Calibration of surface temperature of thin ZrN buffer layers for GaN nanowire growth by plasma-assisted MBE

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Introduction

- optical pyrometry is commonly used in MBE as the non-contact temperature measuring tool; however, quite often results remain unreliable since:
  - the emissivity of the substrate must be precisely known which is an issue if nonconventional substrate materials or multilayer substrates are used
  - correction of pyrometer reading due to a partial coating of the viewport window must be taken into account

⇨ periodic calibration of optical pyrometer under real growth conditions and for a particular substrate is obligatory for successful MBE growth

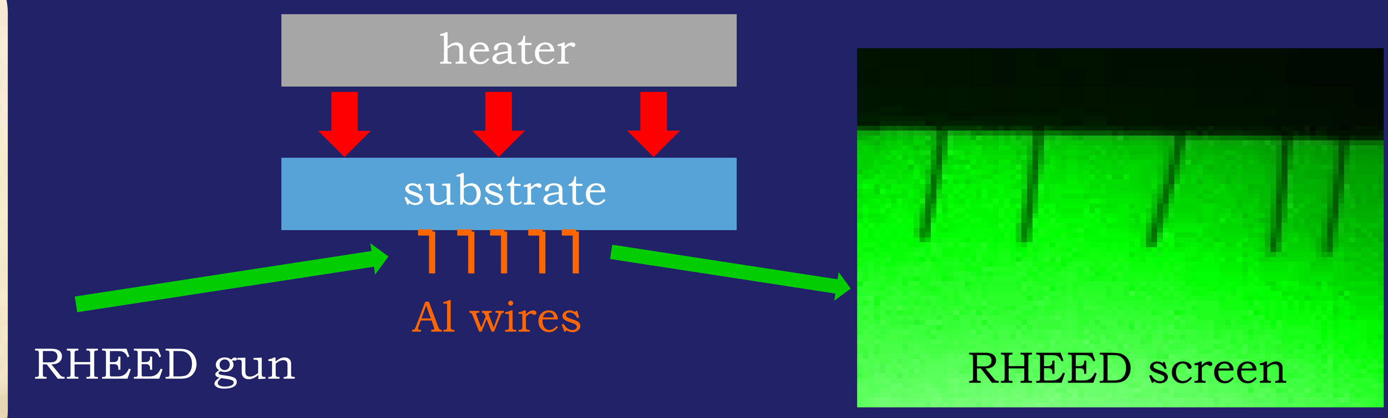
Aim of the work:

1. to determine emissivity of ZrN/Si and ZrN/Al_2O_3 substrates vs. ZrN buffer thickness for measurements of substrate temperature by optical pyrometry
2. to elucidate an influence of ZrN buffer on temperature of substrate surface

Samples and tools

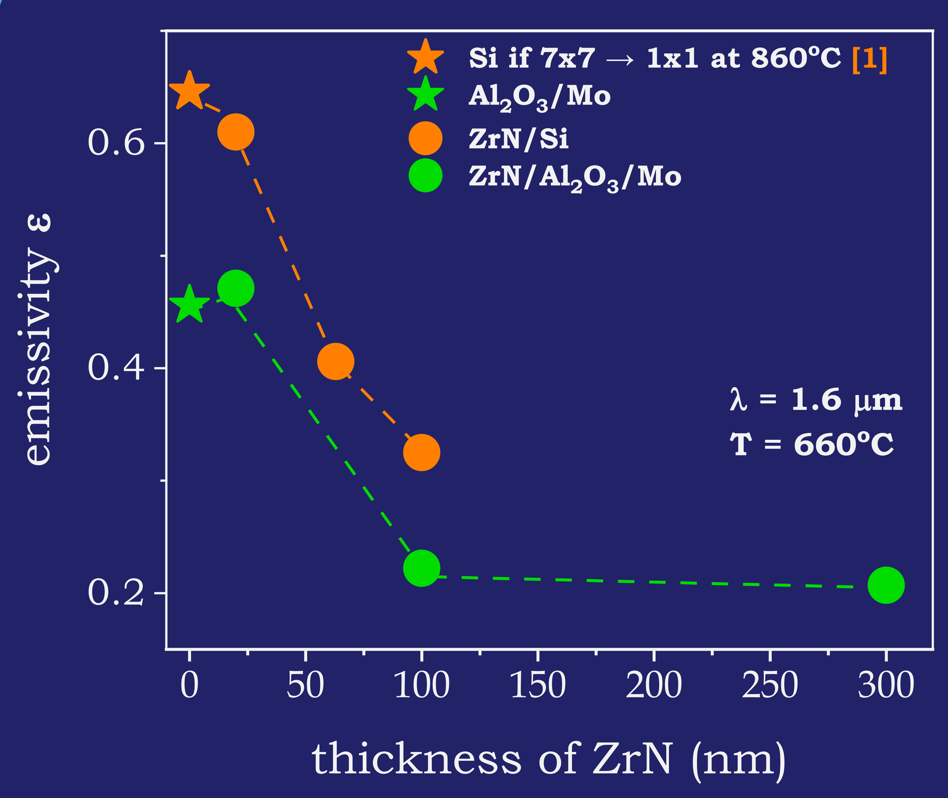
1. **for emissivity measurements:** ZrN buffer layers with thicknesses 20 – 300 nm deposited on 2” Si(111) or c-Al_2O_3/Mo wafers using DC sputtering from a ZrN target
2. **for GaN NW growth:** 3” Si(111) wafer with one half covered by 40 nm thick ZrN
3. optical pyrometer Raytec Marathon MM2ML operating at λ =1.6 µm
4. GaN growth by plasma-assisted MBE using Riber Compact 21 system

Calibration of substrate emissivity



- thin Al wires bonded to the substrate surface
- temperature slowly ramped while the shadows of the Al wires illuminated by a defocused e-beam from the RHEED gun observed on the fluorescent screen
- as soon as the wires melted the emissivity of the optical pyrometer adjusted to get the surface temperature reading of Al melting point (660.3°C)
  - → the method is compatible with clean UHV environment of MBE and works at the temperature close to the temperature range ~ 700°C commonly used to grow nitride semiconductors by PAMBE
  - → the method can be applied for various substrates as long as no reaction of Al with the substrate occurs before Al melting
  - → the procedure automatically takes into account correction of pyrometer reading due to a partial coating of the MBE viewport window

Emissivity of thin ZrN buffers



Planck radiation law

$$E_\lambda(\lambda, T) = \varepsilon \frac{8\pi hc}{\lambda^5} \frac{1}{\exp(\frac{hc}{\lambda kT}) - 1}$$

$E_\lambda(\lambda,T)$ – spectral energy density - energy radiated per unit volume in the wavelength interval λ to λ + Δλ
ε – emissivity
k – Boltzmann constant

[1] T. Suzuki, et al., Jpn. J. Appl. Phys. 32 (1993) L610–L613
[2] S. Edalatpour, et al., J. Quantitative Spectroscopy & Radiative Transfer 118 (2013) 75

- the emissive power ε of substrates depends on the thickness of ZrN buffer as well as on the choice of the bulk wafer material
- the emissivity ε decreases significantly when the thickness of ZrN increases until it converges to the emissivity of the bulk ZrN at thickness of ~100 nm
- such behavior is in agreement with numerical simulations [2] that predict:
  - ⇨ initial decrease of emissivity with an increase of metallic layer thickness as the result of the extraneous contributions of IR light waves experiencing multiple reflections within the thin layer, which are usually internally absorbed in metallic bulks
  - ⇨ the critical thickness above which no size effect is observed for metals is about a hundred of nanometers
- for the same thickness of ZrN buffer the substrate emissivity is larger on Si than on sapphire/Mo wafer

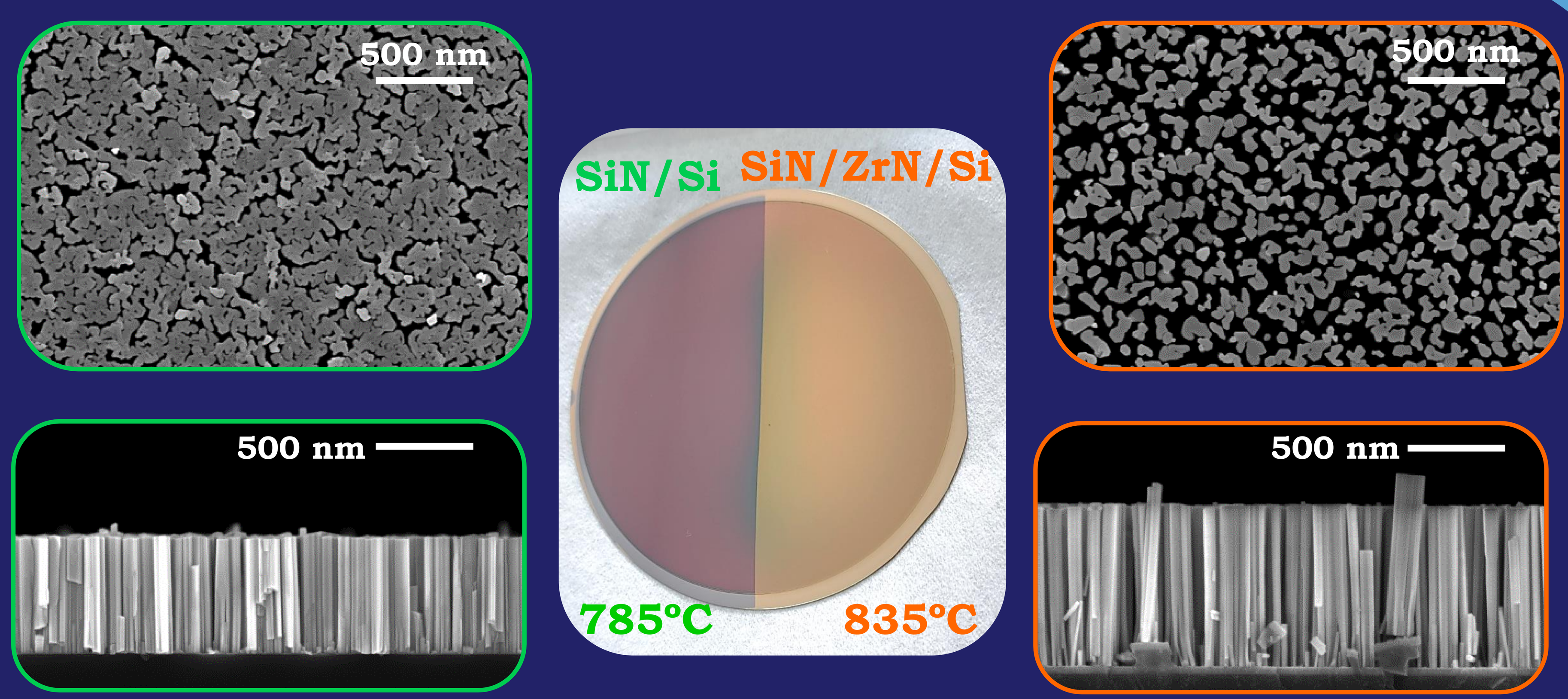
Emissivity of the substrate depends on ZrN thickness, consequences:

<u>from the Planck’s law:</u> if the bulk value of ZrN emissivity (ε = 0.207) is used instead of the real value (ε = 0.466) the temperature reading on 20 nm thick ZrN/Al_2O_3/Mo substrate is 800°C instead of real 705°C – **the error of 95°C**

Self-assembled growth of GaN nanowires on ZrN/Si substrates

The growth procedure:

- 3” Si(111) substrate thermally deoxidized and next exposed to nitrogen flux (nitridized) in MBE growth chamber
- 40 nm thick ZrN buffer layer deposited on a half of the wafer
- ~2 nm thick SiN layer deposited on the entire substrate surface using Si and N sources in MBE growth chamber; the reason – to ensure the same GaN nucleation mechanisms on both parts of the substrate
- self-assembled GaN NW growth at 785°C (as measured on the SiN/Si part), with N and Ga fluxes Φ_N = 16 nm/min and Φ_{Ga} = 8 nm/min, respectively. The growth time 60 min.
  More details in [K. Olszewski et al., Nanomaterials 13 (2023) 2587]



Results:

- using measured ε value for 40 nm thick ZrN the temperature difference between ZrN/Si and Si parts of the substrate ~ 79°C is determined despite uniform heating of the whole wafer
- RHEED shows very short (below 1 min) GaN NW incubation times on both parts, even if so large temperature differences exist; this agrees with our calculations of GaN incubation time on SiN [M. Sobanska et al., Nanotechnology 30 (2019) 154002]

- higher substrate temperature on ZrN-covered part of the substrate
- amount of GaN deposited (fill factor × NW length) nearly the same on both parts of the substrate
  - ⇨ at higher temperature faster Ga adatom diffusion on NW sidewalls towards the NW top facet
  - ⇨ lower radial growth rate on ZrN-covered part of the substrate → longer and thinner GaN NWs, lower degree of coalescence

Summary

- the method of optical emissivity determination of nonconventional substrate materials or multilayer substrates used in MBE growth has been developed and tested for ZrN-buffered Si and sapphire substrates
- values of emissivity ε of ZrN/Si and ZrN/c-Al_2O_3/Mo substrates determined for wavelength of 1.6 µm and T = 660°C as a function of ZrN layer thickness
- emissivity decreases significantly when the thickness of ZrN increases until it converges to the emissivity of the bulk ZrN at thickness of ~100 nm – in agreement with results of simulations reported in the literature
- proper calibration of optical pyrometer crucial for controlling nucleation and PAMBE growth of GaN NWs on ZrN-buffered substrates

National Science Centre Poland

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