HIPIMS discharge analysis by energy resolved mass spectrometry in a reactive atmosphere with Oxygen content

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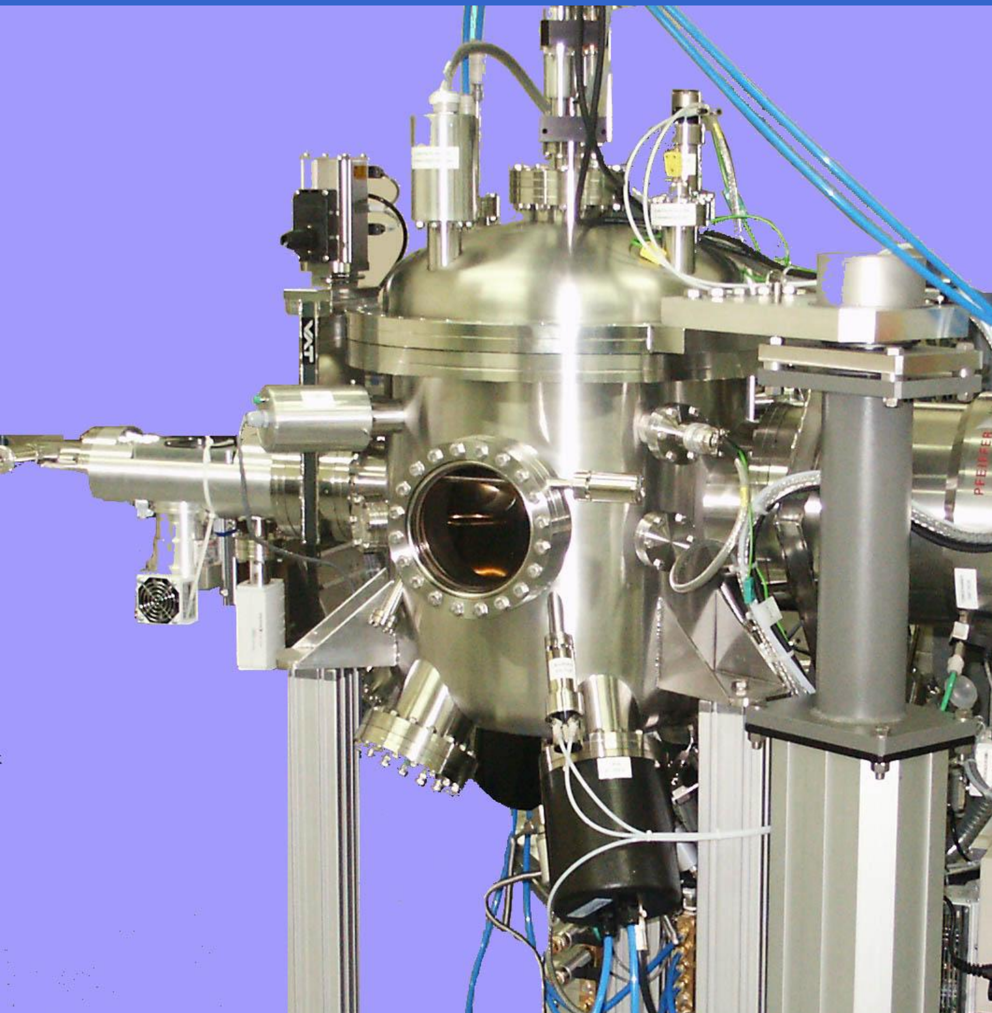
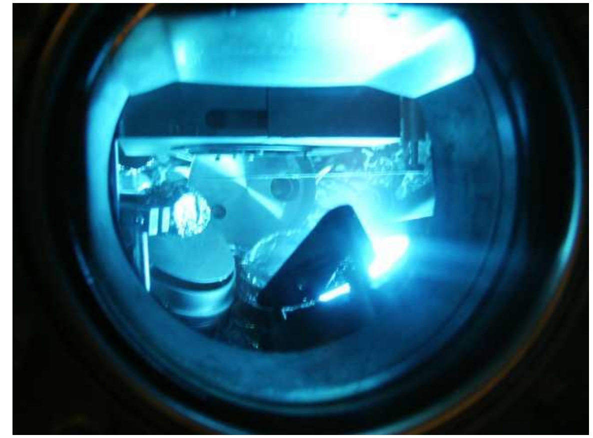
Introduction

This presentation includes details of:

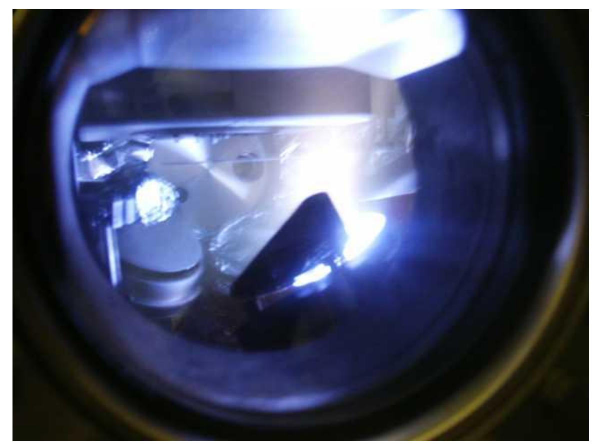
- **Description of the experiments in HIPIMS.**
- **Mass spectrometry analysis. Study of the ion energies by time average for Negative and Positive ions.**
- **Mass spectrometry analysis of the ion concentration depending on the air flow.**
- **Cross-sectional TEM analysis and electron diffraction pattern analysis of the films deposited by HIPIMS.**

Experiment set up



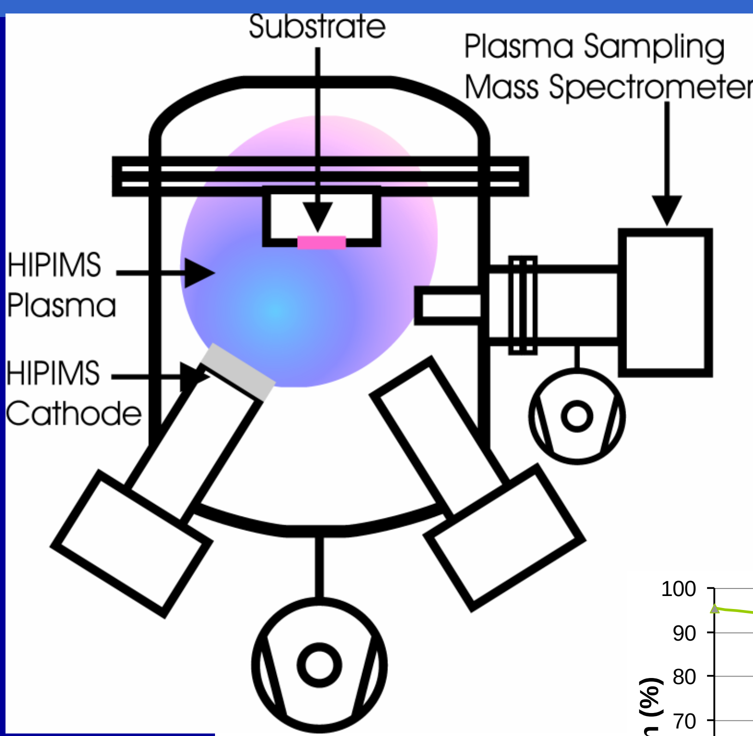


HIPIMS



conventional DC- sputtering

Experiment conditions



- Pressure:1-1.7 Pa

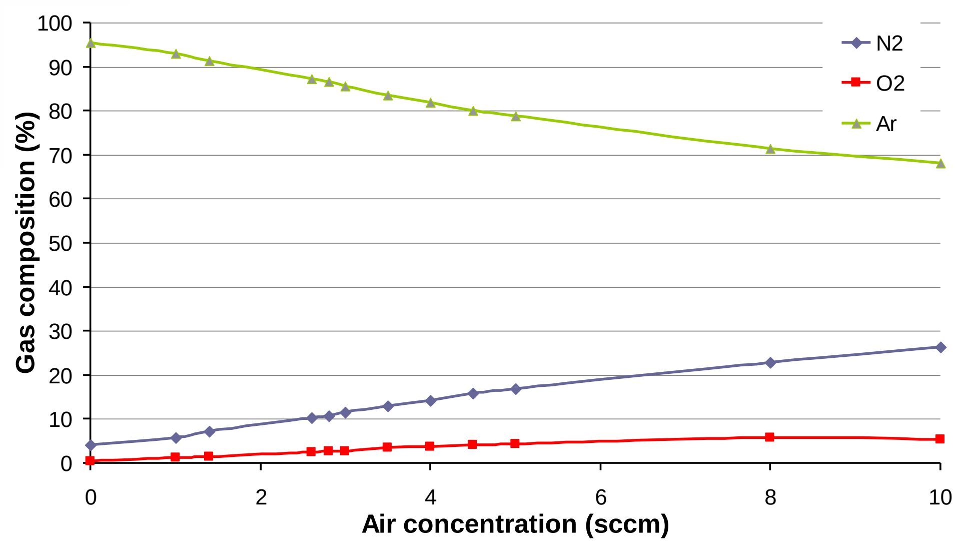
Ar:Air partial pressure ratio ranging from 45:1 to 45:10

- Peak current: from 3 to 10 A.

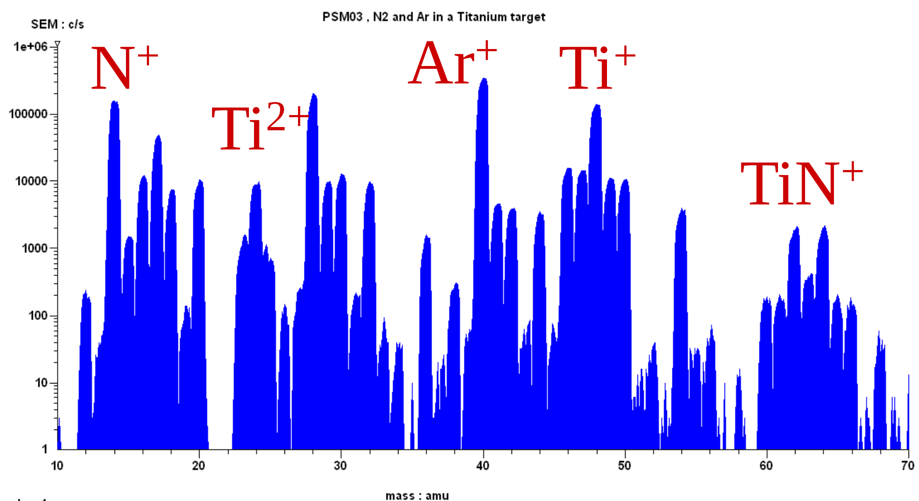
With a pulse duration of 200 μs.

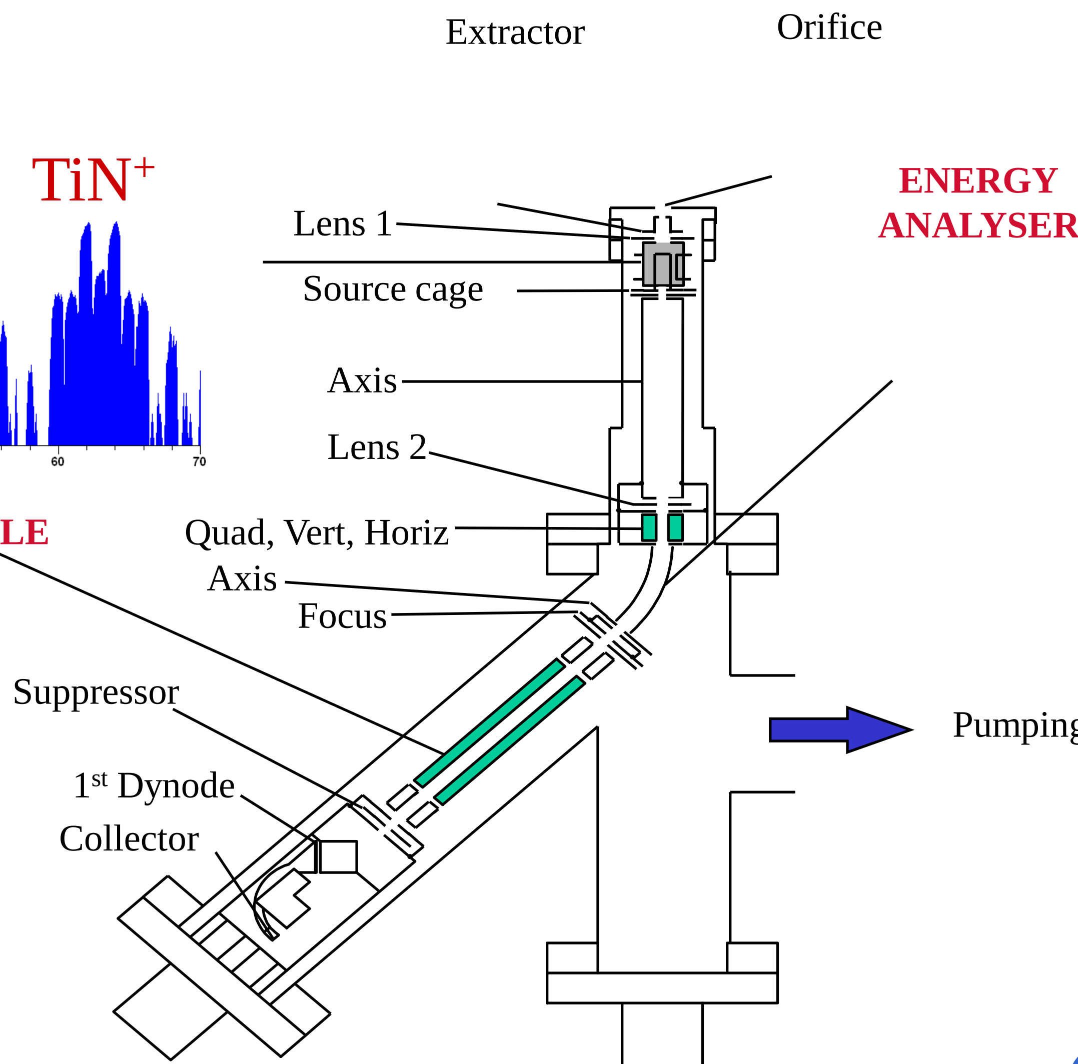
- Frequency: from 100 to 400 Hz

- Average power: 0.6 kW

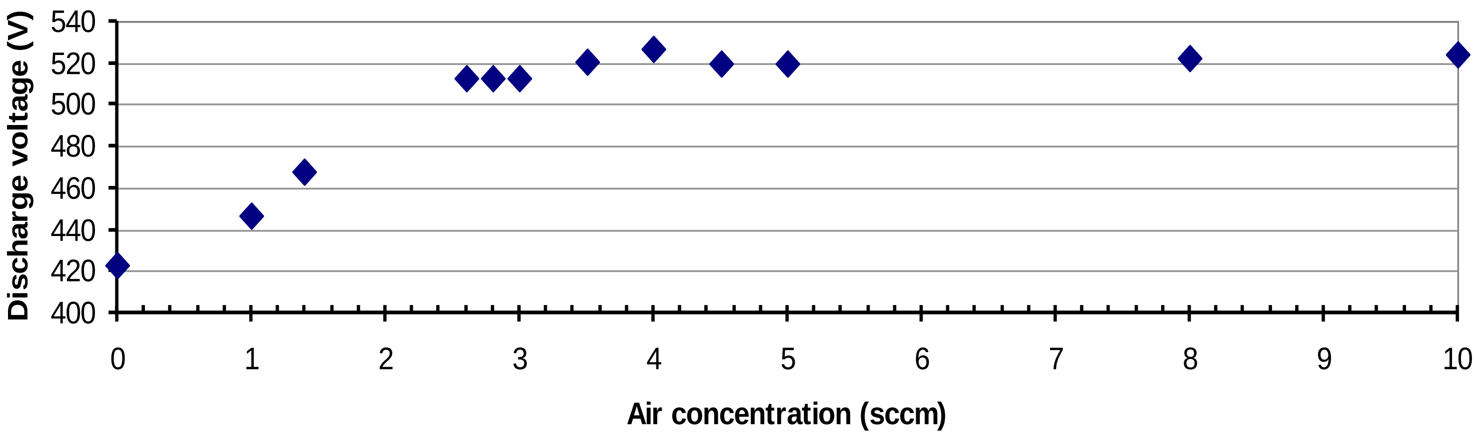


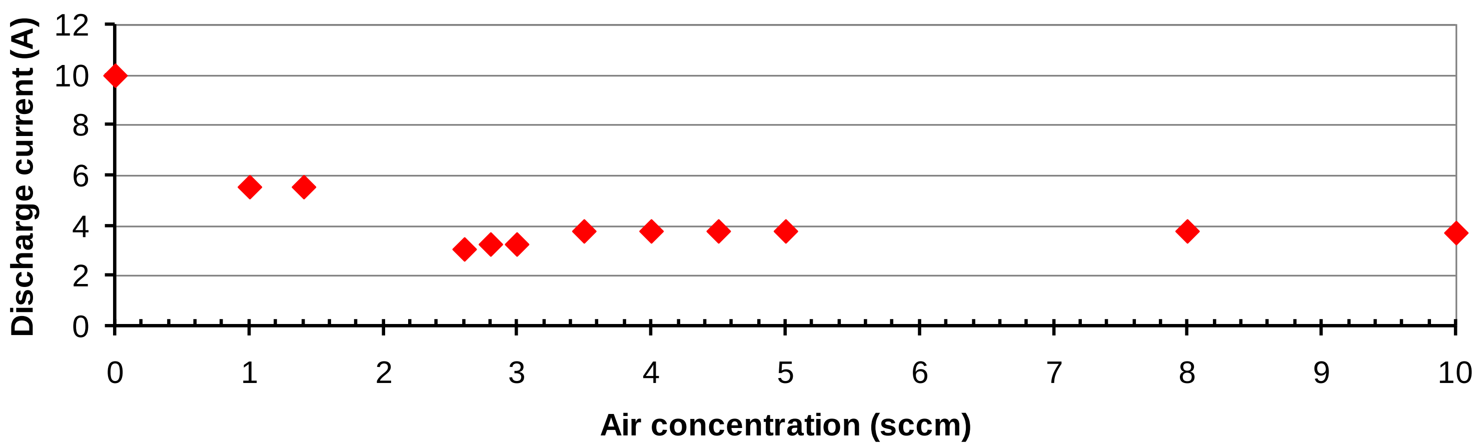
The Hiden EQP Mass/Energy Analyser



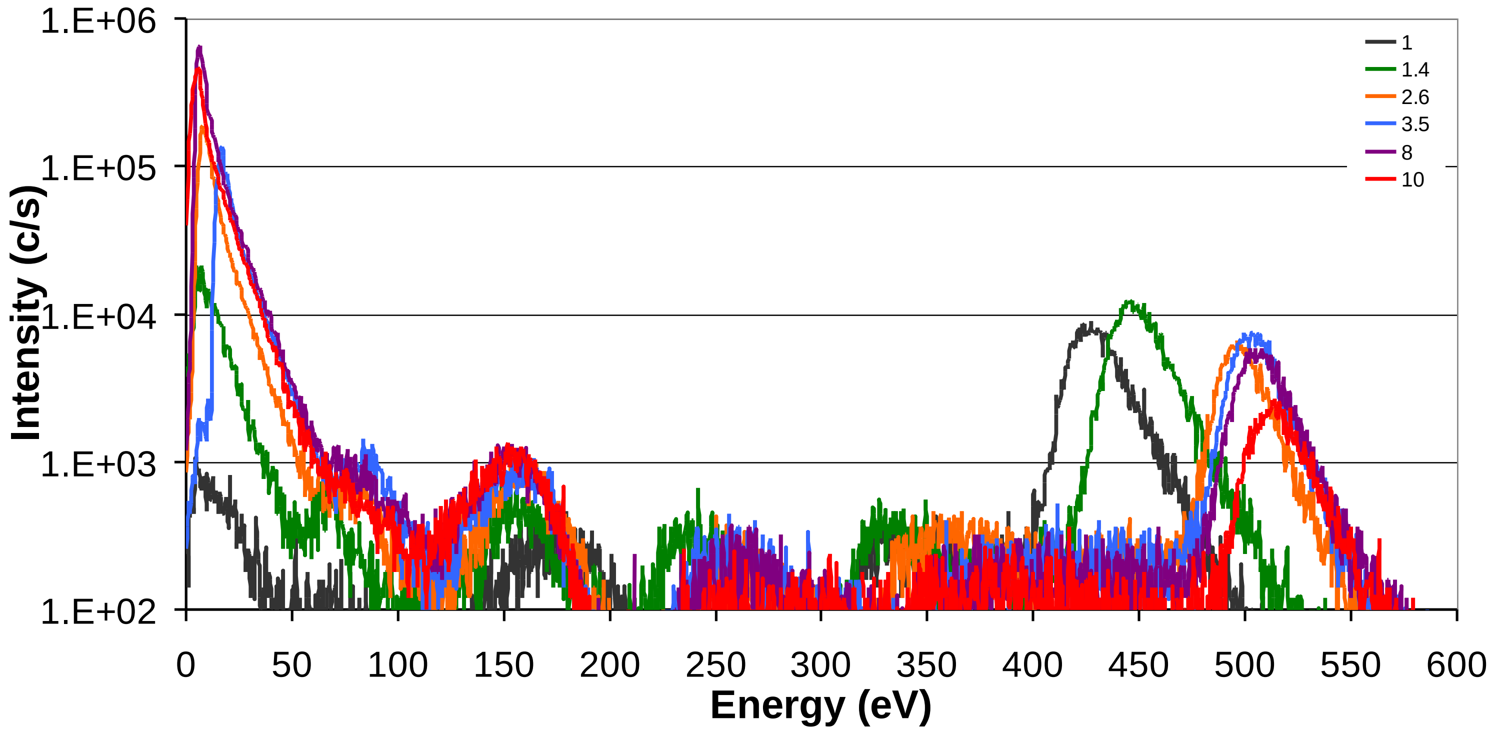


Discharge current and voltage in function of air concentration



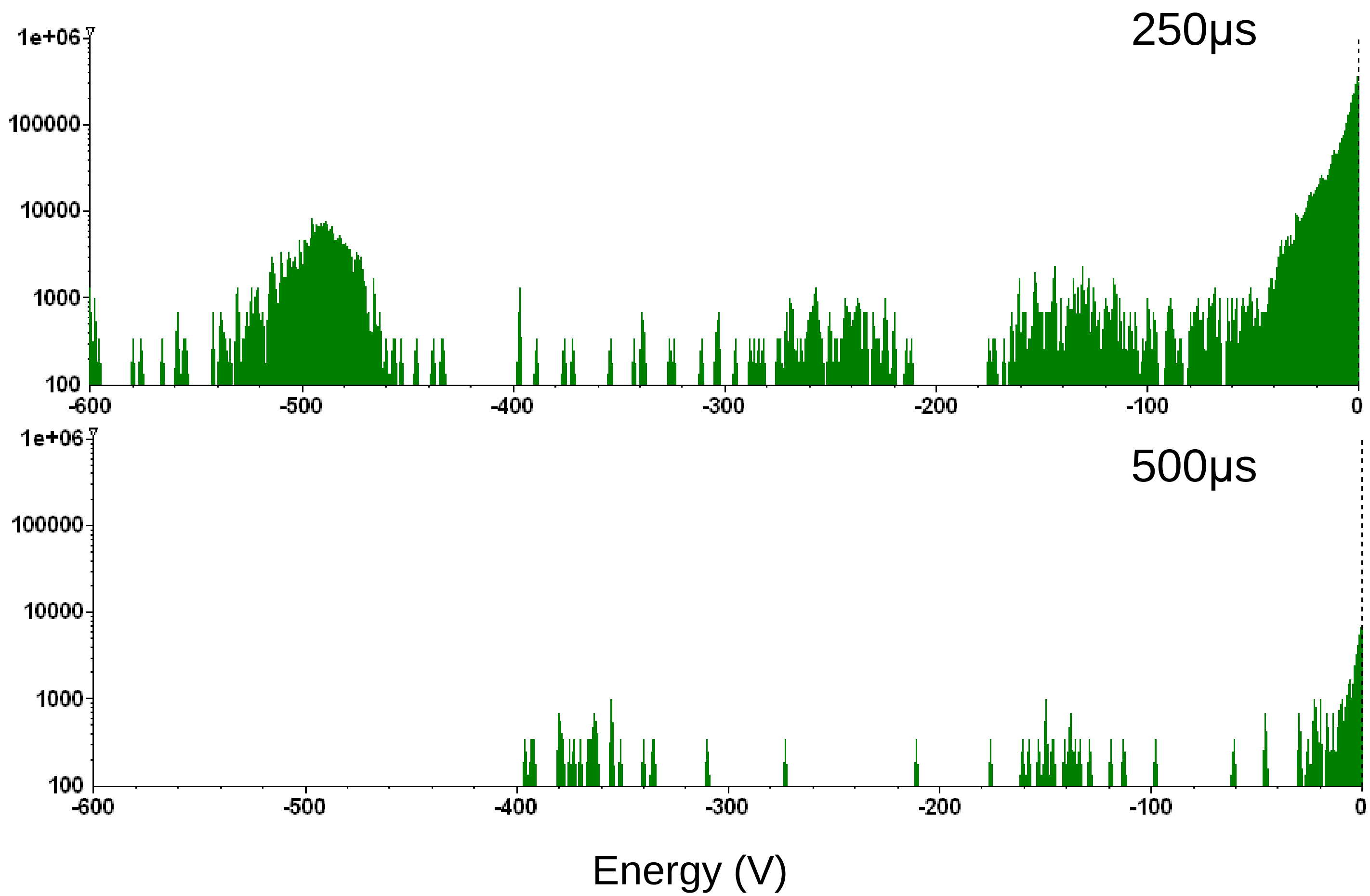


IEDF for Negative ions O^-



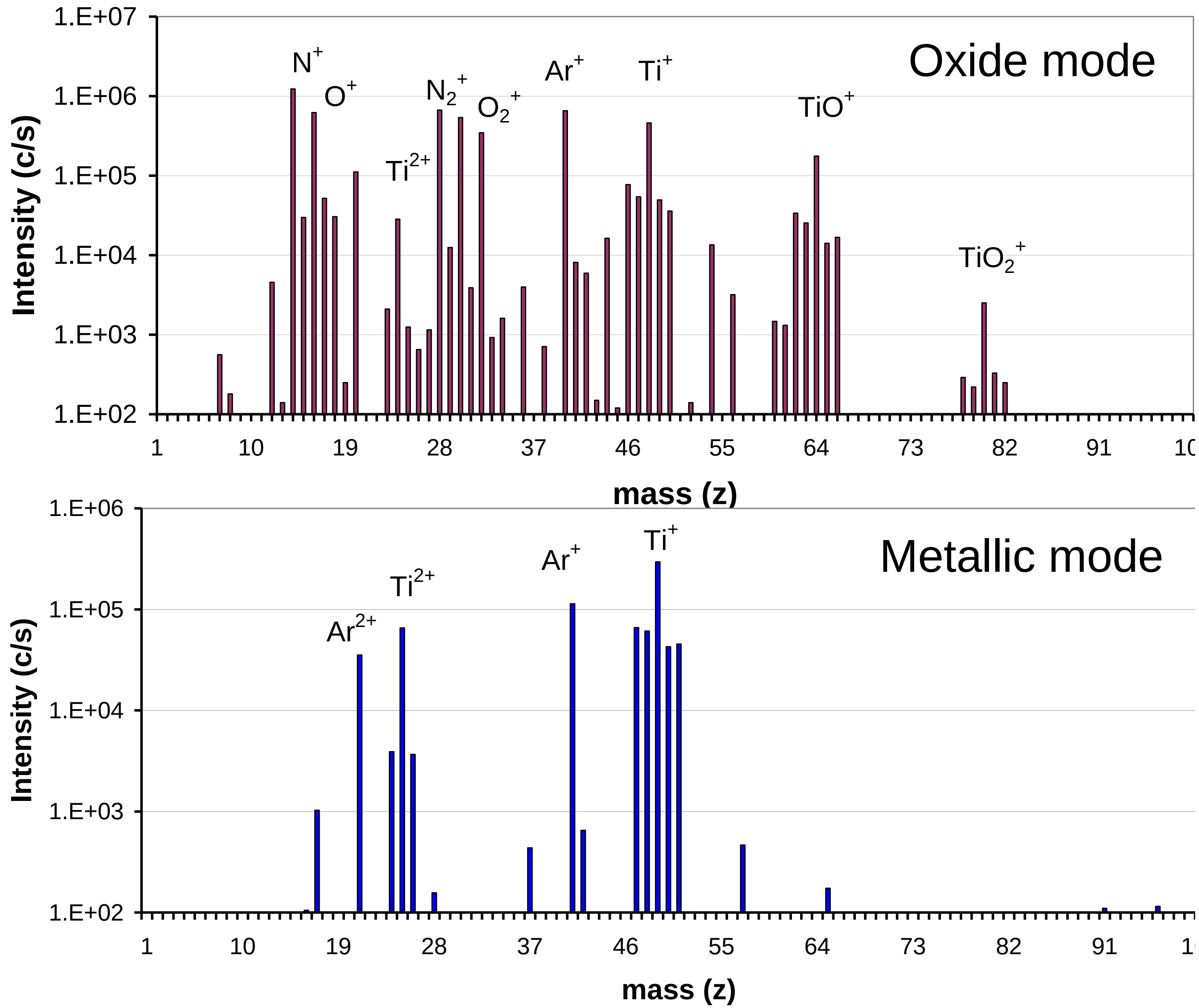
The high-energetic tail represents negative ions, O^-, which have been created close to the target

Time resolved measurements for negative ions, at 3.5 sccm air flow



Gate width 250 µs

Mass spectra of positive ions for metallic and oxide mode



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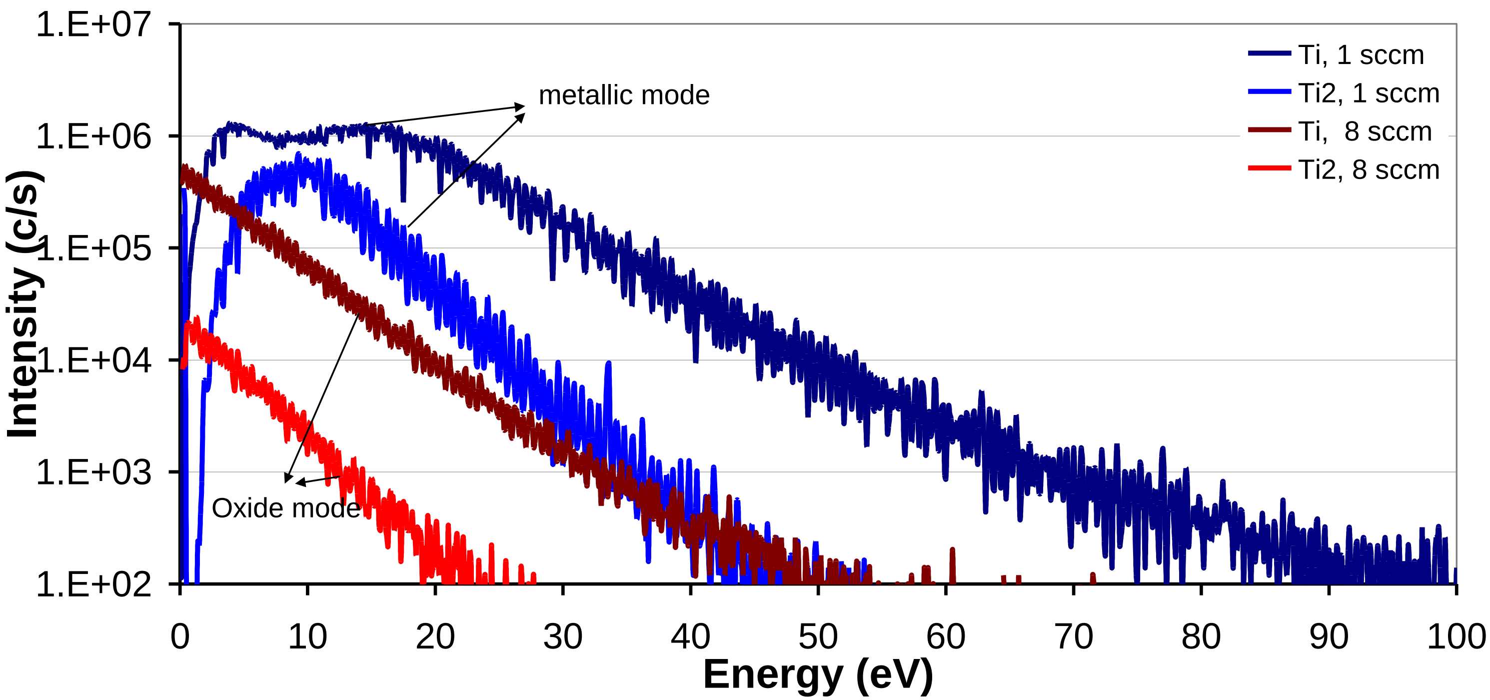
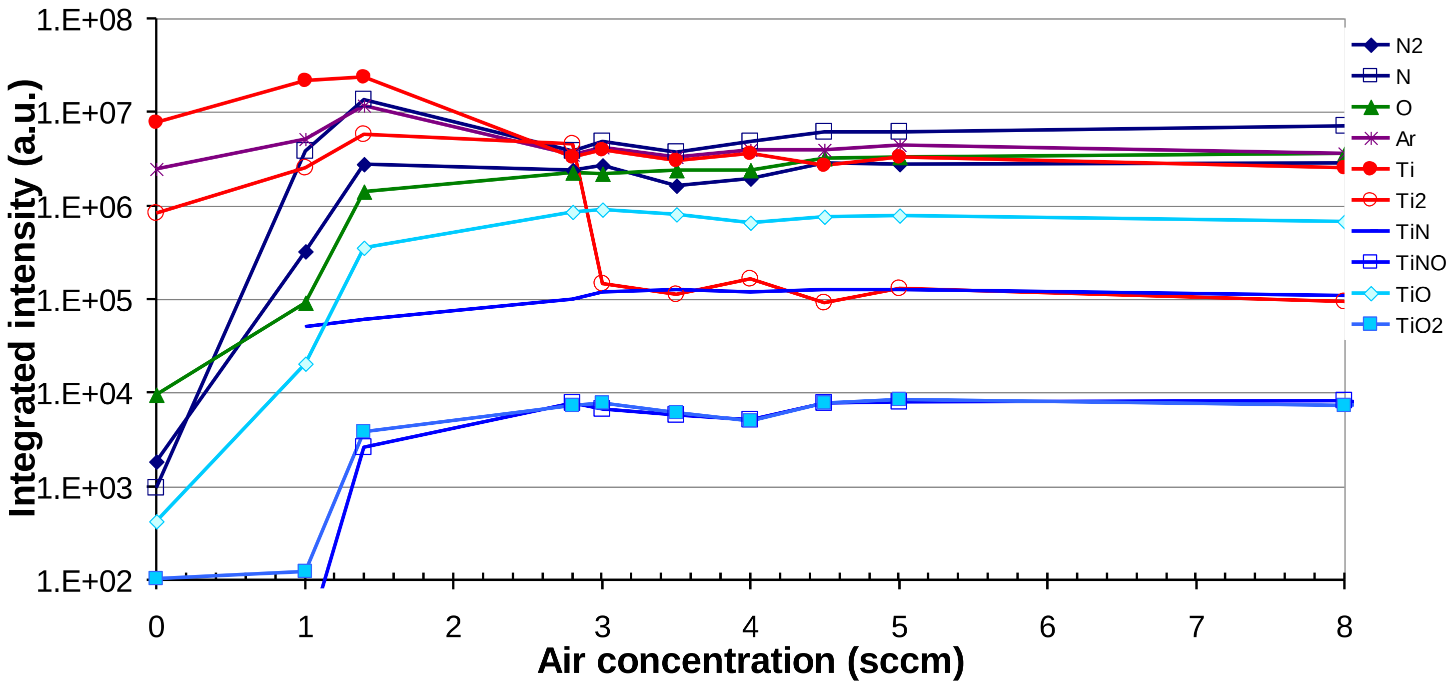
IEDF, positive ions



Energy of the ions change with air concentration. Note, 1sccm contains 1.18% O_2 and 5.7% N_2 and 8sscm contains 5.7% O_2 and 22.8% N_2.

Positive ions



Noticeable change in Ti^{2+} intensity by increasing the air concentration at 3sscm (2.75% O_2 and 11.55% N_2).

Ratio of ion concentration for positive ions

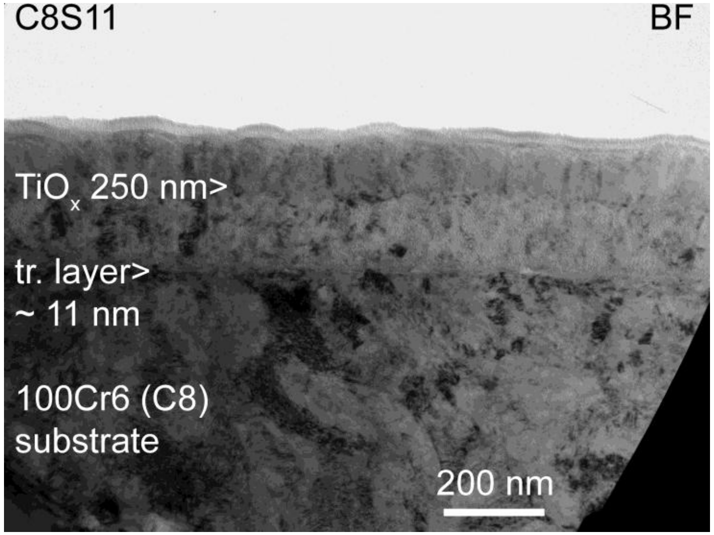
	Air Flow (sccm)									
	0	1	1.4	2.8	3	3.5	4.5	5	8	10
N^+/N_2^+	0.52	11.67	4.94	1.60	1.77	2.29	2.14	2.19	2.51	3.04
T^{2+}/Ti^+	0.11	0.12	0.25	1.41	0.04	0.04	0.03	0.04	0.04	0.03
Ti^+/Ar^+	3.20	4.17	1.99	0.94	0.95	0.90	0.69	0.75	0.70	0.94
Ti^+/O^+	86.79	27.92	4.08	2.05	0.07	0.05	0.03	0.04	0.03	0.02
TiO^+/Ti	0.00	0.00	0.02	0.27	0.23	0.27	0.28	0.23	0.27	1.03

The secondary electron emission caused by ion impact is slightly modified by a partial poisoning of the target. Above the critical flow of oxygen less target is sputtered. It would be a reduction of the deposition rate . The change to the oxide mode modifies the secondary electron emission coefficient leading to an increase in the discharge voltage. The electric field becomes weaker and the ion densities decreases as observed from the N^+/N_2^+, T^{2+}/Ti^+, Ti^+/Ar^+, Ti^+/O^+ ratios. The contribution of oxide molecules becomes more important as observed for the TiO^+/Ti^+ ratio. This could be a indication of less atomic Ti being sputtered.

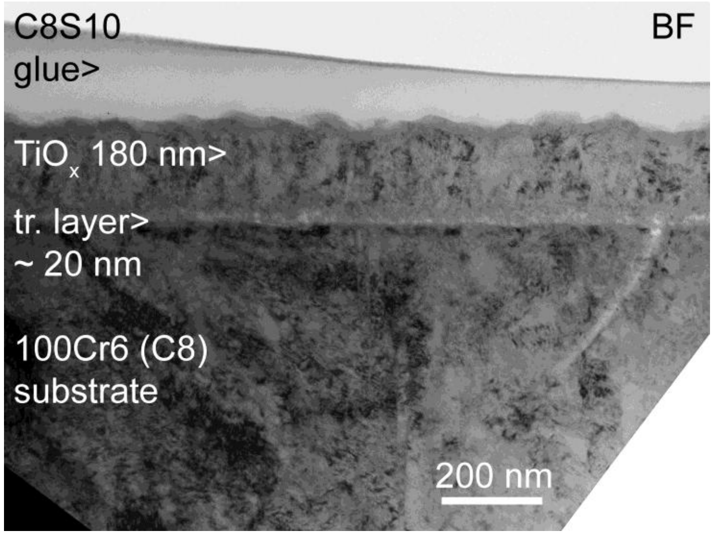
Coatings structure: metallic and poisoned mode

Cross-sectional TEM micrographs analysis of the films present a very dense polycrystalline and columnar structure.

Grain size (lateral): 50-100 nm



2 sccm air
metallic mode



10 sccm air
poisoned mode

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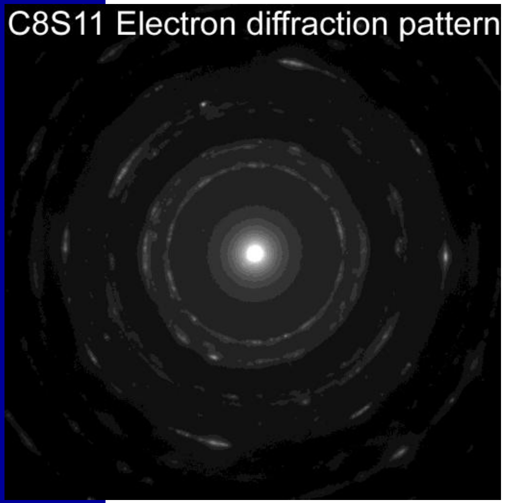
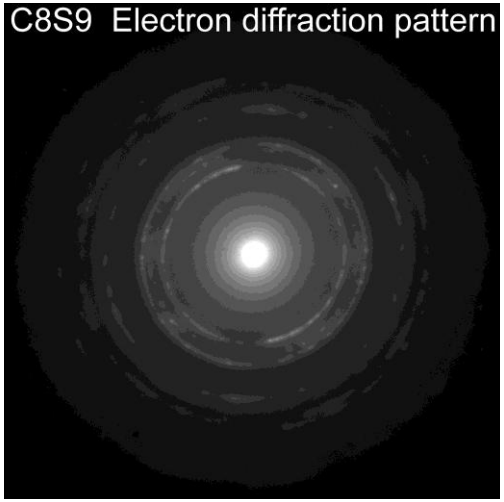
Electron diffraction Pattern

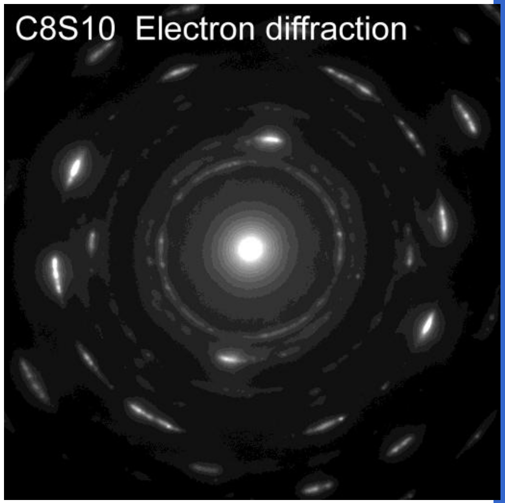
The presence of the stable TiO_2 phases (rutile and anatase) can be excluded. Samples seem to consist of the same phase. The coatings are highly crystalline. The grains are randomly orientated and small in size.



2 sccm air
metallic mode



5 sccm air
transition mode



10 sccm air
poisoned mode

Conclusions

- Plasma in a reactive atmosphere has been characterised by using a mass spectrometry.

- The detailed study of the energy distribution of O^- shows high-energy ions formed at the cathode with energies proportional to the full cathode voltage .

- Increasing the air content in the discharge resulted in an enhanced activation of the oxide species, TiO^+ and TiO_2^+, and a reduction in the atomic ion N^+, Ti^+ and especially Ti^{+2} .The ion energy is reduced significantly from the metallic to oxide mode.

- Films deposited with HIPIMS in the reactive atmosphere for metallic and oxide mode were analysed by TEM. Samples present a very dense polycrystalline and columnar structure. The grains are randomly orientated and small in size 50-100 nm. Electron diffraction pattern shows that the samples present the same phase independent of the metallic or oxide mode operation.