Optimising the thickness determination of homopolymers and diblock copolymers using optical spectral reflectance during solvent vapour annealing

INM - RUC 2019 - Master's Thesis

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Roskilde University

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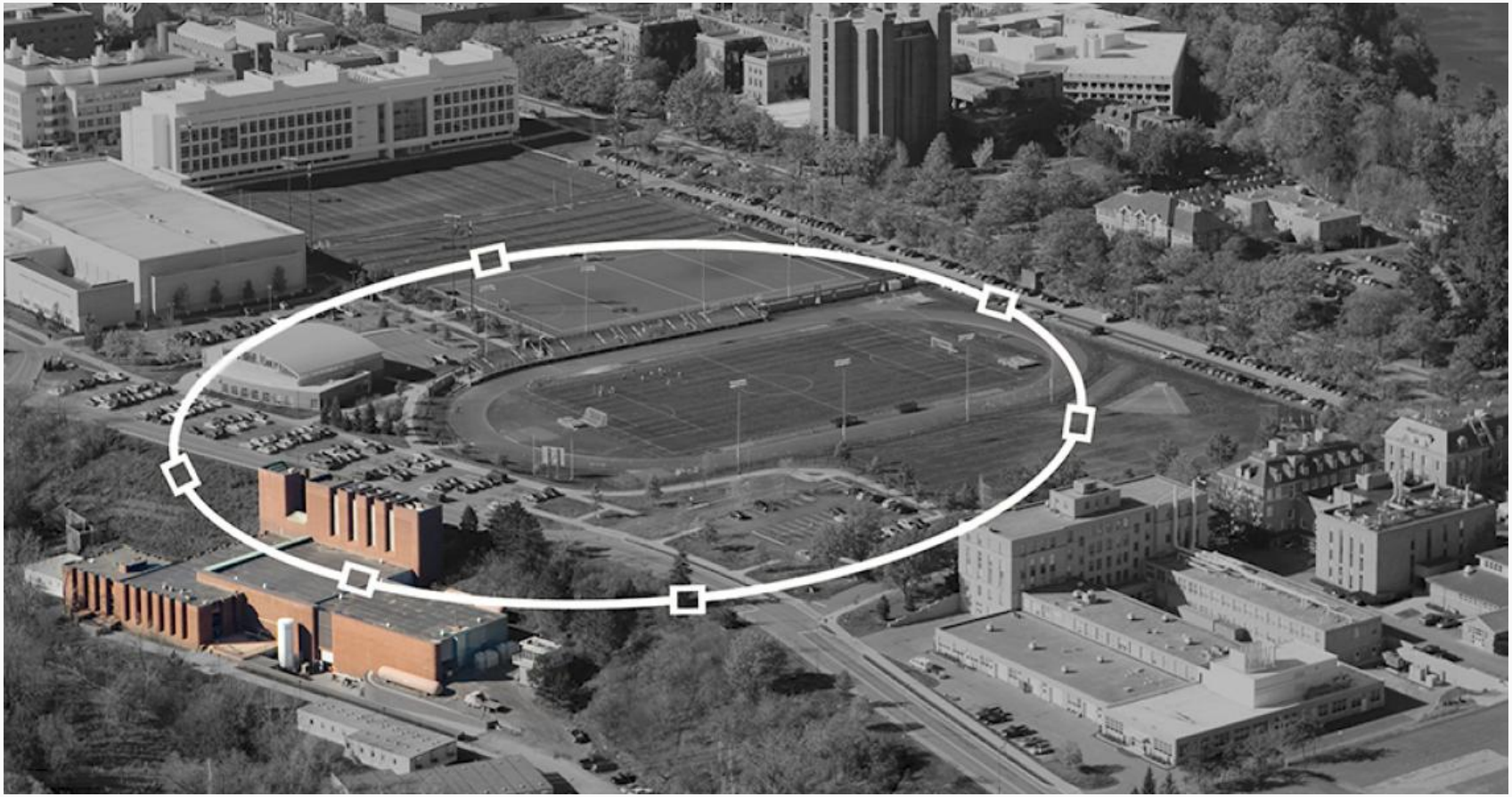
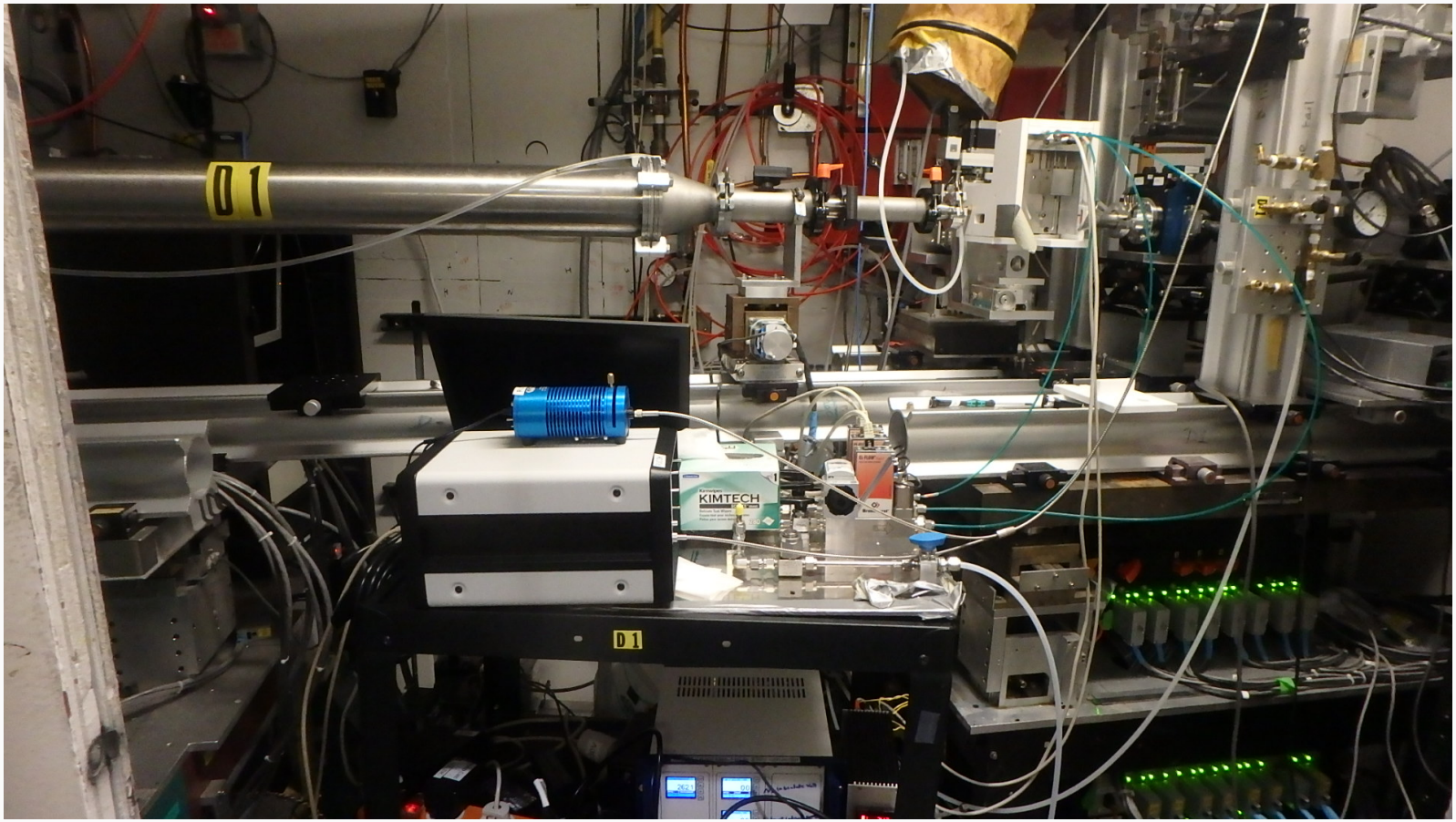
Master Thesis Background

Cornell High Energy Synchrotron Source (CHESS)

December '17 and May '18



D-Line



Problems with the reflectance measurements during swelling

Figure 1: Polystyrene - SVA run used in Master Thesis

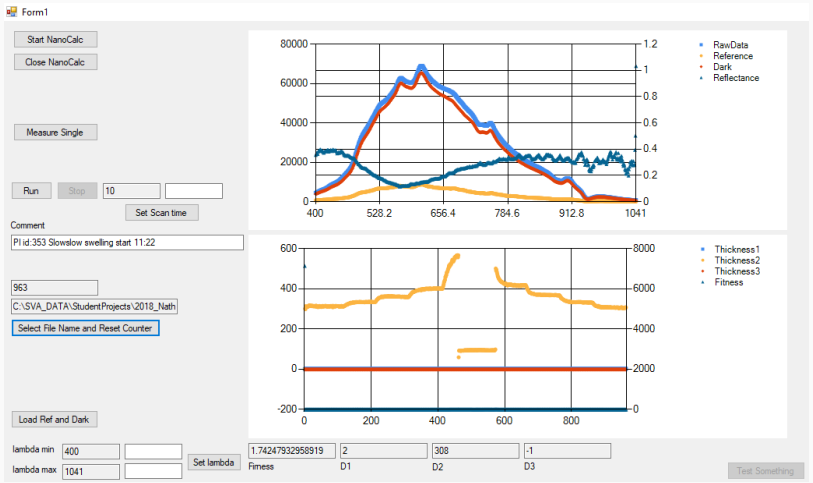


Figure 2: Polyisoprene - SVA run used in Master Thesis

Master Thesis Problem Formulation

Problem Formulation

What are the advantages and limitations when using optical spectral reflectance for determining the thickness of thin polymer films during solvent vapour annealing?

What is the optimal modelling and fitting method for the optical spectral reflectance measurements and thickness determination of the homopolymers, polystyrene and polyisoprene thin films during solvent vapour annealing?

Can the same thickness determination be used on thin films with a horizontal nano scale structure such as the diblock-copolymer Polystyrene-b-Polyisoprene?

Experimental Setup

Experimental Setup Overview

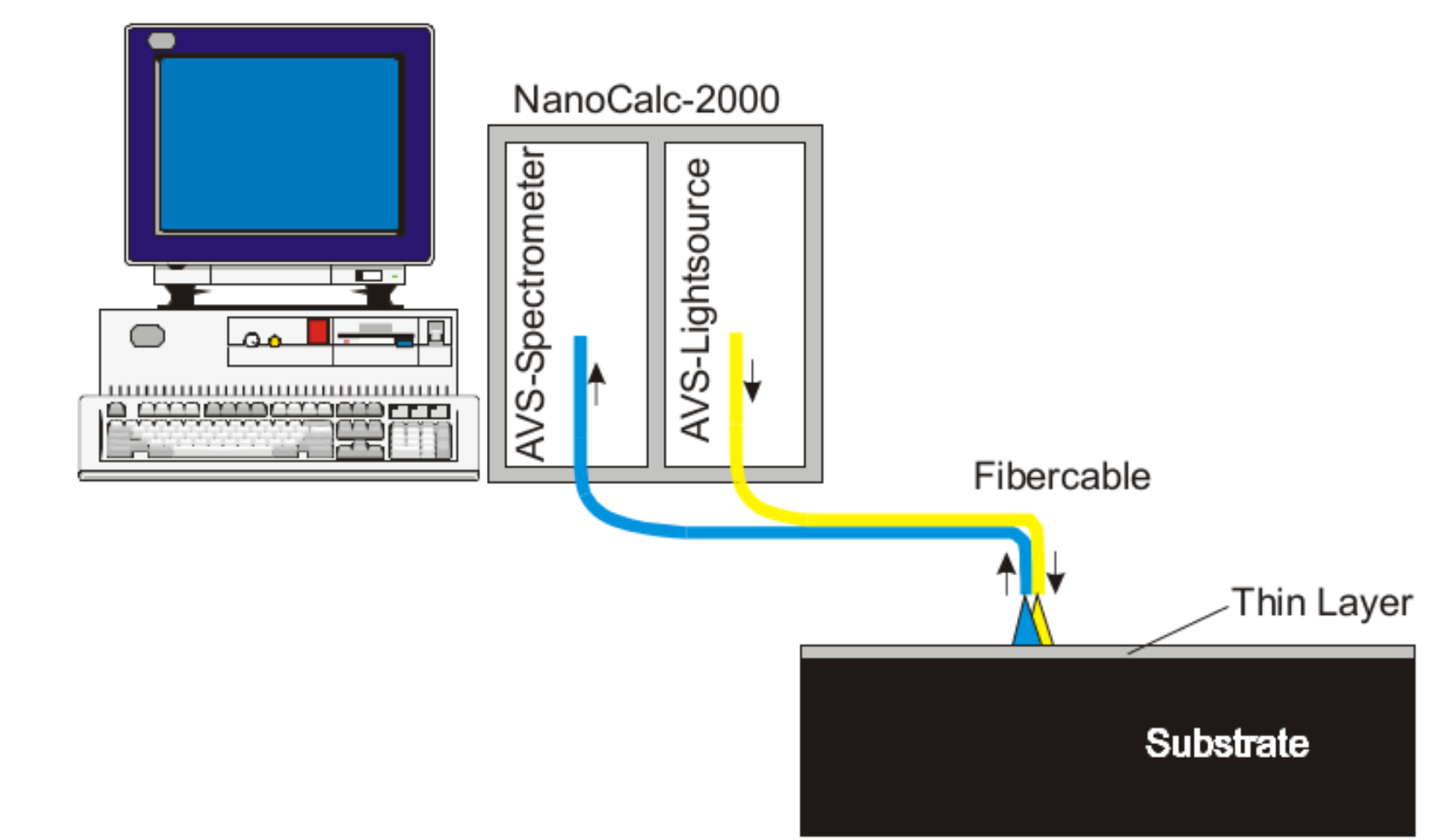
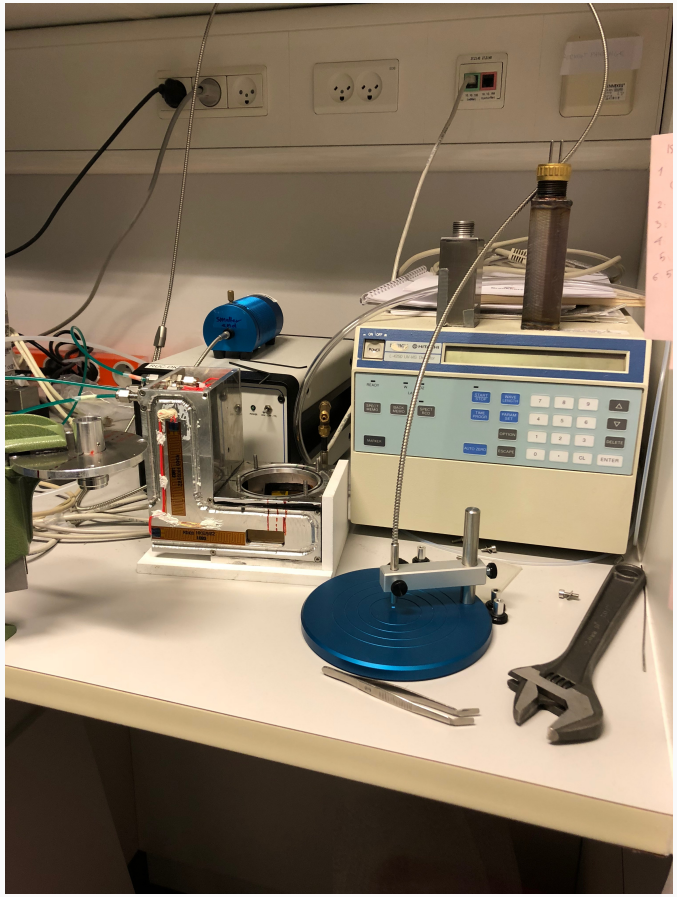
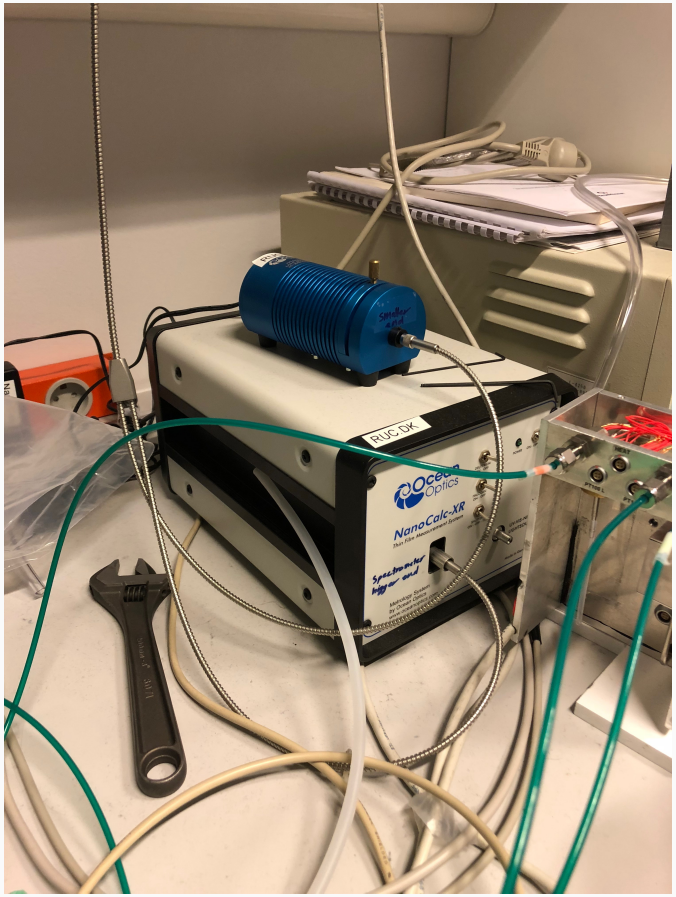


Figure 3: Taken from NanoCalc Software Manual v.4

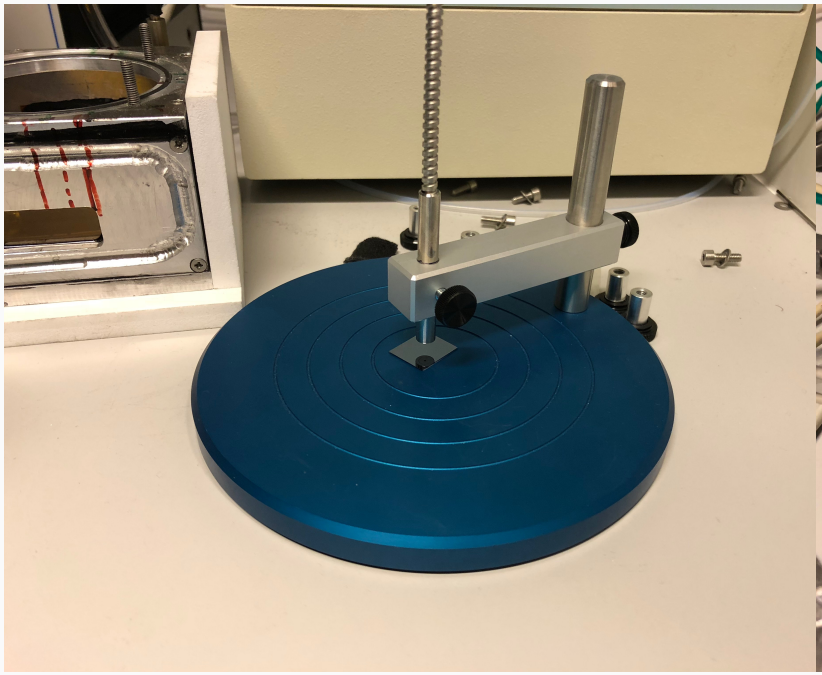
Experimental Components

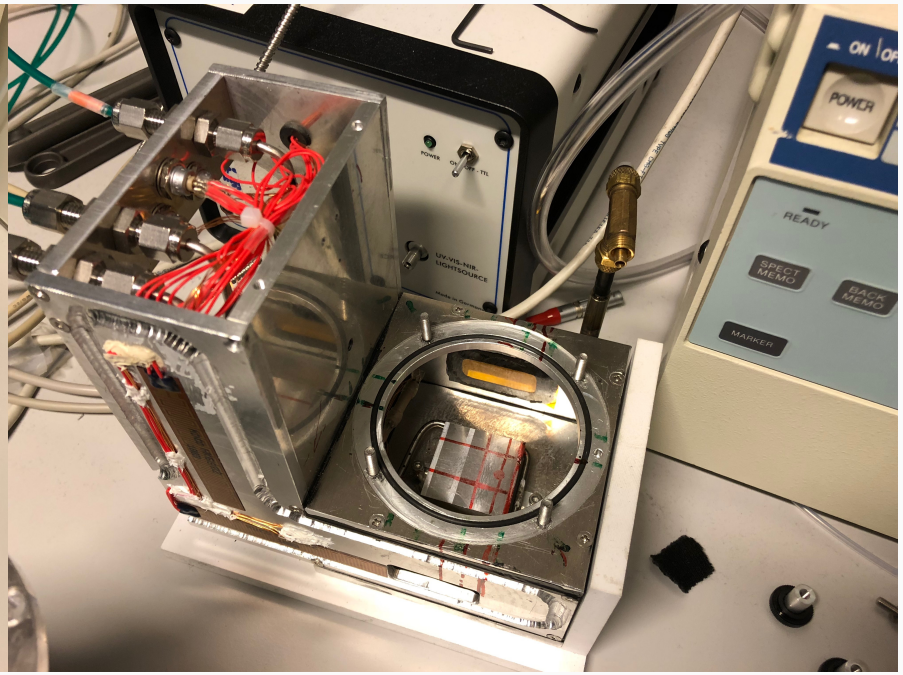
NanoCalc XR, Halogen Light Source, Test Chamber and Single Point Stage





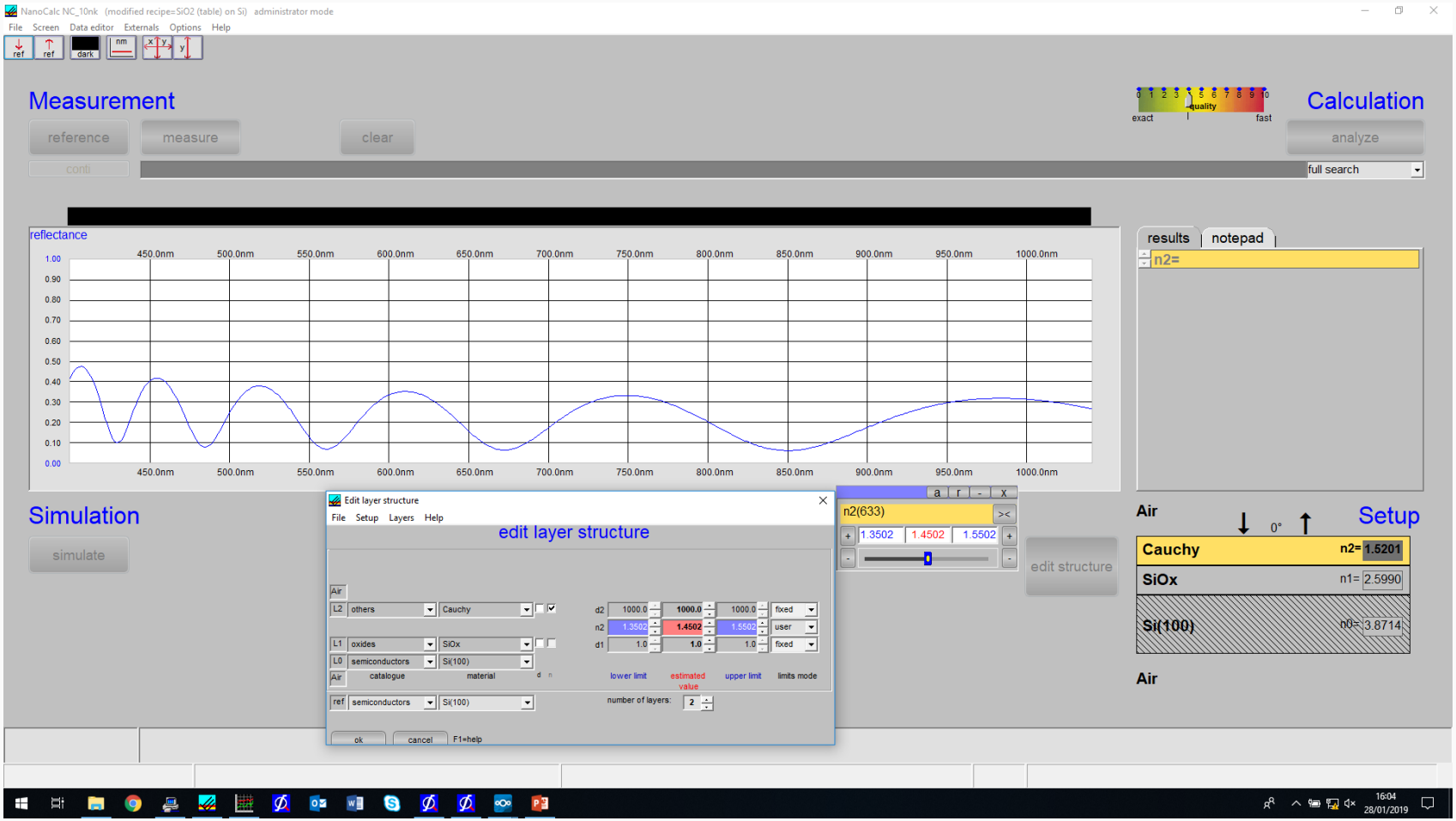
Experimental Components Cont.





Step Wafer

NanoCalc Software



Taking Measurements

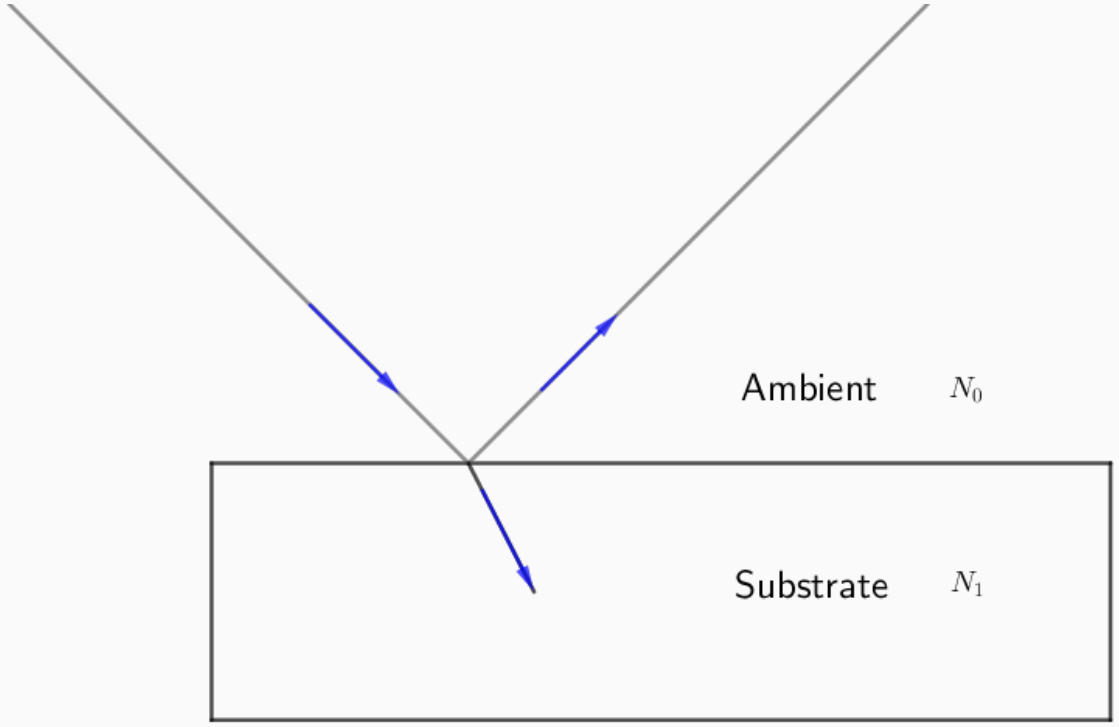
$$Reflectance = \frac{Meas - Dark}{Ref} \cdot R_{sub}$$

- Dark Measurement - *Dark*
  - Measuring stray light.
- Reference Measurement - *Ref*
  - Measuring light from blank wafer - SiOx/Si
  - Ref data - minus dark
- Wafer Measurement - *Meas*
  - Wafer with spincoated polymer
- Reflectance from ambient/substrate model - R_{sub}
  - Calculation - Fresnel model

Polymers

Fresnel Models

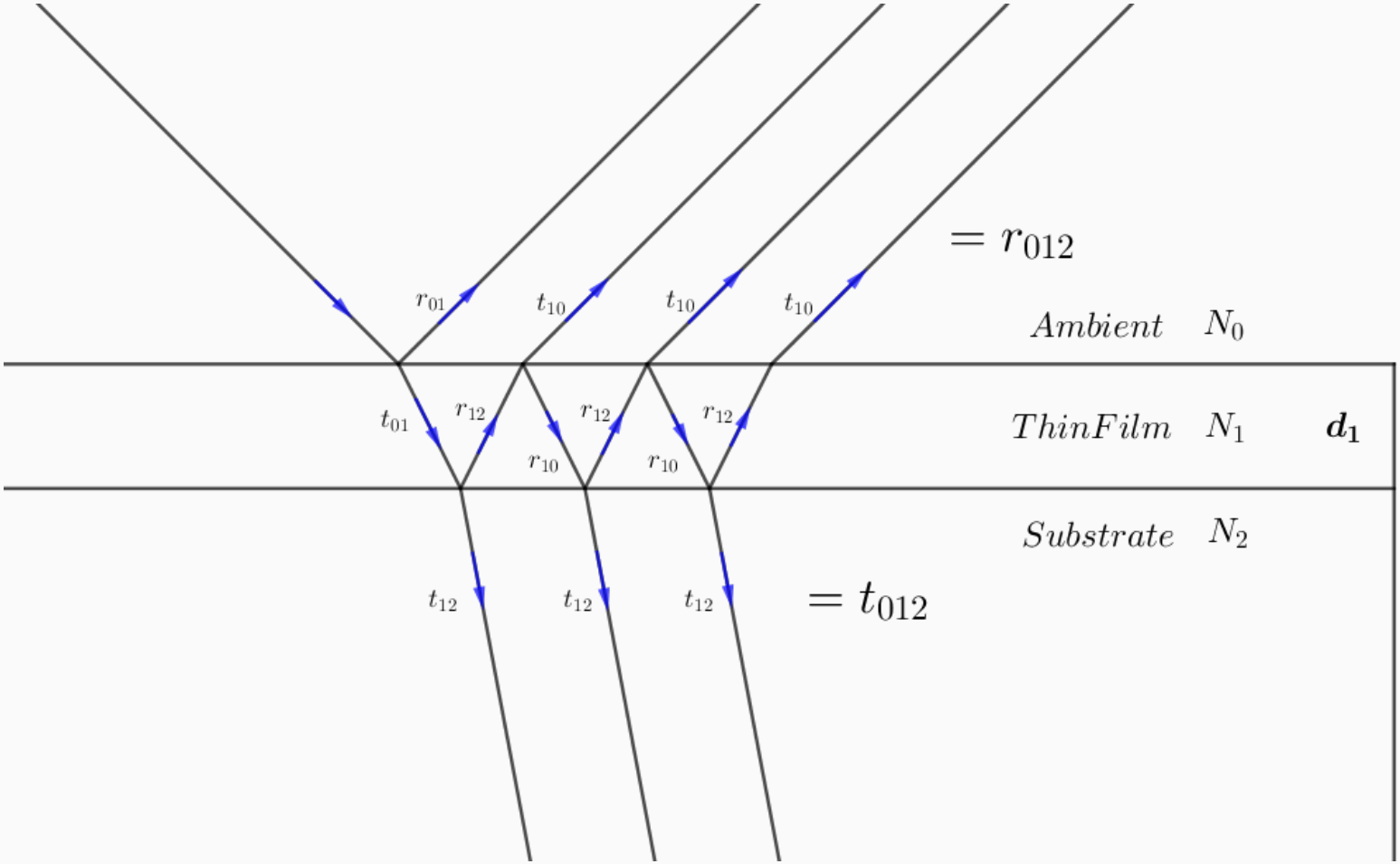
Fresnel Equations - Substrate



$$r_p = \frac{E_{r,p}}{E_{i,p}} = \frac{N_1 - N_0}{N_0 + N_1}$$

$$R_p = \mid r_p \mid^2$$

Fresnel Equations - One layer



$$r_{012} = r_{01} + t_{01}t_{10}r_{12}\exp(-i2\beta) + t_{01}t_{10}r_{10}r_{12}^2\exp(-i4\beta) + t_{01}t_{10}r_{10}^2r_{12}^3\exp(-i6\beta) + \cdots$$

Fresnel Equations - One layer

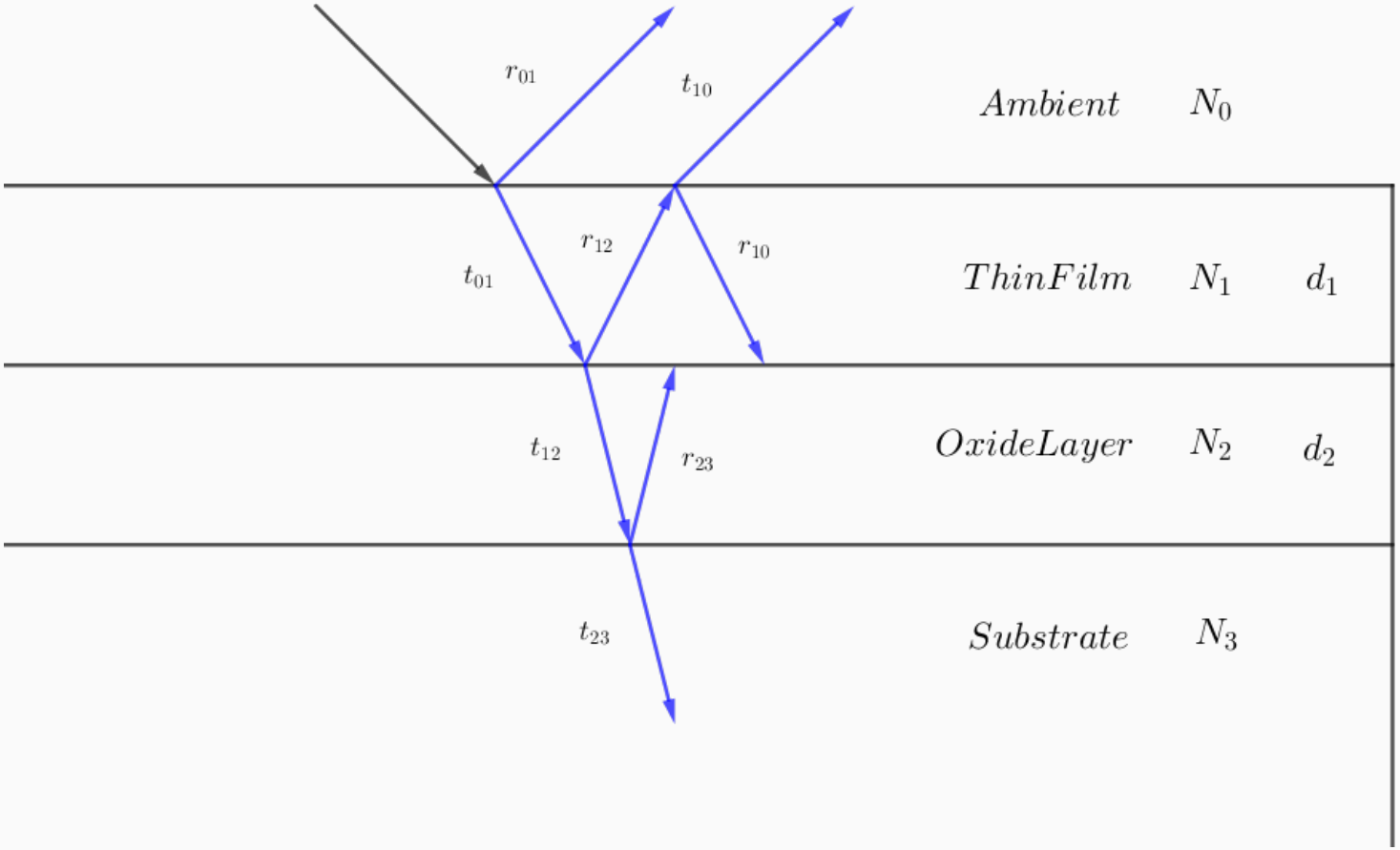
$$r_{012} = \frac{r_{01} + r_{12}\exp(-i2\beta)}{1 + r_{01}r_{12}\exp(-i2\beta)}$$

$$r_{jk} = \frac{N_k - N_j}{N_j + N_k}$$

$$\beta = \frac{2\pi d_1}{\lambda} N_1$$

$$R_{012} = \mid r_{012} \mid^2$$

Homopolymer Fresnel Model



Homopolymer Fresnel equations

$$r_{0123} = \frac{r_{01} + r_{123} \exp -i2\beta_1}{1 + r_{01} r_{123} \exp -i2\beta_1}$$

$$r_{123} = \frac{r_{12} + r_{23} \exp -i2\beta_2}{1 + r_{12} r_{23} \exp -i2\beta_2}$$

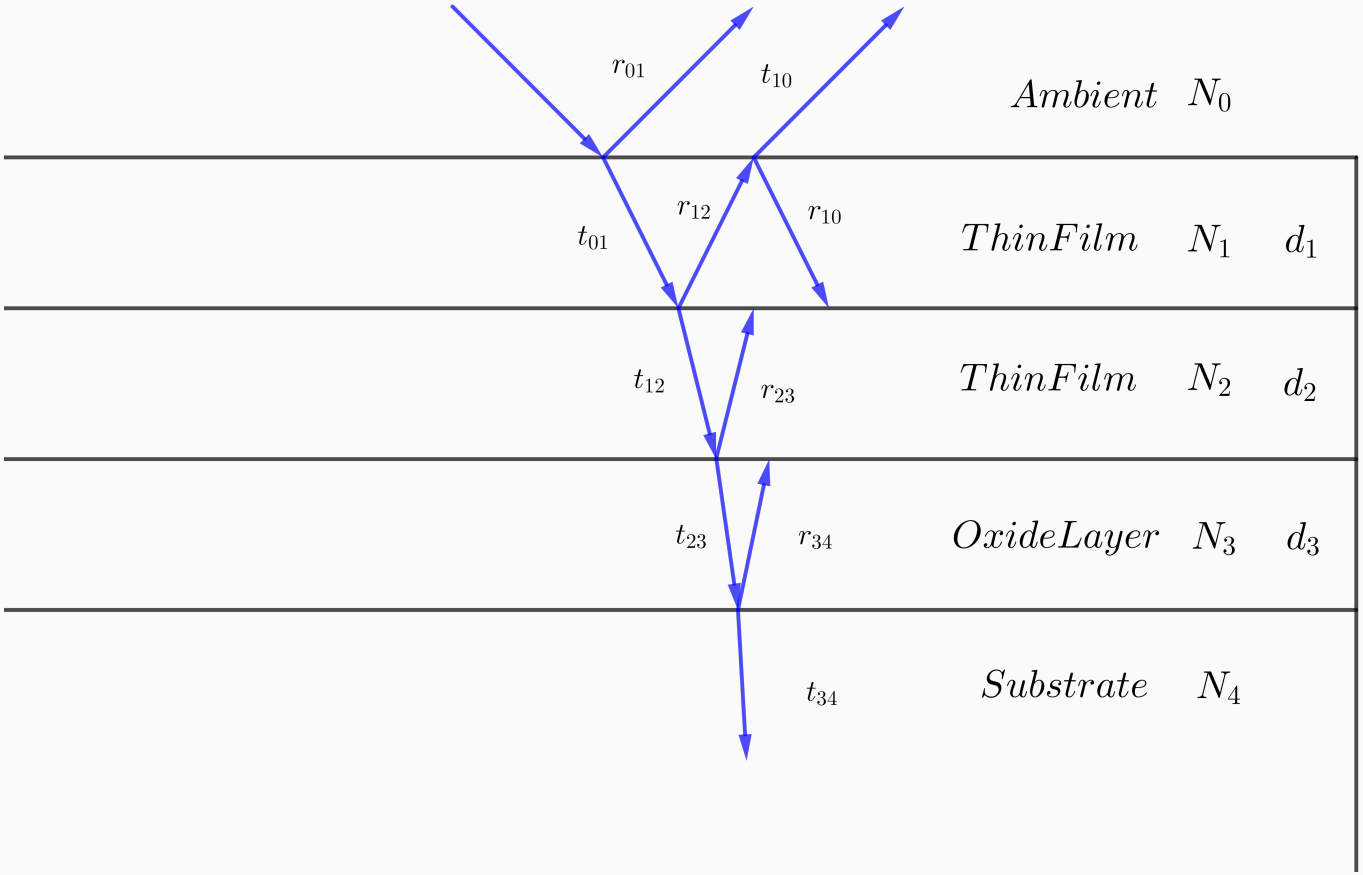
$$r_j k = \frac{N_k - N_j}{N_j + N_k}$$

$$R_{0123} = | \ r_{0123} \ |^2$$

$$\beta_i = \frac{2\pi d_i}{\lambda} N_i$$

N_0, N_1 and d_1 fitted during solvent vapour annealing.

Diblock copolymer Fresnel model



Diblock copolymer Fresnel Equations

$$r_{01234} = \frac{r_{01} + r_{1234}\exp(-i2\beta_1)}{1 + r_{01}r_{1234}\exp(-i2\beta_1)}$$

$$r_{1234} = \frac{r_{12} + r_{234}\exp(-i2\beta_2)}{1 + r_{12}r_{234}\exp(-i2\beta_2)}$$

$$r_{234} = \frac{r_{23} + r_{34}\exp(-i2\beta_3)}{1 + r_{23}r_{34}\exp(-i2\beta_3)}$$

$$r_{jk} = \frac{N_k - N_j}{N_k + N_j}$$

$$\beta_i = \frac{2\pi d_i}{\lambda} N_i$$

N_0, N_1, N_2, d_1 and d_2 fitted during solvent vapour annealing.

Plotted Fresnel models

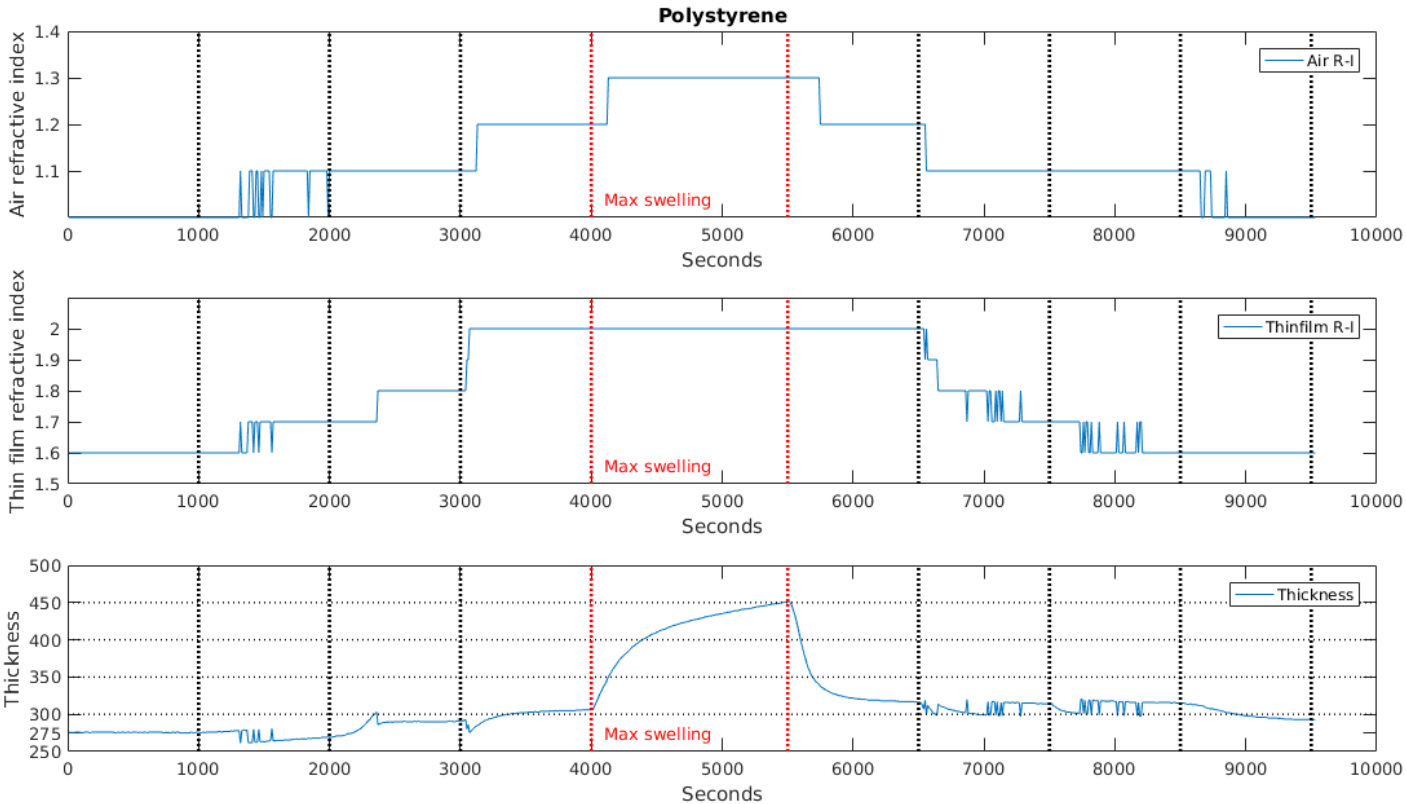
Solvent vapour Annealling

SVA protocol

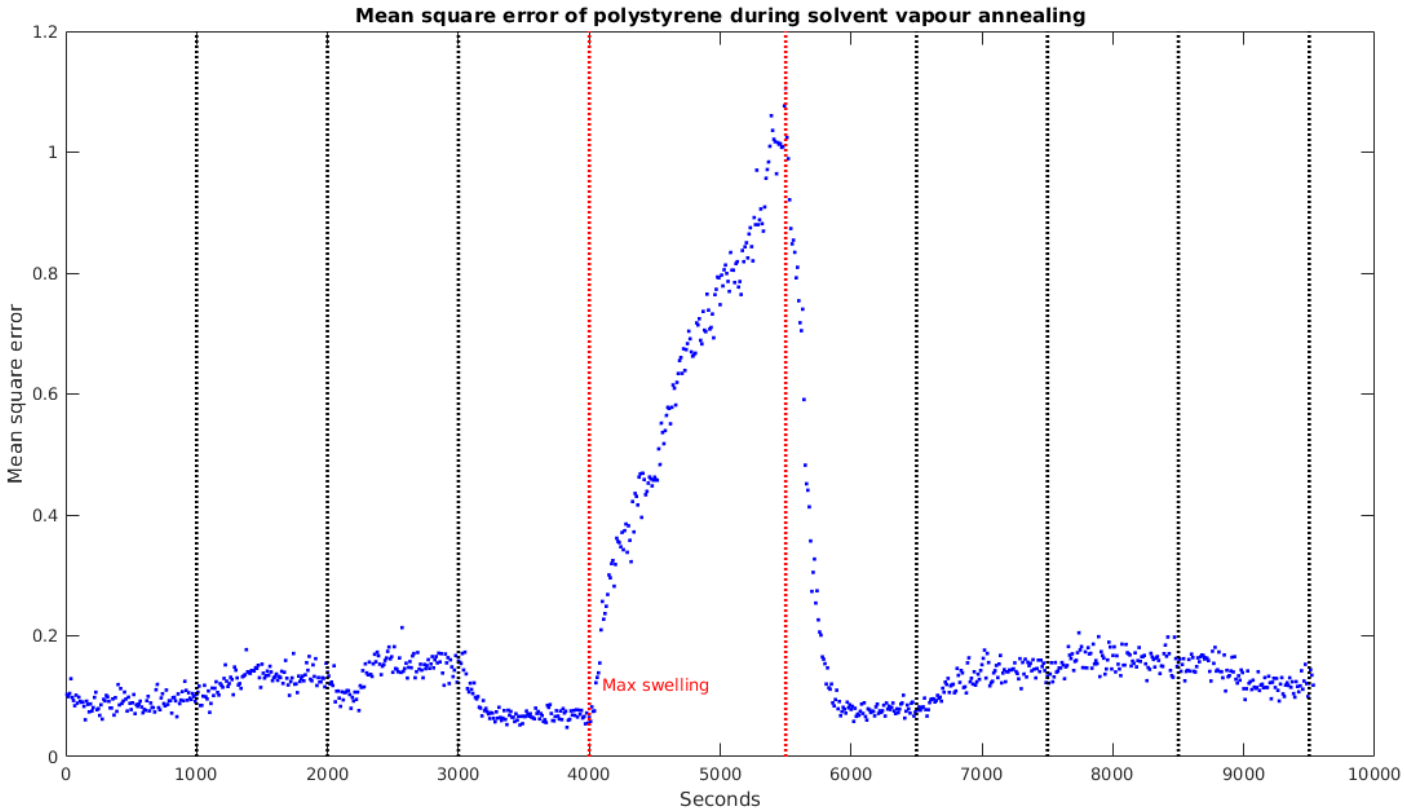
Results - 1 polymer layer

Movie: PSsva.avi

Polystyrene Results



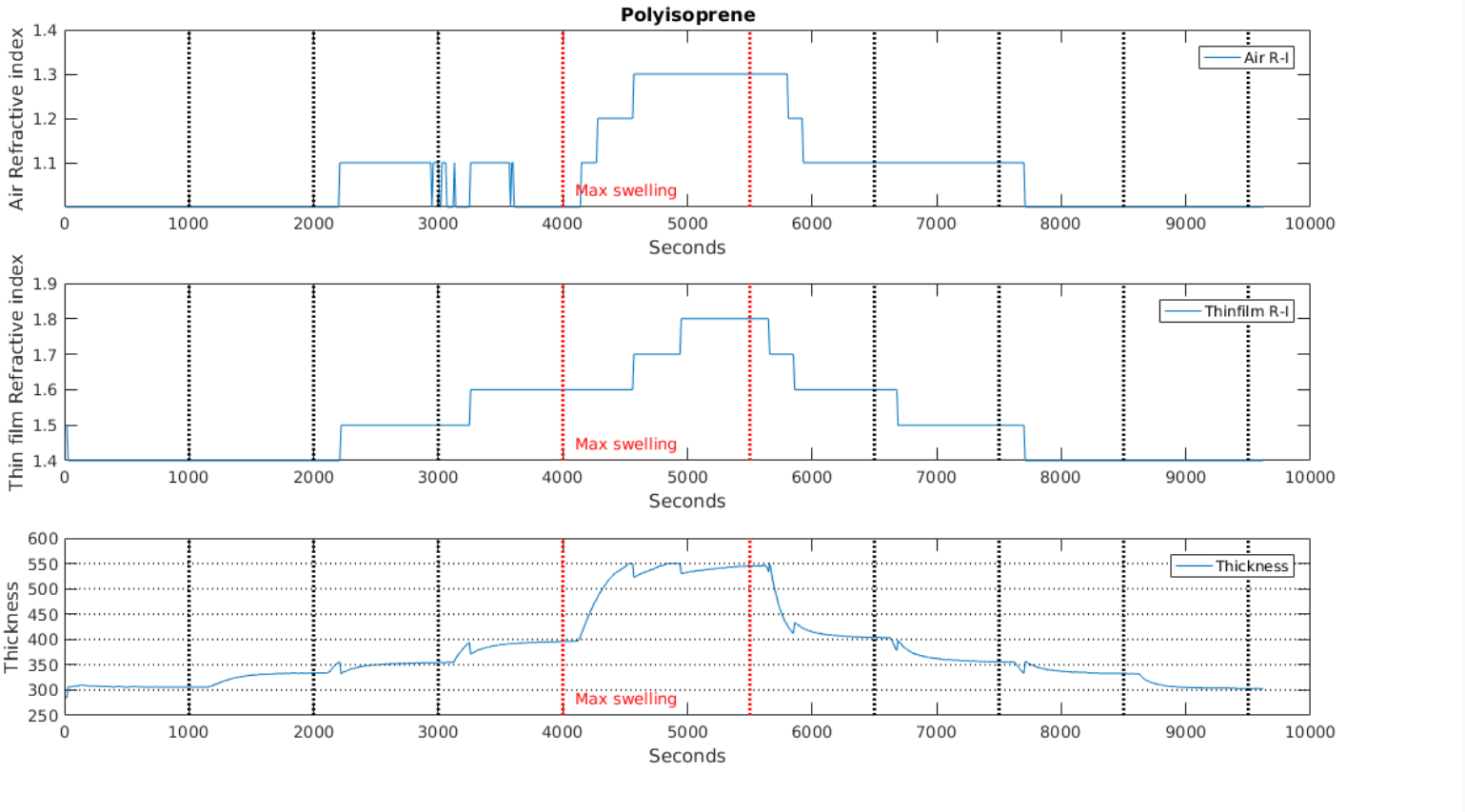
Polystyrene MSE



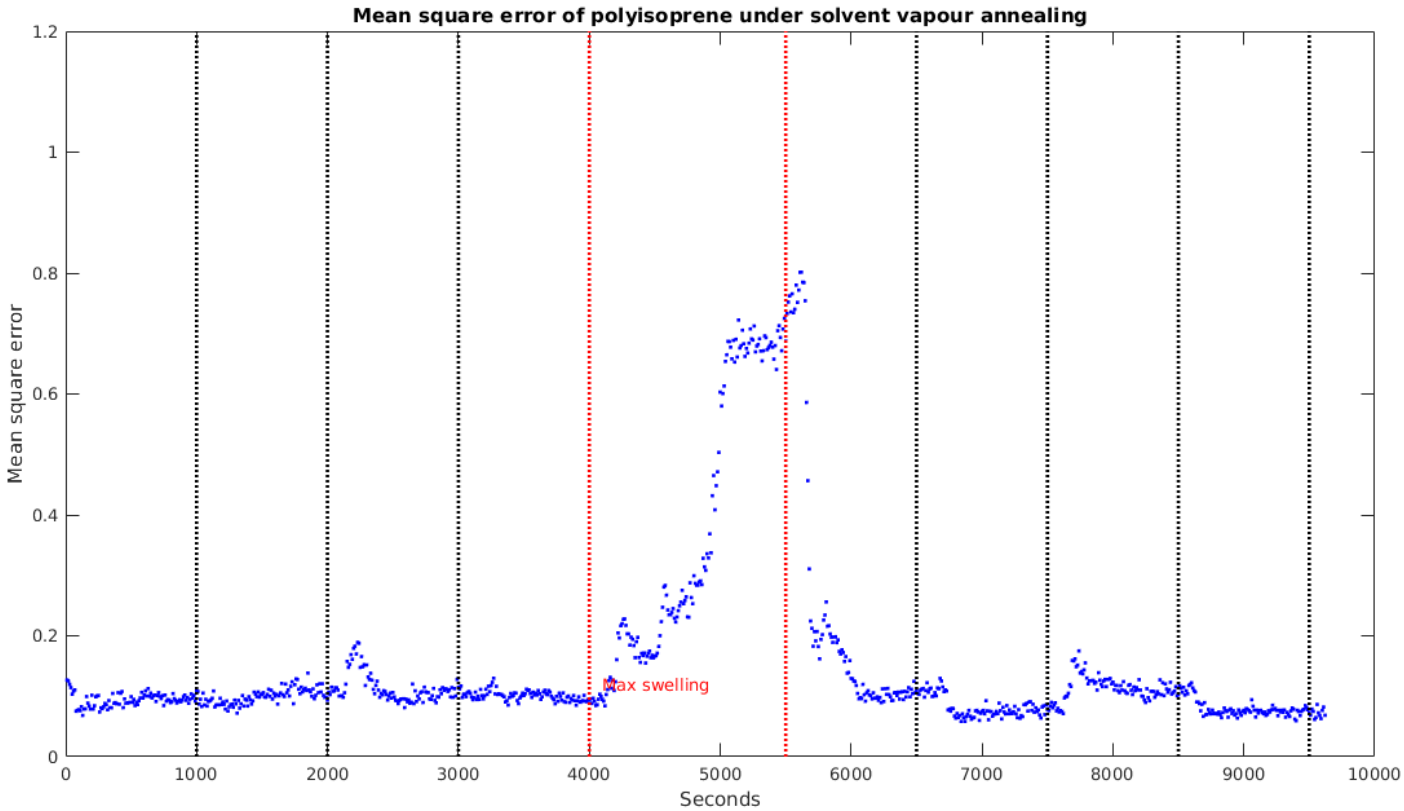
Polyisoprene

Movie:PIsvaframevalue2.avi

Polyisoprene Results



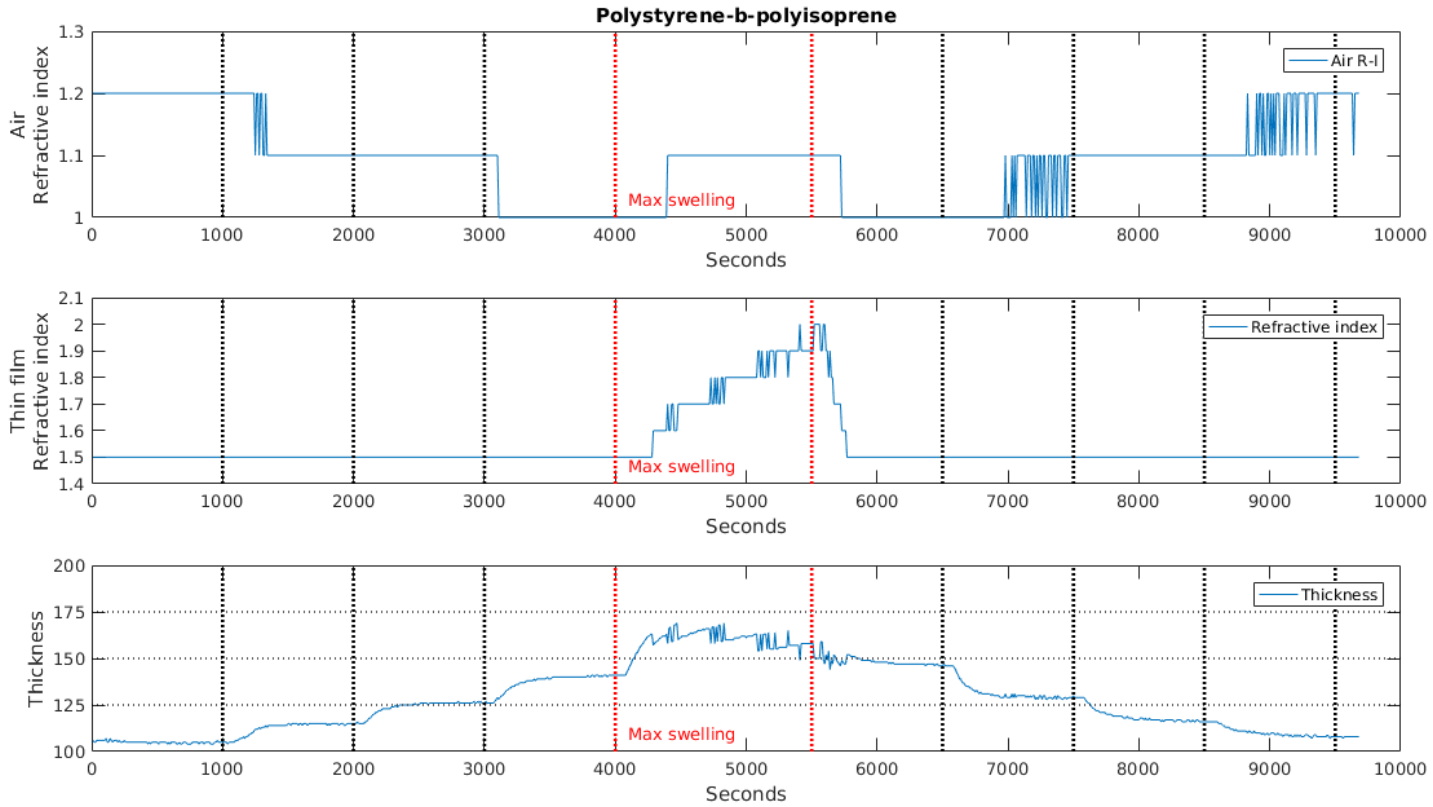
Polyisoprene MSE



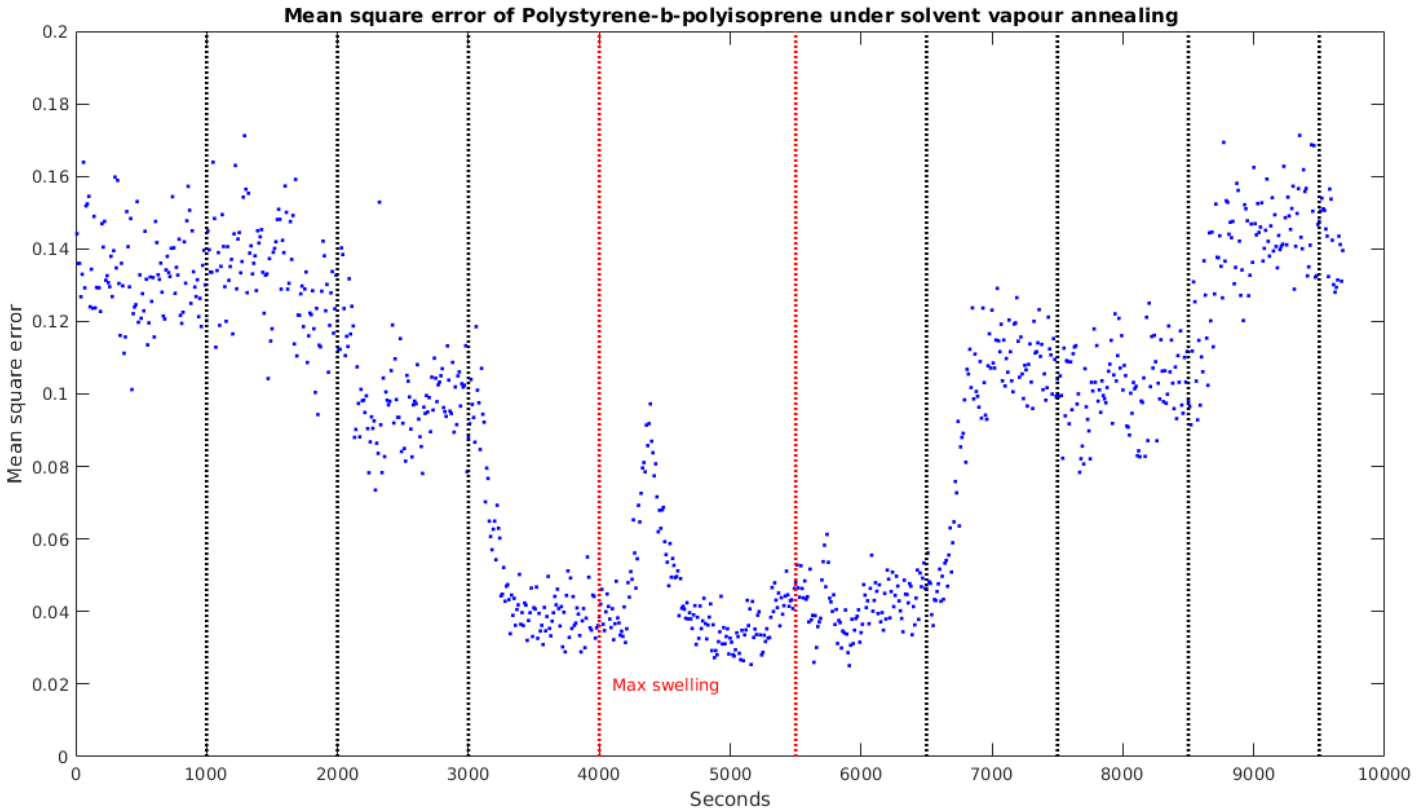
Polystyrene-b-polyisoprene

Movie: PSbPIsinglemodel.avi

Polystyrene-b-polyisoprene Results



Polystyrene-b-polyisoprene MSE



Results - 2 polymer layers

Using a 3rd party apparatus and software

content...

Missing