Automation of Precision Ellipsometry System

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Abstract Precision ellipsometry (PREL) is an optical technique used to measure changes in the thickness of surfaces or thin films. With a sensitivity of up to 0.01 nm, it is used in studies of molecular layers. A compact, low-cost and portable computer-driven system was developed to automate the fluidic processes of PREL. Two fluidic components were designed and built—a syringe pump to control the injection of liquid reagents and a valve to regulate the flow of rinsing liquid for washing. Motors were used to control the flow of liquids with pipes, syringes and other low-cost materials. Circuit networks, as well as programmes in Arduino and Python, were developed and integrated together to provide computer control through a graphic user interface and to simplify setting operation parameters. The automated system reduces operational fatigue and removes inaccuracies in data collection due to human error. It also enables multiple PREL set-ups to be run simultaneously by a single operator. The system can be incorporated into the existing PREL set-up easily, thus allowing PREL to be applied in a research or industrial environment.

Keywords Engineering mechanics · Industrial engineering-processing · Precision ellipsometry · Automation · Valve · Syringe pump · Fluidic control

1 1. Research Problem

A. *Precision Ellipsometry*

Precision ellipsometry (PREL) is an optical technique used to determine changes in the thickness of surfaces or thin films. With a sensitivity of up to 0.01 nm, it is used

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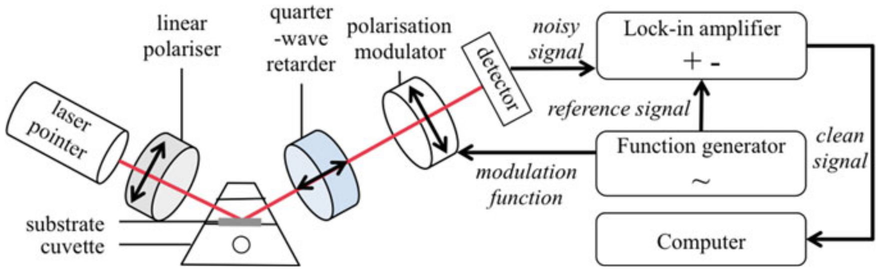


Fig. 1 Electro-optical set-up of liquid-phase precision ellipsometry

in the study of molecular layers and the adsorption and desorption of molecules [1]. It has a wide range of practical applications, including bio-sensors, protein detection systems, the real-time monitoring of materials growth and reaction kinetics.

Presently, a portable and versatile PREL set-up has been developed [2] and used in the study of biomolecules [3]. This project shall focus on automating this set-up.

In PREL, the substrate is placed into the cuvette, which has two transparent sides. The laser pointer shines polarised light onto the substrate. During an experiment, liquid reagents are injected into the cuvette. When the reagent molecules are adsorbed onto the surface of the substrate, there will be a change in the polarisation of the light reflected off the substrate. This change in polarisation is recorded by the modulator, photo-detector and lock-in amplifier, Fig. 1. Washing is then carried out by passing rinsing liquid through the cuvette to remove any reagents that have not attached to the substrate. Each layer to be deposited on the substrate involves one cycle of injection and washing. Presently, a pipette is used to inject reagents manually and manual clamp is used to control the flow of the rinsing liquid. This has several disadvantages.

Firstly, it is labour-intensive, especially since a typical experiment involves multiple cycles of injection and washing. Secondly, manual operation increases noise in data acquisition. As the PREL set-up is sensitive, any vibrations caused by the operator may affect the accuracy of data collected. Thirdly, it is imprecise. Due to human reaction time, it is impossible for fluids to be added to the cuvette at the precise moment when they are needed. Also, with manual operation, the rate at which fluids are added will be different each time. In time-sensitive experiments, this may lead to errors calculating the rate of change of film thickness, which is crucial in kinetics studies.

Automation of the fluidic processes of PREL is beneficial. Firstly, it allows the operator to multitask, since he or she no longer needs to focus entirely on the set-up during an experiment. Secondly, it allows for multiple PREL set-ups to be run by a single operator simultaneously, reducing the duration of experimentation.

B. *Engineering Goal*

This project aims to automate the fluidic components of the present PREL set-up by building a compact syringe pump to inject reagents, and an automated valve without moving parts in liquid to control the flow of rinsing liquid. The new system will combine fluidic control and the respective electronics with the existing data acquisition system. It will be designed such that it can be integrated with the existing set-up easily, to allow PREL to be applied in a research and industrial environment.

2 Design of System

The development of fluidic control will consist of an automated valve and a syringe pump controlled by the same Arduino that is used for data acquisition. The syringe pump and the automated valve were designed to make the system as compact, low-cost and portable as possible (Fig. 2).

C. *Design of Syringe Pump*

Conventional syringe pumps have polished rails for a sliding mechanism to push the plunger, making these syringe pumps complicated and expensive. A pump without rails would usually be three times the distance travelled by the piston (Fig. 3, top). We took a novel approach and designed a pump without rails which is only two times the travel of the piston (Fig. 3, bottom), by incorporating the screw into the plunger (Fig. 4).

When the motor rotates clockwise, the long screw will rotate as well. Since the slider is unable to rotate, it will move forward, pushing the plunger flange of the syringe inwards. Conversely, when the motor rotates counter-clockwise, the slider

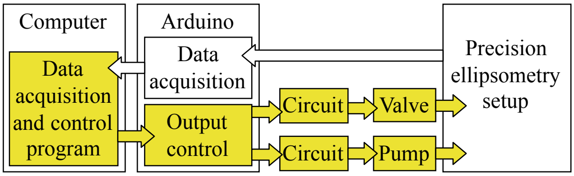


Fig. 2 Schematic of proposed system. The yellow boxes show the components built for fluidic control

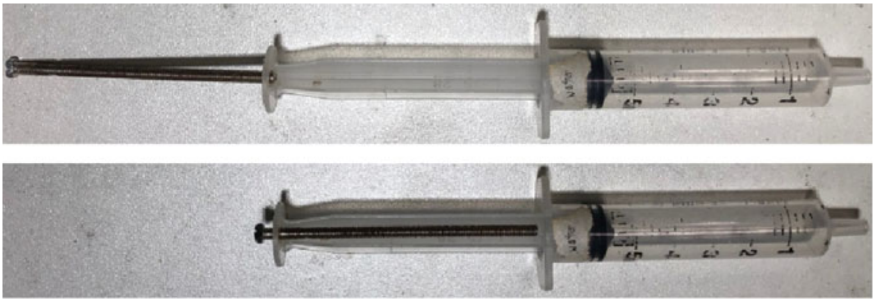


Fig. 3 Length of conventional syringe pumps without rails (top), and our design (bottom)

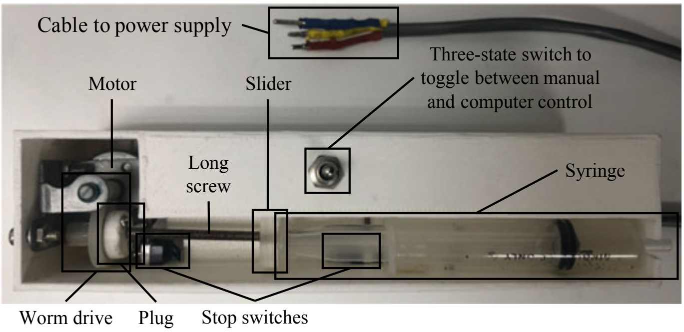


Fig. 4 Diagram of syringe pump

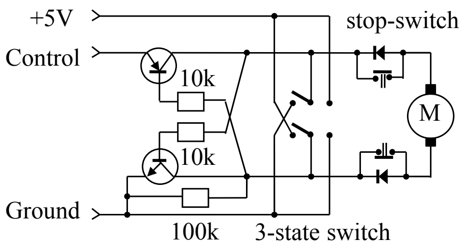
will move back to its original position near the motor, allowing the syringe to be replaced.

The three-state switch allows the operator to choose between manual operation or computer control. In the side positions, the switch applies +5 V power in forward or reverse polarity to the motor for the slider to move forwards or backwards respectively. The transistors are connected with their collector to the three-state switch, so that they are both closed when it is in the side positions. In the middle position, the motor will be controlled by the Arduino. With 5 V at control, both transistors are open and apply voltage to the motor for the slider to move forward; with 0 V at control, no voltage is applied. In this case, the operator's only role is to flick the switch backward to move the slider back to its original position after each syringe has been emptied. The circuit (Fig. 5) has been designed such that manual operation always overrides computer control, without creating any short circuits.

The stop switches break the circuit when pressed by the slider, preventing it from moving towards them any further. However, the motor will still be able to rotate in the opposite direction for the slider to move away from the stop switch being pressed. This allows the motor to be used continuously without needing to be reset each time the slider moves forward or backward.

D. *Design of Valve*

Fig. 5 Diagram of control circuit of syringe pump



A valve was developed to regulate the flow of rinsing liquid into the cuvette. It has no moving parts in the liquid, to prevent any contamination of the rinsing liquid from the moving parts (Fig. 6).

The rotation of the motor was used to twist the pipe, since the motor is not strong enough to close the pipe by pressing it. A customised 3D-printed accessory was attached onto the main gear of the motor. It includes a holder to clamp the pipe to the motor, as well as a finger to press the stop switches at the sides. The ends of the pipe are held in place by clamps, so that the pipe does not shift when it twisted. When the motor rotates clockwise, the pipe is open (Fig. 7, left). When it rotates counter-clockwise, the pipe is closed (Fig. 7, right) (Fig. 8).

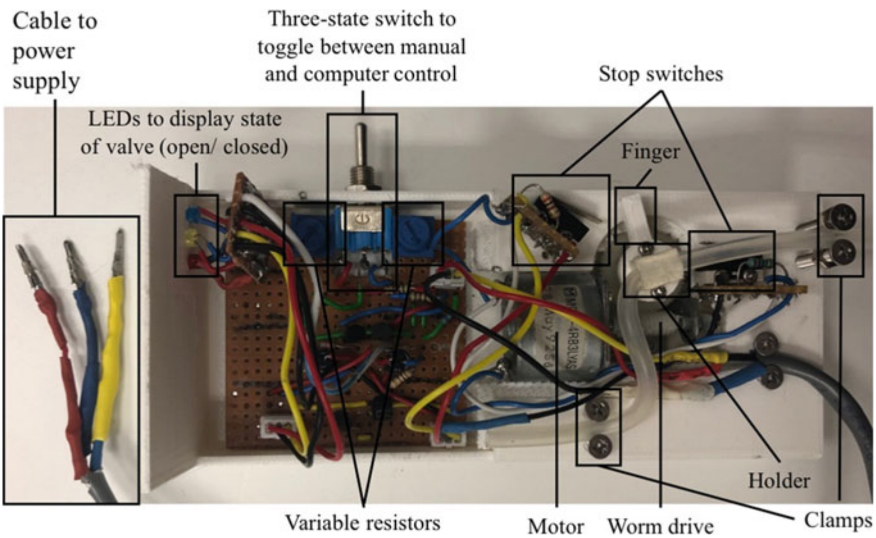
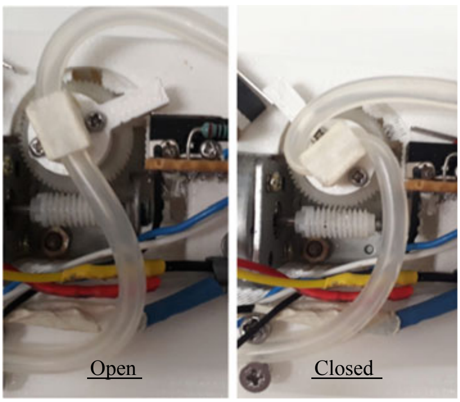


Fig. 6 Diagram of valve

Fig. 7 Shape of pipe when water can flow (left) and cannot flow (right)



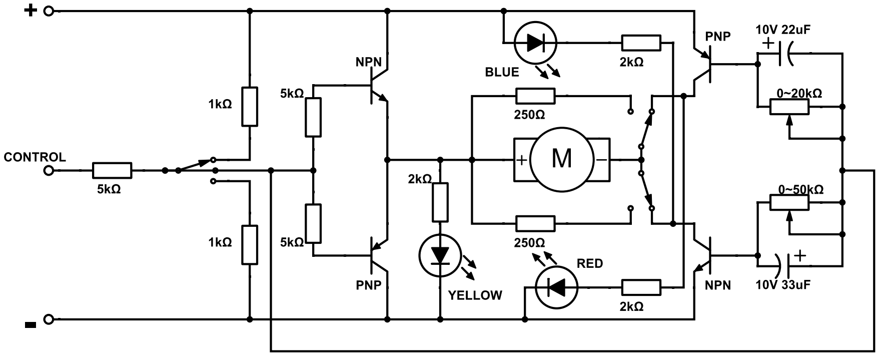


Fig. 8 Control circuit for valve

A circuit was designed to control the rotation of the motor. Stop switches at the side of the motor are used to break parts of the circuit, to stop the motor at the precise position where the pipes are opened or closed. If the motor rotates clockwise to press the switch on the right, only the circuit responsible for clockwise rotation will be broken. Thus, the motor can still rotate counter-clockwise. The same is true for the opposite direction. This feature allows the device to be used continuously without the need to reset it after each rotation.

The three-state switch allows the operator to toggle between manual operation and computer control. When the switch is at the centre, the device is controlled by the computer. When the switch is flicked to the left or right, the motor rotates clockwise or counter-clockwise respectively.

E. *Integration with Computer*

To allow the operator to control the syringe pump and valve with the computer, a graphic user interface (GUI) was built using Python. In the GUI, the operator inputs a schedule for injection and washings, with a time specified for the start and end of each. When it is time to perform an action, Python will send a byte to Arduino through serial communication. Four different bytes are defined in both the Python and Arduino code for each of the four actions—start injection, stop injection, start washing and stop washing (Figs. 9 and 10).

After receiving the byte from Python, Arduino will change the voltage supplied by the ports. Three Arduino ports are used, one for the valve and two for syringe pumps. Each port can either be set to high voltage or low voltage. The six possible Arduino controls are each represented by the six bytes.

The syringe pump and the valve were designed to respond to the Arduino controls. Injection begins when the Arduino port the syringe pump is connected to supplies a high voltage. Similarly, washing begins when the Arduino port the valve is connected to supplies a high voltage. The reverse is true when injection and washing ends. For data acquisition, the sensors connected to Arduino writes back to Python, which saves the data. In this way, the system simultaneously records data and performs actions.

Fig. 9 GUI upon entering the system

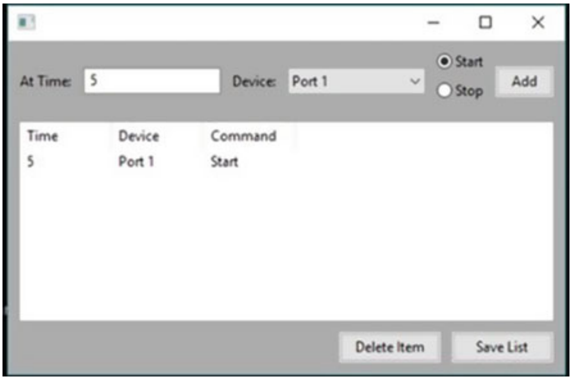


Fig. 10 GUI for scheduling of tasks

3 Evaluation

Our automated system was evaluated by deposition of poly-electrolyte multilayers from water solutions onto silicon substrate and simultaneous recording of deposited thickness. The molecules used here were: poly-allylamine-hydrochloride (PAH), which is positively charged in water solution, and sodium poly-styrene-sulfonate (PSS), which is negatively charged. Thus depositing one above the other creates a stable film by electrostatic attraction.

The process of multilayer deposition was as follows: silicon substrates were cleaned by wet chemistry installed into optical cuvette and optical system was aligned. While rinsing water was flowing through the cuvette, data acquisition started. At 590 s after the start, the valve was closed, and at 600 s, the syringe pump injected 1 ml of PAH solution; it is seen that the thickness gradually increased to around 0.5 nm, Fig. 11. At 1000 s, the valve was opened and the solution was rinsed away, but the thickness did not change; it shows that the attachment was permanent. The same process was repeated with PSS solution at 1200 s, then again PAH at 1800 s and so on. The measurement shows that PSS consistently gives increments of around

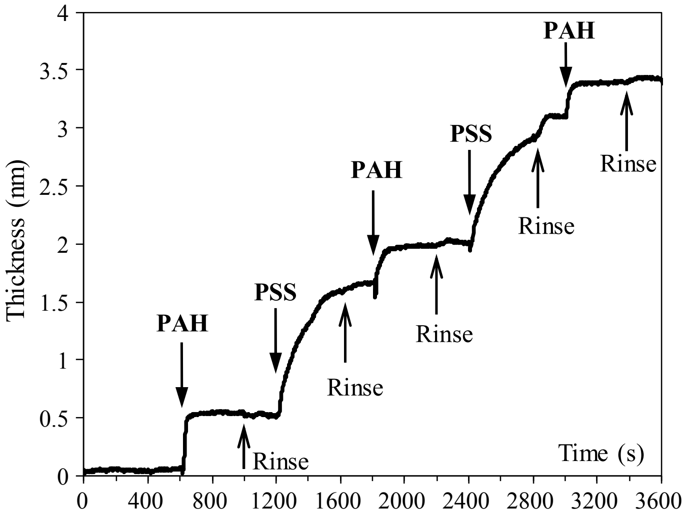


Fig. 11 Example of PREL measurement of sequential attachment of poly-electrolyte layers PAH and PSS on oxidised silicon substrate

1 nm and PAH around 0.5 nm. This proves reproducibility of control and acquisition and confirms stability of the whole system even during the whole hour of the process.

Our automated system brings about several advantages. Firstly, our system is easy to use, with the GUI being the sole interface the operator has to work with. With the syringe pump and valve in place, the operator simply schedules the time for injection and washing. Secondly, the programme designed brings computer precision to the PREL set-up, for injection and washing to be performed on time. With the system, there will no longer be any more mistimed addition of chemicals. Thirdly, our system is portable because it takes power only from a USB power supply, and USB ports are readily available. It is also compact, as the components can be fit into two small 3D-printed boxes—one for the syringe pump and one for the valve, with a total space of 40 mm wide, 165 mm long and 60 mm high. Fourthly, the cost of the system is low. While commercial peristaltic pumps or a standard syringe pumps can cost up to $500, our system was built with low-cost materials, such as relatively cheap motors, circuit boards and 3D-printed bodies. Lastly, the automated system can be easily modified to be compatible with other techniques as it does not have requirements specific to PREL (Fig. 12).

4 Conclusion

We have developed a flow control system to automate the fluidic components of the present PREL set-up. It consists of a syringe pump to inject reagents, and a valve

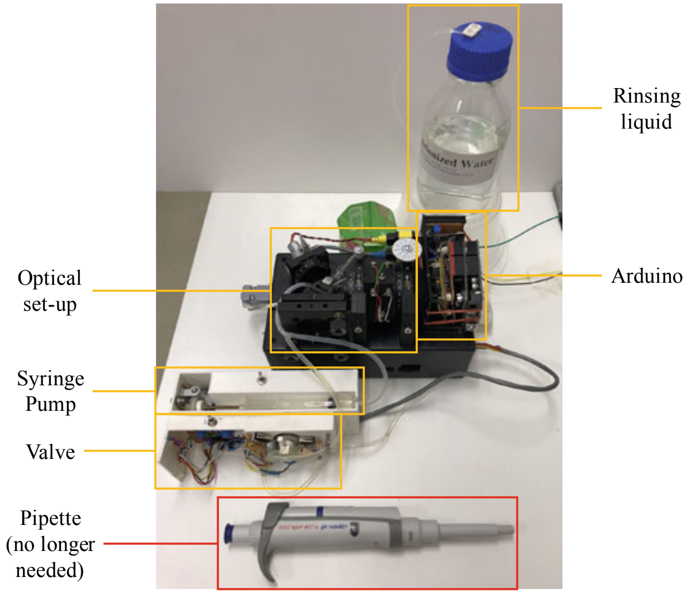


Fig. 12 Automated system developed in this project, integrated with the existing PREL set-up

to regulate the flow of rinsing liquid, respective electronic circuits and software to control the system simultaneously with data acquisition. The specialised automation system can be integrated easily into the existing PREL set-up. Furthermore, the compact, low-cost and portable nature of the system allows it to be used efficiently in a research or industrial environment. Most importantly, it works.

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