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# Development of Microfluidic Cell for Liquid Phase Layer Deposition Tracking

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**Abstract**

This paper shows how microfluidic tools can be used for up-to-date microstructural investigations based on thin film deposition. The construction and production methods of such measuring procedures are introduced, and their application in ellipsometric investigations is shown. By using these tools, the researchers provide the possibility to observe and document the effects of certain fine structural processes in the development of the final microstructure. This paper describes two specific application areas of such microfluidics cells. Microfluidics cells can be used together with both optical microscopy and spectroscopic ellipsometry to understand previously unexplored microstructural changes.

**Keywords**: *microfluidics, material science, rapid prototyping, in situ material testing.*

## 1. Introduction

Nowadays with the advancement of technology the quality of produced materials has begun to improve significantly. As a result, there has been a growing need for advanced material testing methods [1]. However, some of the currently used manufacturing and material testing methods still contain procedures for which the process of the occurring phenomena is not yet fully understood.

A good example of this phenomenon are the different etching processes, especially colour etching. In optical microscopic analysis etching is a common method for revealing the microstructure of previously polished specimens [2]. Despite its frequent use, many microstructural changes during etching have not been fully explained [3], although many attempts have been made to clarify the connection between colour etching and certain metallographic properties [4, 5].

Due to the extensive use of rapid prototyping methods and to the development of polymer materials, a new possibility to monitor microstructural changes has emerged. Additive manufacturing technologies have now achieved the dimensional accuracy needed to create channels up to a few tens of millimeters in diameter [6]. Using these techniques and materials, microfluidic cells can be formed in which the etchant can be circulated under controlled conditions [7]. Depending on the design of the cell, microfluidics may be combined with devices already used in metallography, such as an optical microscope or a spectroscopic ellipsometer. Thus, the etching processes could not only be investigated in discrete moments but also in situ.

## 2. Experimental

### 2.1. Design of microfluidic cells

To demonstrate the versatility of the fabrication methods, two different cells have been designed.

When designing the microfluidic cells, care must be taken to ensure that the completed system can be integrated into the equipment to be used with it. For a cell-compatible geometry, the sample had to be embedded so that it could be examined independently of its original geometry.

The first microfluidic cell was designed to be compatible with an optical microscope. This enables real-time optical tracking of etching and layer-building processes [8].

As the magnification increases, the working distance between the objective and the specimen becomes smaller. Thus, the maximum resolution had to be considered during the design. An Olympus BX51 type LMPlan FI50/0.50 long working distance optical microscope with a minimum focal length of 3.6 mm was selected as an equipment to be designed for. The laminar flow of the etchant is provided by the formed channel system, supported by syringe pumps. Since good quality images require a sufficiently long distance between the objective of the microscope and the sample, the cell is closed as shown in **Figure 1** by a glass sheet. The design considerations are also illustrated in **Figure 1**. There is another chamfer in the finished geometry for the junction between the sample and the cell, which makes it easier to swap the samples (**Figure 2**).

The second microfluidic cell was specifically designed to be used with a spectroscopic ellipsometer. Ellipsometry enables the measurement of surface layer thickness with nano meter accuracy, to study optical refractive index, homogeneity or even the surface's nano roughness[9].

Ellipsometry is particularly sensitive to refraction at interfaces. The cell must be designed so that the beam of light used for the test passes through each crossing interface perpendicular to the surface of the sample. Thus, maximal luminous intensity and low noise can be ensured. For designing the cell, a Woollam M-2000DI ellipsometer was used. The beam of light exiting the transmitter is approximately 3 mm in diameter, decreasing to one-tenth until it reaches the sample's surface. (**Figure 3**).

A moving mechanism must be also provided which allows the beam to be focused at any point on the surface to be examined, while the sample is fixed. Therefore, the microfluidic cell must be created from two parts. The upper cell provides the flow of the etchant and the angles required for the maximal intensity. The lower cell is used to fix and position the specimen. The two halves can be attached to each other as shown in **Figure 4**.

## 2.2. Fabrication of the microfluidic cells

To create microfluidic cells, a chemically inert material that does not react with either the sample or the etchant should be chosen. The material also must be transparent to let the light pass through. Poly-dimethyl-siloxane (PDMS) was chosen as the base material of the cells. It has an already proven quality for microfluidic cell manufacturing. When the monomer of PDMS is mixed with the curing agent in a 10:1 ratio, the high viscosity mixture begins to crosslink. This mixture was filled into a mould, then heat treated at 100 °C for 60 minutes to support polymerisation. The moulds for both cells were made with an Objet Eden 250 type 3D printer.

The created PDMS cell which is compatible with an optical microscope is bonded onto a glass sheet by crown discharge treatment to seal the cell. To strengthen the bond, the finished cell was pressed on the glass sheet and rested in an oven at 60 °C for an additional 30 minutes. The internal channel system of the cell thus formed has a volume of 205 mm$^3$ (**Figure 5**).

In the case of the other cells created to be used with an ellipsometer, the glass sheet could not provide the perpendicular angle of light entry

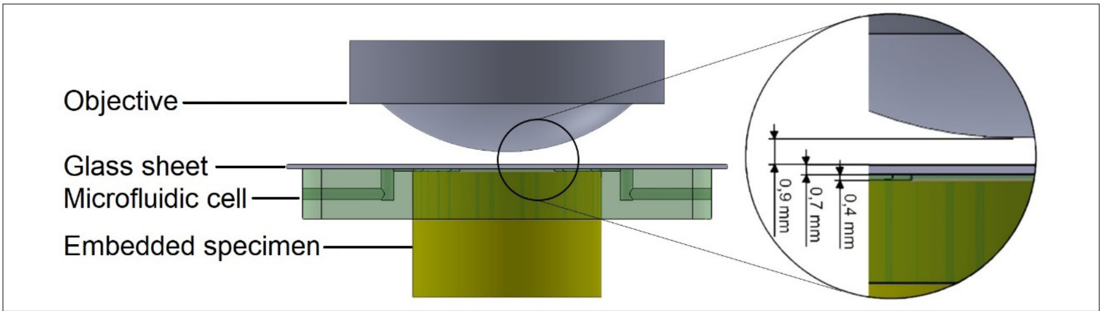


**Figure 1.** *The dimensions of the microfluidic cell, which is compatible with an optical microscope.*

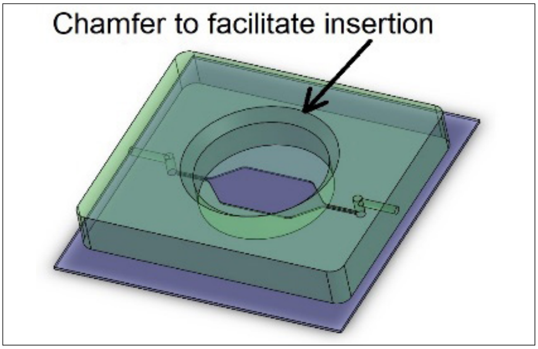


**Figure 2.** *3D design of the microfluidic cell which is compatible with an optical microscope.*

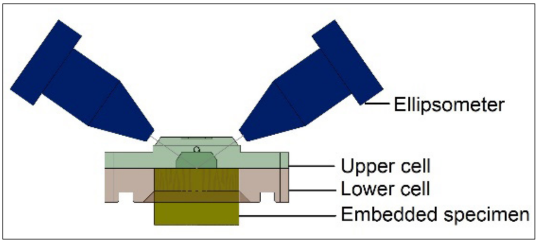


**Figure 3.** *Design of microfluidic cell compatible with spectroscopic ellipsometer.*

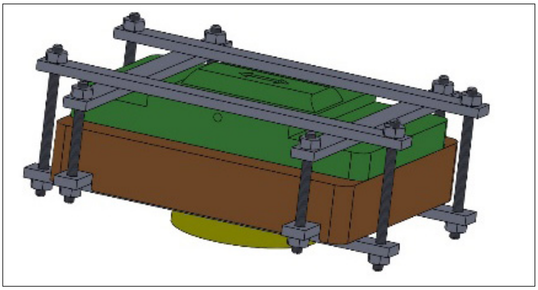

**Figure 4.** *Clamping mechanism of microfluidic cell which is compatible with an ellipsometer.*

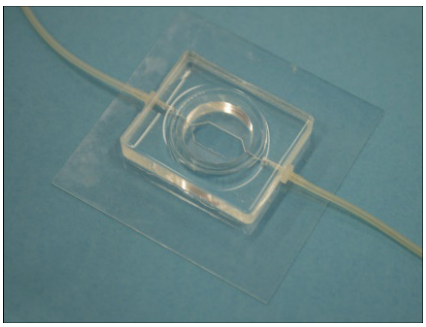

**Figure 5.** *The fabricated microfluidic cell which is compatible with an optical microscope. Silicone tubes are attached to both ends of the cell.*

and exit. Thus, both the top and bottom of the cell should be made by PDMS. They can be secured to each other as shown in **Figure 4.** This cell has an internal channel volume of 851 mm$^3$.

## 3. Application of microfluidic cells

The inlet and outlet openings of the cell can be connected to a syringe pump by means of silicon tubes. The pump can supply the cell with fresh etchant while the laminar flow is ensured. The equipment thus developed is able to track the processes on the sample's surface by various means throughout the etching process. The technique allows digital image capture for the first presented cell and the following of interference change caused by layer growth on the sample surface for the second cell.

Although the use of microfluidics systems extends the capabilities of microstructure testing methods, the limitations of the system should not be forgotten. In microscopic examinations, the magnitude of magnification cannot be increased indefinitely due to the focal length of the used lens. The smallest particle size that can be examined is approximately 3–5 µm. For the other microfluidic cell, developed for ellipsometry, the dimensions of the internal channels of the cell are bound to provide light paths, so that the entire volume must be filled with the desired milling agent prior to measurement. Because the microfluidics system takes time to fill, the initial etching phase cannot be recorded..

## 4. Conclusion

Microfluidic systems enable in situ microstructure investigations, which could significantly improve the monitoring of etching and thin-layer growth processes. Despite the obvious drawbacks of the technology, it is well suited to combine with other microstructure analysing equipment. Furthermore, they can be safely used in corrosion, etching, or coating applications due to their closed system.

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