# Reactive deposition of Vanadium Nitride by High Power Impulse Magnetron Sputtering (HiPIMS)

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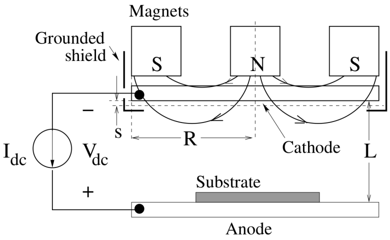
# *Introduction*

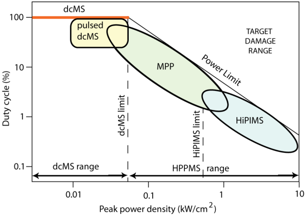
- Magnetron sputtering has been a highly successful technique that is essential in a number of industrial applications

- A magnet is placed at the back of the cathode target to confine the energetic electrons near the cathode
- In a conventional dc magnetron sputtering discharge the power density (plasma density) is limited by thermal load on target
- Low ionization flux fraction of the sputtered material (<10%)



# *High power impulse magnetron sputtering discharge*

- High ionization of sputtered material requires very high density plasma
- High power pulsed magnetron sputtering (HPPMS)
- In a HiPIMS discharge a high voltage pulse is supplied for a
  - low frequency
  - low duty cycle
  - low average power
- Ionization flux fraction of the sputtered materials up to 60%

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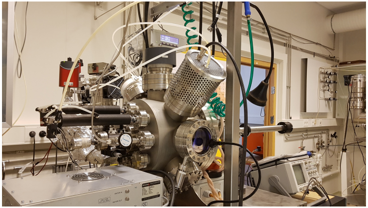
- Power density limits

  $p_t = 0.05$ kW/cm$^2$ dcMS limit

  $p_t = 0.5$ kW/cm$^2$ HiPIMS limit

# Experimental Setup

- Both HiPIMS and dcMS deposition are made at average power of 150W
- All depositions are made at working pressure of 0.9 Pa on 1$\mu$m $SiO_2$/Si substrate
- HiPIMS pulse was 200 $\mu$s long at frequency of 100 Hz repetition
- Two different 3 inches magnets are used for the sputtering



| Magnet | Center diameter [mm] | Ring diameter [mm] | MFD of center (average) | MFD of Ring (average) | magnetic flux of center (total) | magnetic flux of Ring (total) | $K$ |
|---|---|---|---|---|---|---|---|
| Weak | 14 | 10 | 0.322 T | 0.314 T | 49 $\mu$Wb | 690 $\mu$Wb | 0.07 |
| Strong | 30 | 8 | 0.396 T | 0.367 T | 279 $\mu$Wb | 664 $\mu$Wb | 0.42 |

# Deposition of pure Vanadium films

# *Pure Vanadium deposition*

- HiPIMS films are always denser and smoother than dcMS

Extracted data from XRR and AFM measurements of vanadium films

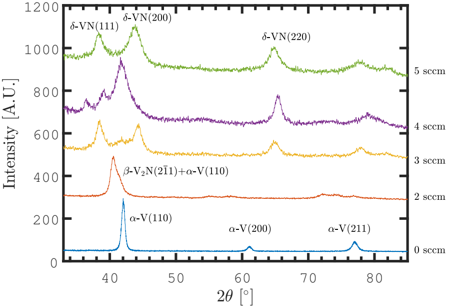
| Method | Magnet | Density [g/cm$^3$] | DR [nm/min] | Roughness [nm] | 111 grain size [nm] | 200 grain size [nm] | 220 grain size [nm] |
|---|---|---|---|---|---|---|---|
| HiPIMS | Strong | 5.79 | 0.98 | 2.41 | 16 | 12.5 | 11.1 |
| HiPIMS | Weak | 5.59 | 2.54 | 5.76 | 15.8 | 12.3 | 10.4 |
| dcMS | Strong | 5.11 | 2.66 | 8.41 | 16.2 | 13 | 10.5 |
| dcMS | Weak | 5.23 | 3.36 | 8.30 | 16 | 12.5 | 11.4 |

- Using the strong magnet results in:
  - Higher film density in HiPIMS, lower density in dcMS film
  - Lower deposition rate for both methods
  - Significantly smother HiPIMS film, slightly rougher dcMS film
- The ratio $DR_{HiPIMS}/DR_{dcMS}$ is 0.37 for strong and 0.77 for weak magnet
- Grain size is independ of deposition method and magnet strength

# Deposition of Vanadium Nitride films

# Introducing Nitrogen into the chamber

- Mixture of 40 sccm Ar and a range of $N_2$ flow rates was injected to the chamber
- 2 sccm nitrogen flow results in mixture of $\alpha$-V and $\beta$-$V_2N$ phases
- 5 sccm nitrogen flow results in stoiciometric cubic $\delta$-VN phase

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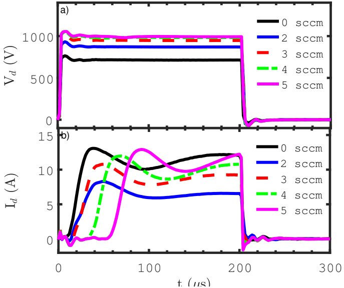
# Introducing Nitrogen into the chamber

- Increasing Nitrogen flow results in:
  - Increase in discharge current
  - Longer delay in current onset
  - Need for higher voltage to keep the power constant
  - Lower deposition rate

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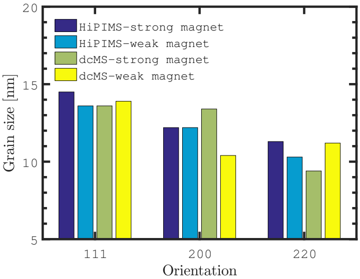
# *Vanadium Nitride deposition using different magnets*

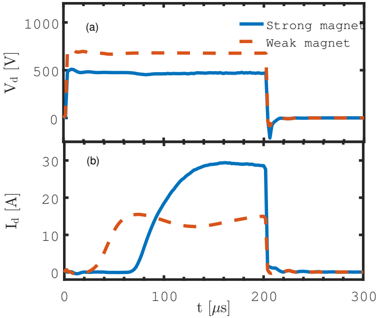
- HiPIMS films are denser and smoother than dcMS film
- $DR_{HiPIMS}/DR_{dcMS}$ is 0.53 for strong and 0.85 for weak magnet
- The ratios is 30% higher for strong magnet and 9% for weak magnet than V deposition ratio
- Strong magnet results in:
  - Higher density for both methods (close to bulk density)
  - Lower deposition rate for both methods
  - Smoother surface in HiPIMS, rougher surface in dcMS

| Method | Magnet | Density [g/cm$^3$] | DR [nm/min] | Roughness [nm] |
|---|---|---|---|---|
| HiPIMS | Strong | 6.137 | 0.83 | 0.63 |
| HiPIMS | Weak | 5.79 | 1.35 | 2.53 |
| dcMS | Strong | 5.25 | 1.55 | 5.44 |
| dcMS | Weak | 5.17 | 1.59 | 4.17 |



# *Vanadium Nitride deposition using different magnets*

- Strong magnet generates about 2 times higher discharge current, thus the deposition is done at lower cathode voltage
- There is a longer delay on current onset when using strong magnet
- Longer delay is probably due to lower cathode voltage

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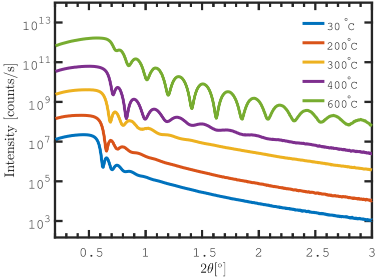
# *Vanadium Nitride deposition at different temperatures*

- Growth temperature varied from room temperature up to 600°C
- Deposition at higher temperatures leads to:
  - Higher film density
  - Smoother film surface
  - lower growth rate up to 400°C but slightly higher rate at 600°C.

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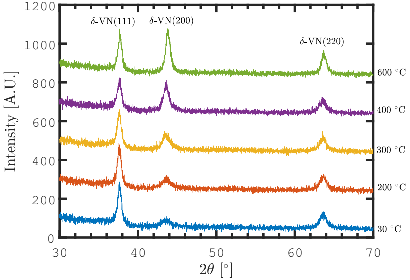
# Vanadium Nitride deposition at different temperatures

- All films are polycrystalline and the (111), (200) and (220) crystal orientations are present in all samples
- Grains oriented in the (111) direction are dominant for all growth temperatures
- The (200) grain size increases steadily with increasing temperature from 5 to 13 nm

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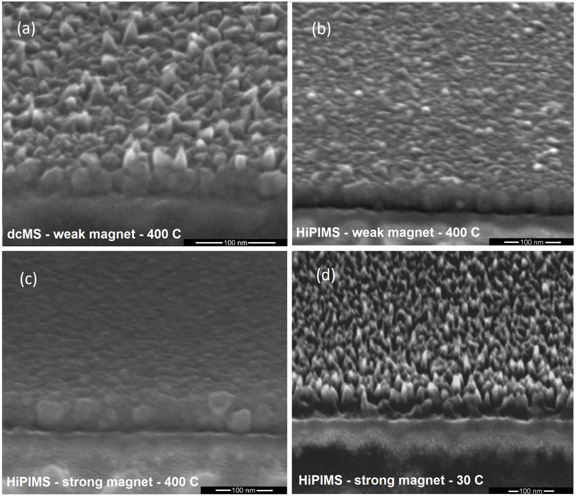
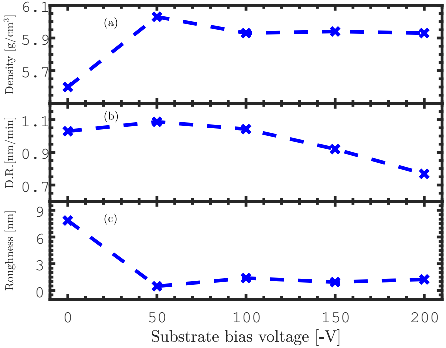
# *Cross section SEM images of VN thin films*



# Effect of substrate bias on properties of HiPIMS deposited vanadium nitride films

# Vanadium Nitride deposition at different temperatures

- In terms of density
  - Grounded substrate - Lowest density (5.60 g/cm$^3$)
  - At -50 V - Highest density (6.03 g/cm$^3$) (close to bulk)
  - -100 V to -200 V - Almost unchanged (5.93 g/cm$^3$)
- Deposition rate
  - At -50 V - Highest DR (1.09 nm/min)
  - At -200 V - Lowest DR (0.77 nm/min)

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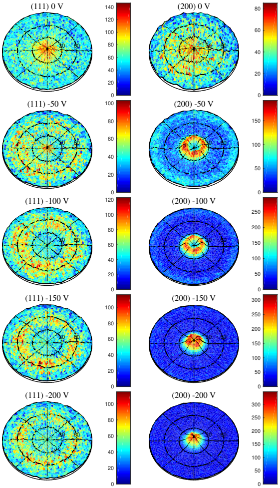
- Surface roughness
  - Grounded substrate - Roughest (8.2 nm)
  - At -50 V - The most smooth (0.47 nm)

# *Substrate bias effect*




- Film deposited at grounded substrate has uniformly distributed (200) planes while the (111) planes show an intense spot around $\psi = 0$ indicating considerable $\langle 111 \rangle$ texture normal to the substrate
- Substrate bias at -50 V, the (200) planes exhibit a ring at $\psi = 20^\circ$ along with an intense spot at $\psi = 0$ for (111) planes

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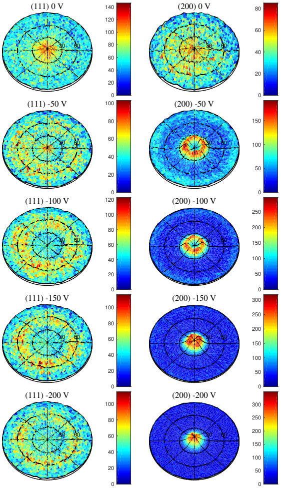
# *Substrate bias effect*




- Thus, there is a competition between these planes to grow normal to the substrate and in this regard the ⟨111⟩ texture is dominant
- Substrate bias up to -100 V causes the ⟨111⟩ texture to disappear but the (200) planes shows up at $\psi = 15°$. Further increase in the substrate bias results in dominant ⟨200⟩ texture normal to the substrate

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# Substrate bias effect

- Nitrogen percentage decreased as substrate bias increased
- Substrate bias of −50 V - lowest resistivity 48.4 $\mu\Omega$ cm
- This is among the lowest resistivities which has been reported for VN thin film
- Substrate bias of −200 V - highest resistivity

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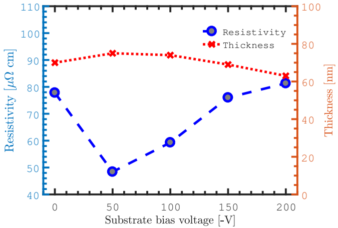
Nitrogen amount in VN films deposited by varying substrate bias

| substrate bias voltage V | Nitrogen content [%] |
|---|---|
| 0 | 54.52 |
| -50 | 51.60 |
| -100 | 50.61 |
| -150 | 49.22 |
| -200 | 48.02 |



# Conclusion

- HiPIMS deposition leads to denser films and smoother surfaces than films deposited by dcMS
- HiPIMS rate is closer to the dcMS rate in reactive mode than in non-reactive mode
- Lowering the magnetic field strength increases the deposition rate significantly for HiPIMS while dcMS rate is weakly dependent on the magnetic field
- Substrate bias of $-50$ V leads to most dense and smooth VN film and the highest deposition rate and conductivity.
- Low substrate bias encourages off-normal $\langle 200 \rangle$ texture and high substrate bias leads to only $\langle 200 \rangle$ texture normal to the substrate

# Thank you for your attention

The slides can be downloaded at
`http://langmuir.raunvis.hi.is/~tumi/ranns.html`
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# References

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