1. Real-Time Micro-Biocontamination Monitor for Ultrapure Water Systems

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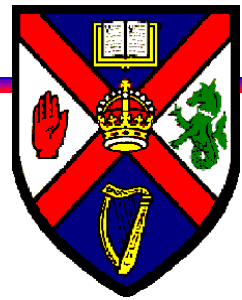
2. International TIE Project:

Detection and Control of Microbiocontamination in Ultrapure Water Processes



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3. Bacterial contamination concerns all high-purity water users.

- Pharmaceutical companies
- Power-generation facilities
- Microelectronics manufacturers
- Others (medical, bio-product, food, etc.)

4. Contamination by surface-attached (sessile) bacteria is significant.

- The advantage of surface attachment, in the harsh ultrapure water (UPW) environment, causes sessile bacteria to far outnumber the planktonic bacteria found suspended in the water.

- Implications of sessile populations:
  1) sloughing events cause product contamination
  2) sessile bacteria that survive biocide application seed microbial repopulation of water system

5. Current methods of detecting biocontamination are inadequate.

- Usually, only planktonic bacteria are sampled.
- Contamination is underestimated, and efficacy of system sterilization is overestimated.
- No real-time instruments are available for industrial detection of sessile bacteria.

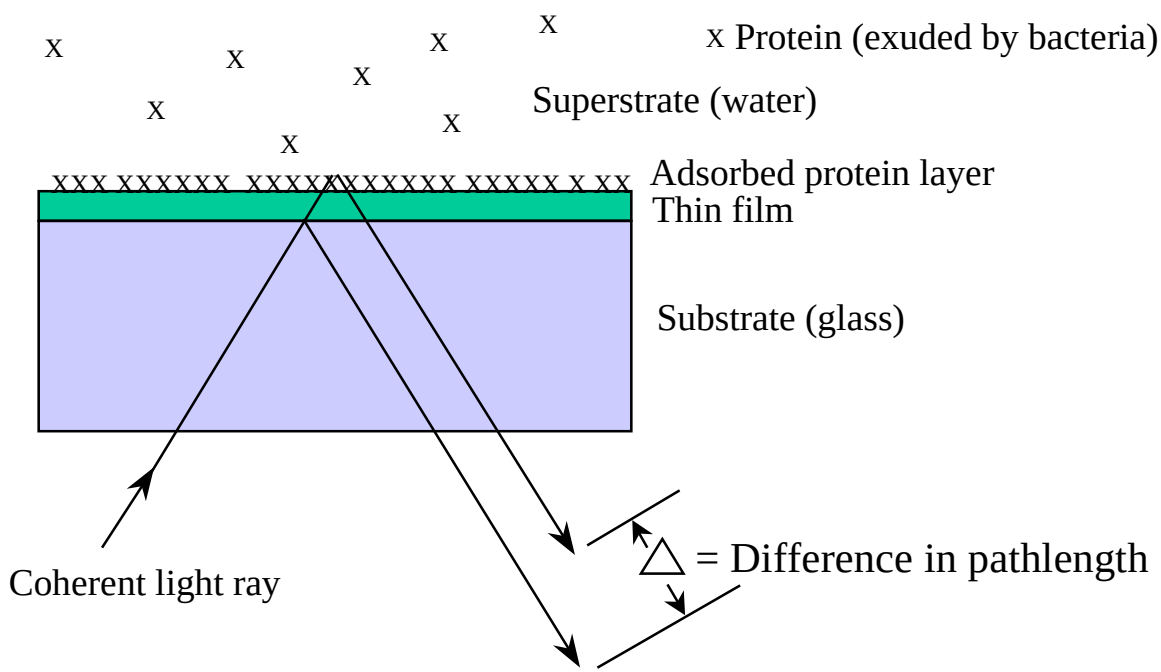
6. A real-time micro-biocontamination monitor is needed.

- Monitor would warn of biocontamination before critical levels occur.
- Monitor would enable corrective measures to be taken before contamination is released.
- Monitor will provide early warning by detecting a protein film that forms on wetted surfaces prior to sessile-bacteria attachment.

7. Various technologies were considered for monitor.

- Surface Plasmon Resonance
- Resonant Wave Modulation
- Optical Fluorescence
- Optical Interference

8. The micro-biocontamination monitor (MBM) being developed uses optical interference.



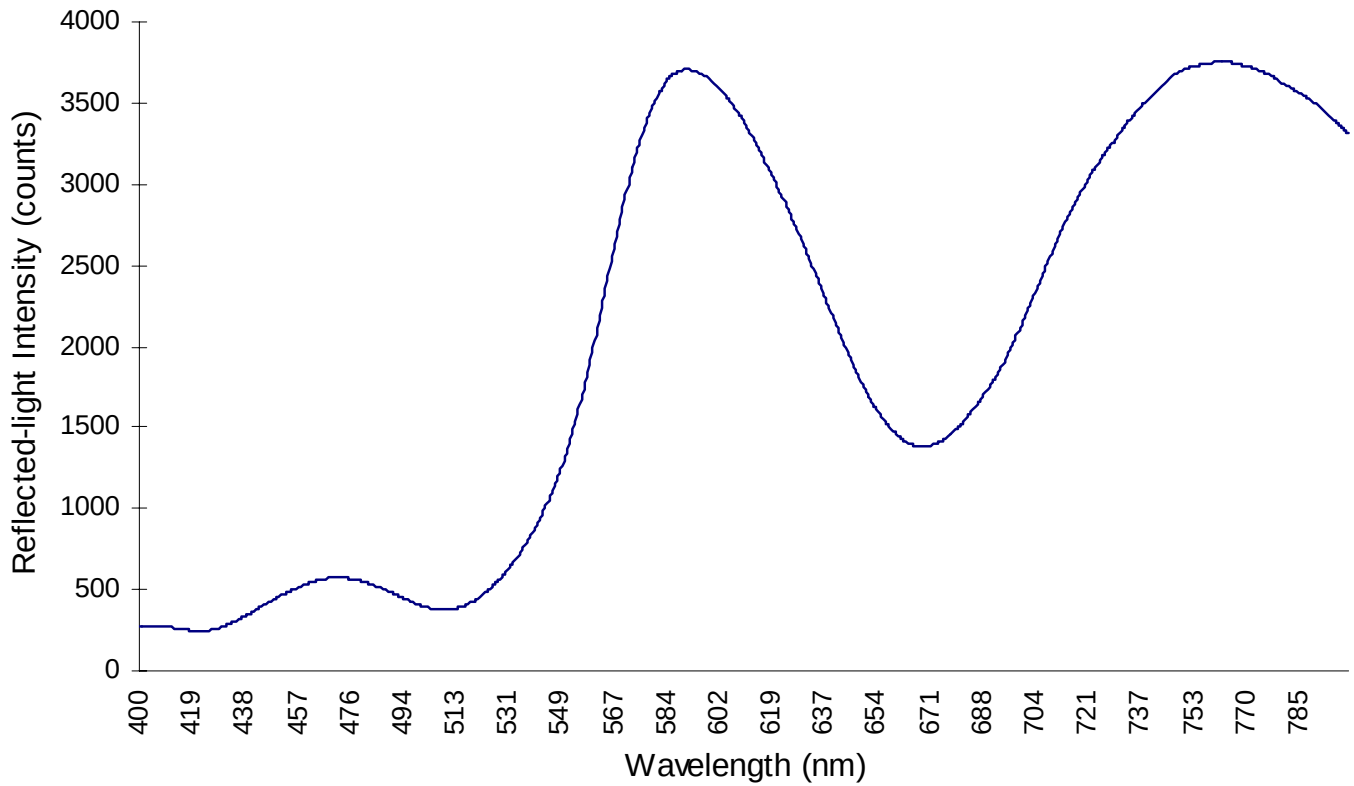
9. Protein deposition onto thin film modulates reflected-light interference.

- Maximum and minimum interference of reflected waves occurs at wavelengths (λ) that are quarter multiples of the thin film's optical thickness (nd; n = refractive index of thin film, d = thickness of thin film):

$$\text{Reflectance} = R = f\{\cos(4\pi)(nd/\lambda)\}$$

- Attachment of bacterial protein to thin film shifts wavelengths of reflectance-spectrum extremes.

10. Reflectance Spectrum of TiO_2 Thin-Film



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11. Shift in wavelength of spectral extreme can be estimated using basic interference relationship.

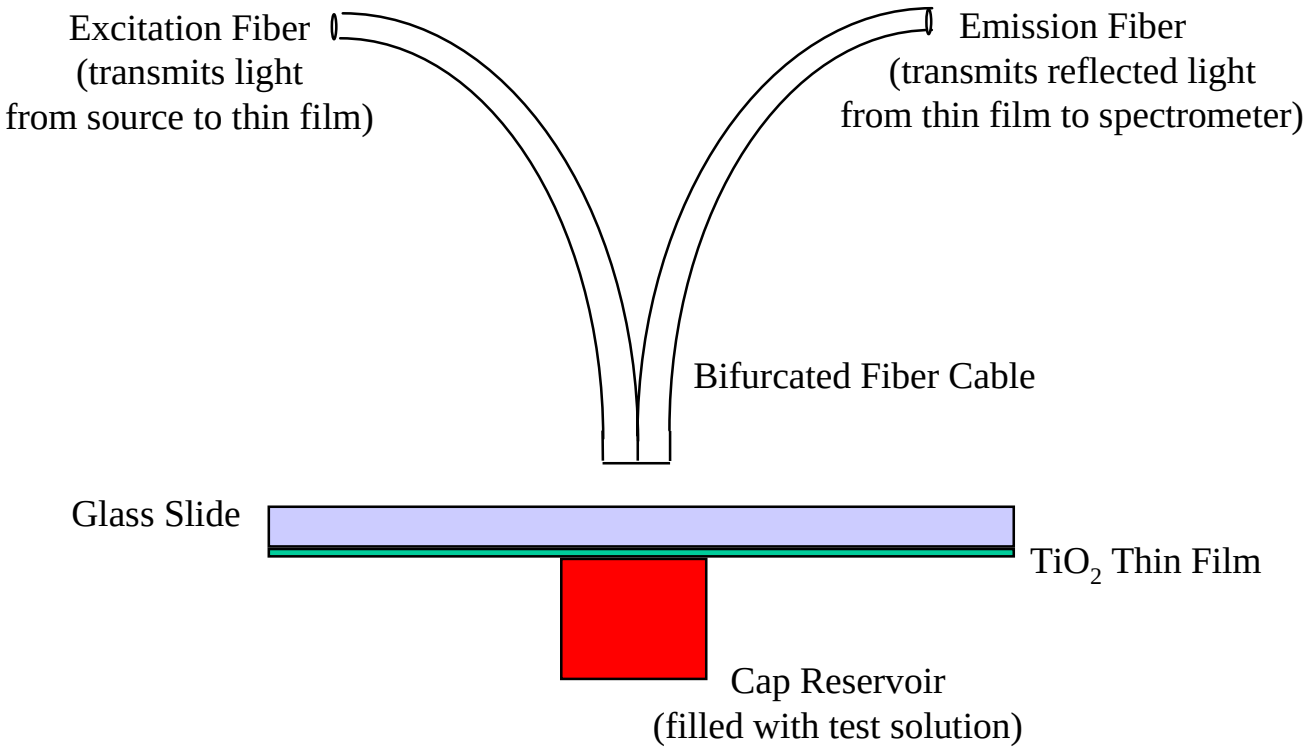
- Thin-film's optical thickness is related to the order (m) in an interference spectrum according to:

$$2nd/\lambda = m$$

- For small thickness changes (constant m), where i and f denote the initial and final conditions:

$$m = (2n_i d_i)/\lambda_i = (2n_f d_f)/\lambda_f; \text{ and, } n_i d_i/n_f d_f = \lambda_i/\lambda_f$$

12. MBM Experimental Setup

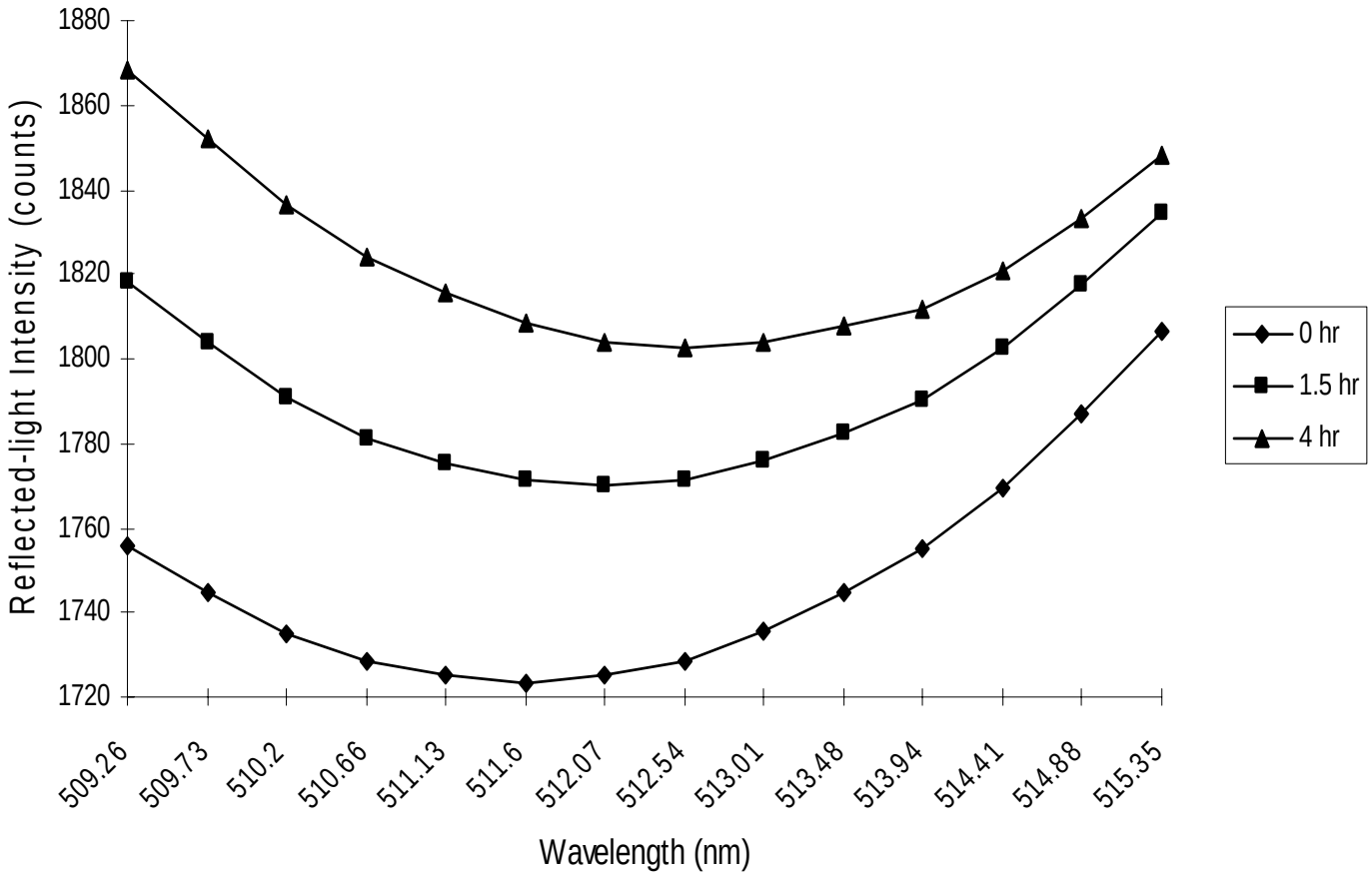


13. Protein deposition onto thin film was detected with MBM experimental setup.

- MBM detected deposition of, what was assumed to be, a monolayer of fibrinogen protein (used as a bacterial-protein analogue).

- The minimum-reflectance wavelength (λ_{min}) near 512 nm, increased by about 1 nm during 4 hrs contact of the thin film with an 8-ppm fibrinogen solution.

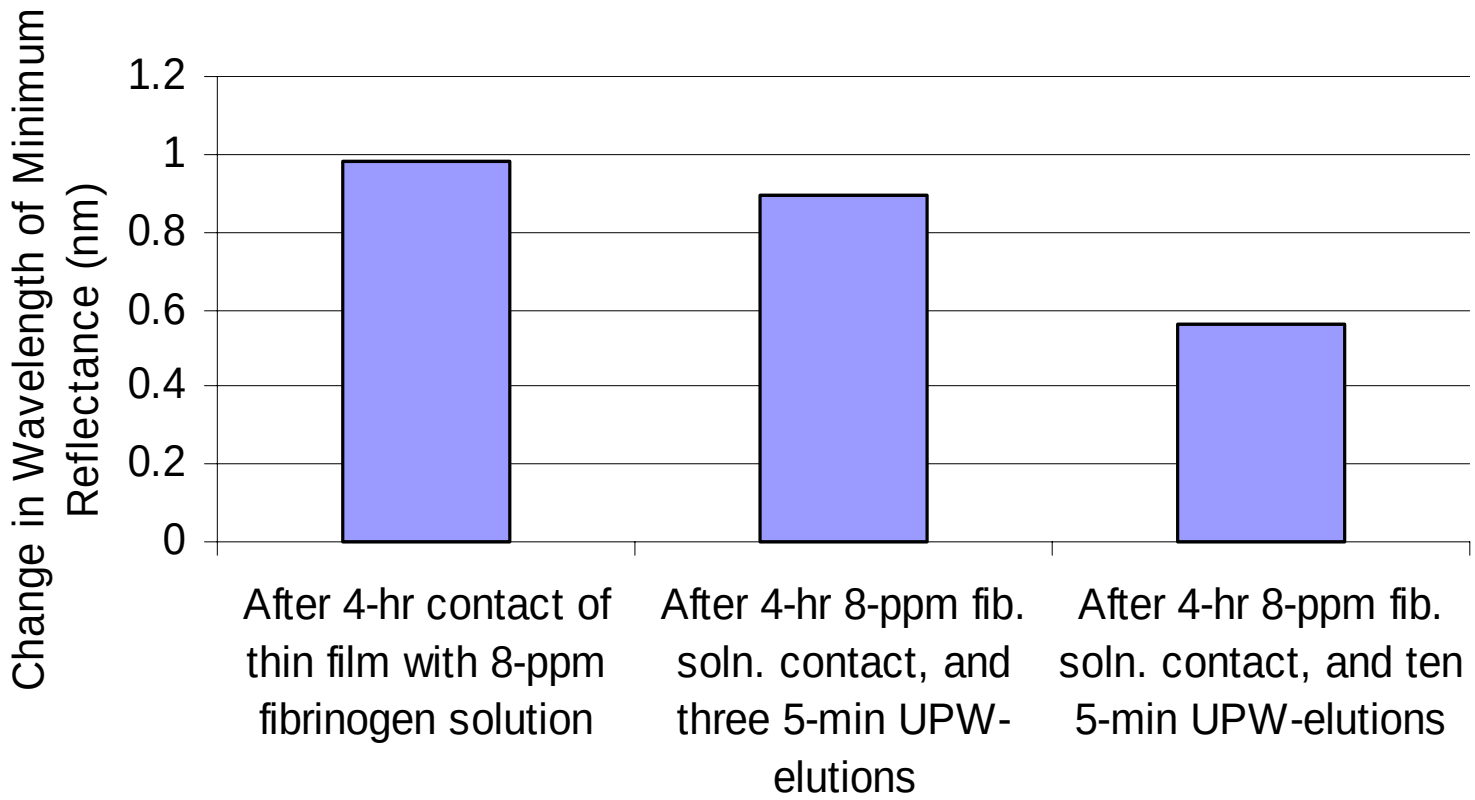
14. Wavelength of minimum reflectance increases as fibrinogen protein attaches to thin film.



15. Additional experiments were done to confirm ability of MBM to detect actual monolayer.

- Fibrinogen monolayer is bound to surface by van der Waals forces, and additional layers are attached by hydrogen bonds.
- Control experiments showed that 10, 5-min UPW-elutions of fibrinogen-contacted surfaces should produce fibrinogen monolayer.
- MBM can resolve at least 0.1 nm shift of λ_{min}, and a shift of >0.5 nm was seen for fibrinogen monolayer.

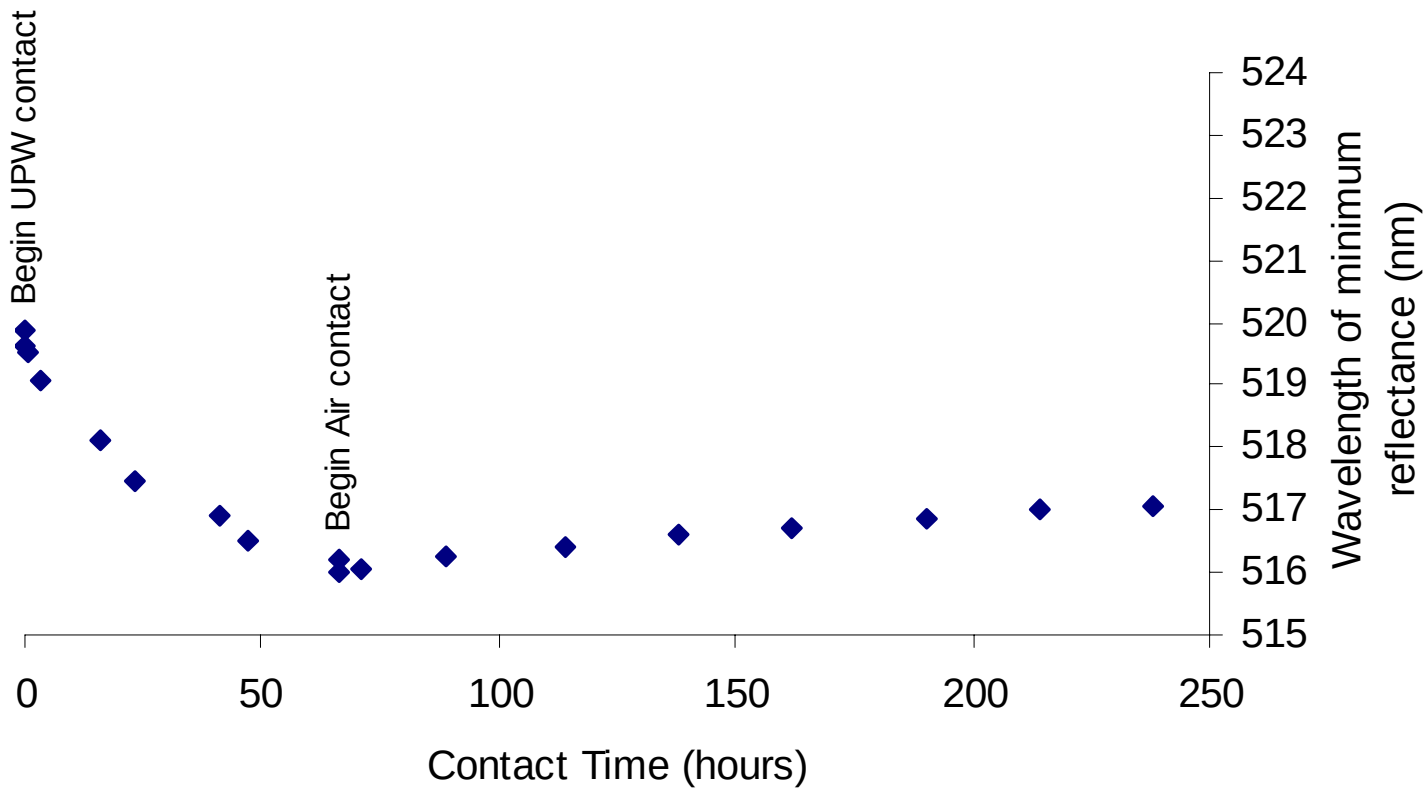
16. Fibrinogen monolayer caused a λ_{min} increase of more than 0.5 nm.



17. Control experiments were done to validate measurement.

- Thermal variation of n_{TiO2} wasn't significant; no shift in λ_{min} > 0.1 nm was seen for 10 °C decrease in thin-film temperature.
- Light-source drift , heating, and photolytic effects caused no change in λ_{min} that was > 0.1 nm.
- λ_{min} decreased significantly during UPW contact (wetting) of TiO_2 thin film, and increased during drying period (air contact).

18. The λ_{min} of the TiO_2 thin-film changed with UPW-wetting and air-drying.



19. Drift of λ_{min}, during UPW contact, probably not caused by redox or hydrolytic reactions.

- Redox conditions, at pH and E_h of UPW, favor TiO_2 stability.
- Hydrolysis of thin film was probably negligible.

$$HTiO_3^- + H^+ = TiO_2 + H_2O$$

$$\lg[HTiO_3^-] = pH - 18.00$$

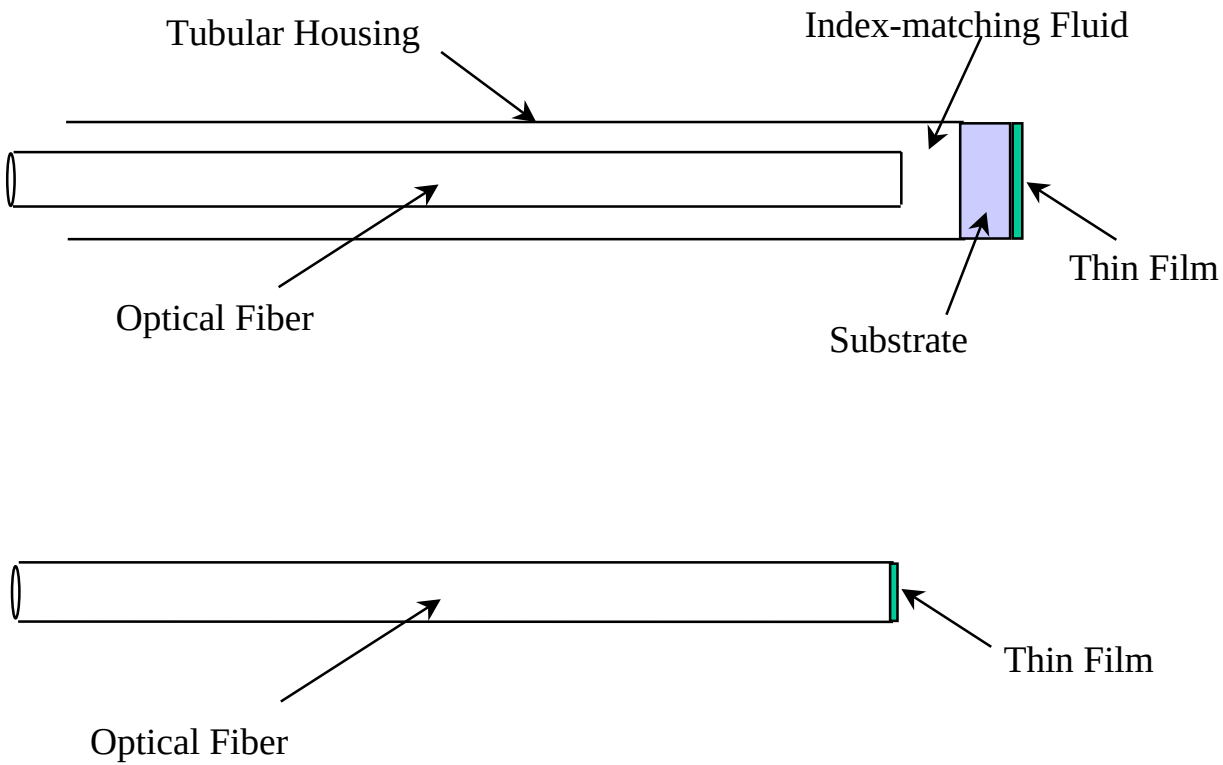
20. Drift of λ_{min} was probably caused by microporosity of the thin film.

- TiO_2 thin film was deposited by e-beam evaporation.
- Possible that n_{TiO2} was changed by the hydration of TiO_2 sites as UPW migrated through micropores.
- Reflectance intensity (R) was observed to decrease and increase, with λ_{min} decrease and increase, respectively. This would be expected for air and water exchange in the micropores; *i.e.,* R would decrease as water displaced air.

21. Drift of UPW-wetted TiO_2 thin films can be addressed in various ways.

- Coat thin film with a material (*e.g.*, protein) that will prevent movement of water into the micropores.
- Use CVD to deposit TiO_2 onto substrate.
- Use a material such as PVDF for the thin film. The low n of PVDF can be tolerated by using: a thin film of Ta_2O_5 at the substrate/PVDF interface, to improve reflectivity; and, a photodiode-array detector, to improve signal resolution.

22. Possible Sensors



23. Design Options

- Various thin-film materials can be used.
- Software aids include signal averaging and polynomial regression.
- Hardware options include optical-grating modification, photodiode use, and source-light referencing.

24. Conclusions

- Monitor shows promise for industrial use.
- Experiments show ability to detect protein deposition onto thin film.
- Design modifications are available to improve performance.