

# High Power Impulse magnetron sputtering of Ti/TiO<sub>2</sub> thin films: a plasma emission spectroscopy study



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#### **Abstract**

The popularity of thin film deposition in manufactured products is experiencing rapid growth while plasma-based techniques are used to create innovative products in different field of area: glass, plastic or steel industries, etc. In the framework of the Plasmagen project (2021-2023), we have performed an in-depth plasma emission spectroscopic analysis using different techniques such as DC magnetron sputtering (DCMS) or High-Power Impulse Magnetron Sputtering (HiPIMS). The HiPIMS plasma source developed during the Plasmagen project was used. As a result, we have successfully identified optimized electrical parameters for increasing the ionization degree of the plasma during the synthesis of Ti and TiO<sub>2</sub> thin films. This study relied solely on the plasma emission spectrum and the information provided by the power supply unit.

## **Experimental setup**

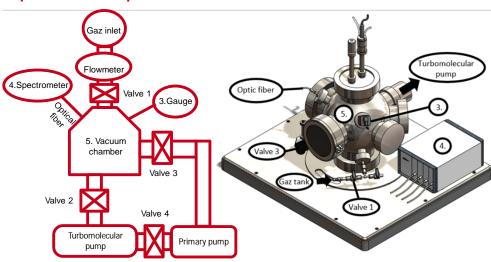


Fig. 1: Schematic view of the vacuum system

# Research Strategy

The HiPIMS power supply is firstly characterized (maximum working voltage, maximum peak current, maximum frequency, etc.)

Different parameters such as the sputter gaz composition, the cathode voltage/current and pulse length are used in various combination.

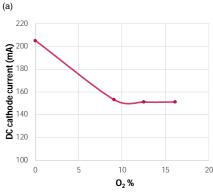
A custom Python-based data analysis software is used to analyse the spectrum data. The intensity peak of each measurement are highlighted resulting in the identification of the corresponding element.

After compiling and analyzing all available data, the experimentation setup can be concluded.

Transition from metallic to poisoned mode

## Results

Fig. 2: 3D Model of the vacuum system



The transition between metallic and oxide modes is observed in figure 3. Due to a lack of precision of the gas injection system, we conclude that the transition occurs between below 9% of  $\rm O_2$  injected in the vacuum chamber. Nonetheless, other studies have shown that the transition could happen at low percentages of oxygen [1.2]

Fig. 3: Transition between metallic and poisoned modes in DCMS at 50W

[1] D. Boivin et al., Towards control of TiO2 thickness film in R-HiPIMS process with a coupled optical and electrical monitoring of plasma, Surface & Coatings Technology, 433, 128073 (2022).

[2] C. Nouvellon et al., Deposition of titanium oxide films by reactive High Power Impulse Magnetron Sputtering (HiPIMS): Influence of the peak current value on the transition from metallic to poisoned regimes, Surface & Coatings Technology 206, 3542–3549 (2012)

### Optical emission spectrometry

An Avantes multichannel optical emission (OES) spectrometer (ULS2048CL) is used to collect various spectra from 305 nm to 900 nm as shown in figure 4. Data analysis was achieved using a custom Python script.

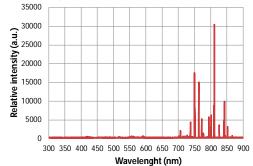


Fig. 4: Example of optical emission spectrum obtained with our Avantes spectrometer

# 1. Influence of the pulse width on the cathode peak current

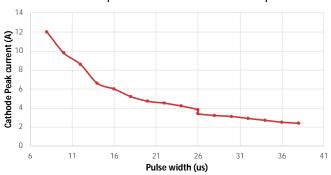


Fig. 5: Pulse width influence on the peak current

The key finding of this work is the observation in HiPIMS regime of a decrease in the current peak as the pulse width increases while keeping the average power constant. Furthermore, we consistently observe a proportionality between current and pulse length. This led us to conclude that self-sputtering is the underlying phenomenon responsible for these observations. Figure 3 visually depicts this relationship, revealing an optimal parameter range where the amount of pulverized titanium in our vacuum chamber is significantly higher. However, as the current drops too much, the ratio of pulverized titanium decreases once again.

#### 2. Influence of the cathode peak current on the Ti/Ar ratio

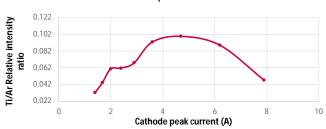


Fig. 5: Ti/Ar ratio as a function of the cathode peak current

#### **Conclusions**

- > Using our system, we identified a set of parameters that were optimal to efficiently pulverizing a titanium target.
- The HiPIMS plasma generation method is significantly more efficient than DC plasma generation.

