for samples 1 and 2. As will be described, however, this apparent change in peak wavelength is larger than would be expected due to the seven degree change in temperature.

The shift in PL peak wavelength with temperature has been measured for AlGaAs [5]. For Al_{0.3}Ga_{0.7}As near room temperature, a 10 degree change in temperature was found to shift the photoluminescence peak wavelength only 3 nm [5]. The bandgap of Al_{0.3}Ga_{0.7}As [6] has a dependence on temperature similar to but slightly higher than that of InGaAsP [4]. Thus, it would be expected that the PL peak for InGaAsP would shift only 3 nm or less for a 10 degree change in temperature. It seems unlikely, therefore, that the 12 to 23 nm differences in peak position found for measurements in this study are solely due to temperature differences.

The change of the PL peak position with incident power density is plotted in Fig. 2. Here again there is no obvious correlation, although there is a slight trend for the peak to shift to shorter wavelengths at increased power density. This is opposite to what would be expected if the primary effect of increased power were to heat the sample, in which case higher power densities would shift the PL peak to longer wavelengths. Such a shift, to longer wavelengths with increased power, has been observed for AlGaAs [5].

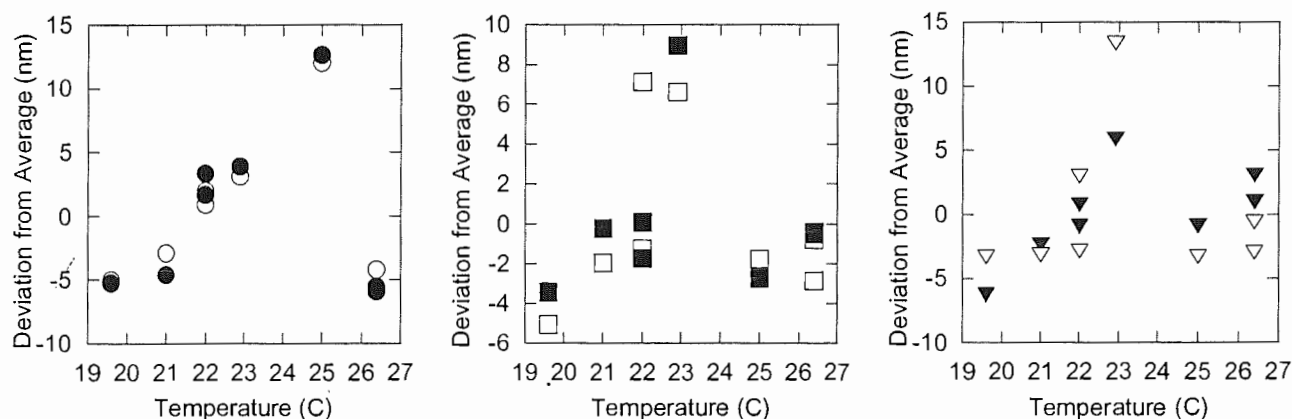


Fig. 1 Deviation of the PL peak wavelength from the average, as a function of the measurement temperature reported by the participants, for the six samples: ● sample 1, ○ sample 2, ■ sample 3, □ sample 4, ▼ sample 5, and ▽ sample 6.

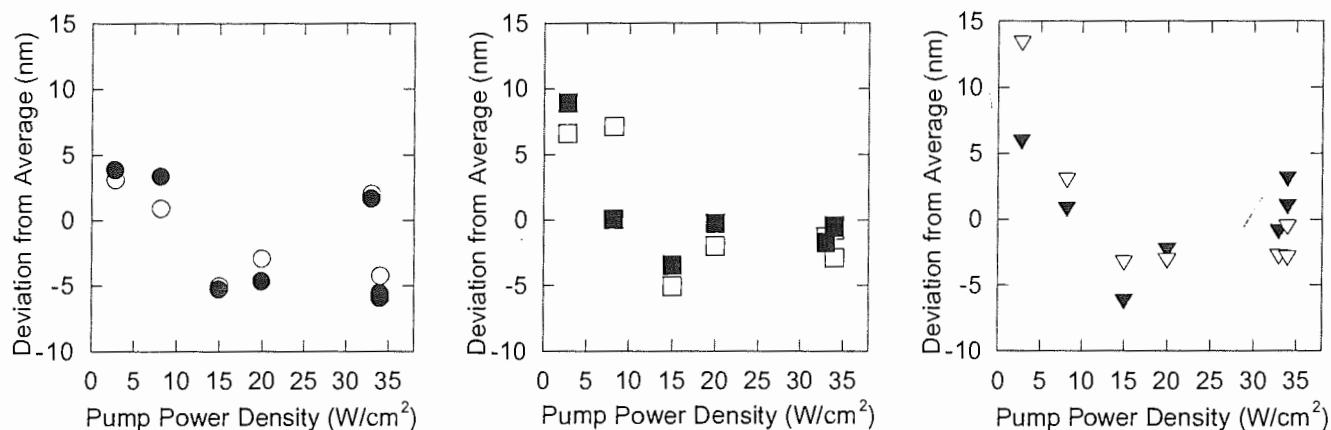


Fig. 2 Deviation of the PL peak wavelength, from the average, as a function of the pump power density reported by the participants, for the six samples: ● sample 1, ○ sample 2, ■ sample 3, □ sample 4, ▼ sample 5, and ▽ sample 6.

As already mentioned, the PL peak identification method may also contribute to offsets. Several different methods of peak identification were used by the participants. To test the influence of this, raw data, supplied by the participants, was fit at NIST using two different methods: a fourth-order polynomial and an asymmetric bell curve [5]. For all but two of the instruments there was little difference in the peak identified using different methods [3]. The largest difference was found when the data point with the maximum intensity was reported to be the peak for samples that were measured with a 20 nm step size. This method did not produce such a problem when the measurement step size was reduced to 2 nm. Overall, small differences were found between the peak wavelengths identified by different methods, indicating that peak identification is not a major source of variation.

No definitive correlations were found between measurement conditions and PL peak position. Since the variables were measured in different labs with different tools and differing degrees of precision, this does not necessarily mean that real correlations do not exist. Future work at NIST will address this through systematic measurements in a controlled environment. Future work will also be directed at obtaining samples with laterally uniform XRD. Fairly thick InGaAsP layers were used for this study, in order to eliminate complications due to thickness fringes in the spectra. It may be necessary to use thinner samples in the future.

4. Summary

Ex-situ characterization of 1 μm thick InGaAsP films by XRD and PL has been investigated through an interlaboratory comparison. Lateral nonuniformity in the specimens dominated the variation in the XRD measurements, causing the results to be more sensitive to beam positioning than to measurement differences. The PL variation across each sample was smaller than the variation between measurements and allowed differences between 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.

Acknowledgements

The authors acknowledge the NIST Advanced Technology Program for funding this work and thank the industrial laboratories that participated in the interlaboratory comparison: Agere, Agilent, IQE, JDS Uniphase, and Nortel.

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