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Manual analysis: When software is useless

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Manual analysis:

When software is useless

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Overview

- Introduction
- Example 1: Comparison of simulations with data not accurate
- Example 2: Models not good enough
- Example 3: Data highly ambiguous
- Conclusions

Why is software useful?

- Efficient way of doing many repetitive calculations
  - Direct comparison of simulation with data
  - Automated fitting
- Easy access to data bases (stopping, cross sections)
- Integration of advanced physical models
- Recognise when data is insufficient or ambiguous
- Help design experiments
- Education
- Many more !!!

So when is it not useful?

- Direct comparison of simulation with data; automated fitting
  - May not be accurate enough!
- Easy access to data bases (stopping, cross sections)
- Integration of advanced physical models
  - If reality (beam/sample interaction or sample description) is not well modelled, or data bases are wrong, good simulations are wrong
- Recognise when data is insufficient or ambiguous
  - Sometimes no further data can be obtained, and still results are needed

Example 1:
RBS study of thin oxide layers prepared by reactive magnetron sputtering

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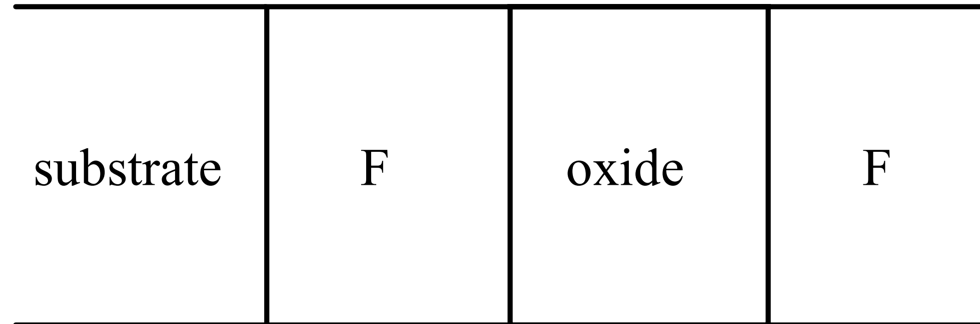
Overview

Introduction
Experimental details
Results
Conclusions

Introduction

- Ferromagnetic/oxide/ferromagnetic junctions:



- Electron tunneling through the insulator oxide
- Related magnetoresistance could be used in devices

- The structure of the system affects its properties:

- Oxide formation at the interfaces
- Layer thickness
- Layer composition

- This work:

- Si / NiFe 60Å / Al_2O_3 t / Co 65Å $t \in [0,200 \text{ Å}]$
- Si / NiFe 60Å / MgO t / Co 65Å $t \in [0,160 \text{ Å}]$

Aim: determine the oxygen content of each layer.

Sample preparation

- Magnetron sputtering:
  - Pbase = $1x10^{-7}$ Torr
  - $Ni_{81}Fe_{19}$, Co: 1Å/s in Ar atmosphere
  - Oxide layers: from Al or Mg metallic targets, 0.1 to 0.2 Å/s in 1.5 mTorr Ar-10 vol% O_2 atmosphere

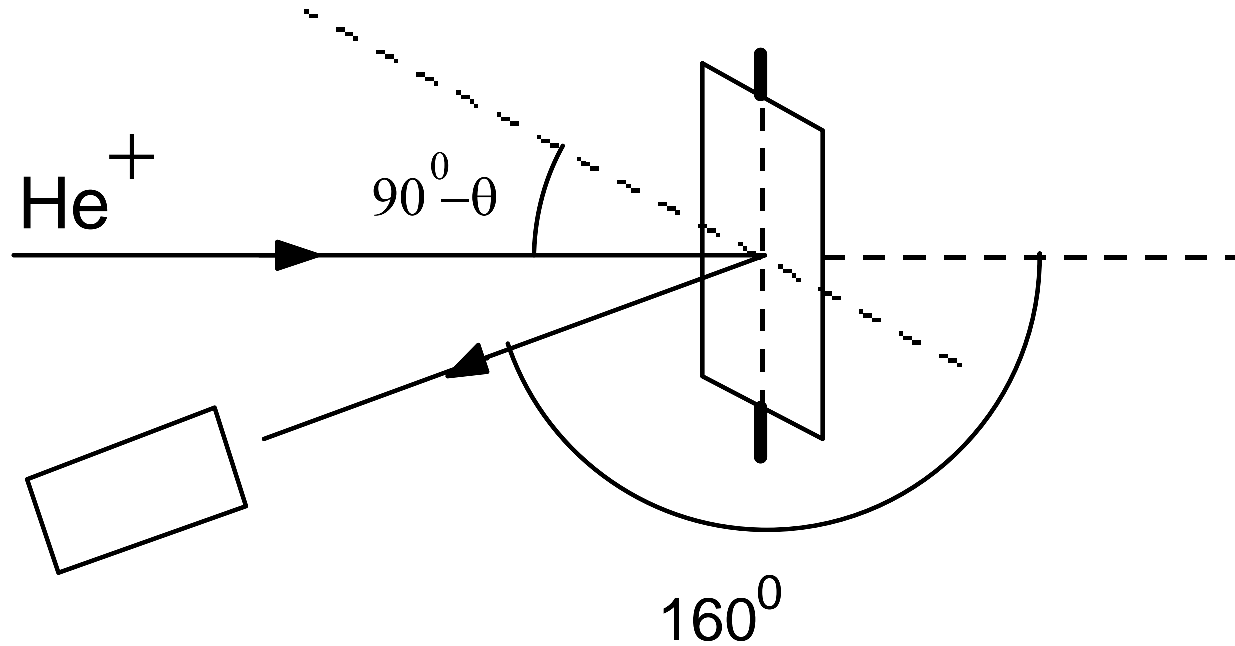
- Deposition process:
  - NiFe is deposited on the Si wafer
  - O_2 is introduced in the chamber
  - Al or Mg is deposited
  - O_2 is removed
  - Co cap layer is deposited

Vacuum is never broken during the process

In samples with t=0 Å, no O_2 is introduced

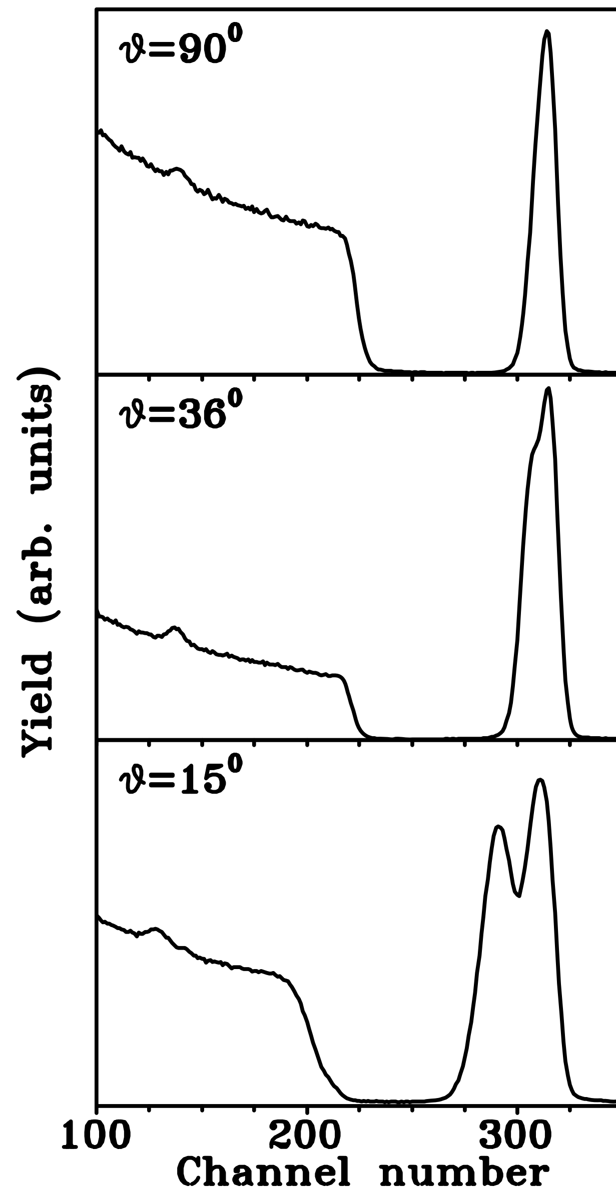
RBS

- 1.0 MeV He^+
- Cornell geometry:



- Tilt angles used:
  Al_2O_3: 90°, 45°, 36°, 15°
  MgO: 90°, 45°, 36°, 20°

- Si / NiFe 60Å / Al_2O_3 25Å / Co 65Å

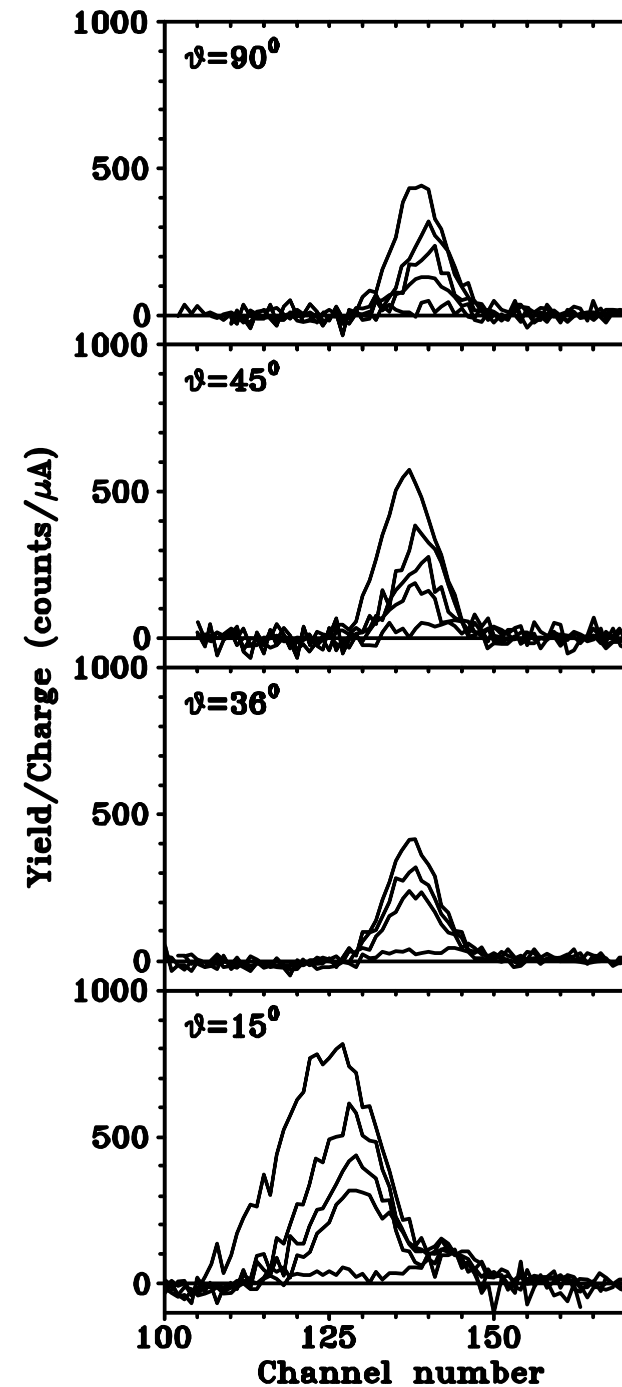


125 Å depth resolution

Channeling: reduced background

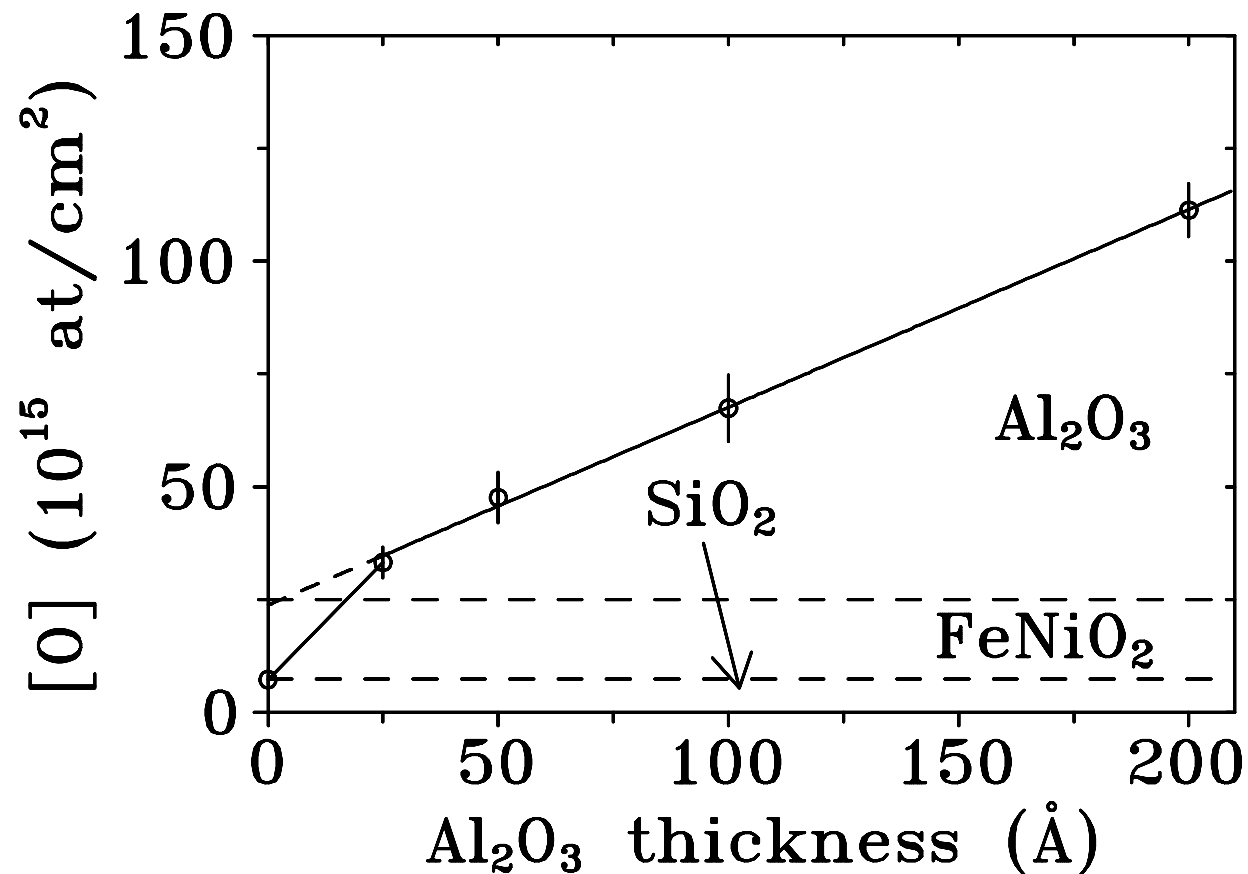
33 Å depth resolution

- Si / NiFe 60Å / Al_2O_3 t / Co 65Å



- Larger Al_2O_3 thickness ⇒ larger oxygen peak
- At t=0 Å there is still oxygen:
  Si native oxide, Co surface oxide
- At 15° these contributions are separated

● Si / NiFe 60Å / Al_2O_3 t / Co 65Å



- t = 0 Å: Si native oxide thickness is 24(4) Å
- t ≥ 25 Å: [O] is proportional to t ⇒ $Al_2O_{2.7(6)}$
- Excess oxygen ⇒ extra oxide layer: NiFe 18(6) Å

- Si / NiFe 60Å / Al_2O_3 t / Co 65Å

$\vartheta = 15^0$

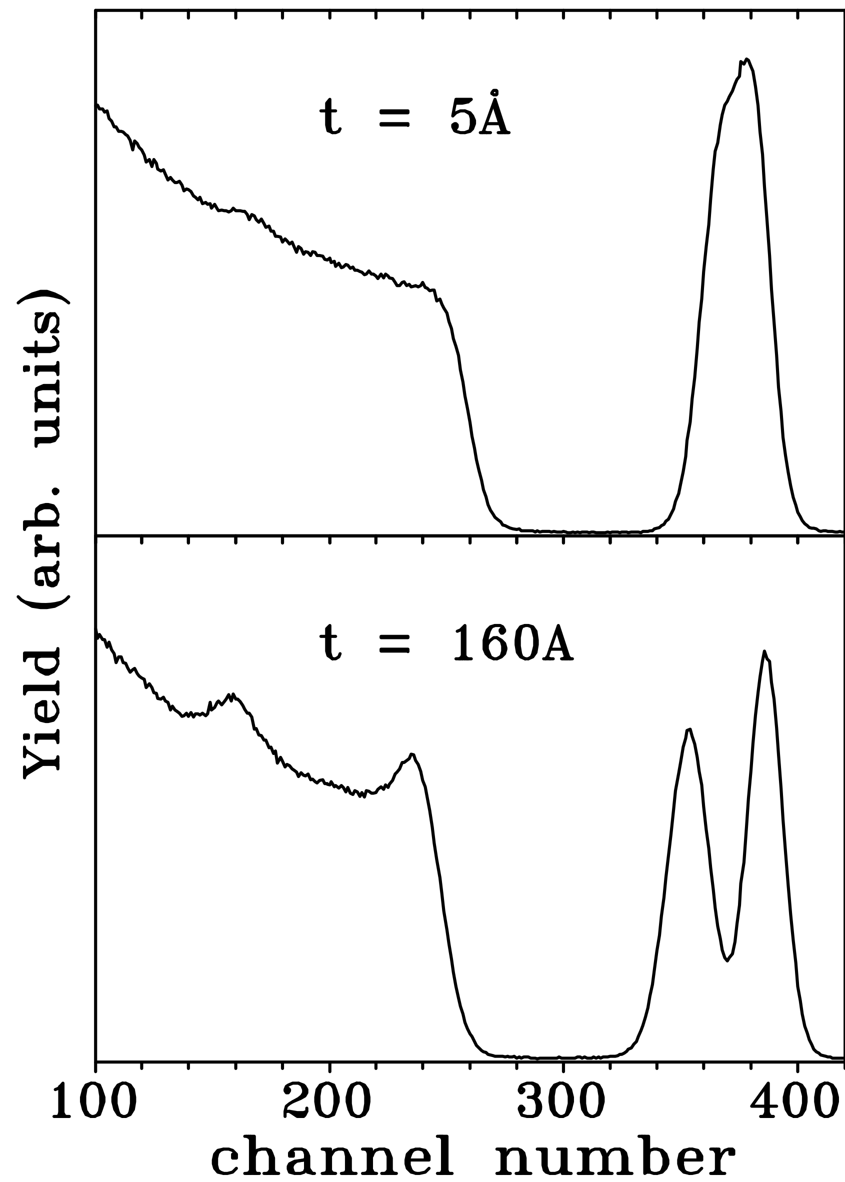
Yield (arb. units)

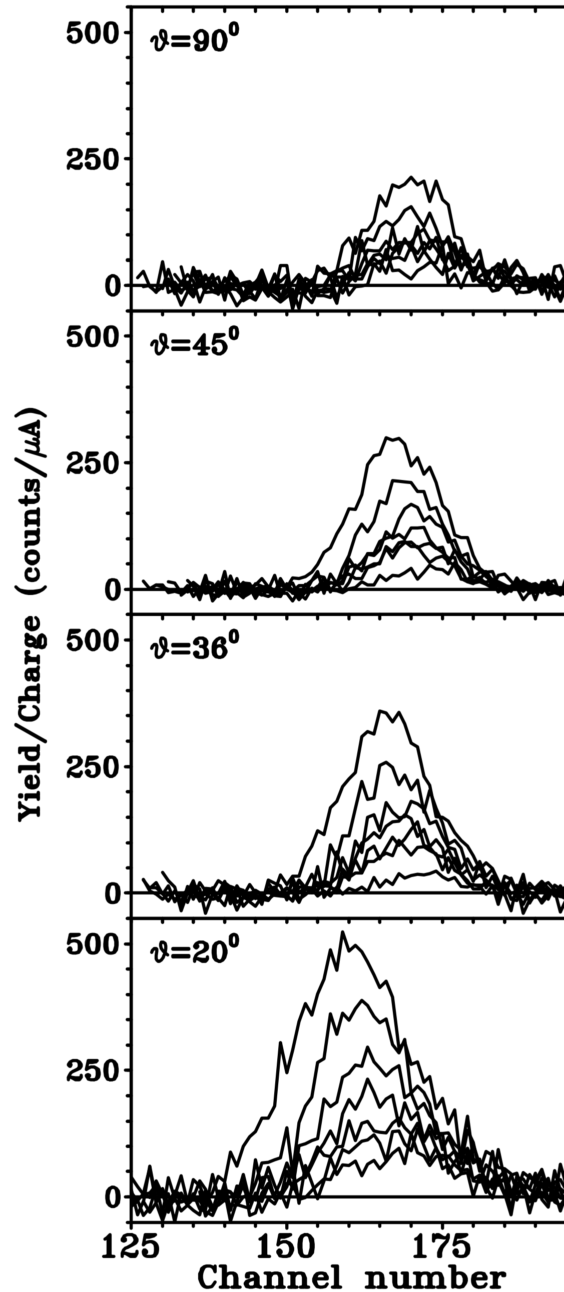
100 200 300

Channel number

- No further fitting: good agreement.

● Si / NiFe 60Å / MgO t / Co 65Å
t = 0, 5, 10, 20, 40, 80, 160 Å





- Si / NiFe 60Å / MgO t / Co 65Å

- Larger MgO thickness ⇒ larger oxygen peak
- At t=0 Å there is still oxygen:
  Si native oxide, Co surface oxide

● Si / NiFe 60Å / MgO t / Co 65Å

[O] (10^{15} at/cm^2)

150
100
50
0

0 50 100 150 200

MgO thickness (Å)

MgO
SiO_2
$FeNiO_2$

- t = 0 Å: Si native oxide thickness is 36(6) Å
- t ≥ 20 Å: [O] is proportional to t ⇒ $MgO_{1.3(4)}$
- Excess oxygen ⇒ extra oxide layer: NiFe 19(4) Å
- Transition regime between t = 0 and 20 Å

Example 2:
Grazing angle RBS analysis of Si/Re 50 Å/(Co 20Å/Re 5Å)$_{16}$

Sample:

Si/Re 50 Å/(Co 20Å/Re 5Å)$_{16}$

1 MeV He^{+} beam detected at 160º in the Cornell geometry

Grazing angle: down to 6º with surface of the sample

Bohr+Chu straggling:

Bad simulation for nominal structure

Impossible to fit all spectra with the same profile

Chu straggling, no DS

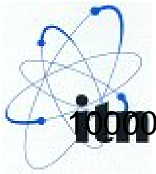
θ=6º

θ=8º

θ=12º

Yield (counts)

Channel



Chu straggling, no DS, independent fits

θ=6⁰

θ=8⁰

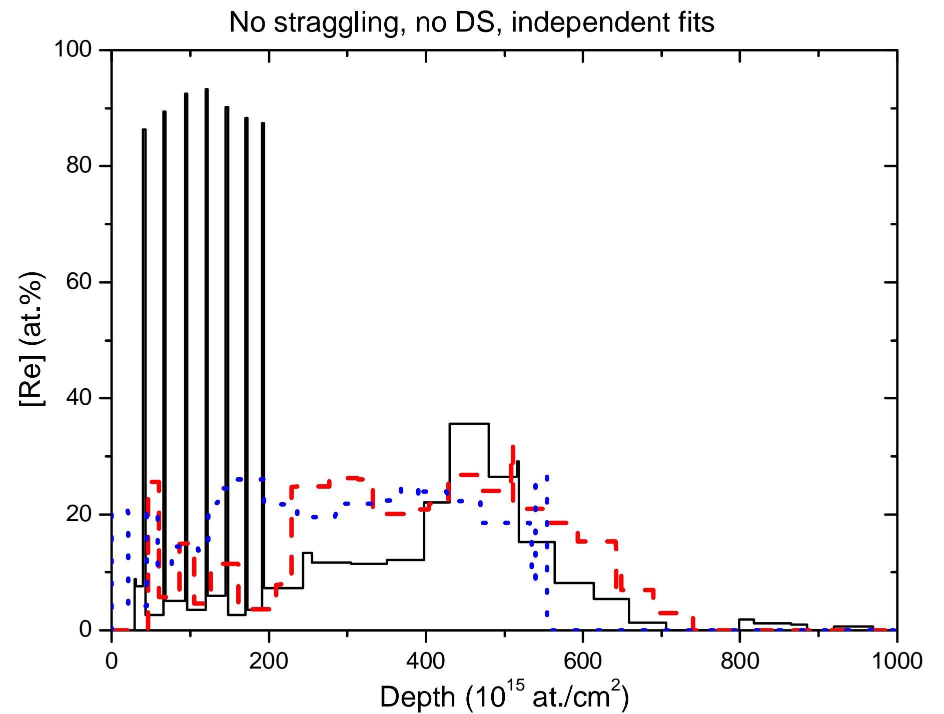
θ=12⁰

Yield (counts)

Channel

Fit the spectra one by one:

Perfect fits with no meaning



No DS: poor simulation at low energy (no problem!)

No roughness: simulation requires Co/Re diffusion:

TEM shows interfaces are sharp but not flat!

Proper straggling, no DS, no roughness

θ=6°

θ=8°

θ=12°

Yield (counts)

Channel

Best analytic simulation available:

Pretty good, but not perfect

A fit would lead to a wrong depth profile

Proper straggling, DS, roughness

$\theta=6^{o}$

$\theta=8^{o}$

$\theta=12^{o}$

Yield (counts)

Channel

Example 3:
RBS analysis of InGaN/GaN quantum wells for hybrid structures with efficient Förster coupling

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Overview

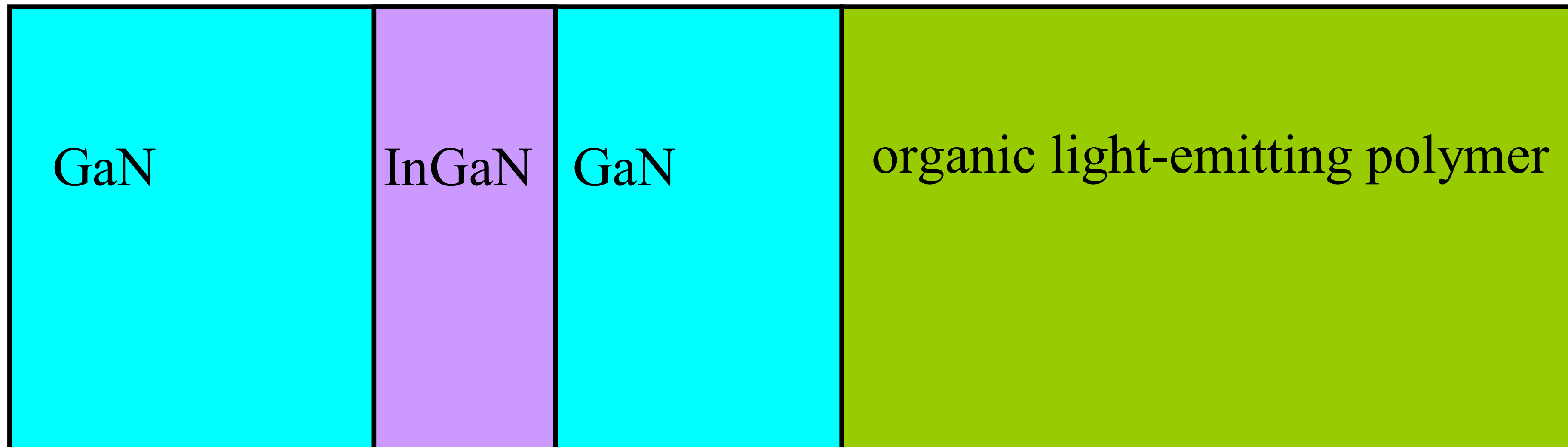
Introduction

Experimental details

RBS analysis

Förster coupling

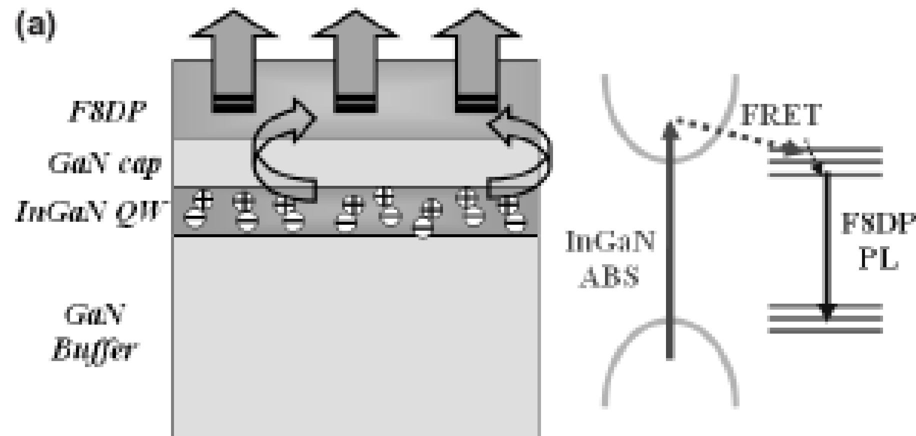
Förster resonant energy transfer (FRET)

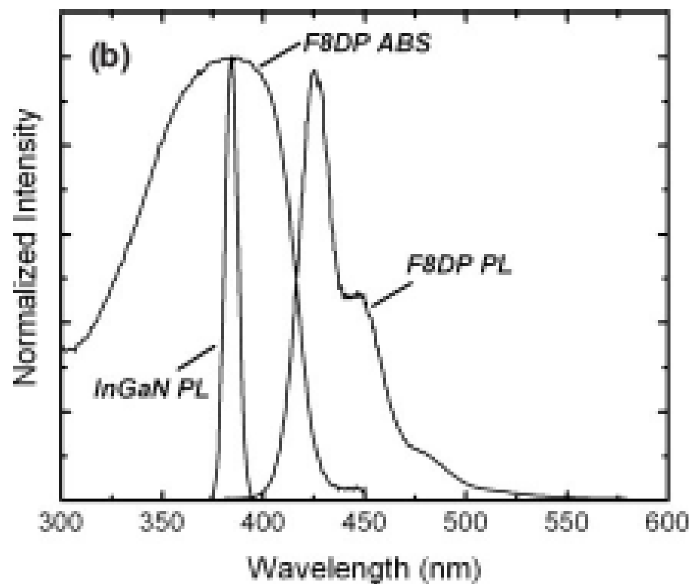


semiconductor QW: donor	LEP: acceptor
excellent electrical properties	cheap, high throughput
complex and costly	entire visible spectrum
near-UV to the green visible	direct electrical injection is issue

FRET can lead to higher overall luminescence efficiencies than in comparable systems involving fluorescence-based radiative energy transfer

FRET depends on dipole-dipole interactions





- *non-radiative* transfer of an excitation generated in an InGaN/GaN QW into an overlayer of LEP
- higher overall luminescence efficiencies than in comparable systems involving fluorescence-based radiative energy transfer
- characteristic range is only a few nanometres.
- GaN cap layer must be of nanometre-scale thickness <10 nm. Its thickness determines the effiency of the FRET process

Experimental details- sample growth

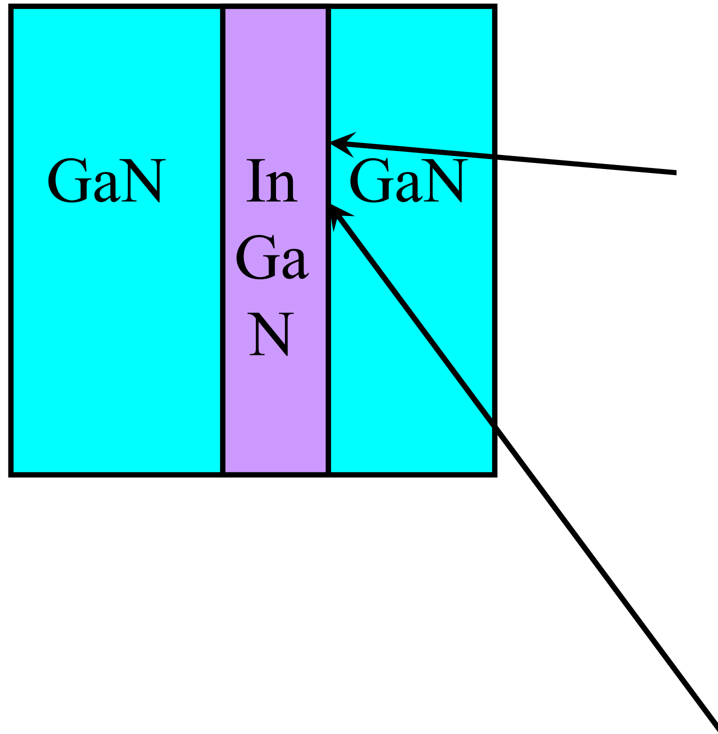
- $In_xGa_{1-x}N/GaN$ QWs with nominal t_{QW}= 2.5, x=0.075 nm were grown by metal-organic vapour phase epitaxy on ~2 mm thick GaN buffer layers on (0001) sapphire substrates.
  - Sample A: t_{GaN}=15 nm
  - Sample B: t_{GaN}=4 nm
  - Sample C: t_{GaN}=2.5 nm

- 5nm thick polymer F8DP overlayers were spin-coated from an F8DP 4 mg/mL toluene solution.

Experimental details- RBS

- 2.0 MeV $^{4}He^{+}$ beam detected at 160º backscattering (Cornell geometry)
- near-normal incidence (5º), and grazing angle (78º for sample A and 80º for samples B and C; the accuracy of the goniometer is 0.02º)
- beam with 0.02º angular divergence, collimated by square slits designed to reduced slit scattering, 0.6 mm high and 0.2 mm wide, leading to beam spots on the sample around 1 mm.
- The detector was located at 70 mm from the sample, and had an aperture 5 mm tall and 1.5 mm wide, to reduce the scattering angle spread.
- The beam current measured by a transmission Faraday cup with precision around 2% was kept at 2 nA.
- The pressure during the experiments was around 10^{-7} mbar.

Results



5º
grazing
c)
b)
a)
In (InGaN SQW)
Ga (GaN cap)
$\Delta E_{In,peak}$
Yield (counts)
Energy (keV)
0
50
100
150
1650
1700
1750

$E_{In,surf}(5^{o})-E_{In,surf}(80^{o})$

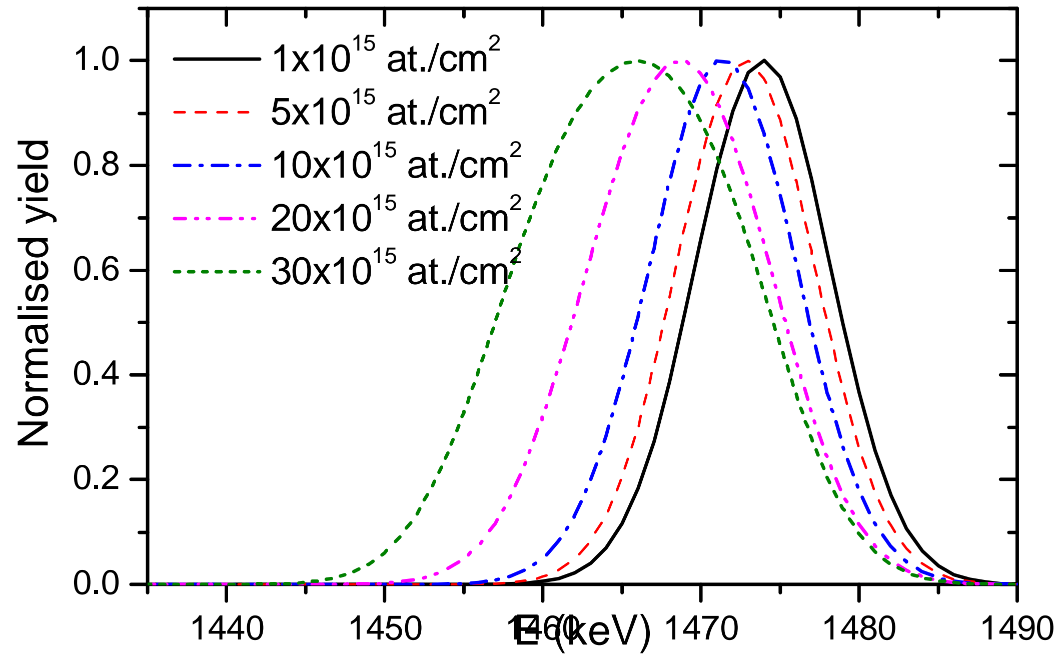
$E_{In,surf}(5^{o})-E_{In,surf}(78^{o})$

$\Delta_{In,surf}$ (keV)

0 10 20 30 40 50 60 70

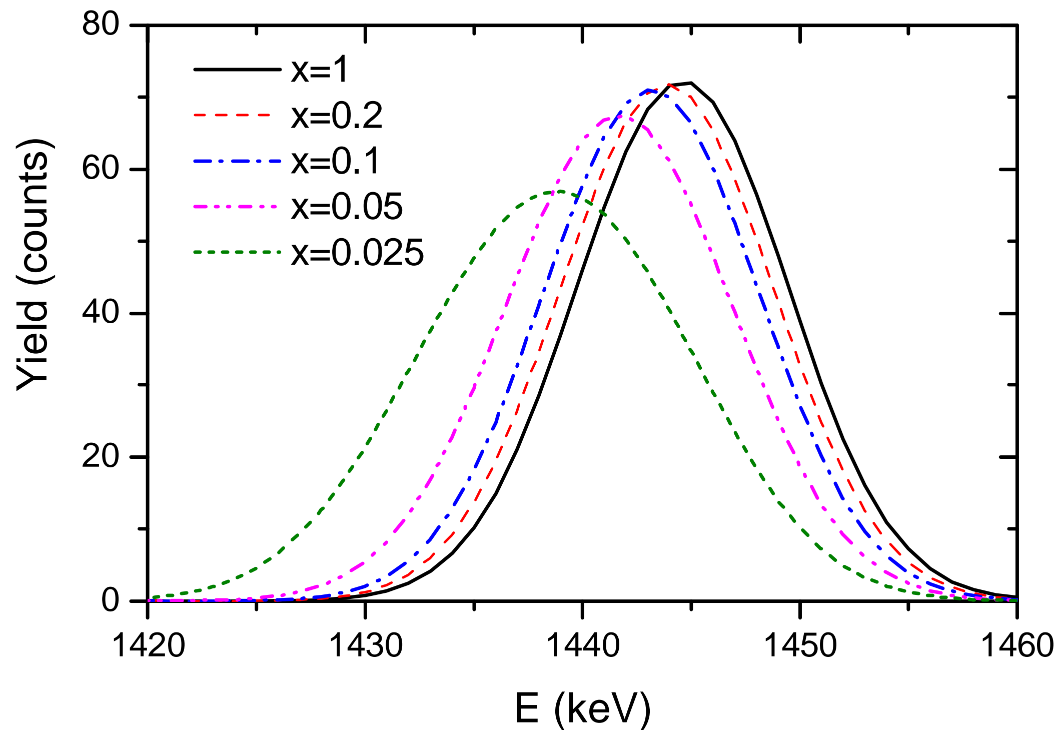
0 20 40 60 80 100 120

GaN cap layer thickness (10^{15} at./cm^2)



Surface pure In layers

$$E_{In,surf} \neq E_{In,peak} \text{ !!!}$$



$Ga_{1-x}In_xN$ with same In total amount, different x, below same GaN thickness

$$\Delta E_{In,surf} = E_{In,surf}\,(5^{o}) - E_{In,surf}\,(\text{grazing})$$

$$E_{In,peak} = E_{In,surf} - \Gamma_{In}\,/2$$

$$\Delta E_{In,surf}(x) = \Delta E_{In,peak} + \Delta\Gamma_{In}(x)\,/2$$

And from $\Delta E_{In,surf}(x)$, $t_{GaN}(x)$ is derived

Table 2. Results for sample A (nominally t_{GaN} =15 nm). The symbols are explained in the text. The shaded lines lead to poor simulations.

x	t_{QW} (10^{15} at/cm^2)	t_{QW} (nm)	$\Gamma_{In}(5^o)$ (keV)	$\Gamma_{In}(78^o)$ (keV)	$\Delta E_{In,surf}$ (keV)	t_{GaN} (10^{15} at./cm^2)	t_{GaN} (nm)	**t_{GaN} / $t_{nominal}$**
0.02	50	5.7	5.16	24.91	33.41(1.03)	85.65(2.81)	9.73(30)	0.65
0.03	32	3.7	3.31	15.98	36.95(1.03)	94.67(2.81)	10.76(30)	0.72
0.05	19	2.2	1.97	9.55	39.49(1.03)	101.14(2.81)	11.49(30)	0.77
0.07	14	1.6	1.46	7.07	40.47(1.03)	103.63(2.81)	11.78(30)	0.79
0.09	11	1.1	1.16	5.59	41.07(1.03)	105.15(2.81)	11.95(30)	0.80
1	1	0.2	0.14	0.64	43.23(1.03)	110.64 (2.81)	12.57(30)	0.84

Table 3. Results for sample B (nominally tGaN =4 nm). The symbols are explained in the text. The shaded lines lead to poor simulations.

x	t_{QW} (10^{15} at./cm^2)	t_{QW} (nm)	$\Gamma_{In}(5^o)$ (keV)	$\Gamma_{In}(80^o)$ (keV)	$\Delta E_{In,surf}$ (keV)	t_{GaN} (10^{15} at./cm^2)	t_{GaN} (nm)	$t_{GaN} / t_{nominal}$
0.03	47	5.4	4.86	27.97	6.63(69)	13.65(1.42)	1.55(16)	0.39
0.05	28	3.2	2.90	16.73	11.27(69)	23.20(1.42)	2.64(16)	0.66
0.07	20	2.3	2.09	12.02	13.21(69)	27.19(1.42)	3.09(16)	0.77
0.09	16	1.9	1.64	9.40	14.30(69)	29.43 (1.42)	3.34(16)	0.83
0.11	13	1.5	1.37	7.90	14.91(69)	30.68(1.42)	3.49(16)	0.87
1	1.4	0.2	0.19	1.06	17.31(69)	35.61(1.42)	4.05(16)	1.01

Table 4. Results for sample C (nominally tGaN =2.5 nm). The symbols are explained in the text. The shaded lines lead to poor simulations.

x	t_{QW} (10^{15} at./cm^2)	t_{QW} (nm)	$\Gamma_{In}(5^o)$ (keV)	$\Gamma_{In}(80^o)$ (keV)	$\Delta E_{In,surf}$ (keV)	t_{GaN} (10^{15} at./cm^2)	t_{GaN} (nm)	t_{GaN} / $t_{nominal}$
0.025	55	6.3	5.67	32.65	0(1.01)	0(2.06)	0(0.23)	0
0.03	45	5.1	4.65	26.74	2.45(1.01)	4.95(2.06)	0.56(23)	0.22
0.05	27	3.1	2.81	16.12	6.85(1.01)	14.11(2.06)	1.60(23)	0.64
0.07	19	2.2	1.98	11.40	8.79(1.01)	18.11(2.06)	2.06(23)	0.82
0.09	15	1.7	1.58	9.06	9.76(1.01)	20.10 (2.06)	2.28(23)	0.91
1	1.35	0.2	0.17	1.02	13.07(1.01)	26.90(2.06)	3.06(23)	1.22

Data grazing angle
Simulation grazing angle
Data 5º
Simulation 5º

c)
b)
a)

Yield (counts)

Energy (keV)

Some solutions rejected due to wrong width of In peak

Sample A: 15 nm
Sample B: 4 nm
Sample C: 2.5 nm

thickness$_{GaN\ cap}$ (10^{15} at./cm^2)

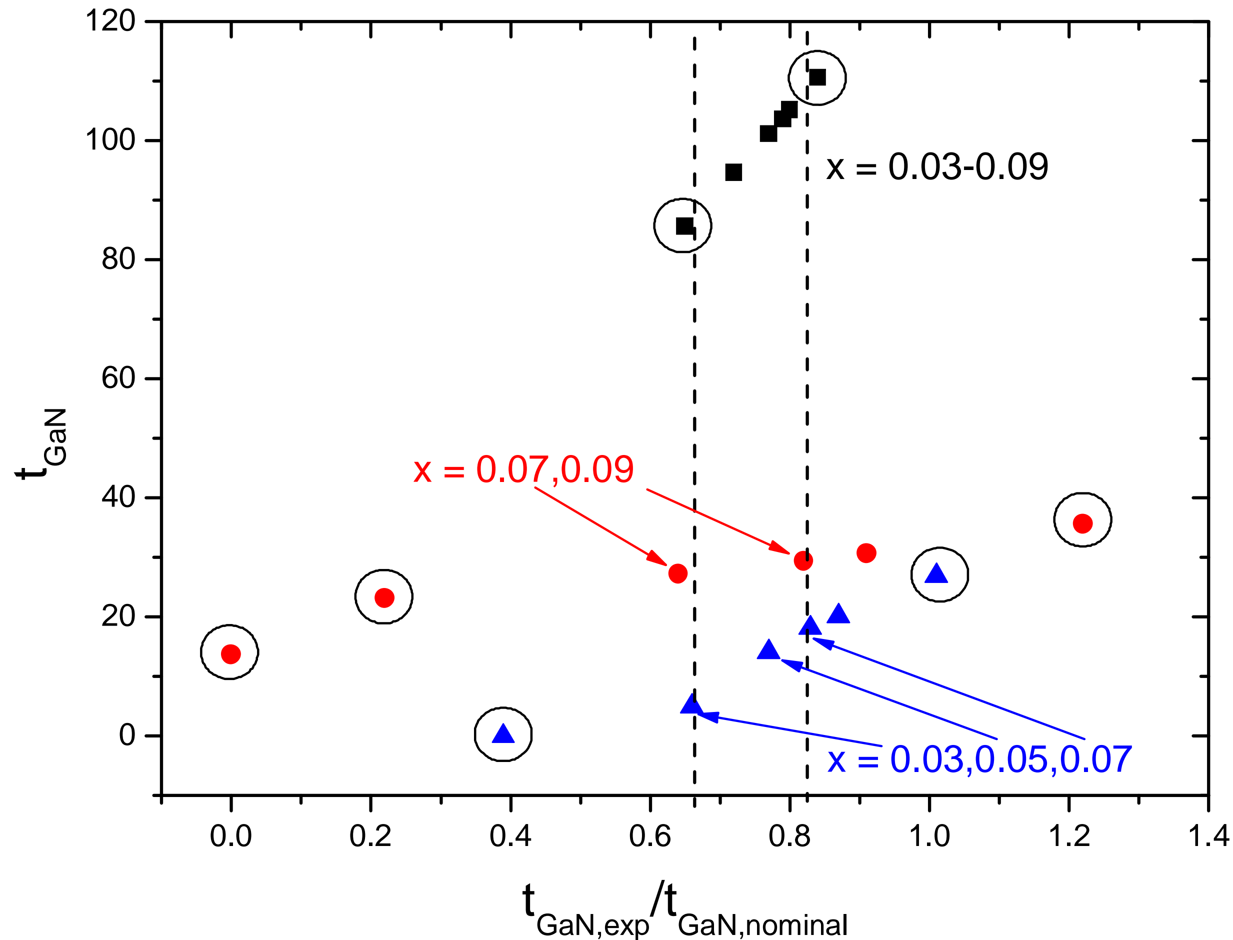
thickness$_{QW}$ (10^{15} at./cm^2)

$$t_{GaN} = -t_{QW}/2 + \Delta E_{In,peak} / S(1/\cos(\text{grazing}) - 1/\cos(5^{\circ}))\,(k - 1/\cos(\pi - \alpha_{scatt}))$$

All QWs grown in exactly the same way

Control and reproducibility of growth excellent

- x must be the same in all samples
- $t_{GaN,exp}/t_{GaN,nominal}$ must be the same in all samples



$x=0.07(1)$, $t_{GaN,exp}/t_{GaN,nominal}=0.79(5)$, $t_{QW} = 2.0(4)$nm

GaN cap layer thickness of samples A, B and C: 11.8(8) nm, 3.2(5) nm, 1.9(5) nm

PL at QW peak with and without LEP

sample A: 11.8nm

(a)

sample B: 3.2nm

(b)

sample C: 1.9nm

(c)

(d)

Normalized PL Intensity

Time (ps)

k_F/k_{QW}

Cap Thickness d (nm)

Förster radius: 6.1(0.8) nm typical for 2D-2D FRET; efficiency at LN: 60%

Conclusions

- Do not trust data analysis codes
  - Good fits are meaningless when data are ambiguous
- Learn how to do manual analysis
  - Sometimes it's the only way of doing things right
- Analysis codes are nevertheless essential
  - They make your life much easier