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Research Article

A NEW METHOD FOR THE MEASUREMENT OF SOFT MATERIAL THICKNESS

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ABSTRACT

Thickness measurement is very critical especially in fabrication of micro and nano devices to determine the thickness of the layers. Stylus measurement is the easiest and most common technique that is being employed among the other thickness measurement methods. Micro-nano fabrication processes requires the usage of both rigid and soft materials. While thickness of a rigid material can be easily detected, thickness measurement of the soft materials presents some difficulties for standard stylus thickness measurement devices. Since the soft materials are deformed by the stylus due to the applied pressure, correct thickness measurement cannot be realized. Here, PDMS (Polydimethylsiloxane) is used as soft material for thickness measurement. By taking the replica of the soft material with liquid plastic which becomes rigid after curing, the depth can be measured easily via conventional stylus thickness measurement devices.

Keywords: Casting, PDMS, Thickness Measurement, Soft Materials, Stylus Thickness Measurement.

1. INTRODUCTION

Soft materials are widely used in research and manufacturing (Marcali *et al.* 2016; Guner *et al.* 2017; Tavakoli *et al.* 2017; Bakan *et al.* 2018). Microfluidics community is one of the biggest users of the soft materials since PDMS has been the primary supply in fabrication of the microchannels after the invention of the soft lithography (Duffy *et al.* 1998) which changed the microfluidics research dramatically. Soft lithography introduced a very useful fabrication way for microchannels. In soft lithography, following the fabrication of the molds from SU8, PDMS is poured on the mold and cured. This process enables the replication of the mold with PDMS and saves the mold providing multiple replications. It removes the requirement of cleanroom fabrication every time for the acquisition of the microchannel.

There are also other fabrication methods for the microchannels which decrease the fabrication cost and complexity providing more access to microfluidics for the research community. These methods include xurography (Bartholomeusz *et al.* 2005; de Santana *et al.* 2013), micromilling (Lopes *et al.* 2015; Singhal *et al.* 2015) and laser machining (Romoli *et al.* 2011; Mohammed *et al.* 2016; Prakash *et al.* 2018; Benton *et al.* 2019). These methods proved themselves in several lab-on-a-chip applications. For example, a micro milled chip is employed in ESR (erythrocyte sedimentation rate) measurement (Isiksacan *et al.* 2017; Isiksacan *et al.* 2018) and laser machined PMMA chip is employed in PT (prothrombin time) measurement (Guler *et al.* 2018). Some of these methods realize microchannel fabrication by machining a bulk PMMA material while some of the fabrication methods require machining of PDMS after the spin coating (Isiksacan *et al.* 2016). Machining of spin coated PDMS endows controlling the channel thickness through the speed of spin coating process.

Soft materials like PDMS is also used for other applications like flexible devices (Kudo *et al.* 2006; Lei *et al.* 2012) and wearable devices (Moon *et al.* 2010; Gao *et al.* 2017). PDMS is also used for coating the surface of other materials for changing the specific properties such as turning a conductive metallic surface into an insulator (Isgor *et al.* 2015). Coating the electrode surface with PDMS to eliminate the conductivity in order to make a total capacitive sensor is a very precise process. Because a thick PDMS may kill the sensor totally or a thin PDMS may not be enough to disable resistive effects, measurement of PDMS thickness is very critical.

Another advantage of the PDMS is that it can be plasma bonded to the glass. Since many kind of clean room fabrication methods can be realized on glass, like the fabrication of microelectrodes (Bilican *et al.* 2016; Guler *et al.* 2018), PDMS becomes more crucial for lab-on-chip (LOC) community. In addition, it is also easy to cut and punch the PDMS that enables easy integration of LOC environmental elements like valves (Guler *et al.* 2017).

Thickness is also important for machining of the PDMS layer aiming channel production (Gitlin *et al.* 2009; Li *et al.* 2012; Isiksacan, Guler, Aydogdu, Bilican and Elbuken 2016) as mentioned before. This method depends on to cut out a thin line from the PDMS layer which is followed by sealing of the layer from both sides. Thus, the layer thickness becomes equivalent to the

channel height and the width of the line becomes equivalent to channel width. Examples can be verified addressing the significance of the thickness measurement of soft materials like PDMS.

We mention PDMS so much due to our microfluidics background where we encountered the problem of measuring the thickness of spin coated PDMS. Therefore, in this study, PDMS thickness is measured as an example of soft material which is one of the most basic material of the research in several fields thanks to its transparency (Isgor, Marcali, Keser and Elbuken 2015; Marcali and Elbuken 2016; Serhatlioglu *et al.* 2019) and biocompatibility (Sun *et al.* 2019). However, PDMS deforms under the pressure of the stylus which make it impossible to realize a correct measurement with devices employing the stylus method. While its flexibility has some benefits in fabrication (Guler *et al.* 2015) and in application (Kudo, Sawada, Kazawa, Yoshida, Iwasaki and Mitsubayashi 2006; Moon, Baek, Choi, Lee, Kim and Lee 2010; Lei, Lee and Lee 2012; Zhu *et al.* 2014; Gao, Ota, Schaler, Chen, Zhao, Gao, Fahad, Leng, Zheng and Xiong 2017), it also puts a challenge in measuring the thickness.

There are other methods for thickness measurement such as optical profilometer and SEM but they are very expensive devices limiting the accessibility for numerous researchers. There are also some other and less known alternatives to SEM and optical profilometer for measuring the thickness of soft tissues which are laser displacement sensors (Lee *et al.* 1988) and Vernier capillaries (Delgadillo *et al.* 2010). Unfortunately, these measurement setups are complex and expensive limiting their accessibility for standard labs.

In this study, a new methodology is developed for the thickness measurement of the soft materials. Here the method is tested on PDMS yet it is also applicable for other materials. For measurement of the thickness, first, a little part of the spin coated PDMS is cut with a razor blade. This part might be in shape of a line, a square or anything else. Afterward, liquid plastic is poured on the PDMS following the removal of the cut piece. After the hardening of the liquid plastic, it becomes a rigid material. The hard plastic enables the measurement of the thickness as it is a non-deformable material to stylus pressure. Since it is the negative replica of the PDMS, the difference of the two points, where one point is taken from the removed part and the other point is taken from the anywhere else on the surface, gives the thickness of the PDMS. A representative drawing for soft and hard material thickness measurement with stylus is shown in Fig. 1.

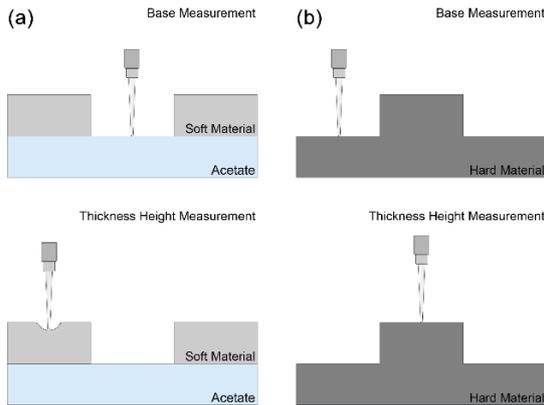


Fig. 1. Measurement of the thickness with stylus from a) soft and b) hard material

2. MATERIALS AND METHODS

PDMS is prepared by mixing 1/10 w/w ratio with its curing agents. For providing better mixing, the two additives are harshly mixed. The mixture quality can be evaluated by the naked eye. When the bubbles are all over the mixture, it means a good mixing quality. Hence the two additives are mixed until the bubbles cover all over the mixture. Then the mixture is put under the vacuum for degassing which removes the air bubbles. This process takes nearly 45 minutes to make the mixture totally bubble free. Hence the PDMS becomes ready for spin coating. Since the PDMS by itself is very viscous, it is not possible to have a thickness less than 10 μm by spin coating. Yet there is a little trick to thin the PDMS adding some toluene into the PDMS which reduce the viscosity and enables thickness down to 1 μm by spin coating. However, this is out of scope of this study.

Glass slides used in spin coating are first covered with an acetate sheath via a double sided tape. After putting the glass slide in the spin coating chamber, the PDMS is poured over it from a cup directly and the spinning process is started. The spinning is done at different rates from 1000 rpm to 5000 rpm for 2 minutes. The spin coated slides are left on a hot plate at 100 $^{\circ}\text{C}$ for 2 hours long. The heat treatment provides curing of the PDMS. Then a rectangular piece is cut with a razor blade from the PDMS layer. Plasma treatment is applied to a previously produced PDMS slab and the PDMS layer. The plasma enables the chemical bonding of PDMS. Thus, spin coated PDMS layer is bond to PDMS slab and it is easily peeled off from the acetate when the slab is removed.

Liquid plastic (Smooth-On, Smooth-Cast), which consists of two parts, is prepared at the same volume ratios. Both the PDMS slab and the liquid plastic solutions are put in the vacuum chamber for 15 minutes long. Due to its porous structure (Zhou *et al.* 2007), cured PDMS has gas molecules trapped inside. During the casting process, some of the trapped gas molecules escape which causes bubble formation on the interface between the liquid plastic and PDMS. Hence, it is a must to degas PDMS before the casting. After taking the PDMS and the liquid plastic from the vacuum chamber, the two parts of smooth on are mixed. It is very critical to pour the two parts into the same cup simultaneously. Afterward, the solution is mixed slowly. It needs to be done very gently avoiding bubble formation in the Smooth-On mixture.

PDMS is put in a container and the liquid plastic is poured over slowly. Pouring needs to be very gentle and slow to prevent bubble formation on the PDMS-liquid plastic interface. Waiting overnight ensures the hardening of the plastic however a few hours like 2-3 h is also enough. Hence PDMS can be separated from the hard plastic. Thickness measurements can be done on the hard plastic. As it is a negative replica of the PDMS, it would give the correct thickness for the mother mold. A representation of our method is shown in Fig. 2.

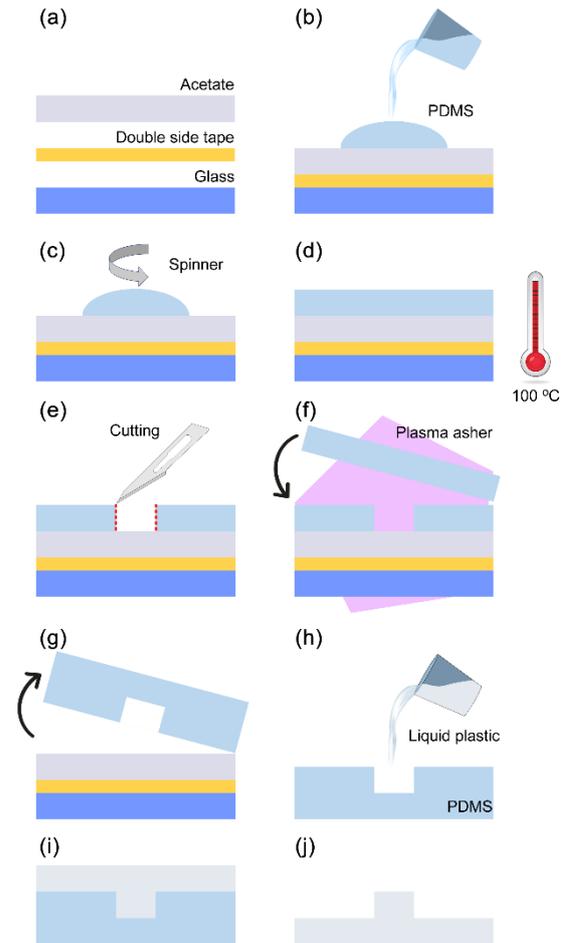


Fig. 2. Liquid plastic casting. a) An acetate sheet is stuck to glass slide with double sided tape b) PDMS is poured c) PDMS is spin coated d) It is put in hot plate at 100 $^{\circ}\text{C}$ e) a little piece is cut out with a razor blade f) Plasma treatment is applied to PDMS slab and spin coated PDMS. g) PDMS slab and spin coated PDMS is peeled off from the surface following the plasma bonding h) liquid plastic is cast-over the PDMS i) after 3 hours of waiting hardened plastic is taken from the PDMS.

Thickness measurements are done with a precision stylus digital micrometer (Mitutoyo). The device realizes the measurement by pushing down on the substrate with the stylus. Displacement of the stylus is detected giving the thickness result. The measurement is done as follows: First, the stylus is put on the base area and the number on the screen is set to zero by pushing the yellow button shown in Fig. 3. And then the stylus is moved upward by pushing the metal arm over the digital head placed at the

right side of the head. The substrate is moved and the stylus is put on the protrusion or the intrusion on the sample. The thickness corresponds to the number that is shown on the screen of the device.



Fig. 3. Picture of thickness measurement device.

Smooth-On consists of two parts which are written on the yellow and blue bottle as part A and part B. These are yellowish and whitish liquids and they can be kept in their own air proof bottles for years without any decay according to our experience. Before dispensing the liquids, the two bottles should be mixed harshly. Then, two parts are put into different cups at the same volume. The precision of the naked eye is sufficient in preparation of liquids at the same volume. Two cups need to be degassed at the vacuum chamber at least for 15 mins. Degassing remove all the bubbles inside the liquids. Then, the two parts are poured in another cup simultaneously and gently. Afterwards, the mixture is stirred slowly and gently to make sure that the two parts are mixed well. Here, the last two steps are critical in terms of avoiding bubble formation, if bubbles form at these steps, it is not possible to get rid of them and the bubble shapes remain in the mold after the hardening of the plastic as well. Degassing the liquids after mixing is not possible since a chemical reaction between each part towards hardening is already started off after the first contact of each liquid. After hardening, the plastic takes the shape of the material to which it is in contact when it was in liquid form. For better seeing, a demonstration example is done in a plastic cup as shown in Fig. 4.

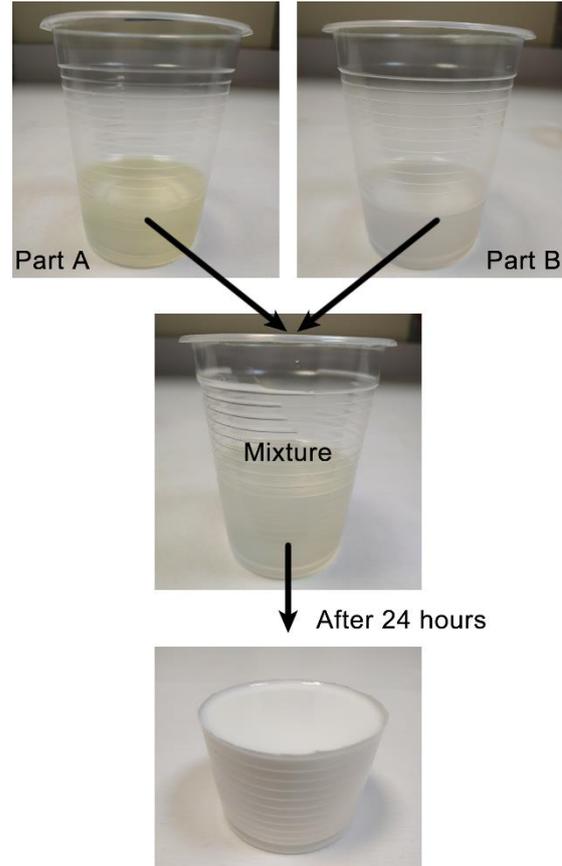


Fig. 4. Preparation of Smooth-On: first Part A and Part B is taken to different cups, after degassing, the two parts are mixed gently in the same cup at the same time. After waiting a few hours, the liquid plastic hardens taking the shape of whatever it is in.

3. RESULTS AND DISCUSSION

Measurements are carried out with three different tips of the thickness measurement device. Pink points in Fig. 5 shows the results of those tips for spin coated PDMS layers. According to the results, tip 3 gives the least while tip 2 and tip 3 nearly give the same height for PDMS in soft material measurement experiments.

After casting with smooth on from the PDMS slab, the same measurement is realized for the hard plastic. The thickness measurement results are shown in Fig. 5 with the blue points for the hard plastic. The tip type does not make any difference for the measurements which are done from the hard plastic.

Here, the same thicknesses are measured from the soft PDMS and the hard smooth on which gives totally different results. When the tip pushes to the PDMS, deformation takes place at the material and it gives less height than normally the PDMS layer has. However, the same fact does not arise for the hard material since the tip cannot cause deformation at the hard material. It proves that the soft materials are not suitable for stylus type thickness measurements due to deformation.

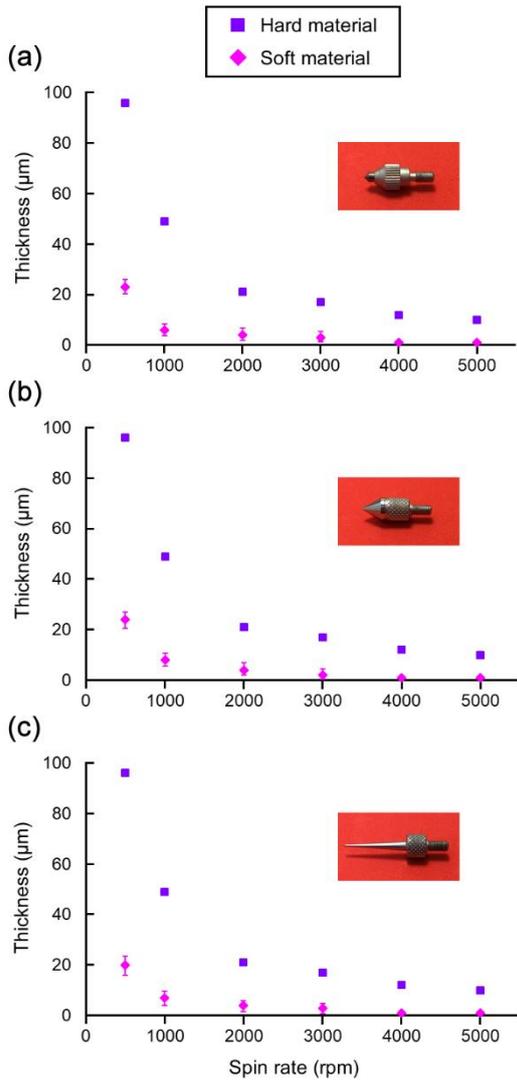


Fig. 5. Result of thickness measurement from PDMS and cast hard plastic for 3 different stylus types. Measurements are done directly from the spin coated PDMS layer at several rpm's and the hard plastic which was cast on the same PDMS layers.

To make sure that our results are correct, PDMS pieces that are cut from the original spin coated layer, as shown in Fig 2e, are sent to the SEM for inspection. The SEM images are shown in Fig. 6. The SEM images prove that the measurements which are done from the smooth on are accurate.

We accept the SEM results as the most valid since it shows every detail without any unclarity. It shows that the 500 rpm spin coated PDMS has 96.8 µm thickness which is the same with the result achieved by the stylus thickness measurement over the hard plastic. However stylus device gives nearly 21 µm thickness on the PDMS which is too different than the correct thickness. In 1000 rpm, PDMS has 49.8 µm thickness according to the SEM photo. Stylus thickness measurement gives 50 µm thickness over the hard plastic which is consistent with SEM image. However, stylus thickness measurement gives nearly 5 µm thickness when the measurement is

realized over the PDMS directly. The trend goes on the same overall PDMS samples. When the stylus measurement is done over hard plastic, the results are consistent with SEM. When the stylus measurement is done from PDMS directly, it is far away to be consistent with the SEM. Besides, tip type does not make any difference when the measurement is realized from the hard plastic while it affects the results when it is done over the PDMS directly. Since PDMS is a soft material, sharper tips cause more deformation than the flat tips. In addition to giving wrong results at stylus measurement, soft material thickness measurement is also dependent on stylus type. Hence stylus measurement loses its reliability for soft materials.

Our method removes the disadvantages of the stylus measurement method for soft material. By casting the soft material with liquid plastic, it can be measured with a stylus after hardening. Since the liquid plastic hardens taking the shape of the PDMS, stylus cannot make any deformation by pushing the material. In addition, it totally takes the shape of the PDMS without any significant shrinkage enabling correct thickness. We prove our thickness results coming from the hard plastic by comparing with SEM images of the PDMS.

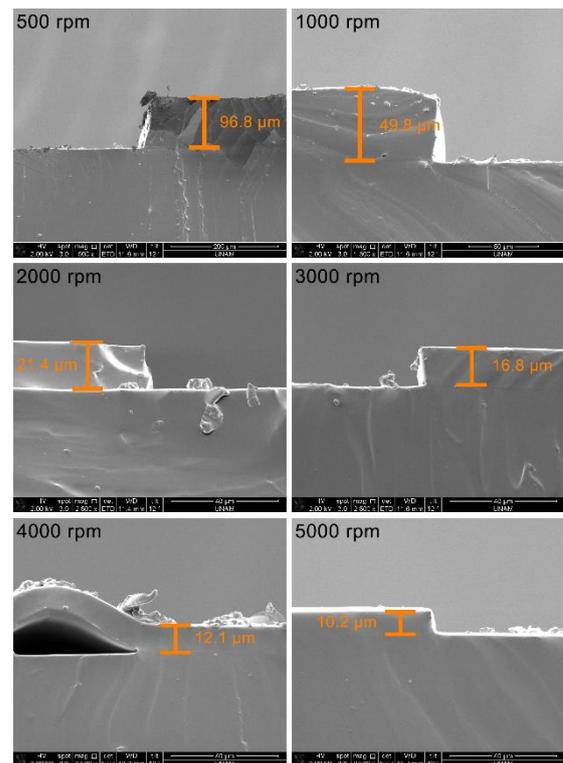


Fig 6. SEM pictures of PDMS pieces which are cut from the original spin coated PDMS layer with razor blade. Spin coating rpm is written on the upside of the left corner of every photo.

For clarity, we cut and inspected the hard plastic with SEM. The SEM picture of the smooth on cast over the PDMS is shown in Fig. 7 from the top view and side view. Here the hard plastic is a negative copy of PDMS. Thus, the protrusion and intrusion at the PDMS surface are found at trans located positions in smooth on. Hence the stylus is placed over the base area and the monitor is set

to zero and then the stylus is placed over the protrusion to measure the thickness. Since the material is hard, stylus cannot make any deformation so the device gives the correct thickness result.

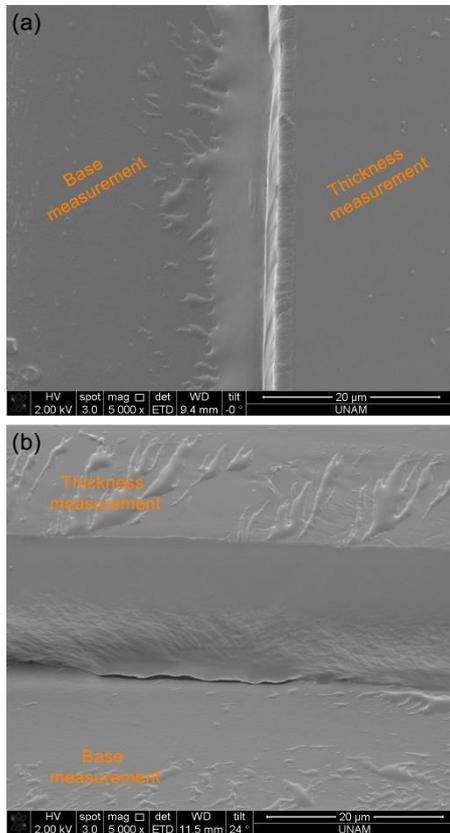


Fig. 7. SEM picture of smooth on casted over the PDMS
a) Top view of the hard plastic b) side view of the hard plastic.

5. CONCLUSION

We show that stylus thickness measurement is not a suitable way for soft materials with serial experiments. Those experiments prove that, when the stylus pushes down on the soft material, a deformation takes place disabling accurate measurement of the thickness. Here, PDMS is chosen to carry out the experiments as soft material. A new method is offered to measure the thickness of soft material overcoming the deformation problem. In this new method, first, the soft material is cast by liquid plastic. After a few hours, the liquid plastic hardens and takes the shape of the soft material. Hence stylus can push the surface of the hard plastic without any deformation enabling correct measurement of the thickness. To make sure that our results are correct, PDMS pieces, which are cut from the original spin coated layer, are investigated with SEM microscopy. SEM images show that the thickness measured from the hard plastic is the same with the real thickness of PDMS.

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