BY Phd Student

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Combinatorial Preparation and Characterization Methods for High Through-put Study of Advanced Functional Materials

Aim of the Research

To understand and optimize the electrochromic behavior of mixed metal oxides deposited by reactive sputtering.

Research Work

We will prepare thin films of mixed Titanium, Tin Oxide and WO_3-MoO_3 mixed layers on glass by reactive DC magnetron sputtering. The deposited $A_xB_{1-x}O_n$ type films will be Characterized by a variety of methods.

Research methods:

Preparation methods

Pulsed mode reactive DC magnetron , Biased RF sputtering systems (see Fig. 1) and Laser ablation deposition system

Characteri-zation methods

Spectroscopic Ellipsometry, Rutherford Backscattering Spectrometry, Transmission Electron Microscopy, Scanning Electron Microscopy and Atomic Force Microscopy

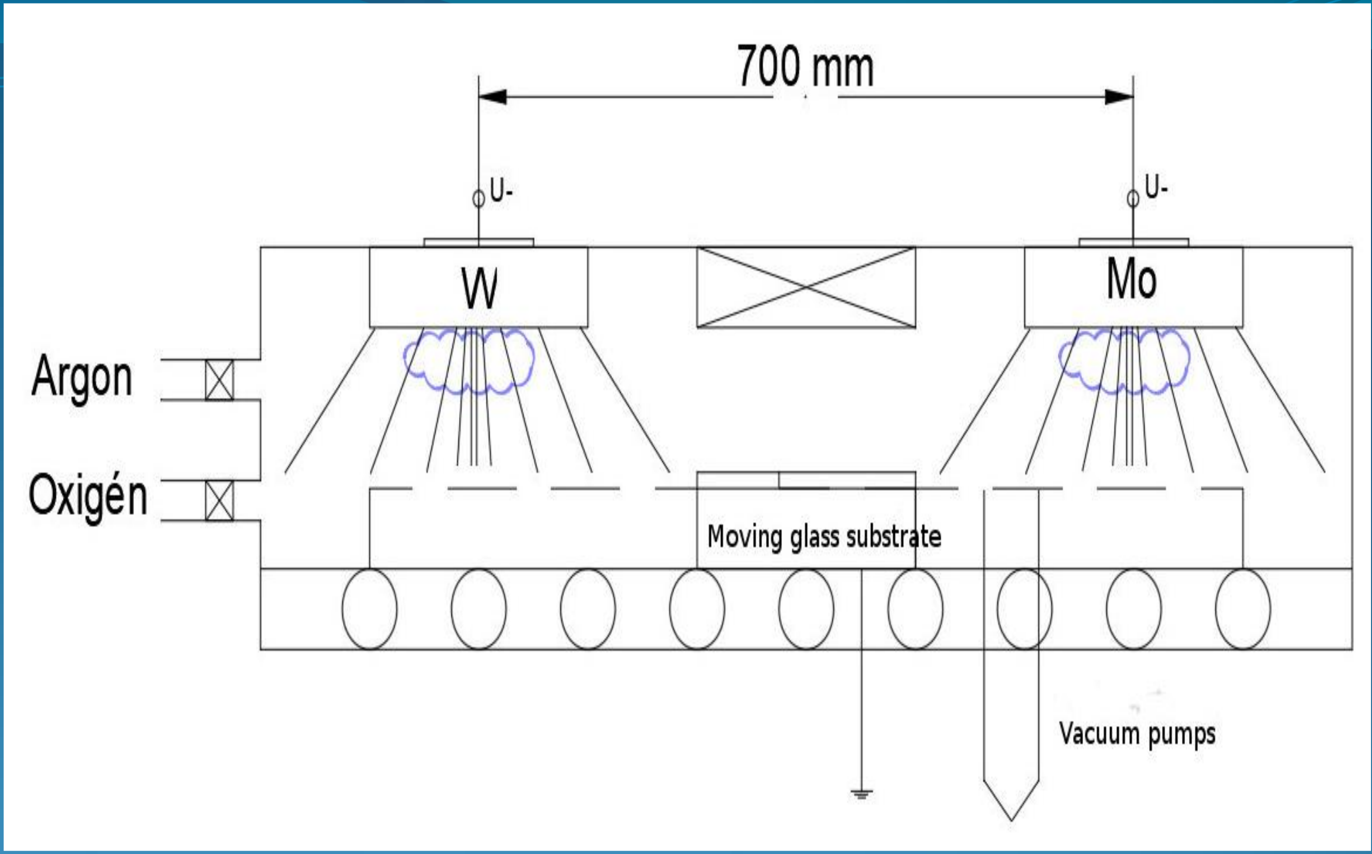
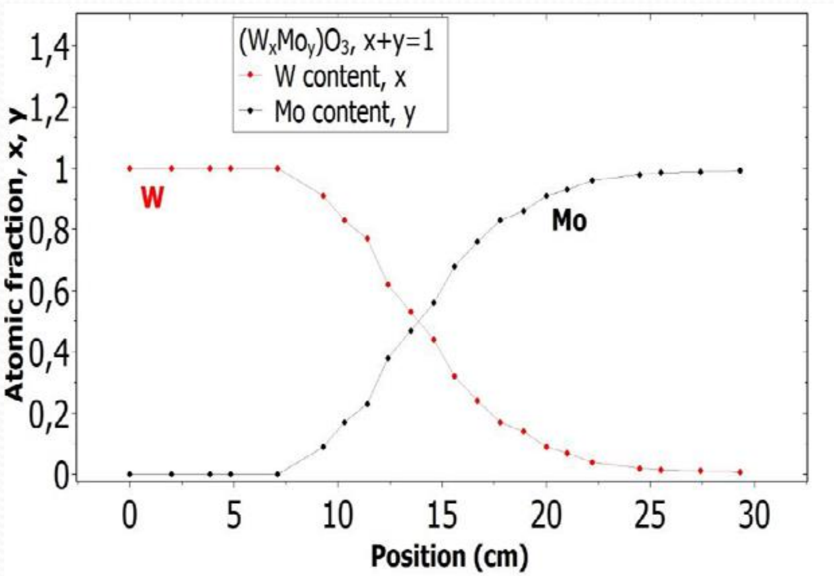


Figure (1) Schematic picture of the deposition arrangement and the photograph of 30x30 cm substrate with composition-gradient layers. Colored bands show thickness and composition gradient.

The research evaluations (The Result)

First half year we measured the WO_3/MoO_3 samples (like the one on Fig. 1) and fit the measurement by the CompleteEASE Wollam program shown in Figure (2).



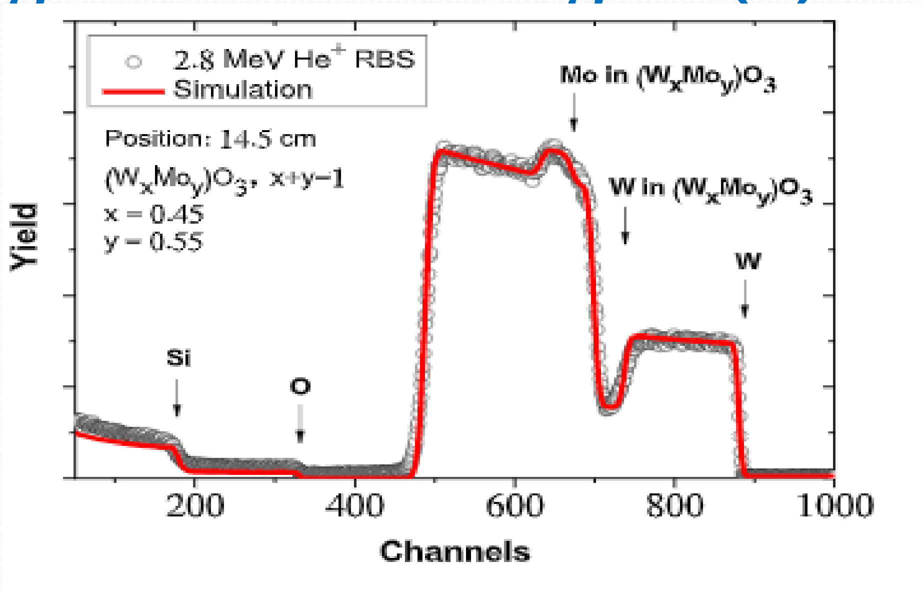


Fig. 2 a., Composition-map along a line by Rutherford Backscattering Spectrometry.

b., One Rutherford Backscattering Spectrometry example near the centre position.

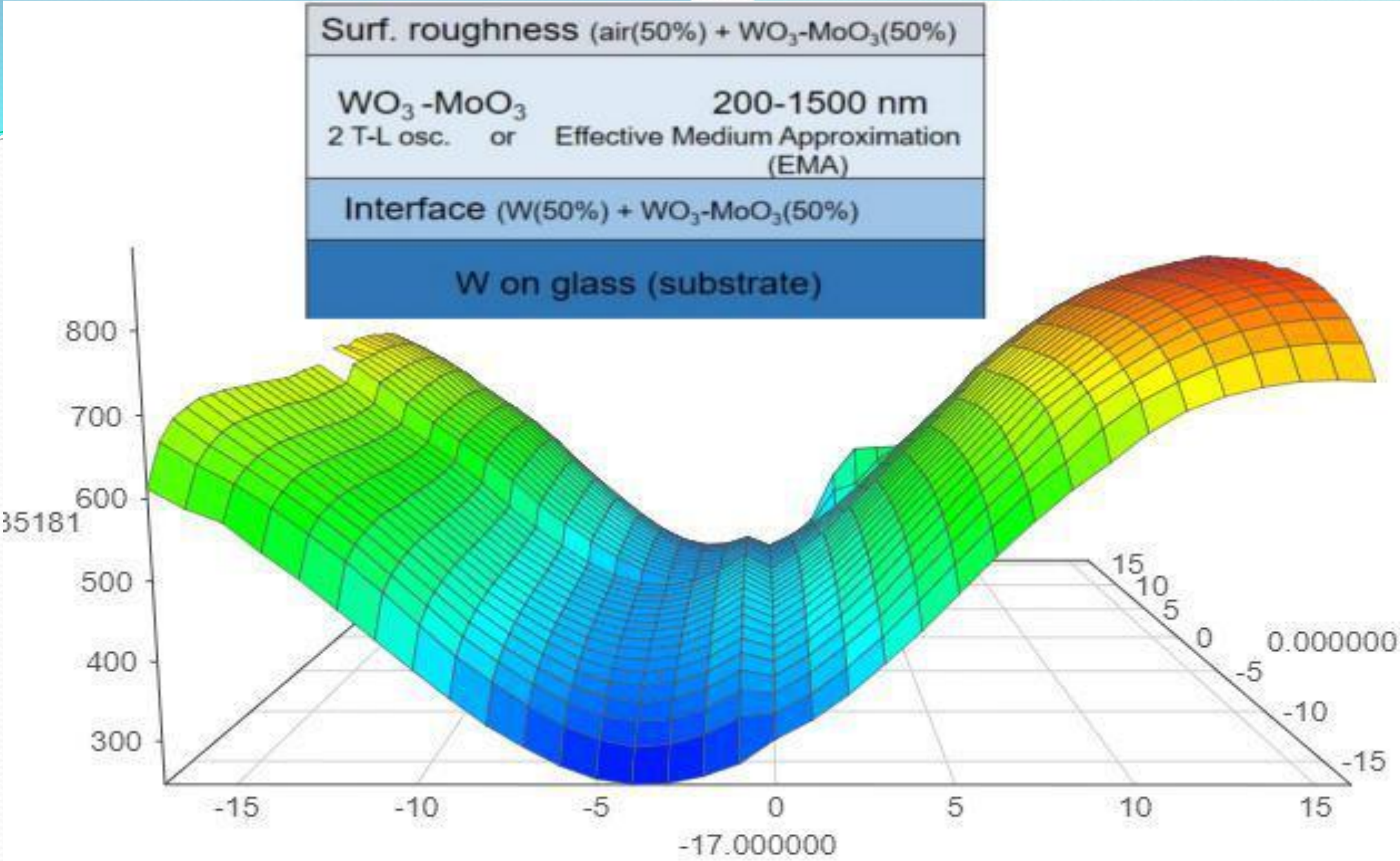


Fig. 2 c., Ellipsometric thickness mapping of the WO_3/MoO_3 combinatorial layer.

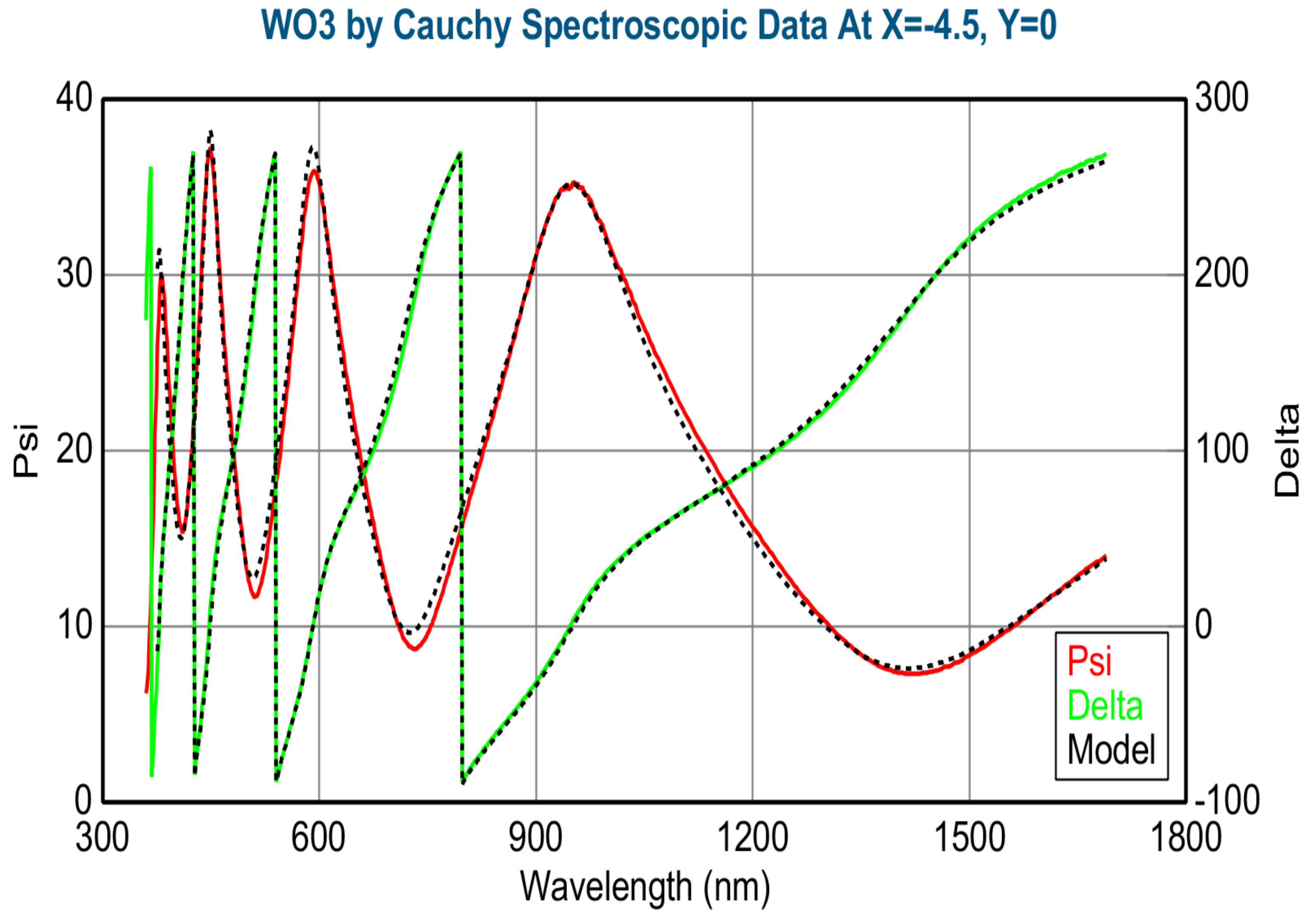


Figure (3) presented Ellipsometry mapping of the 100% WO_3 (left side of the sample) by Cauchy-Dispersion Formula Spectroscopic from (375-1700) nm.

- Layer # 2 = Cauchy Thickness # 2 = 386.61 nm (fit)

A = 2.041 (fit) B = 0.01603 (fit) C = 0.00450 (fit)

k Amplitude = 0.02065 (fit) Exponent = 3.146 (fit)

Band Edge = 400.0 nm

Layer # 1 = INTR_JAW Thickness # 1 = 1.00 nm

Substrate = SI_JAW

Angle Offset = -0.068

MSE = 25.2

A = 2.041 ± 0.002

B = 0.0160 ± 0.0007

C = 0.00450 ± 8.9924E-05

k Amplitude = 0.02065 ± 0.0006

Exponent = 3.15 ± 0.2

Total Thickness = 387.6 ± 0.5 nm.

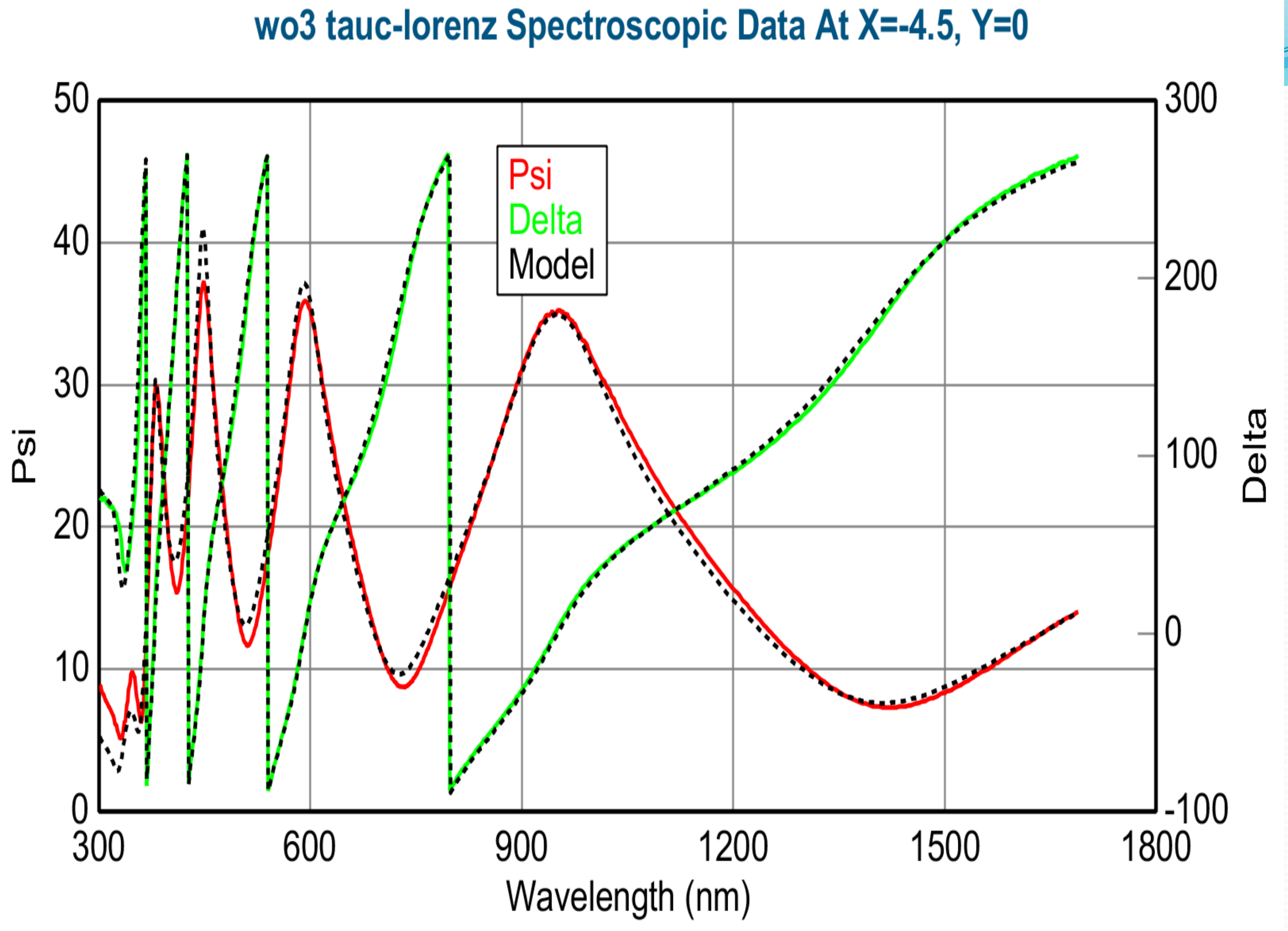


Figure (4) presented Ellipsometry mapping of the 100% WO_3 (left side of the sample) by Tauc-Lorenz Function Spectroscopic from (300-1700) nm.

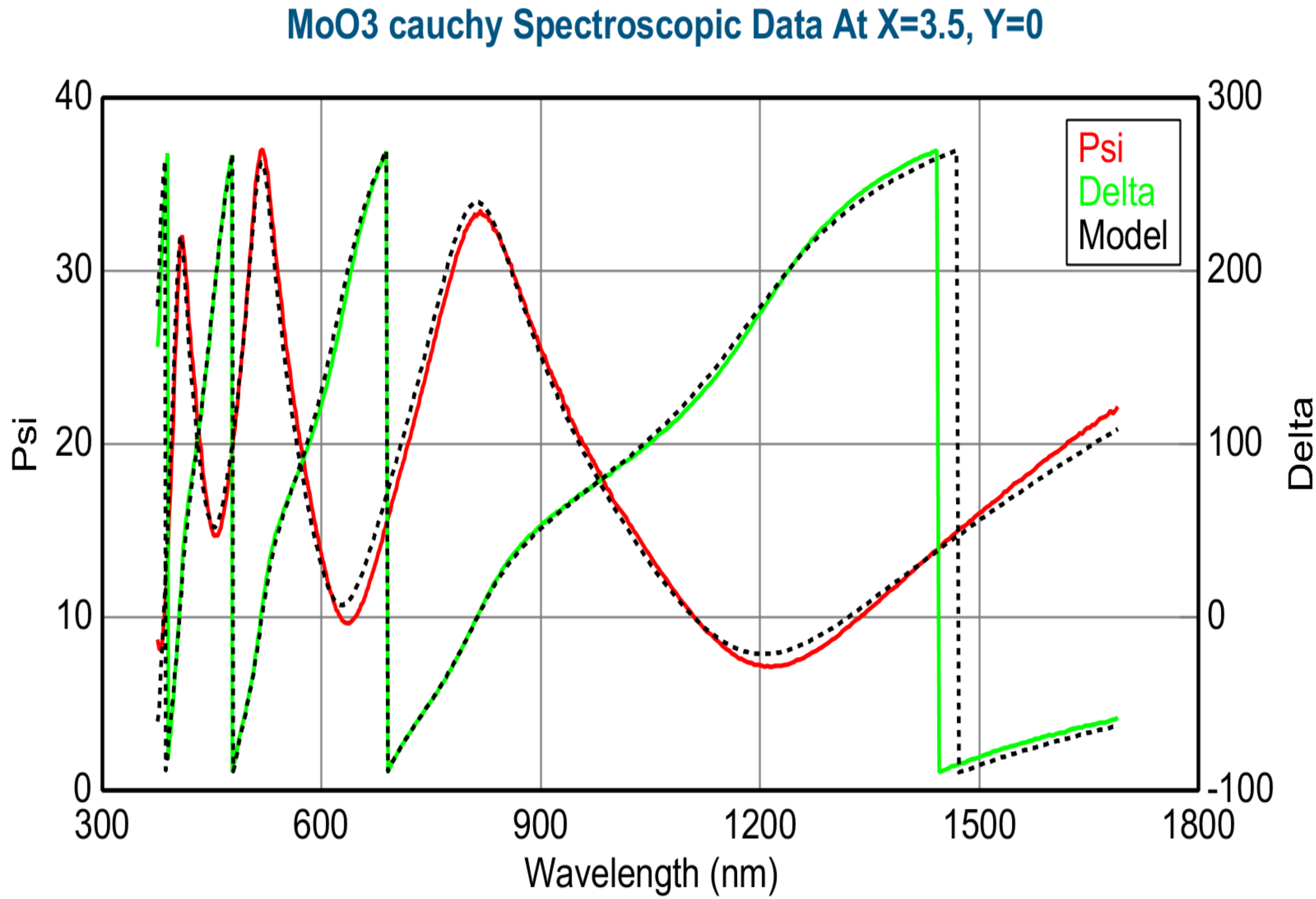


Figure (5) presented Ellipsometry mapping of the 100% MoO_3 (right side of the sample) by Cauchy-Dispersion Formula Spectroscopic from (375-1700) nm.

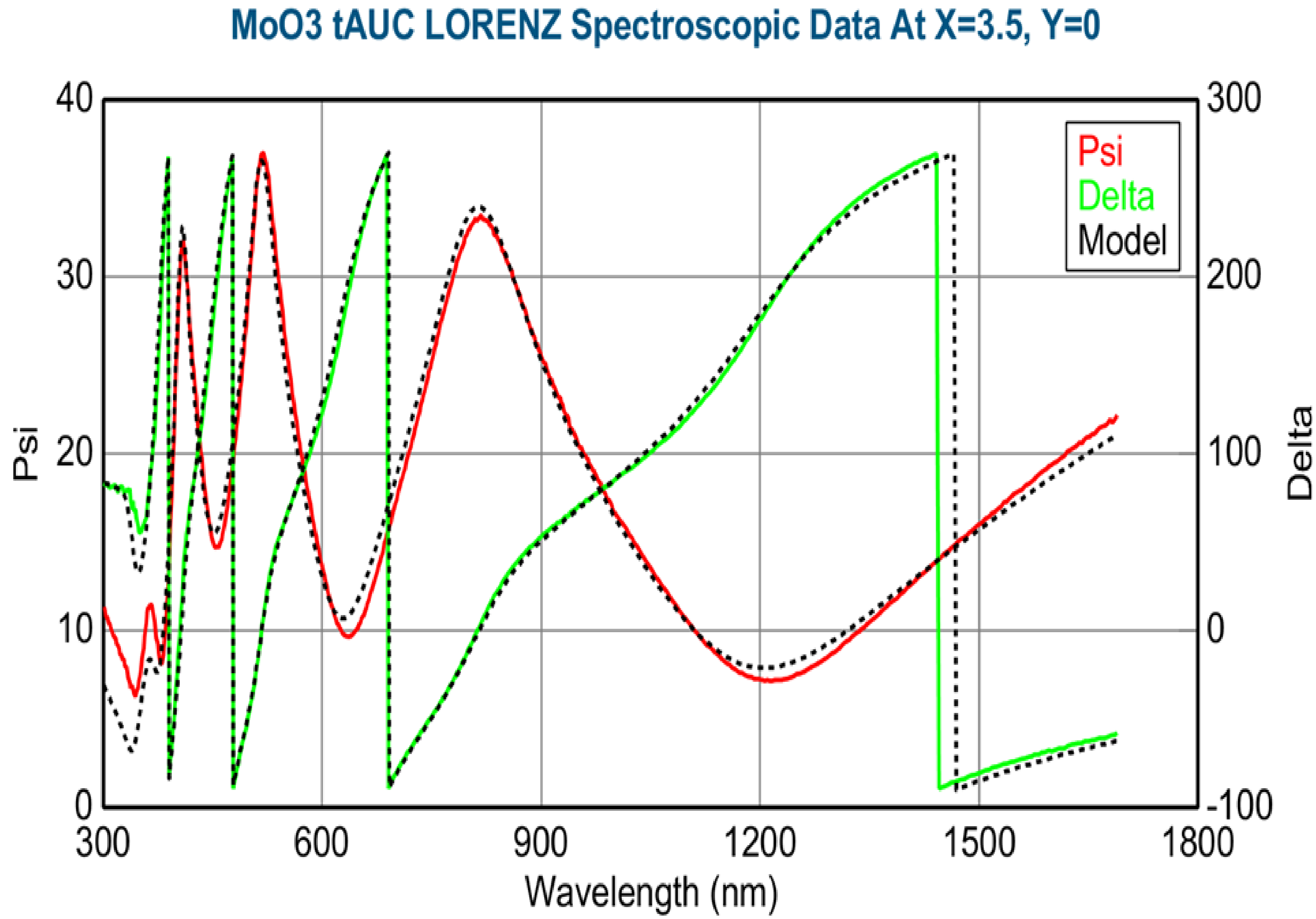


Figure (6) presented Ellipsometry mapping of the 100% MoO_3 (right side of the sample) by Tauc-Lorenz Function Spectroscopic from (300-1700) nm.

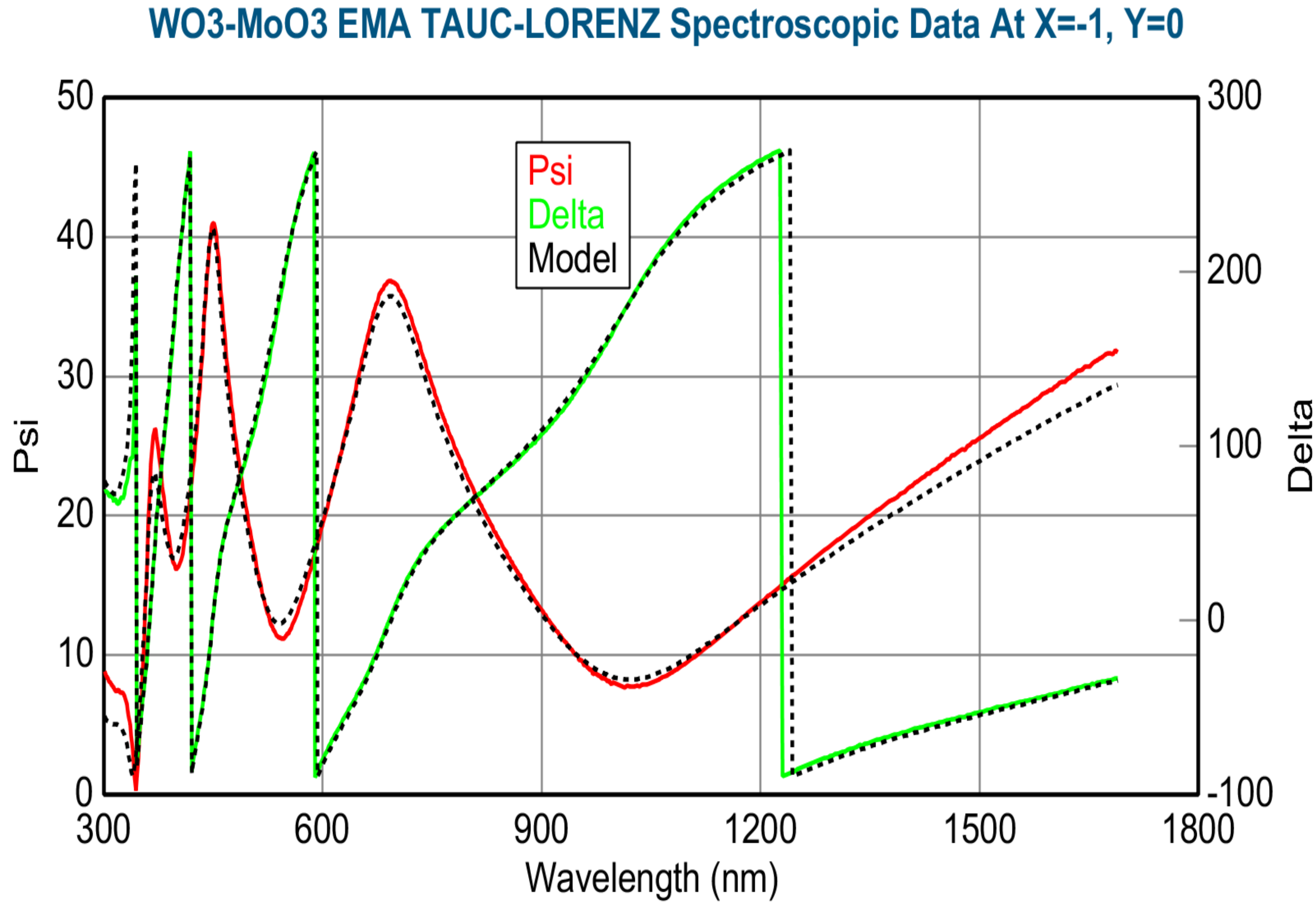


Figure (7) presented Ellipsometry mapping of the Sample: WO_3-MoO_3-on-10cm-long-siliconin by EMA Tauc-Lorenz Function Spectroscopic from (300-1700) nm.

EMA % (MoO3 %) Thickness [nm] MSE vs Position [cm]

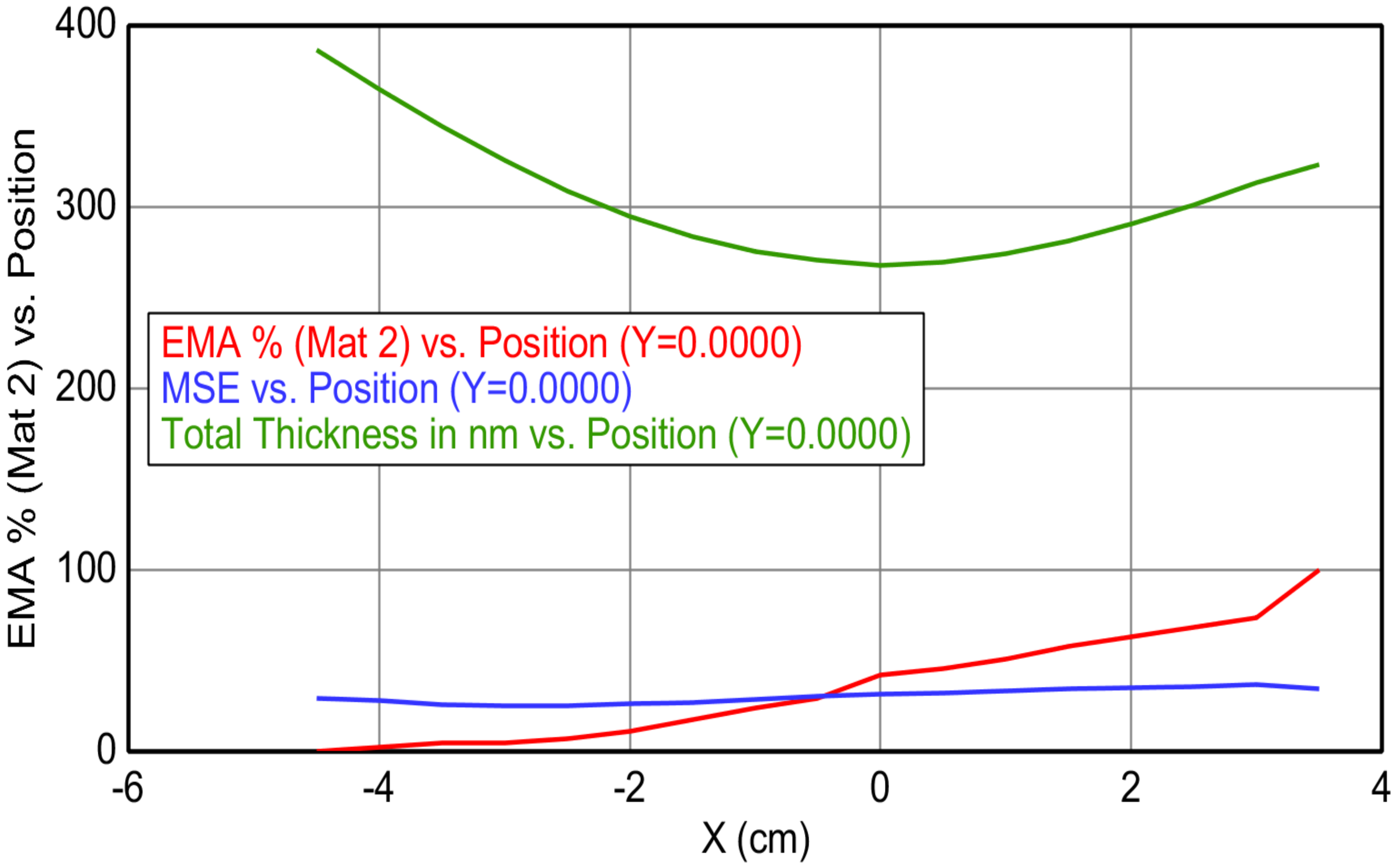


Figure (8) presented Ellipsometry mapping of the EMA% (MoO_3) Thickness (nm), MSE vs Position(cm).

Layer Commands: Add Delete Save

Include Surface Roughness = OFF

- Layer # 2 = EMA Thickness # 2 = **9906.98 nm** (fit)
  - # of Constituents = 2
  - + Material 1 = wo3 tauc-lorenz
  - + Material 2 = MoO3 TAUC LORENZ
  - EMA % (Mat 2) = **100.0** (fit)
  - depolarization = 0.333 Analysis Mode = Bruggeman
- Layer # 1 = INTR_JAW Thickness # 1 = 1.00 nm
- Substrate = SI_JAW

Angle Offset = -0.068

+ **MODEL Options**
+ **FIT Options**
+ **OTHER Options**

Configure Options

Turn Off All Fit Parameters

- MSE = 598.049
- EMA % (Mat 2) = 100.0 ± 5.40
- Total Thickness = 9907. ± 10.870 nm.

Plans for the future:

The physical, chemical and structural properties of the cutting-edge materials are strongly dependent on their composition.

The common procedure to reveal the properties of concentration dependent phases is the preparation of numerous two (or more)-component samples, one for each $C_A/C_{B=1-A}$ composition, and the investigations of the individuals.

This is a low efficiency procedure that costs enormous time of man and machine.

Contrarily, using the combinatorial material synthesis approach, materials libraries can be produced in one experiment that contain up to several hundred or thousands of samples on a single substrate.

In order to identify optimized material structures in an efficient way, adequate automated micro-spot material characterization tools have to be applied.

Planned steps forward:-

1. Electrochromic measurements on stoichiometric WO_3/MoO_3 samples (Fig. 9).

2. Further investigation of stoichiometric and sub-stoichiometric oxides for sensoric purposes (Fig. 10 and 11)).

3. Ti and Tin Oxide (Sn oxides) combinatorial deposition (Fig. 12) and their measurements.

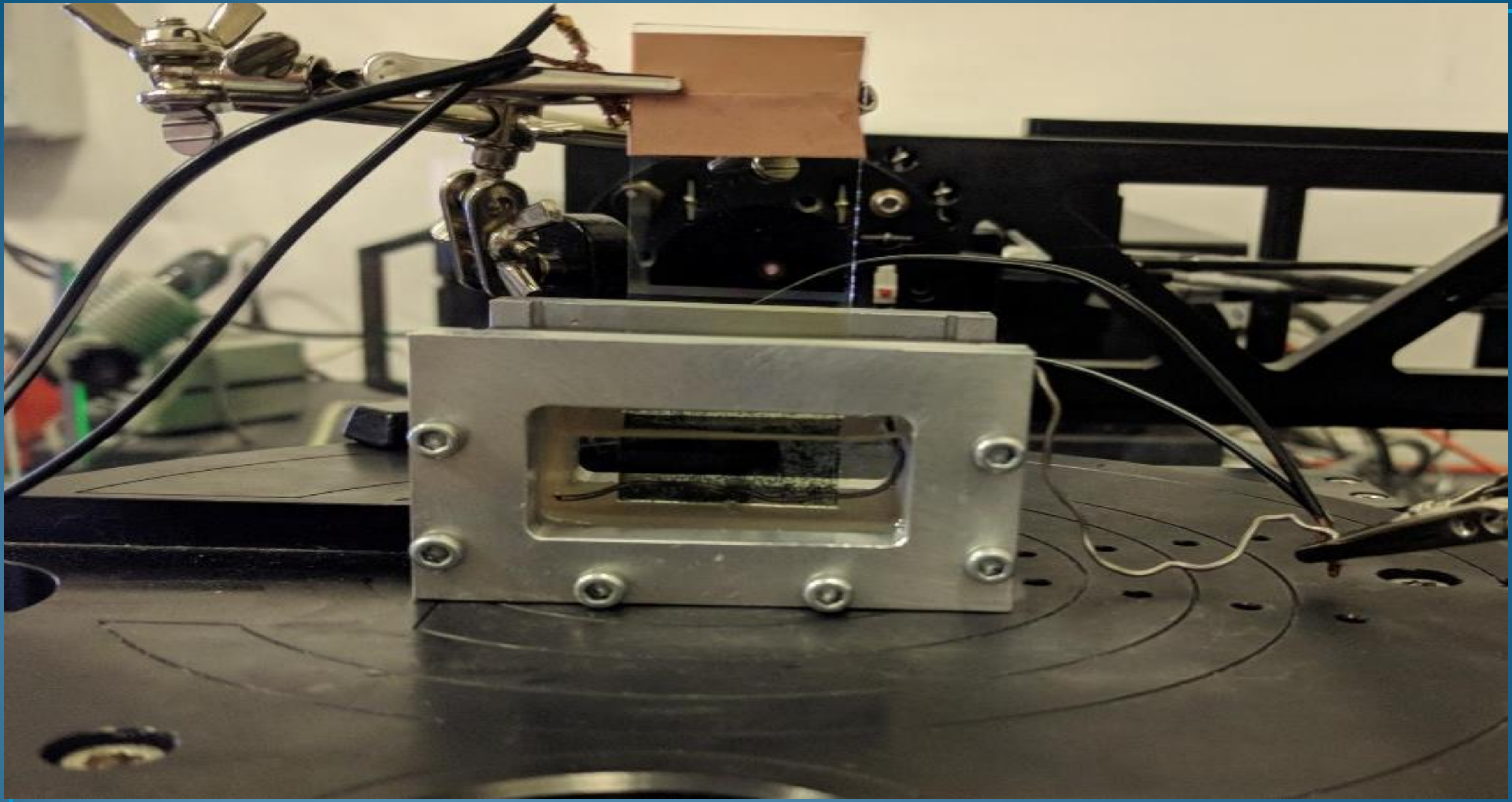
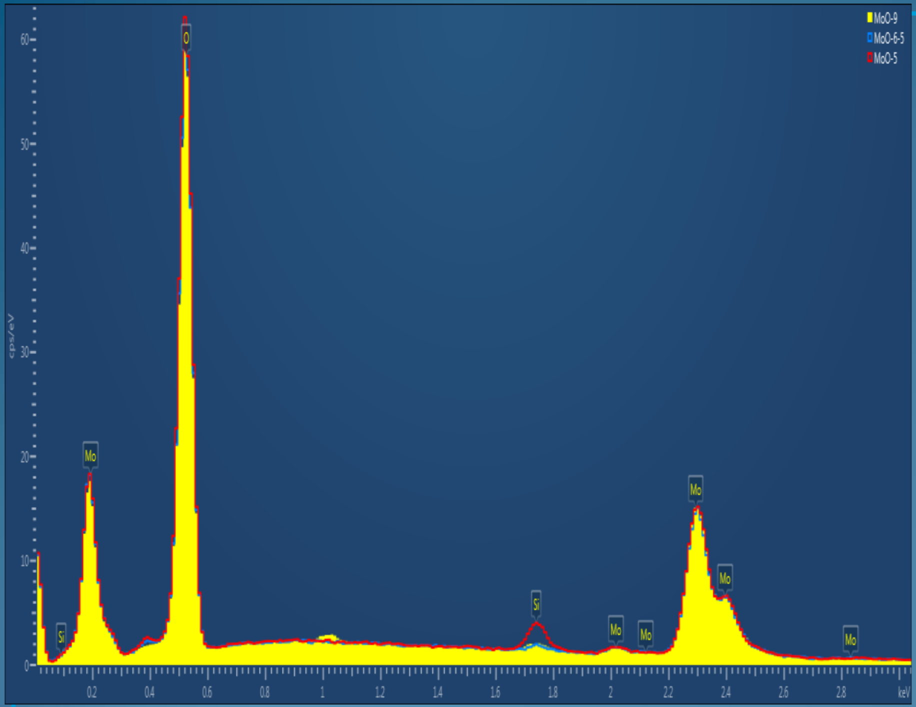


Figure (9) Liquid cell for elctrochromic measurements in transmission mode

Fig. 10 ***Photographs (from different view-angle) of WoO_3/MoO_3 (lower) or WoO_{3-x}/MoO_{3-x} (upper) combinatorial sets on heat-able sensor chips. Left hand side is W-rich, right one is Mo-rich. No. 6 (middle one) is expected to be 50-50% in both cases. The upper rows show sub oxides (semi-transparent layers, No. 2 was broken during tweezer handling) The bottom rows show oxides (transparent layers).***



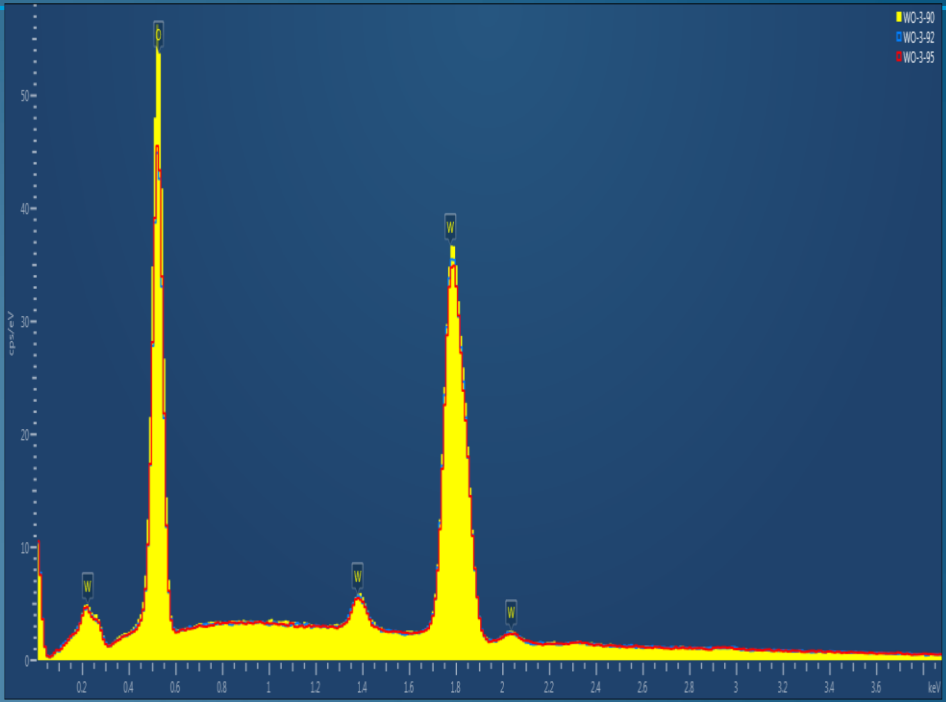


Fig. 11 EDS spectra of stoichiometric oxides and suboxides of W and Mo Semi-quantitave analysis shows 10% oxygen vacancy.

***Fig. 12** Ti and Sn oxides combinatorial deposition. Silicon probes to measure the thickness and composition map.*

Subjects fulfilled in the semester:


Thanks for your attention