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Design and Manufacture of a Microfluidic Cell To Be Used With a Spectroscopic Ellipsometer

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Abstract

In material testing and manufacturing processes, creating thin layers is a widely used method for structure development or for surface treatment purposes. Despite its widespread use, the physical background of the layer development process is currently under-researched. Its examination requires the development of procedures and tools that, in combination with the existing tools, can help to understand these processes. The development of microfluidic cells is a way to solve this problem. In this paper, a newly developed microfluidic cell is presented, which also offers a solution to several problems encountered when using previous designs.

Keywords*: microfluidic cell, design, manufacture, spectroscopic ellipsometry.*

1. Introduction

Various etching methods are widely used in metallography. One of the most common types of these is colour etching [1]. During colour etching, the etchant reacts with the surface of the polished sample to form a thin transparent layer. The light reflected from the surface of this film interferes, causing a visible, cyclic colour change of the individual grains. The orientation of these grains can be calculated using the etching speed [2], [3]. During the etching process, microfluidic cells are used to record surface colour change. The phenomenon occurring on the surface can be observed not only with an optical microscope but also with a spectroscopic ellipsometer [3]. Concepts aimed at achieving this were previously put forward, but they could not be put into practice due to manufacturing limitations [1]. In our work, we aimed to design and manufacture a microfluidic cell that allows the observation of the rapid exchange of the surface and extends the use for nanometer-precision spectroscopic ellipsometry [4] while eliminating the errors of previous concepts. In addition, the positioning of the sample can be performed with an optical microscope.

2. Design and manufacture

The work began with defining the requirements. The dimensions of the embedded sample to be examined and the geometrical parameters of the Woollam M-2000DI spectroscopic ellipsometer used for the tests were given. The following size constraints posed a major design challenge. The height of the device could not exceed 45 mm. A channel at least 3 mm in diameter had to be provided along the path of the light, in which the border between different phases had to be perpendicular to the path of the light. Otherwise the ellipsometer could not detect the previously diffracted light. Finally, the aim was to create the smallest possible internal cavity, as it had to be filled with etchant before any measurement. However, the production of mechanical parts becomes cumbersome within this small size range.

2.1. The device

The schematic structure of the device is shown in Figure 1. It can be divided into three main parts: the polydimethylsiloxane (PDMS) cell, the welded frame structure, and the sample tray.

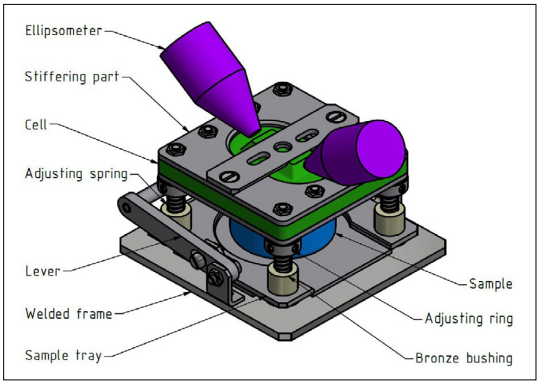

Figure 1. *The schematic structure of the device.*

2.2. Microfluidic cell

The most important part of the device is the cell, where the etching process and the examination occur (**Figure 2**). It must be made of a chemically inert, preferably transparent, dimensionally stable material. The choice has been made for the previously used PDMS **[1]**. The PDMS cell was made by molding, so the geometry had to be manufactured with high precision while avoiding undercut surface elements. In addition, the molds must be heat resistant up to 180 °C. Due to their design, molds can be made by different methods such as additive manufacturing **[5]** or 5-axis CNC machining **[6]**. For economic reasons, the mold created with additive manufacturing was chosen.

Several aspects had to be considered while designing the cell. One of the main disadvantages of the previous models was that the initial stage of the etching process was not observable, as the filling of the cavity took a relatively long time **[1]**. Its volume has been reduced from 851 mm^3 to 90 mm^3. Thus, filling the main cavity under the same flow conditions takes only a few seconds within the new design. By forming surfaces perpendicular to the path of the light beam, refraction at the boundaries of the different media can be avoided or minimized.

In spectroscopic ellipsometry, the maximum intensity of the light beam reflected from the surface of the sample can be obtained at an angle of incidence of 55° **[7]**, **[8]**. The light beam enters and leaves the cell, as shown in section A-A of **Figure 2.** The inner cavities for the light beam are closed with a laminated glass plate. The channels to introduce and drain the etchant are formed perpendicular to the previous direction, indicated by section B-B in **Figure 2.** The two channels in section B-B are offset relative to each other, which

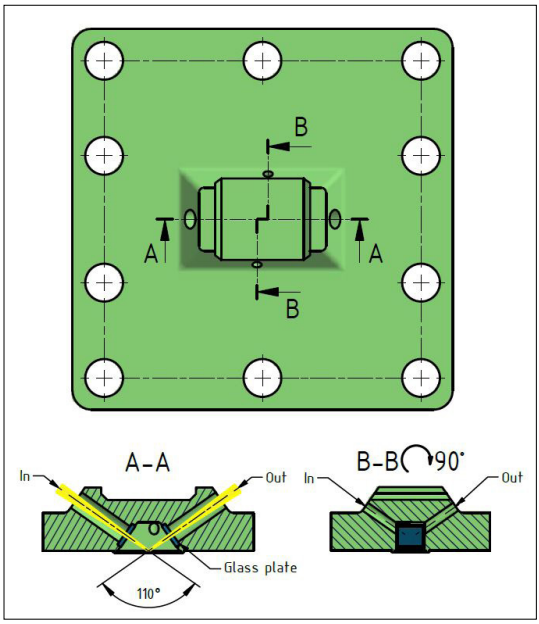

Figure 2. *PDMS cell; Section A-A: the path of the light-beam; Section B-B: the path of the etchant.*

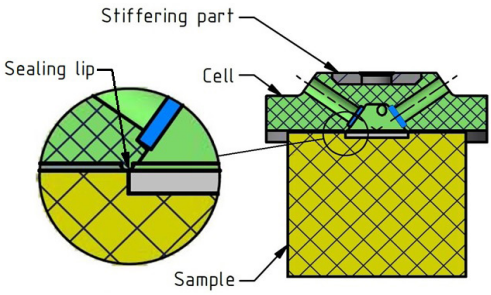

Figure 3. *The sealing lip between the sample and the cell.*

prevents the etchant from stagnating.

The seal between the sample surface and the cell is provided by a 0.3 mm high sealing lip formed in the PDMS. As a result of the spring pressure, the plane of the sample thus enters the point of intersection of the optical axes of the ellipsometer (**Figure 3**).

2.3. Frame structure

The elastic deformation of the cell, and thus the change of the geometrical conditions necessary for the measurement, is prevented by a rigid, welded frame structure made of steel. The precise positioning was ensured by plane grinding the bottom plate after welding, compensating the thermal deformation caused by welding.

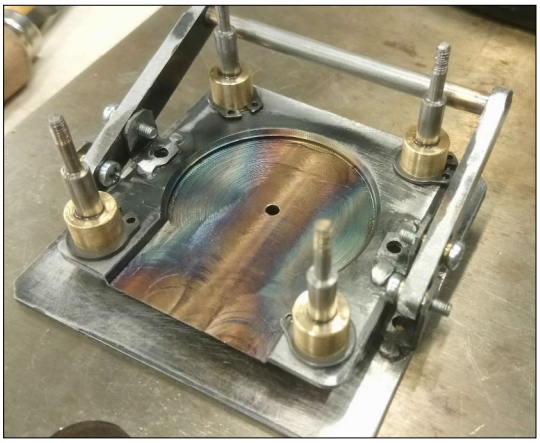

Figure 4. *The sample tray and the lifting mechanism are built into the frame structure.*

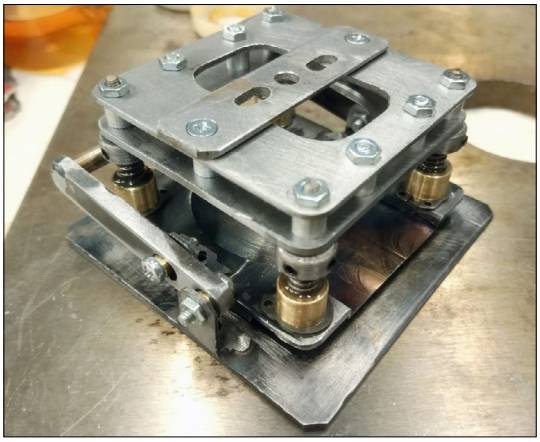

Figure 5. *The assembled device without the PDMS cell.*

The frame ensures the parallelism and precise running of the sample tray with the microfluidic cell. The exchange and positioning of the sample are facilitated by the developed spring mechanism (**Figure 4**). The correct tightness can be adjusted by changing the spring force by prestressing the pairs of springs connected in parallel below and above the sample tray.

The positioning on the surface of the sample is possible by an inspection hole formed in the upper stiffening part. This can be combined even with an optical microscope to increase accuracy. Any arbitrary location can be found on the surface area, so the ellipsometer can precisely target any point on the prepared sample.

Another design consideration was for the frame structure to form a whole assembly unit even without the cell. This allows the combination of the frame with different cell structures as part of a further development process (**Figure 5**).

3. Results

As a result of our work, a prototype of the microfluidics cell was created. After assembling the mechanical parts, the device was adjusted, which meant that the springs ensured the sealing between the sample and the cell. The construction mechanically satisfies the needs arising during the design. The sample tray and the sample can be moved, changed, and positioned without moving the device.

4. Conclusions

Using previous experience of microfluidic cells, a redesigned experimental unit was created to study the etching processes. The new device can be combined with both optical microscopy and spectroscopic ellipsometry. The manufactured frame structure is easy to install, so it can be suitable for inserting new, possibly modified cells. Subsequent, repeated improvements to the construction can provide for metallography, a tool that can open a new chapter in the examination of etching processes.

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References

[1] Renkó J. B., Bonyár A., Szabó P. J.: *Mikrofluidika cella fejlesztése folyadékfázisú vékonyréteg leválasztás nyomonkövetésére*. Acta Materialia Transylvanica, 3/2. (2020) 94–98. https://doi.org/10.33924/amt-2020-02-08

[2] A. Bonyár, P. J. Szabó: *Correlation between the grain orientation dependence of color etching and chemical etching*. Microscopy and Microanalysis, 18/6. (2012) 1389–1392. https://doi.org/10.1017/S1431927612013554

[3] Bonyár A., Renkó J., Kovács D., Szabó P. J.: *Understanding the mechanism of Beraha-I type color etching: Determination of the orientation dependent etch rate, layer refractive index and a method for quantifying the angle between surface normal and the <100>, <111> directions for individual grains*. Materials Characterization, 156. (2019) 109844. https://doi.org/10.1016/j.matchar.2019.109844

[4] Tompkins H. G.: *A User's guide to Ellipsometry*. Academic Press, Inc., Mesa, Arizona, 1993. XIII–XV.

[5] Bonyár A., Sántha H., Ring B., Varga M., Kovács J. G., Harsányi G.: *3D Rapid Prototyping Technology (RPT) as a powerful tool in microfluidic development*. Procedia Engineering, 5. (2010) 291–294. https://doi.org/10.1016/j.proeng.2010.09.105

[6] H. D. Cho, Y. T. Jun, M. Y. Yang: *Five-axis CNC milling for effective machining of sculptured surfaces*. International Journal of Production Research, 31/11. (1993) 2559–2573. https://doi.org/10.1080/00207549308956883

[7] Tompkins H. G. , Hilfiker J. N.: *Spectroscopic ellipsometry: Practical application to thin film characterization.* Momentum Press, LLC, New York, 2016. 31–55.

[8] Fujiwara H.: *Spectroscopic Ellipsometry: Principles and Applications.* John Wiley & Sons Ltd, Chichester, 2007. 81–120.