

# EQP energy and mass analyser

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RF Superconductivity

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UNIVERSITY OF  
LIVERPOOL

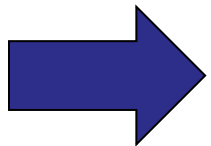


# Overview

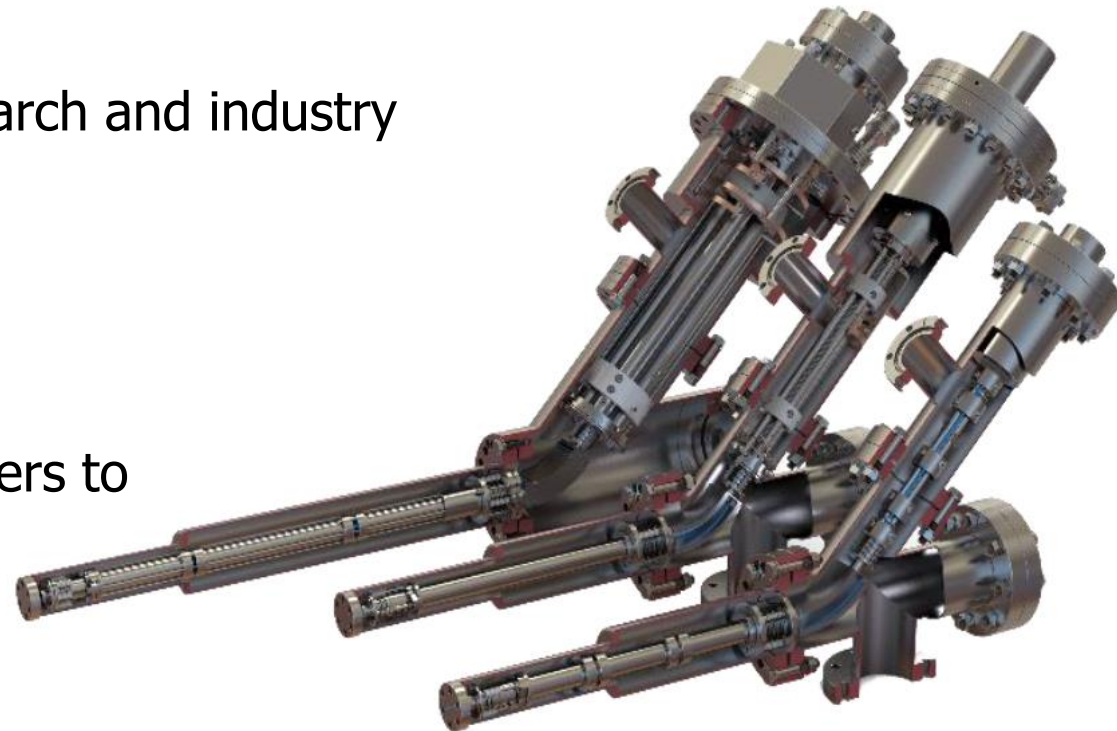
- Introduction
- Technical details of an EQP system
- Coating of Nb thin films using HiPIMS and bipolar HiPIMS
- Coating of alternative superconducting materials
- Summary

# Introduction

- Development of thin films and their deposition methods significantly improved many industries over the past century
- Various techniques available, crucial role in research and industry
  - Magnetron sputtering
  - CVD
  - PECVD, etc.
- Each technique require specific process parameters to create the desired surface/film properties



EQP mass spectrometer

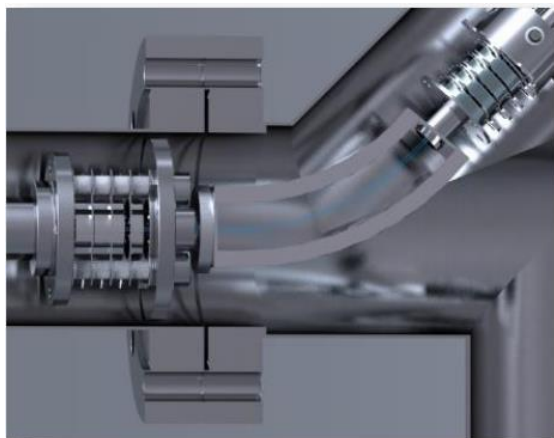
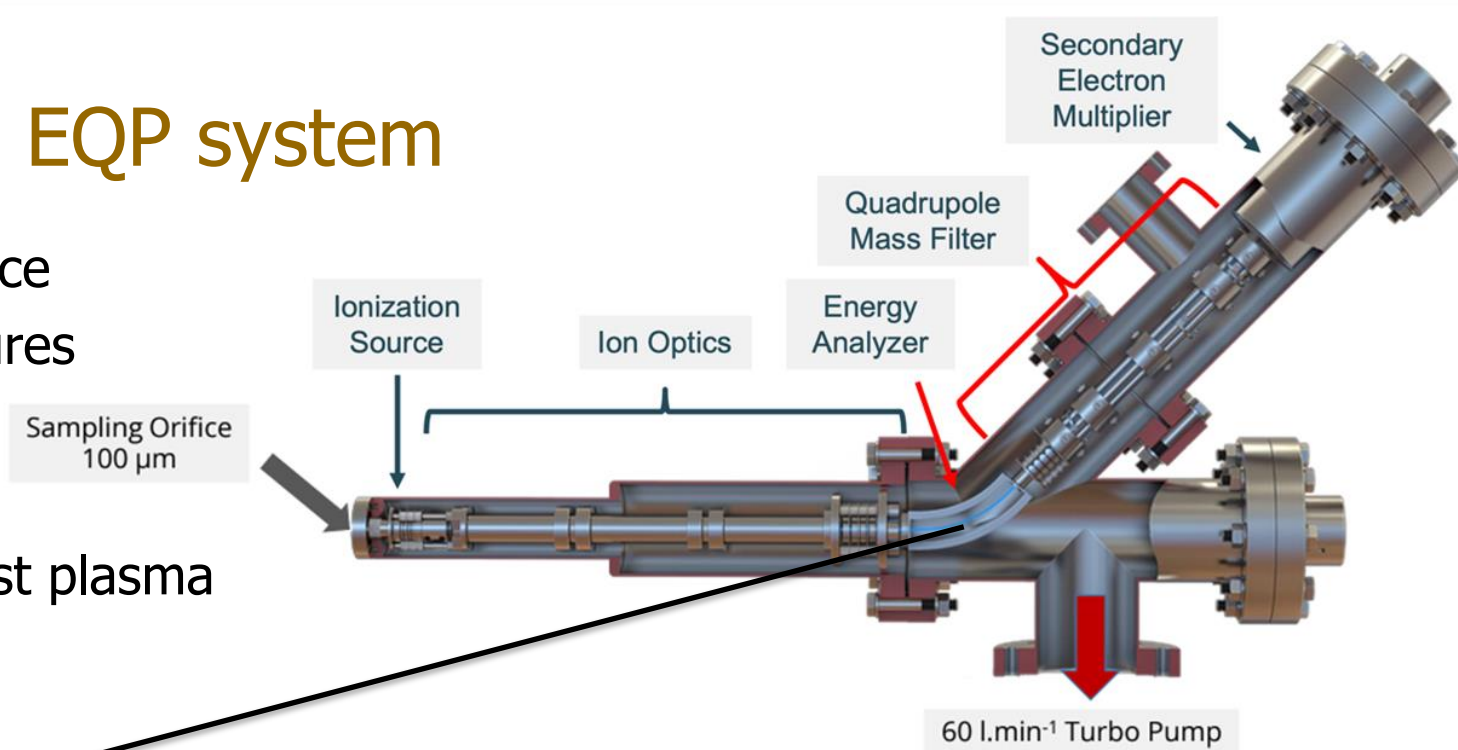


# Introduction

- Electrostatic quadrupole plasma (EQP) mass spectrometer are designed for direct analysis of plasma ion mass and energy in both plasma characterisation and process diagnostic applications
- EQP systems measure both positive and negative ions but can also perform neutrals analysis using an electron bombardment ion source
- Various field of applications
  - DC glow discharge
  - HiPIMS
  - Parallel Plate – RF plasma
  - ICP

# Technical details of an EQP system

- Interchangeable sampling orifice
- Operational at different pressures
- Differential pumping
- Versatile and adaptable to most plasma chamber

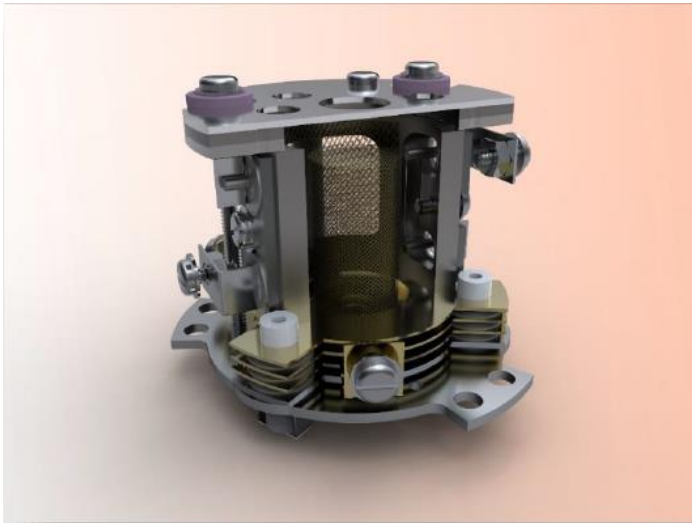


- 45° Electrostatic Sector Energy analyser
  - Constant transmission at all ion energies
  - Minimum perturbation of ion flight path
  - Energy resolution – 0.25 eV FWHM
  - Energy scan at increments from 0.05 eV
  - Possibility to float up to 10 KeV

\*Pictures courtesy of Hidden Analytical, Ltd

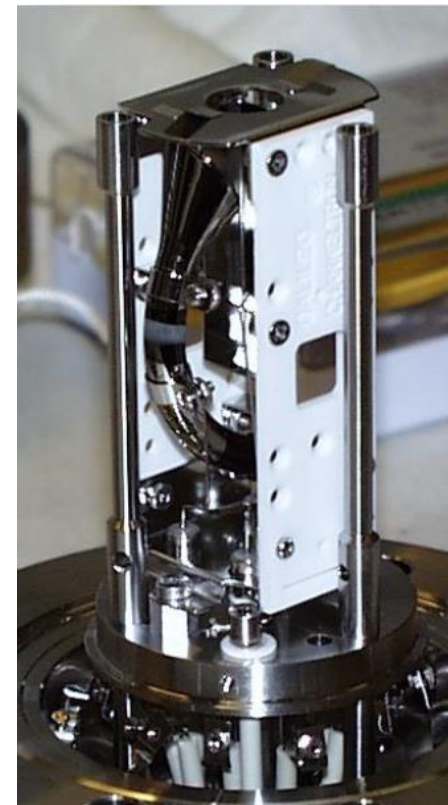
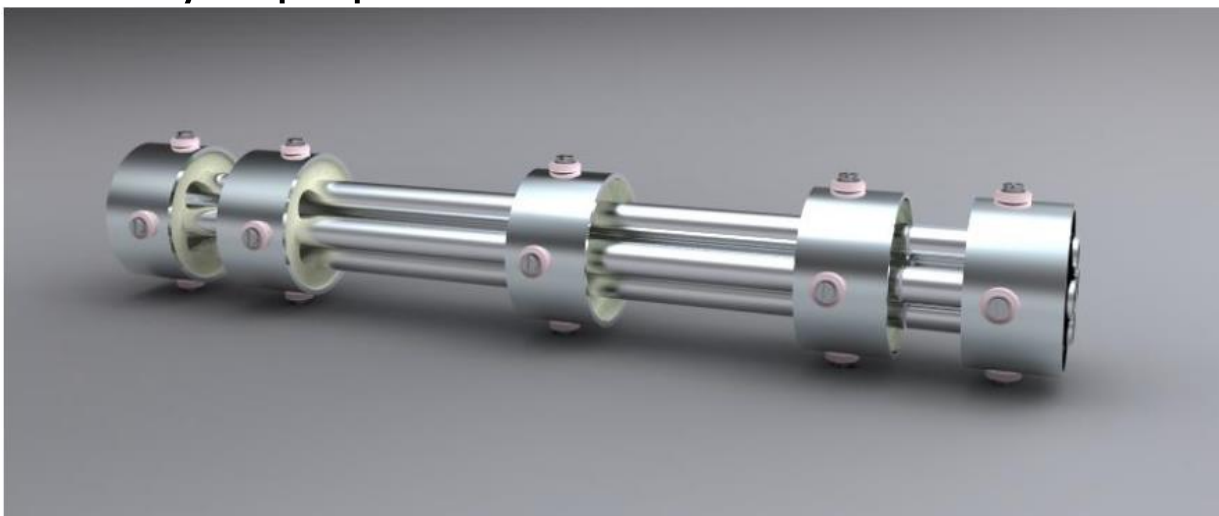
# Technical details of an EQP system

- For the analysis of the neutrals
  - Electron energy (0-150 eV)
  - Thermionic emission (0.2 – 2000  $\mu\text{A}$ )
- Extraction optics fully controllable by software discriminates +ve and –ve ions as well as e- and radicals



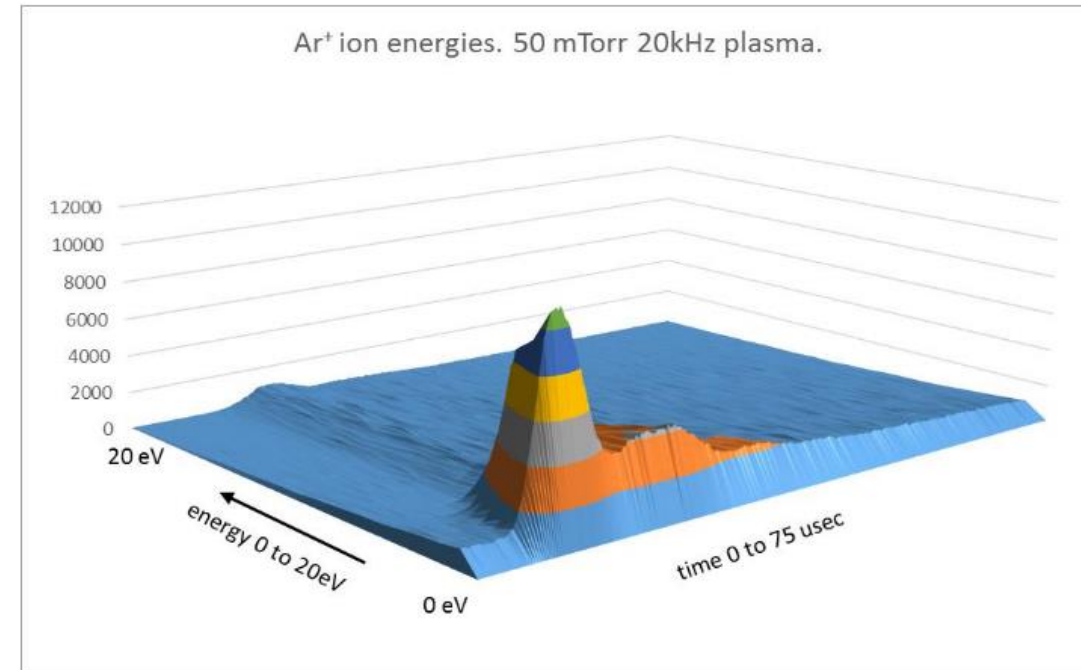
# Technical details of an EQP system

- Control over the quadrupole entrance and exit fields provides enhanced sensitivity for high mass transmission and increased abundance sensitivity
  - Enhanced long-term stability
- High sensitivity secondary electron multiplier (SEM)
- Faraday cup option



# Technical details of an EQP system

- Programmable Signal Gating
  - Signal gating input with 0.1 us resolution
  - Enhanced signal gating modes including programmable signal gating and MCS
  - Automatic background subtraction
  - Ion flight time measurements
- Multi-Channel Scalar (MCS) Device
  - Allow transient event analysis
  - Plasma ignition/modulation/extinction experiments
  - Ion flight time measurements

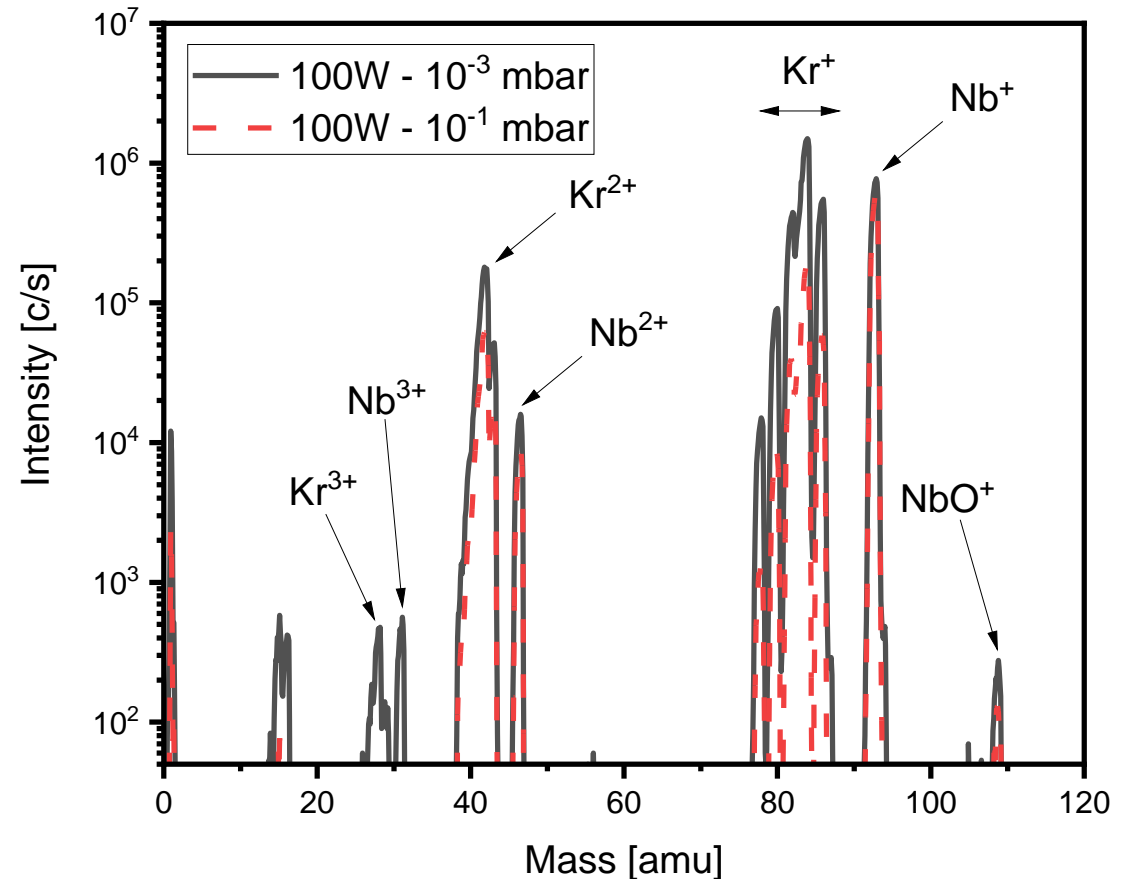


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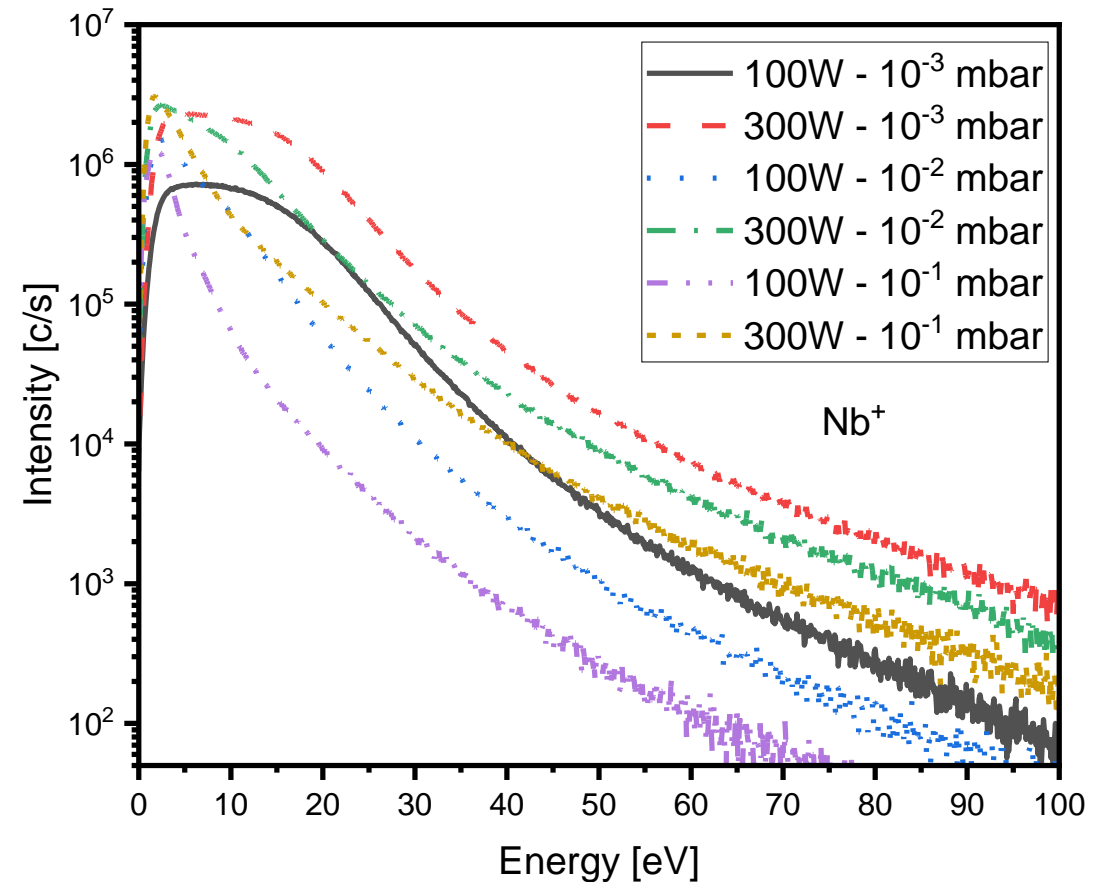
# Coating of Nb thin films: mass scan in HiPIMS

- Influence of pressure
  - Main ions: Nb<sup>+</sup> (93 amu), Kr<sup>+</sup> (84 amu), Nb<sup>2+</sup> (46 amu), Kr<sup>2+</sup> (42 amu) and NbO<sup>+</sup>(109 amu)
  - Formation of NbO<sup>+</sup> due by recombination with residual H<sub>2</sub>O
- At lower pressure, formation of Nb<sup>3+</sup> (31 amu) and Kr<sup>3+</sup> (28 amu)
- Presence of few residues (water)  
"rough" measurement (no baking)



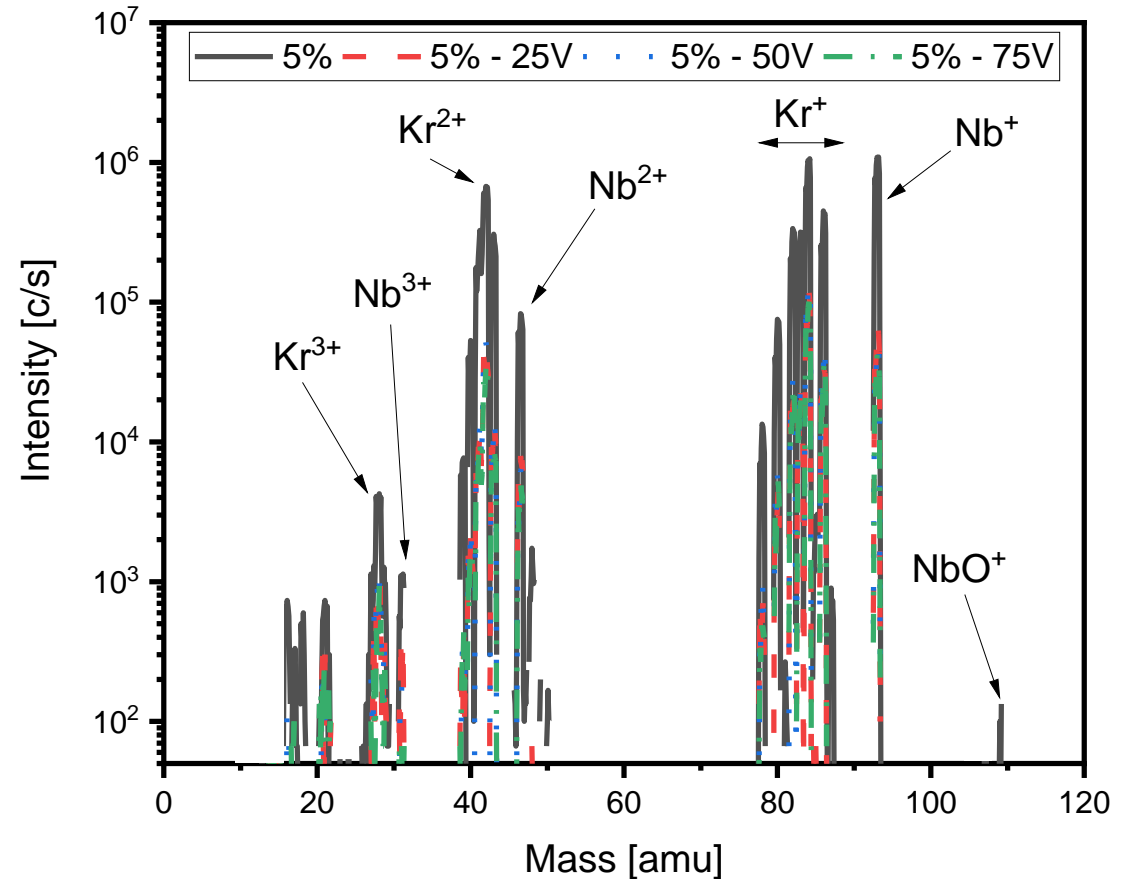
# Coating of Nb thin films: IEDF in HiPIMS

- Increase of pressure directly impact the ion energy
- At lower pressure, increase of input power benefit ion intensity. Whereas, at higher pressure the increase benefit both ion intensity and maximum energy
- Within a pressure range no significant variation

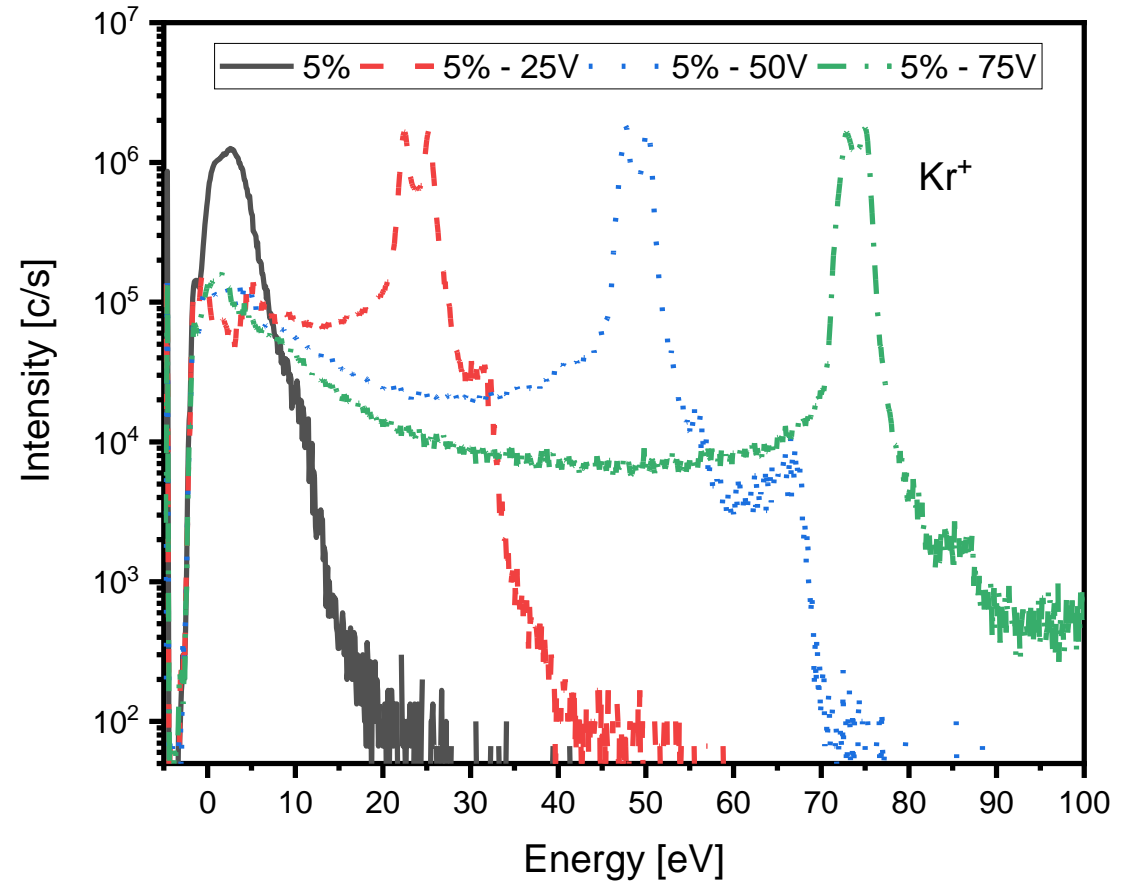
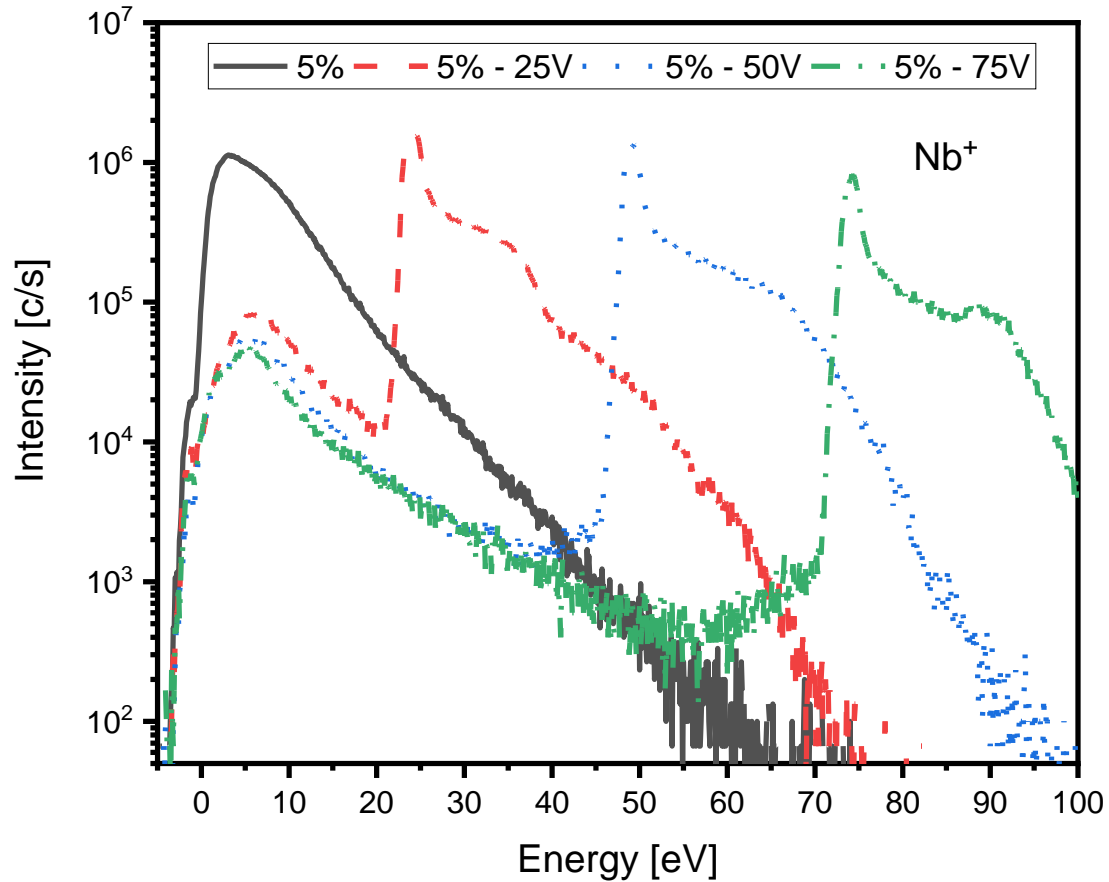


# Coating of Nb thin films: mass scan in bipolar HiPIMS

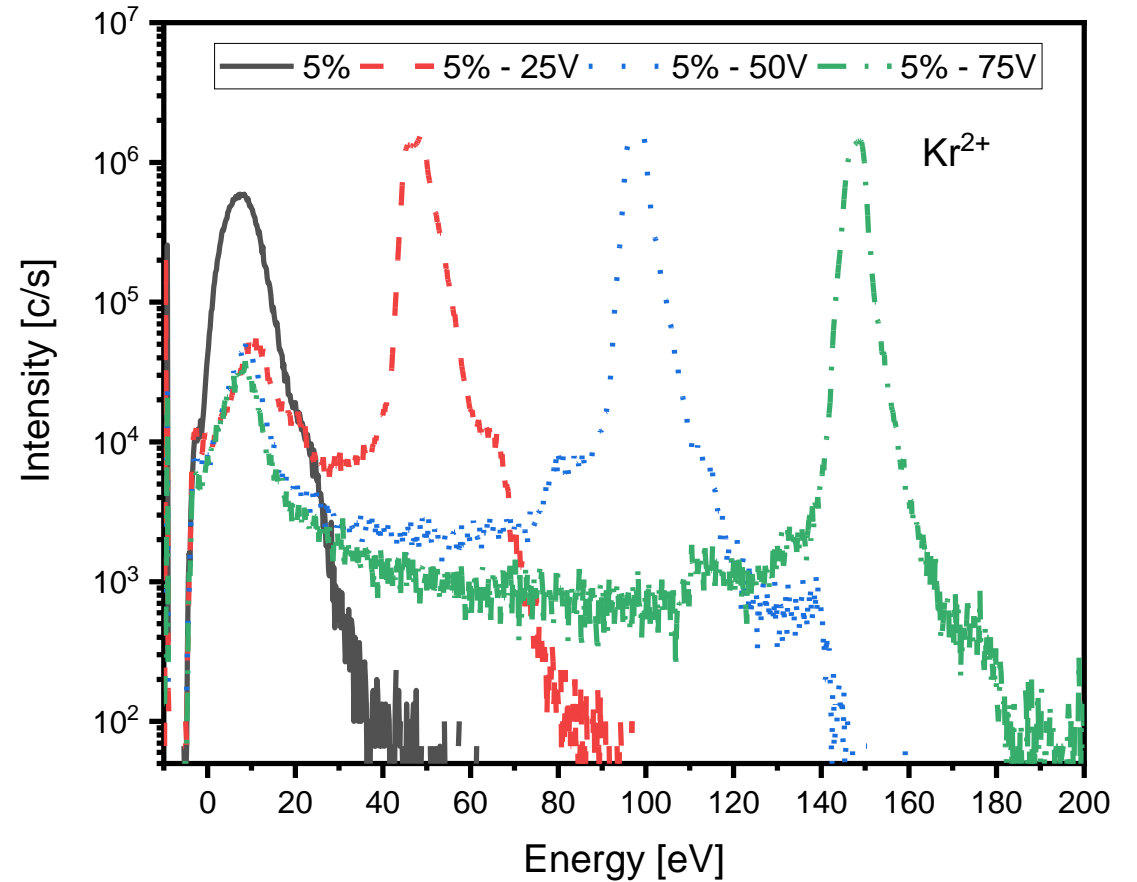
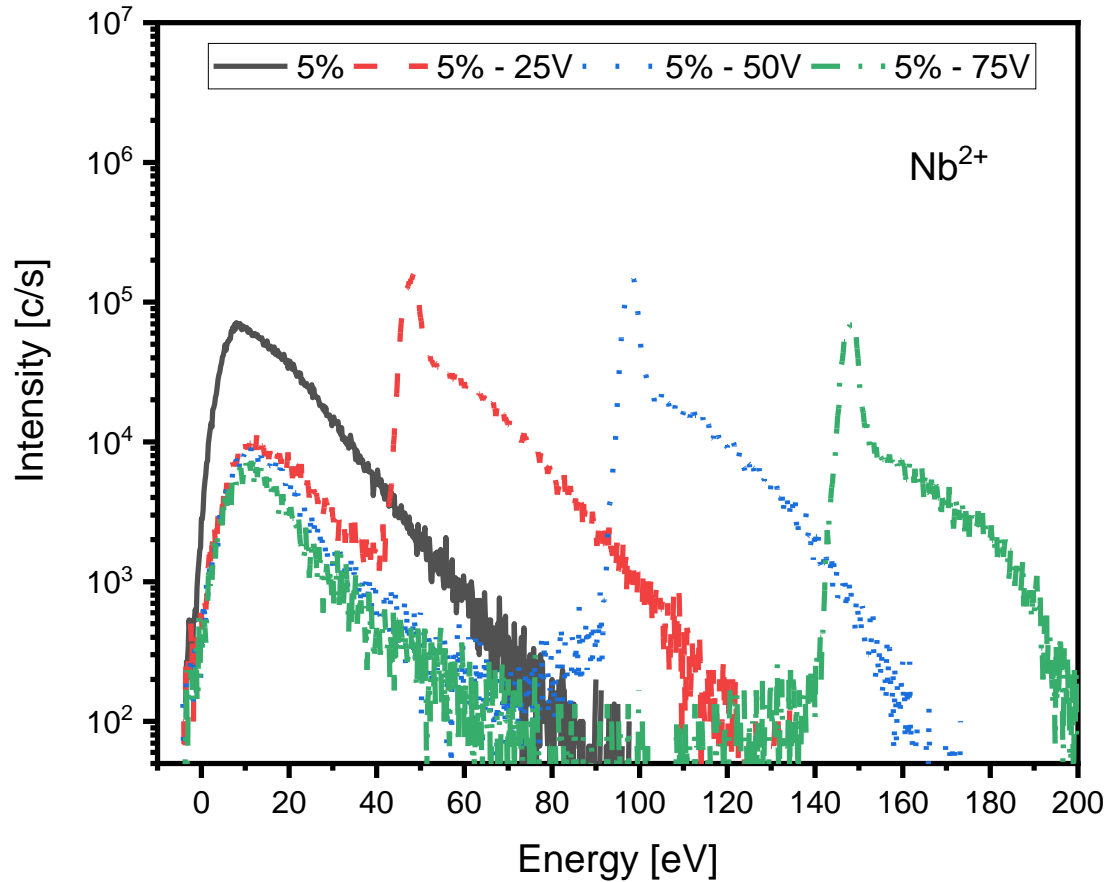
- Main ions:
  - $\text{Nb}^+$  (93 amu)
  - $\text{Kr}^+$  (84 amu)
  - $\text{Nb}^{2+}$  (46 amu)
  - $\text{Kr}^{2+}$  (42 amu)
  - $\text{Nb}^{3+}$  (31 amu)
  - $\text{Kr}^{3+}$  (28 amu)
  - $\text{NbO}^+$  (109 amu)
- Formation of  $\text{NbO}^+$  by recombination with residual O from water dissociation
- Decrease of intensity in bipolar mode



# Coating of Nb thin films: IEDF in bipolar HiPIMS

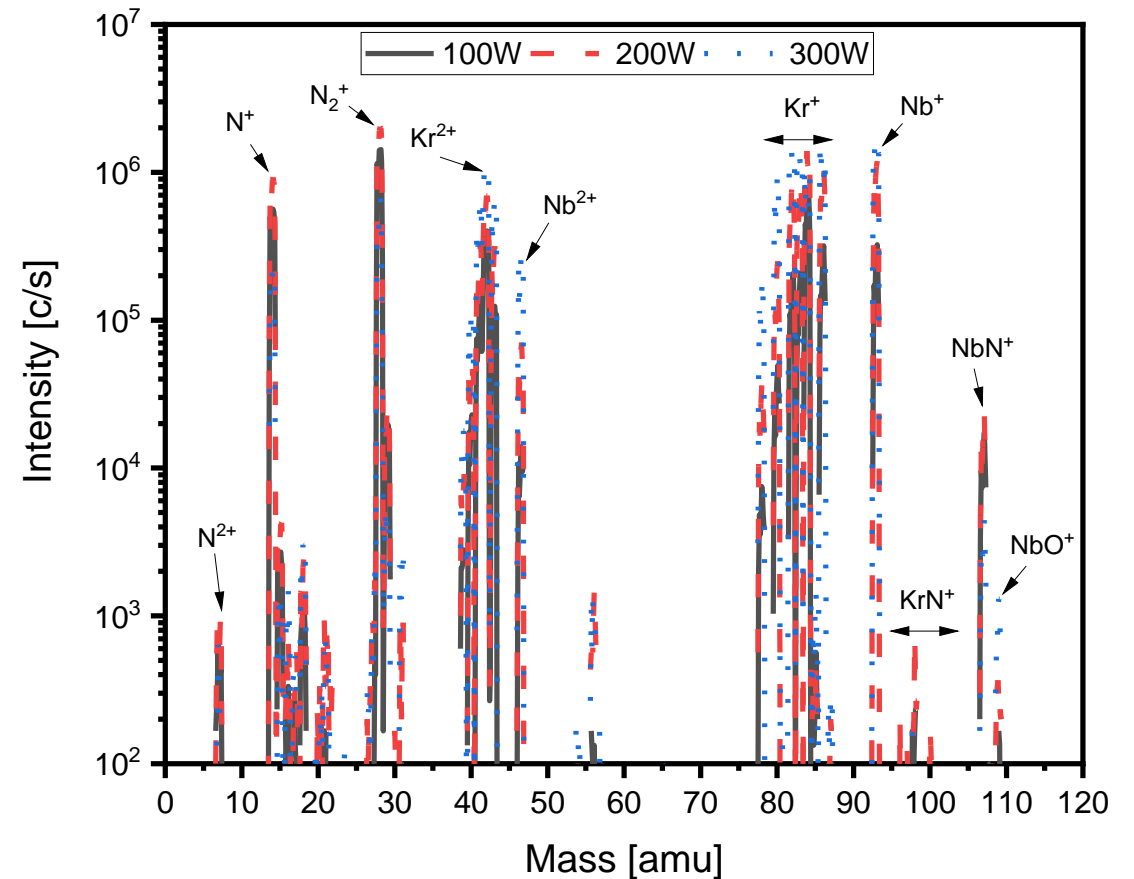


# Coating of Nb thin films: IEDF in bipolar HiPIMS

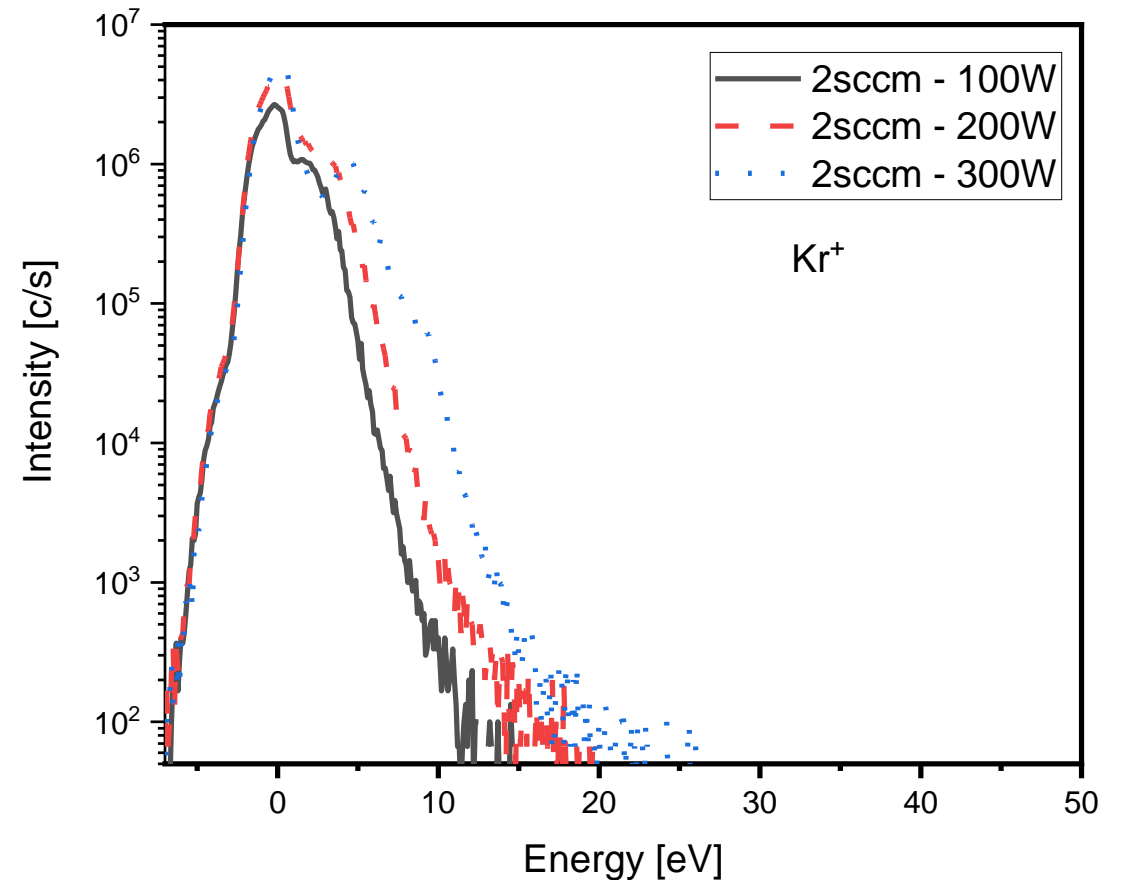
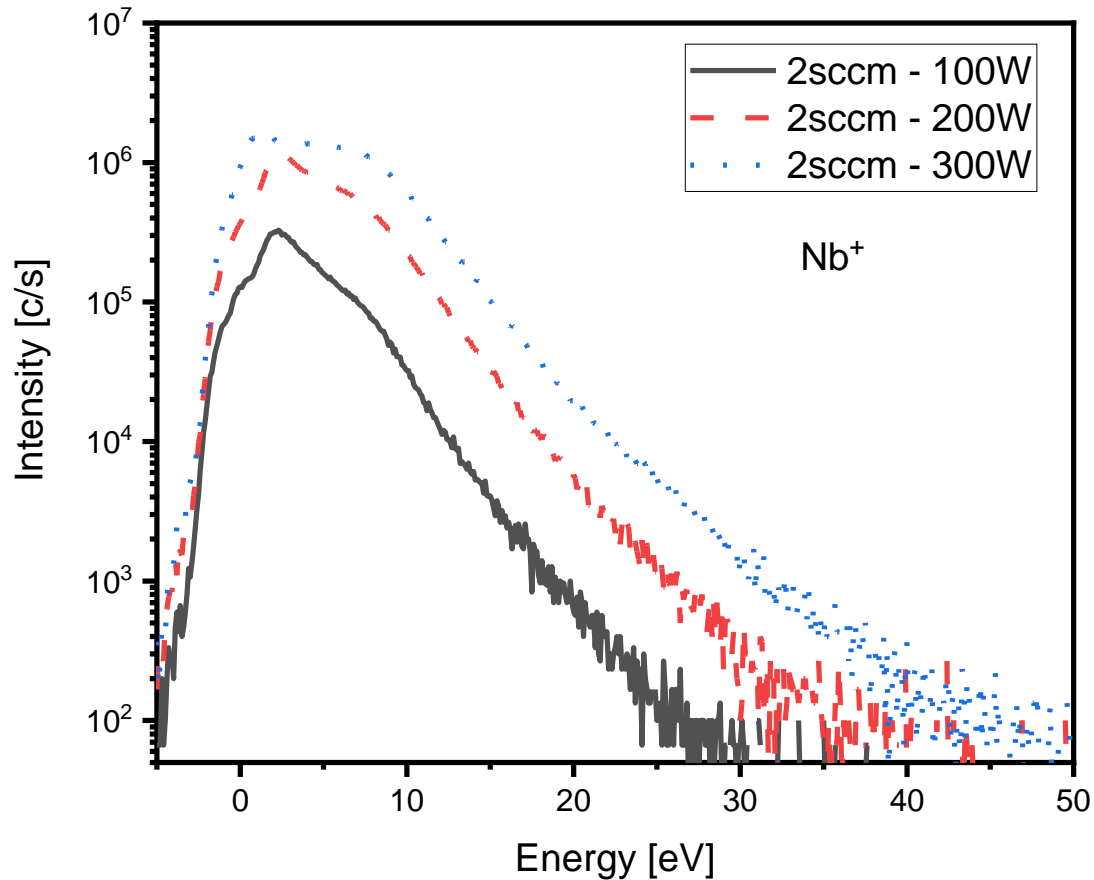


# Coating of other superconducting materials

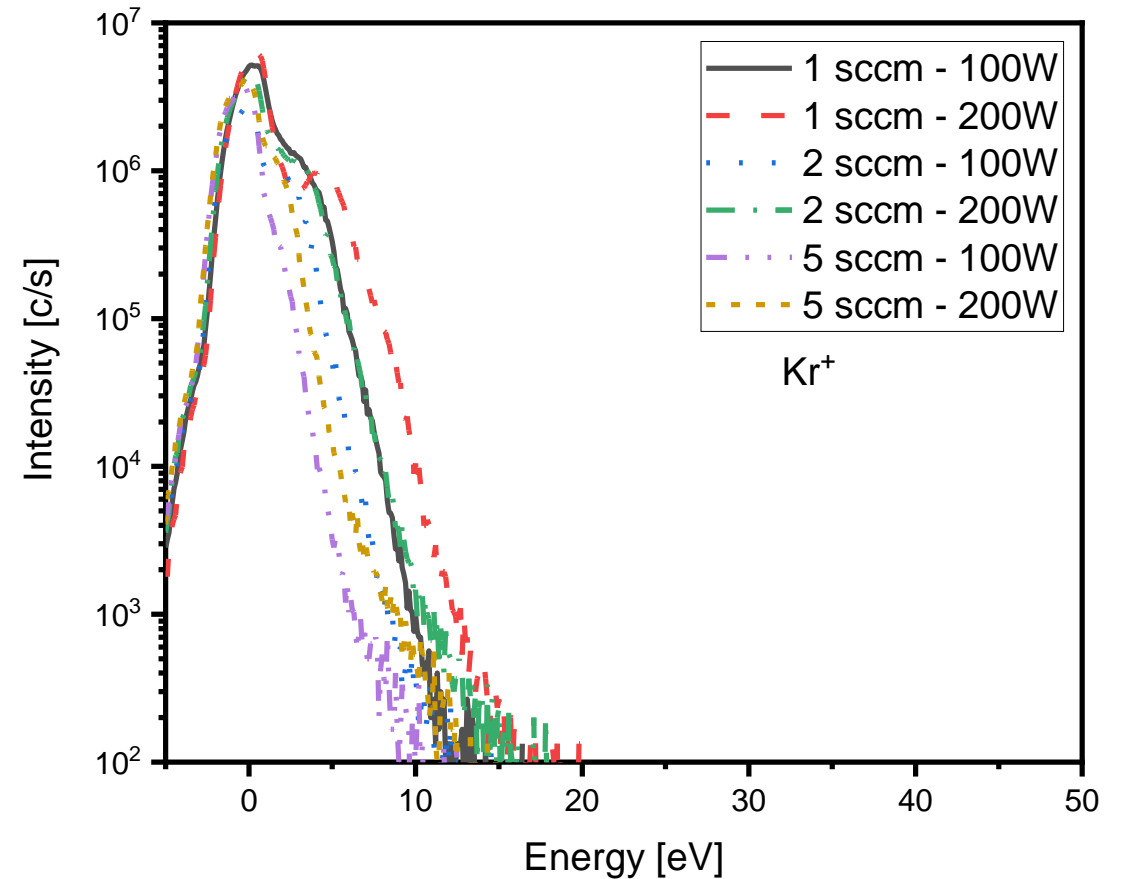
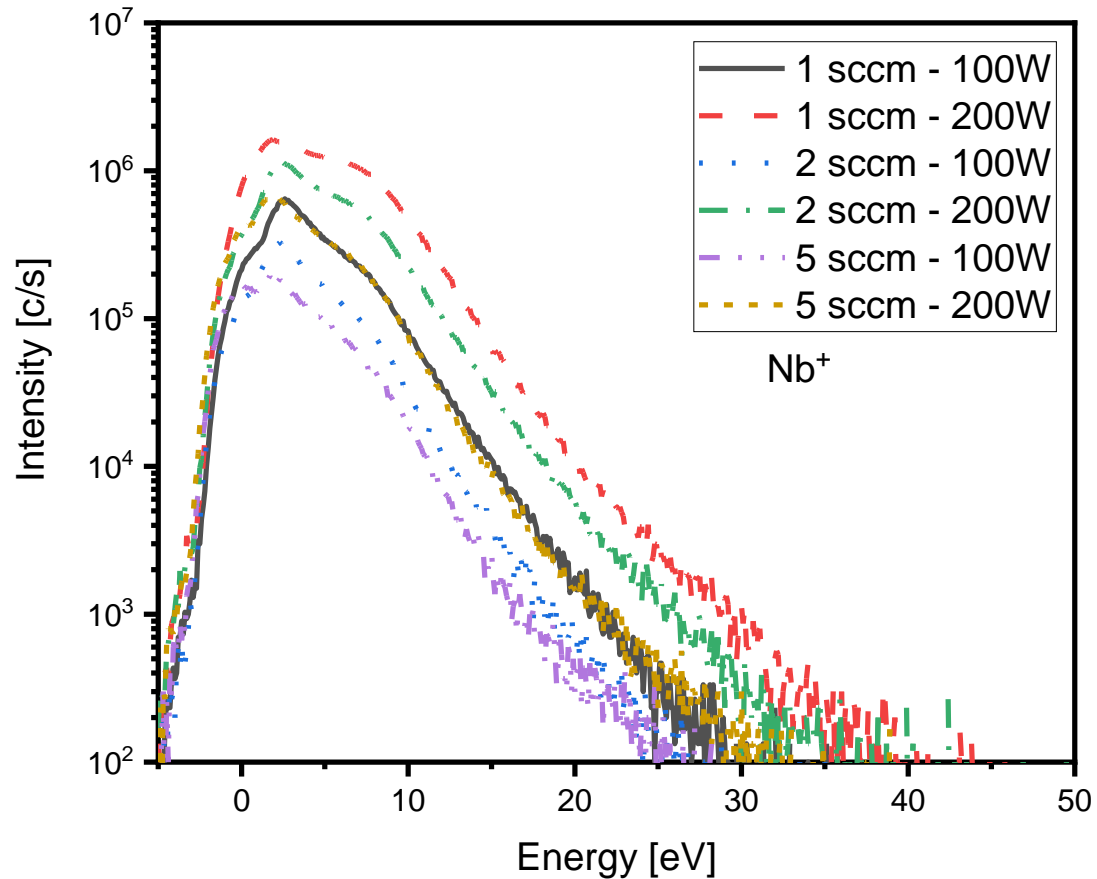
- Adapting results for the investigation of other superconducting materials such as NbN or NbTiN
- Presence of  $N_2$  induces a reduction of the main ions ( $Nb^+$ ,  $Kr^+$ ,  $Nb^{2+}$  &  $Kr^{2+}$ ). This can be compensated by increasing input power
- Recombination between nitrogen ions with niobium and krypton



# Coating of other superconducting materials: NbN



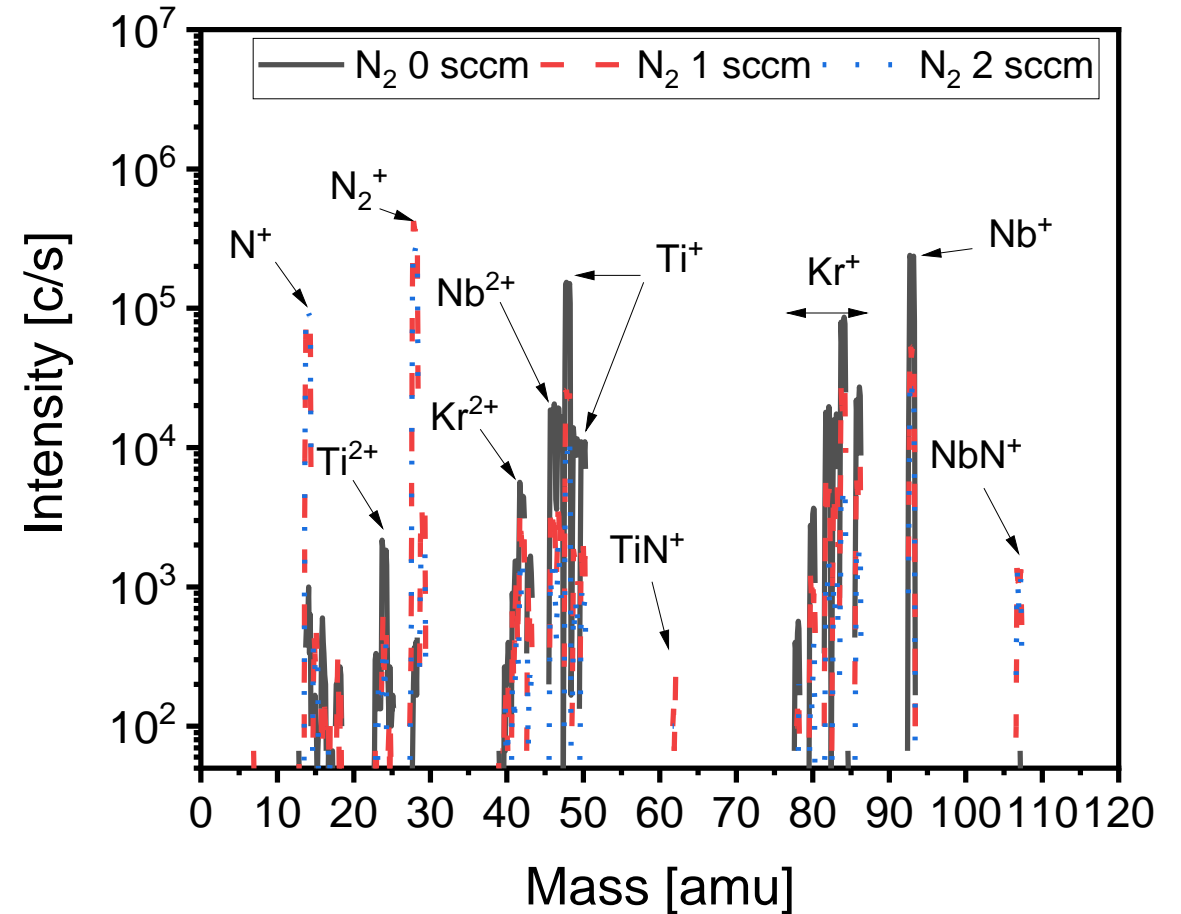
# Coating of other superconducting materials: NbN



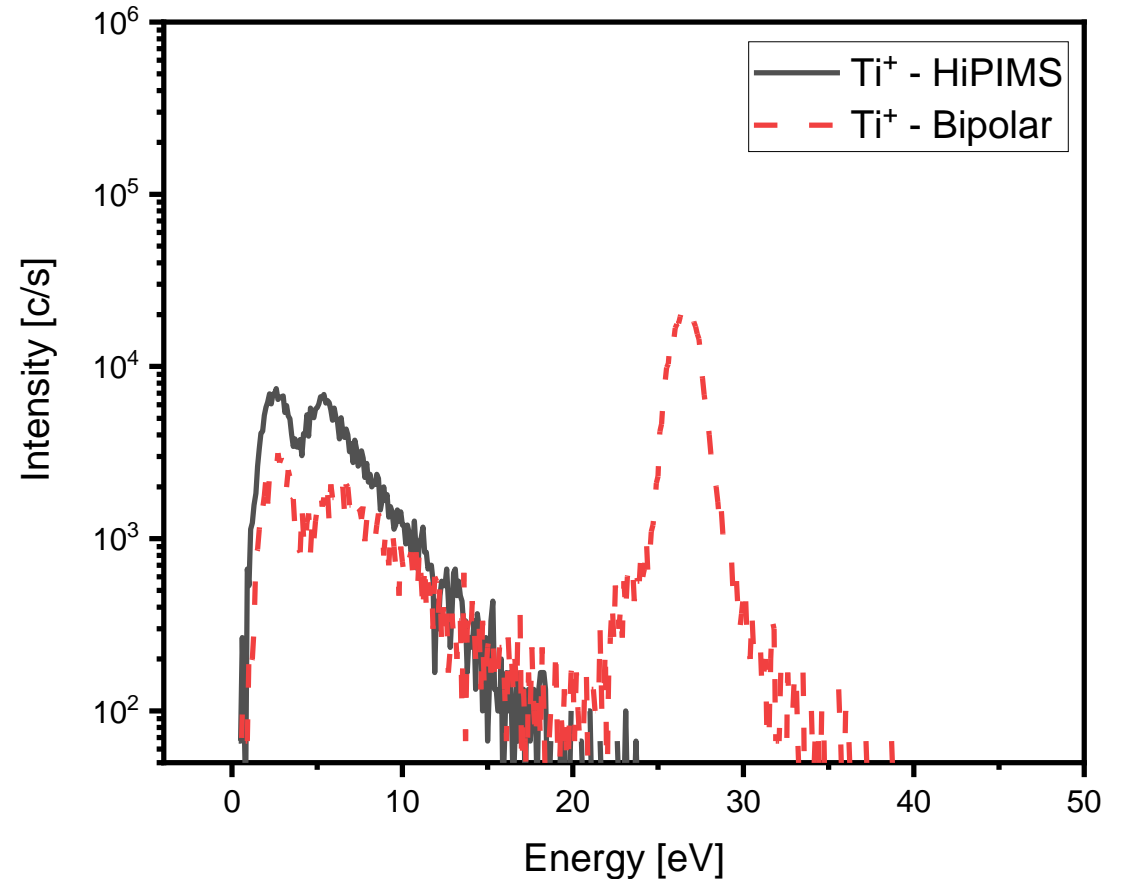
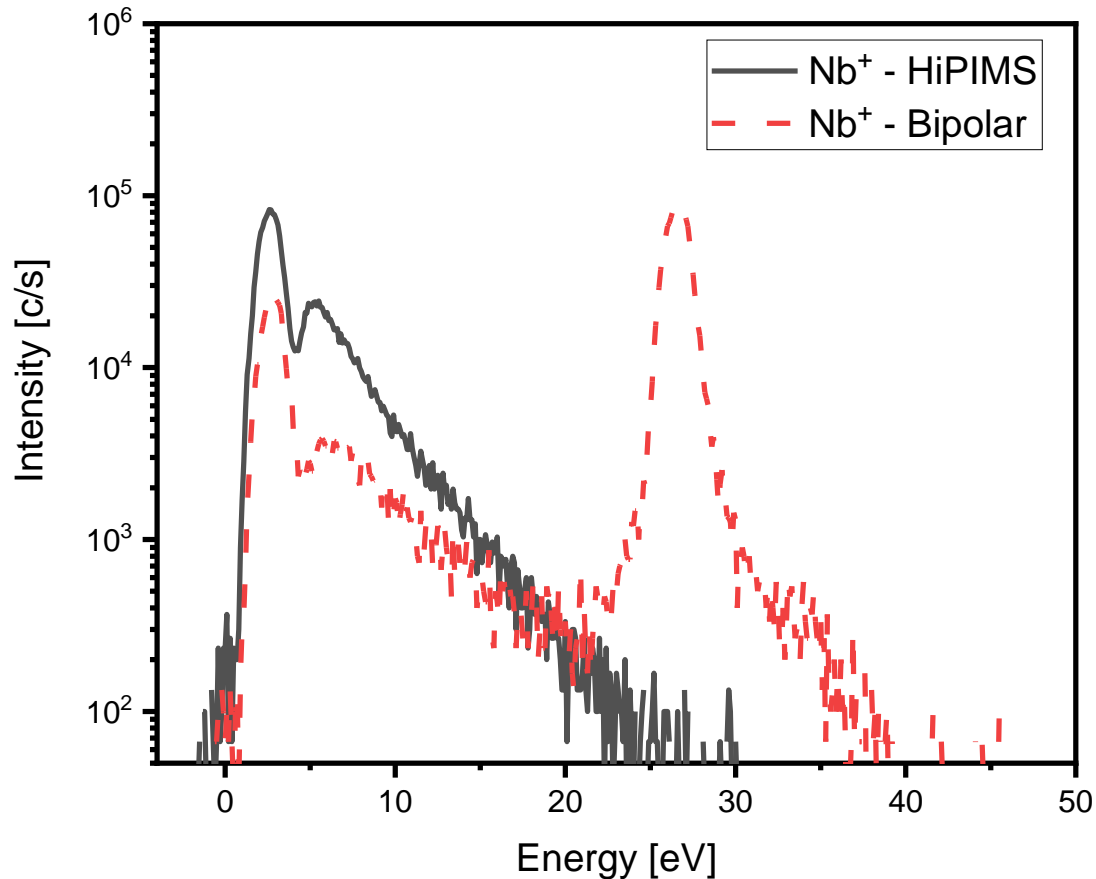


# Coating of other superconducting materials: NbTiN

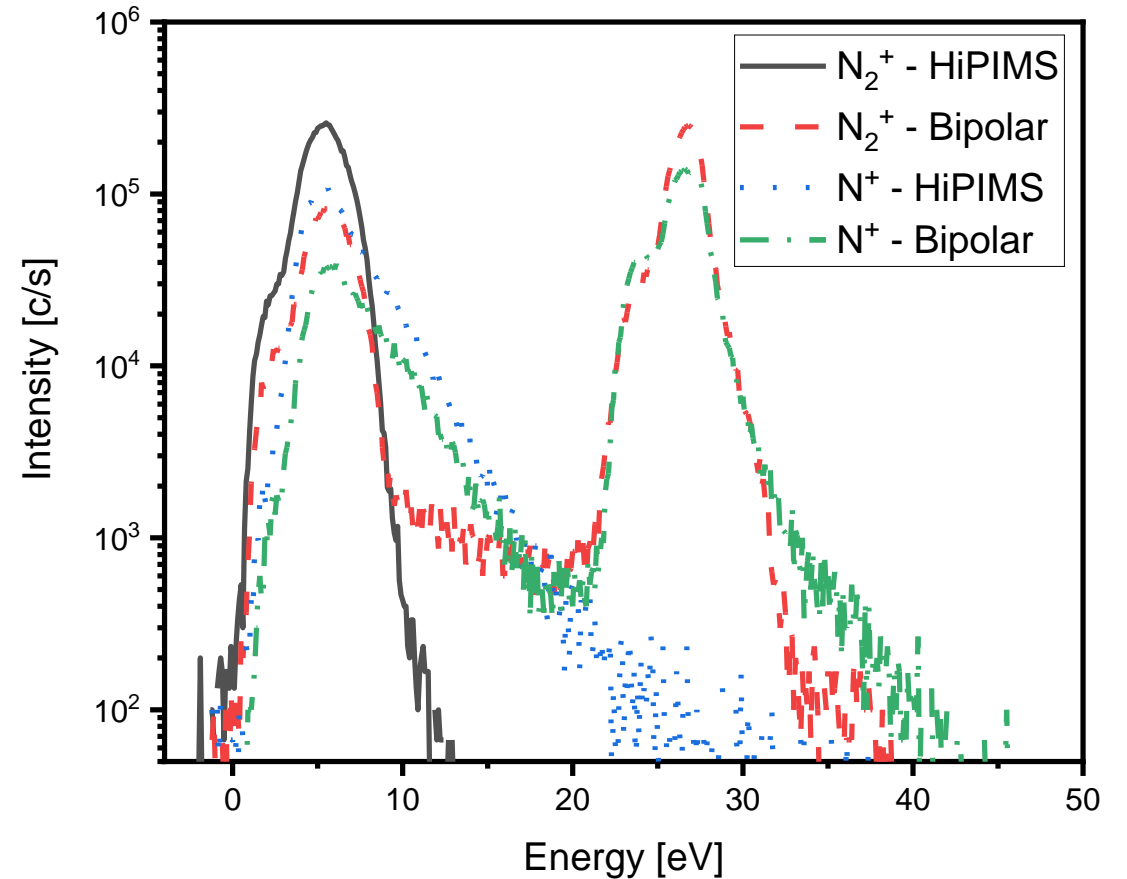
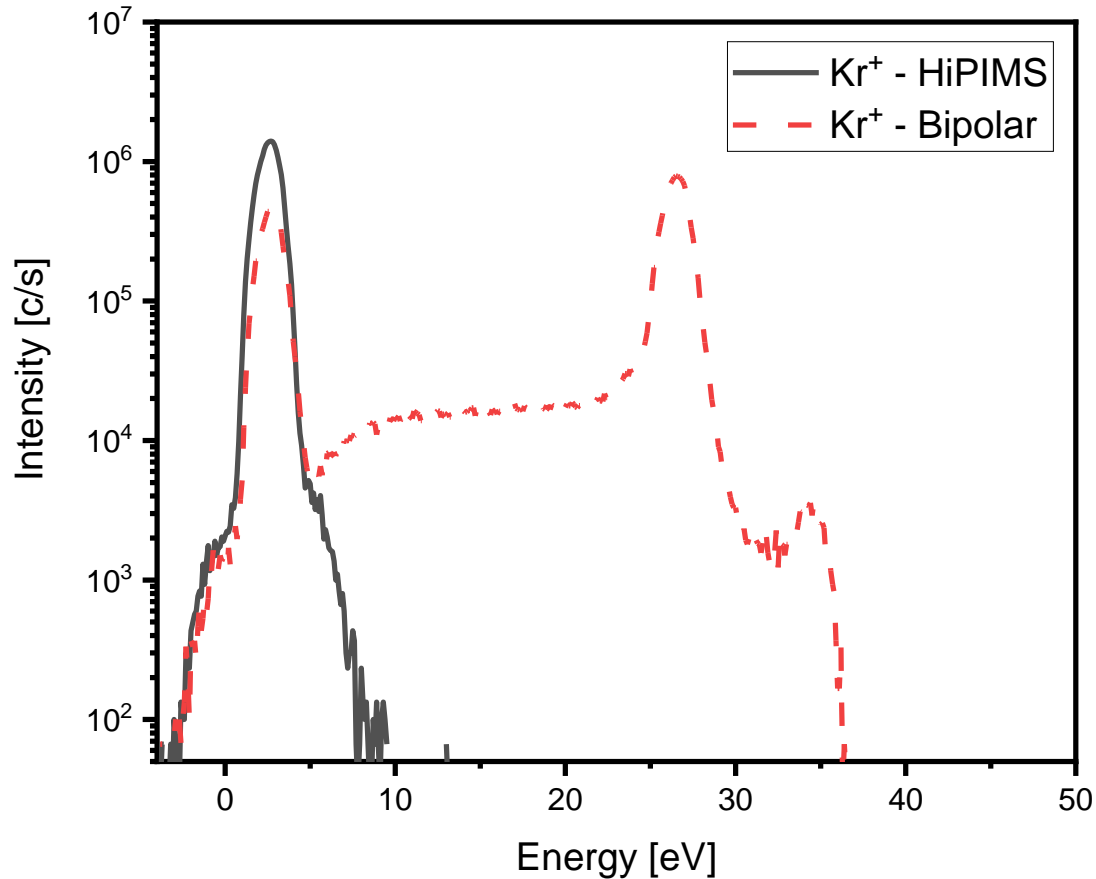
- Similarly to NbN,  $N_2$  impacts other ions intensity
- Increase of  $N_2$  flow rate favour its recombination with Ti
- Same fashion for Nb
- Unlike NbN study, Kr is not recombining with  $N_2$



# Coating of other superconducting materials: NbTiN



# Coating of other superconducting materials: NbTiN



# Summary

- EQP analyser can be customised for the mass/energy analysis of +ve/-ve ions and neutrals in plasma process used for thin film coating
- Both time –averaged / -resolved measurement available – capable to measure continuous or pulsed process
- Compatible (upon upgrade) with heated medium and reactive environment
- Ability to measure ion energies prior or during deposition could enhance thin film properties
- Addition of plasma diagnostic technique is key to improve thin film coating

# Acknowledgements

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# Coating Nb thin films: IEDF in HiPIMS

