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# Combination of electron energy-loss spectroscopy and energy dispersive X-ray spectroscopy to determine indium concentration in InGaN thin film structures

X Wang<sup>1</sup>, MP Chauvat<sup>2</sup>, P Ruterana<sup>2</sup> and T Walther<sup>1\*</sup>

<sup>1</sup>Department of Electronic and Electrical Engineering, University of Sheffield, Mappin Building, Mappin Street, Sheffield S1 3JD, UK

<sup>2</sup>CIMAP, UMR 6252, CNRS-ENSICAEN-CEA-UCBN, 14050 Caen, cedex, France

\*Email: [t.walther@sheffield.ac.uk](mailto:t.walther@sheffield.ac.uk)

**Abstract:** We demonstrate a method to determine the indium concentration,  $x$ , of  $\text{In}_x\text{Ga}_{1-x}\text{N}$  thin films by combining plasmon excitation studies in electron energy-loss spectroscopy (EELS) with a novel way of quantification of the intensity of X-ray lines in energy-dispersive X-ray spectroscopy (EDXS). The plasmon peak in EELS of InGaN is relatively broad. We fitted a Lorentz function to the main plasmon peak to suppress noise and the influence from the neighbouring Ga 3d transition in the spectrum, which improves the precision in the evaluation of the plasmon peak position. As the indium concentration of InGaN is difficult to control during high temperature growth due to partial In desorption, the nominal indium concentrations provided by the growers were not considered reliable. The indium concentration obtained from EDXS quantification using Oxford Instrument ISIS 300 X-ray standard quantification software often did not agree with the nominal indium concentration, and quantification using K and L lines was inconsistent. We therefore developed a self-consistent iterative procedure to determine the In content from thickness-dependent  $k$ -factors using, as described in recent work submitted to Journal of Microscopy. When the plasmon peak position is plotted versus the indium concentration from EDXS we obtain a linear relationship over the whole compositional range, and the standard error from linear

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3 least-squares fitting shows that the indium concentration can be determined from the plasmon  
4 peak position to within  $\Delta x = \pm 0.037$  standard deviation.  
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## 7 8 **1. Introduction**

9 III-V nitride semiconductor materials have great potential for optoelectronics due to their  
10 direct and adjustable band-gap and their relative insensitivity to dislocations [1]. During the  
11 past decade, several types of Light Emitting Diodes (LED) and Laser Diodes (LD) have been  
12 fabricated based on  $\text{In}_x\text{Ga}_{1-x}\text{N}$  quantum wells or nanowires [2, 3]. Wurtzite structure GaN and  
13 InN have direct band-gaps of 3.4eV and 0.7eV, respectively, so  $\text{In}_x\text{Ga}_{1-x}\text{N}$  compounds cover  
14 the band-gap range from 0.7eV to 3.4eV, which includes emission wavelengths corresponding  
15 to red, green and blue light. As the indium concentration changes, a morphological instability  
16 may be induced during growth of nanowires [3] or thin films. In order to clarify the effect of  
17 indium concentration on growth and optical properties, a reliable and highly accurate  
18 determination of indium concentration in  $\text{In}_x\text{Ga}_{1-x}\text{N}$  will be required.  
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20 As Narukawa *et al.* [4] suggested in 1997, transmission electron microscopy (TEM) and  
21 energy dispersive X-ray spectroscopy (EDXS) techniques can be used to determine the  
22 localization of excitons at deep traps originating from In-rich regions in the quantum wells [5],  
23 however, due to the image observed in TEM being essentially a result of averaging through  
24 the thickness of the sample, a quantitative determination of the indium composition is difficult  
25 to obtain from conventional TEM [2]. In order to solve this problem, we have combined a  
26 self-consistent iterative procedure developed for EDXS in TEM with plasmon energy  
27 measurements by electron energy-loss spectroscopy (EELS). [As the scattering cross-section  
28 for plasmon scattering is much higher than that for ionization core-losses \(K, L or M\), our  
29 EELS experiments could be conducted at dose levels sufficiently low to neglect  
30 beam-induced changes of the In/Ga ratio in InGaN. In \[3\] such studies have already been  
31 performed, however, mostly confined to the compositional range  \$0 \leq x \leq 0.5\$ .](#)  
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## 39 **2. Experimental**

### 40 **2.1 $\text{In}_x\text{Ga}_{1-x}\text{N}$ thin film growth**

41 The investigated InGaN samples have been grown by metalorganic vapour phase epitaxy  
42 (MOVPE) in a close-couple shower head AIXTRON reactor using trimethyl-gallium (TMGa),  
43 trimethyl-indium (TMIn) for the metals and  $\text{NH}_3$  for nitrogen. Three important parameters have  
44 been particularly investigated and monitored for the control of the indium incorporation:  
45 growth temperature, chamber pressure and III/V ratio. The growth of highest indium content  
46 layers has been carried out at the lowest temperature of 550°C, as well as highest III/V ratio  
47 (>40000). For the layers with In composition below 25%, the growth temperature could be  
48 above 700°C. The nominal indium concentration was first assessed by high resolution X-ray  
49 diffraction (XRD). For TEM investigation, cross-sectional specimens were prepared by  
50 conventional grinding and dimpling, followed by ion milling. In order to minimize the ion  
51 beam damage, the samples were maintained at liquid nitrogen temperature during ion milling  
52 using a Gatan PIPS at 5 keV, with a final **polishing** step at 0.6 keV. During the whole procedure,  
53 the beam angle was set at 5° for the two guns. Prior to analytical TEM by EDXS and EELS,  
54 conventional TEM investigations were performed by high resolution TEM using a JEOL 2010  
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3 microscope, for defect investigation in order to determine the strain relaxation mechanisms in  
4 these layers which may involve the formation of typical defects such as dislocations [6, 7, 8],  
5 stacking faults [9, 10, 11] and, in the worst cases, inversion domains [12, 13] as well as phase  
6 ordering and phase separation [14, 15].  
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## 10 **2.2 EDXS and EELS characterization**

11 Analytical TEM was carried out on a Schottky field-emission JEOL 2010F transmission  
12 electron microscope operated at 197kV (this voltage allows the user to increase the high  
13 tension by up to 3kV for energy-filtered imaging, cf. [16]). The microscope is equipped with a  
14 Gatan Imaging Filter (GIF 200) that allows for an energy resolution of  $\sim 0.9$  eV, measured as  
15 the full width at half-maximum (FWHM) of the zero-loss peak (ZLP). All spectra were  
16 collected in diffraction mode with a collection semi-angle of  $\sim 20$  mrad using a dispersion of  
17  $0.0502$  eV per channel (calibrated by drift tube offsetting),  $\sim 5$  nA beam current and  $\sim 50$  nm  
18 probe size to avoid electron beam damage of the sample. Several spectra were recorded for  
19 each thin film sample, from different regions of different specimen thickness. At this high  
20 dispersion, the spectrometer dispersion is constant over the whole field of view, as confirmed  
21 earlier by others [17], and drift of the high tension or the magnetic prism strength is not a  
22 problem as the zero loss is recorded within all low loss spectra and it is only the distance of  
23 the plasmon peak from the zero loss peak that matters.  
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29 For EDXS, an Oxford Instruments Si:Li detector with ultrathin window and the slowest pulse  
30 processor settings in the ISIS300 software package were used. The energy resolution of the  
31 EDXS system depends on the X-ray energy as well as on the pulse processor settings and  
32 varies between  $60$  eV (FWHM of *strobe*) and  $136$  eV (FWHM of Mn  $K\alpha$  at  $5895$  eV). In some  
33 cases, a Cu signal was observed due to the copper support, however, this did not influence  
34 quantification. Care was taken to avoid strong diffraction conditions that are known to  
35 influence the X-ray yields, particularly near zone-axis and two-beam conditions [18]. Tilting  
36 the layers by a few degrees off the zone axis while keeping them almost edge-on does not  
37 cause a problem as long as the beam ( $\sim 50$  nm diameter) remains well contained within the  
38 layers ( $>80$  nm thick).  
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## 43 **3. Result and Discussion**

44 A typical low loss EEL spectrum of an  $\text{In}_x\text{Ga}_{1-x}\text{N}$  thin film is shown in Fig 1. Two main  
45 features are observed: the zero-loss peak and the bulk plasmon excitation. The plasmon peak  
46 position has been used to determine the Al concentration in  $\text{Al}_x\text{Ga}_{1-x}\text{N}$  thin film samples [19,  
47 20] and the In concentration in  $\text{In}_x\text{Ga}_{1-x}\text{N}$  [3]. For the latter, however, no calibration points for  
48 the range  $0.5 < x < 1$  were available, so the quality of the linear fit proposed therein could be  
49 questioned.  
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53 For determining the plasmon peak energy highly accurately, Fourier logarithm deconvolution  
54 should be applied to remove the plural scattering [21]. Figure 1(a) shows the comparison of  
55 original low loss EELS and deconvoluted spectra of an  $\text{In}_{0.54}\text{Ga}_{0.46}\text{N}$  thin film. The  
56 deconvolution method changes the height of the plasmon peaks but has no effect on the  
57 plasmon peak energy position, which indicates the specimens are almost ideally thin for  
58 EELS. Both deconvoluted and original EELS contain a high level of noise. Therefore, a  
59 Lorentzian based least-squares regression analysis has been applied to fit the plasmon peak.  
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Figure 1(b) depicts the processed low-loss EELS of  $\text{In}_{0.54}\text{Ga}_{0.46}\text{N}$  thin film, and the peak position  $E_{\text{max}}$  can be obtained with sufficiently high accuracy. Figure 1(c) demonstrates how the plasmon peak evolves and shifts from pure GaN, over two different ternary InGaN alloys, to pure InN.

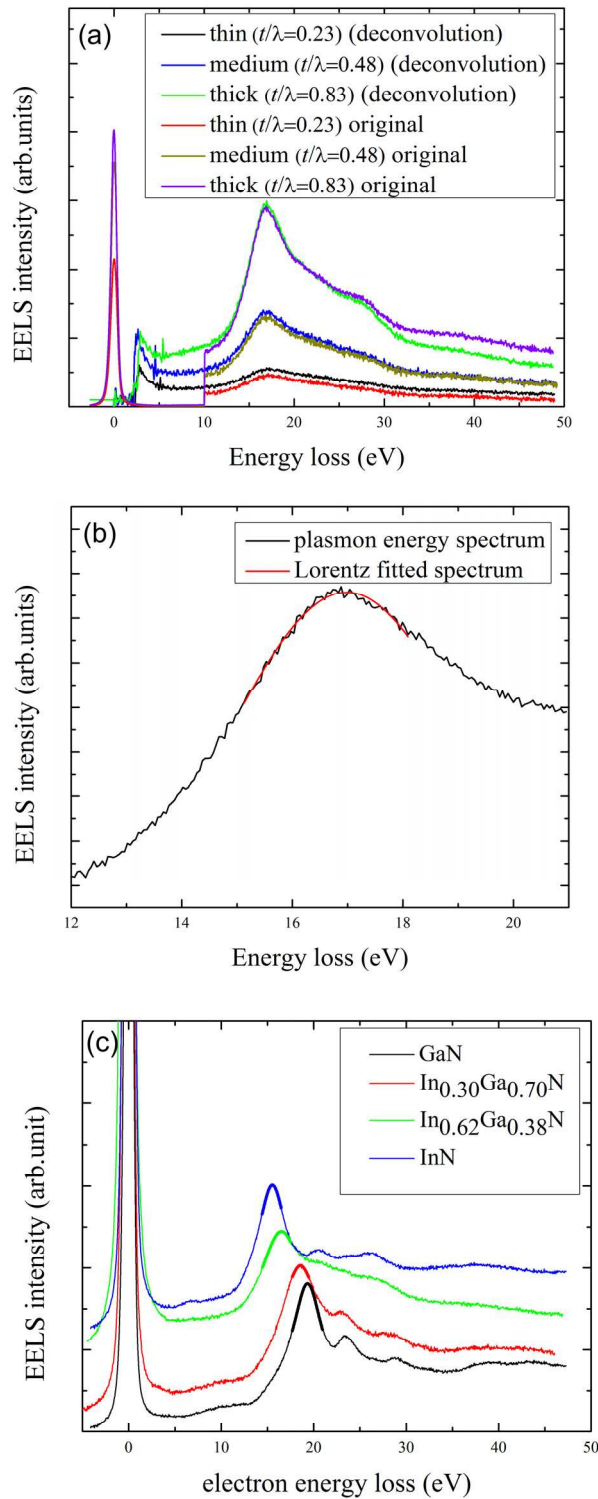


Figure 1: (a) comparison of original and deconvoluted EELS spectrum of  $\text{In}_{0.54}\text{Ga}_{0.46}\text{N}$  thin

film ( $t/\lambda = 0.23, 0.48, 0.83$  where  $\lambda$  denotes the inelastic mean free path). (b) Lorentz curve fitted to central plasmon peak for  $\text{In}_{0.54}\text{Ga}_{0.46}\text{N}$  sample with  $t/\lambda = 0.83$ . (c) Comparison of plasmon peak shift from pure GaN, over two ternary InGaN alloys, to pure InN demonstrates how the peaks evolve. Raw data are displayed and the regions for the optimal Lorentz fits are highlighted in bold.

The EELS experiments were repeated three times for each specimen, using different specimen thicknesses. Then, a weighted average energy was calculated (the ideal specimen thickness for optimal signal-to-noise ratio in an EELS for a single plasmon excitation is given by  $t = \lambda$ ). Table 1 shows the weighted average values of plasmon peak energies for different nominal indium concentrations.

nominal indium concentration, $x_{\text{nominal}}$	weighted average plasmon peak energy (eV)
0	19.33±0.01
0.135	18.69±0.04
0.20	18.42±0.01
0.30	18.27±0.01
0.40	17.66±0.05
0.54	17.00±0.02
0.62	16.76±0.04
0.74	16.14±0.03
0.84	15.88±0.02
1	15.52±0.01

Table 1: weighted average value of plasmon peak energy as function of nominal indium concentration

As the nominal concentration provided by the growers seemed not reliable, we used an additional EDXS analysis to check the indium concentration for each sample. For sufficiently thin samples, the Cliff-Lorimer  $k$ -factor provides a useful approach to determine the indium concentration of a thin film. For thicker specimens, either an additional absorption factor needs to be included or a thickness-dependent  $k_{\text{In,Ga}}^*$  factor be defined which effectively includes absorption and fluorescence effects if the above intensities are recorded from thicker specimen regions [22].

$$k_{\text{In,Ga}}^* = \frac{x I_{\text{Ga}} A_{\text{In}}}{(1-x) I_{\text{In}} A_{\text{Ga}}} \quad (1)$$

where  $k_{\text{In,Ga}}^*$  is the effective  $k$ -factor for weight percent of the In L-line with respect to a Ga line (K or L),  $x$  is the indium concentration,  $I_{\text{Ga}}$  and  $I_{\text{In}}$  are the intensities of Ga and In lines, respectively, and  $A$  are the atomic weights of the corresponding elements.

We have performed Monte Carlo simulations for X-ray generation and detection using the CASINO code [23] to calculate theoretical  $k_{\text{In,Ga}}^*$  values and to compare them to our experiments.

For indium concentrations  $x > 0.5$ , simulations suggest the experimental curves of  $k_{\text{In,Ga}}^*$  vs. Ga K/L ratio should be similar and ideally equal to the theoretical curve. By using the modeled

$k_{In,Ga}^*$  curves and experimental intensities  $I_{Ga}$  and  $I_{In}$ , a new indium concentration  $x$  can be obtained from equation (2). Then the  $k_{In,Ga}^*$  simulation is repeated for this updated indium concentration  $x$  to provide an updated value for  $k_{In,Ga}^*$ , etc. If this is iterated, then the solution for **each** spectral measurement converges to exactly one combination of  $k_{InL,GaL}^*$ ,  $k_{InL,GaK}^*$  and  $x$ , yielding a self-consistent solution for quantification using Ga L or Ga K lines.

$$x = \frac{1}{1 + \frac{I_{Ga} * A_{In}}{I_{In} * A_{Ga} * k_{InGa}^*}} \quad (2)$$

Figure 2 plots the new calibrated indium concentration versus the nominal concentration.

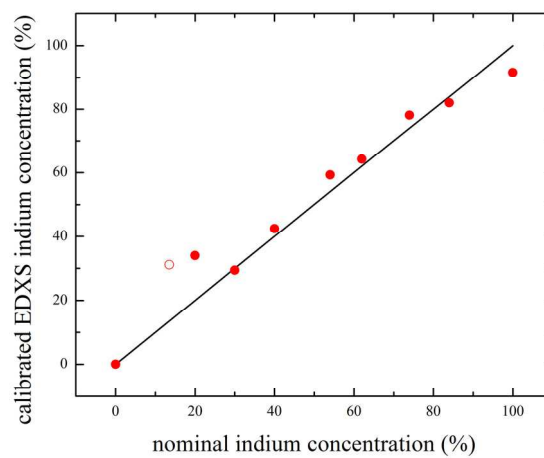


Figure 2: new calibrated indium concentration versus nominal indium concentration. For one sample only (marked by an open symbol) the iteration did not converge properly for both GaL and GaK line quantification, and the intensity ratio of Ga K/L was lower than modeled so this point has been excluded from further analysis.

We did not observe any strain effects on the plasmon peak position, as could be expected for biaxially strained thin quantum wells, however, for strained InGa<sub>N</sub> layers only a few nanometers thin it is almost impossible to disentangle the effects of strain and compositional gradients [24] and our layers were much thicker and partially relaxed.

The relationship between plasmon peak energy and this newly determined indium concentration is shown in figure 3. The relationship between plasmon peak energy and indium content is linear and can be expressed as

$$E_{max}[\text{eV}] = (19.39 \pm 0.06) - (4.02 \pm 0.11)x$$

where  $x$  is the indium concentration and  $E_{max}$  is the plasmon peak energy. The adjusted  $R^2$  ( $R^2=0.9845$ ) confirms the plasmon peak energy versus calibrated indium concentration is linear over the complete compositional range  $0 < x < 1$ , with an uncertainty in the indium concentration (random mean-square error from linear regression) of  $\Delta x = \pm 0.037$ , which indicates an improved accuracy in the determination of indium concentration of InGa<sub>N</sub> compared to previous studies [3].

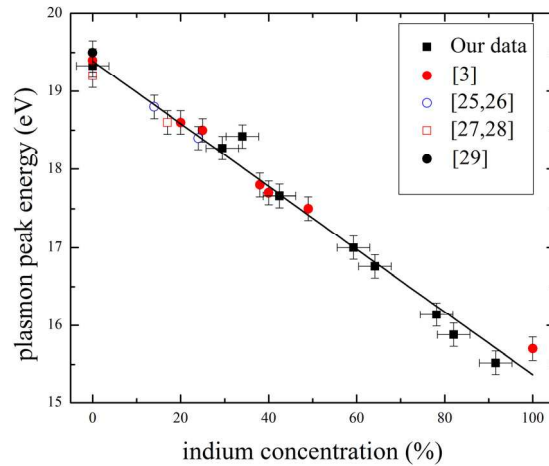


Figure 3: dependence of plasmon peak position on measured indium concentration in InGaN, including our data as well as data from other groups [3, 25-29]. The black line is the linear least-squares regression fit to all data.

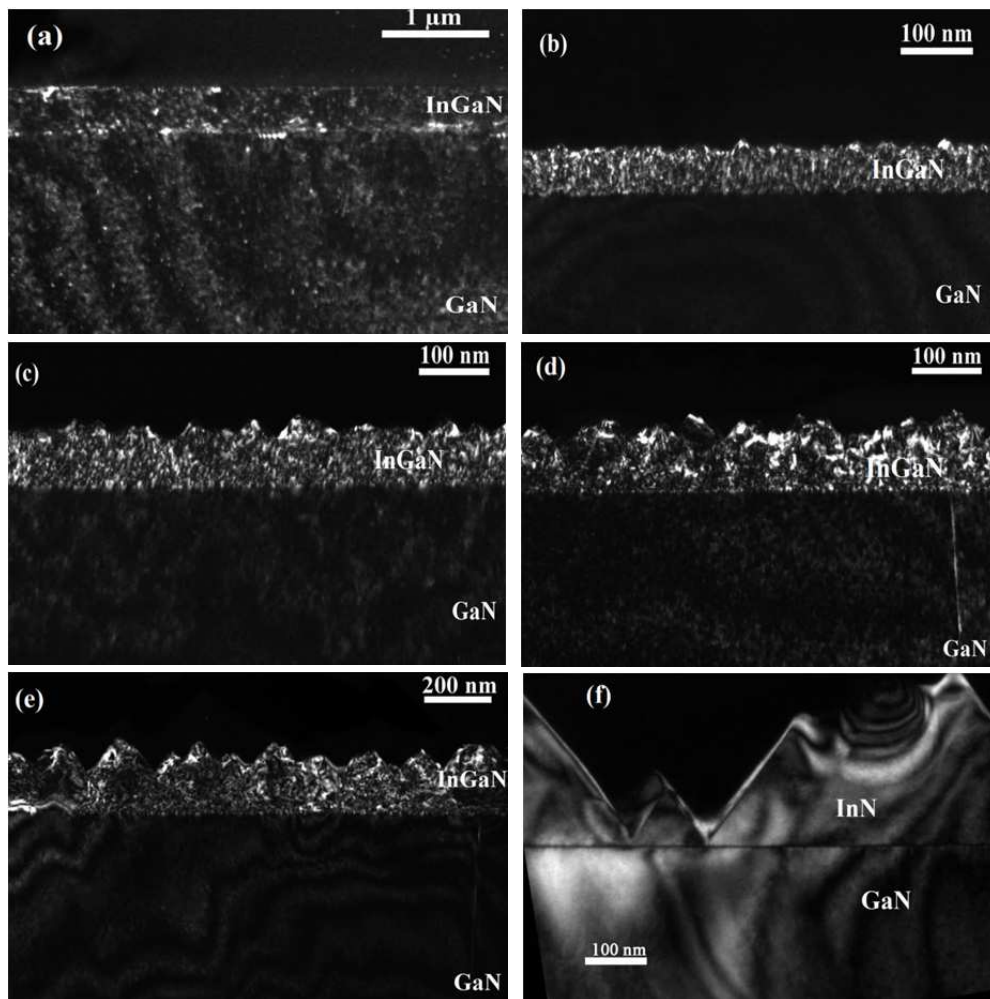


Figure 4:  $g=0002$  weak beam image of (a)  $\text{In}_{0.34}\text{Ga}_{0.66}\text{N}$ , (b)  $\text{In}_{0.42}\text{Ga}_{0.58}\text{N}$ , (c)  $\text{In}_{0.59}\text{Ga}_{0.41}\text{N}$ , (d)  $\text{In}_{0.64}\text{Ga}_{0.36}\text{N}$ , (e)  $\text{In}_{0.78}\text{Ga}_{0.22}\text{N}$ , (f)  $\text{In}_{0.92}\text{Ga}_{0.08}\text{N}$  (InN).



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4 The increase of the indium concentration in InGaN quantum wells often leads to the  
5 formation of V-type defects [30]. Denser V-type defect formation will lead to a higher level  
6 of surface roughness. Figure 4 shows some  $g=[0002]$  weak beam overview images of the  
7 different indium concentration samples investigated in the present study. From the  
8 investigation, it has been noticed that for low indium concentration ( $x < 0.3$ ), even 400nm thick  
9 films of InGaN have a smooth surface (see fig. 4a). When the indium concentration increases,  
10 the surface roughness increases dramatically and layer crystalline quality degrades as shown  
11 in figures 4 (b)-(e). However, for pure InN growth on GaN, the thin film is approximately  
12 100-300nm thick and consists of faceted islands of pyramidal forms, as shown in figure 4 (f),  
13 but it exhibits a perfect crystalline quality, in contrast to the In rich ternary alloys.  
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#### 18 4. Conclusion

19 We have studied the influence of indium content,  $x$ , of  $\text{In}_x\text{Ga}_{1-x}\text{N}$  thin films on EELS plasmon  
20 peak energy over the whole compositional range  $0 < x < 1$ . Our study confirms that the plasmon  
21 peak energy has a linear relationship on the indium concentration in the InGaN ternary system.  
22 By a combination of plasmon peak energy fitting and EDXS as a calibration tool to check the  
23 nominal indium content, the reliability in determination of the **absolute** indium concentration  
24 from EELS has been improved to  $\Delta x = 0.037$ .  
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