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Thickness Measurement of Photoresist Thin Films Using Interferometry

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1. Introduction

Photoresists are polymers sensitive to light, usually in the ultraviolet (UV) range of the electromagnetic radiation spectrum. They are classified into positive and negative. In positive types, the exposed to light region of the photoresist becomes solvable to a developer, while the unexposed one remains unsolvable. In negative types the opposite happens. Photoresists are widely used in micro and nano fabrication such as photo and e-beam lithography. In these techniques the substrate, usually a silicon (Si), wafer, is spin - coated with a thin film of photoresist and then is selectively exposed to the proper radiation. The substrate is then developed with a suitable chemical solution which etches the exposed (positive) or unexposed (negative) regions, resulting in transferring the desired pattern on it. For these, knowledge of the photoresist exact thickness is crucial for the calculation of the necessary radiation energy dose.

Traditionally in photolithography practice, the thickness of photoresist films is deduced indirectly by the volume of the resin dispensed during spin coating, divided by the wafer surface. However, this method is subject to errors due to uncertainty of the solvent evaporation and thickness distribution nonuniformities, e.g. near the wafer edges due to wetting effects etc. Recently, the measurement of liquid crystal (LC) films thickness was achieved using a relative simple processing of common interferometer images (optical profilometer), considering the phase shift of light as propagated through LCs. The technique was tested and verified with atomic force microscopy (AFM) in micron and sub micron film thickness, and was found to be in good agreement to the AFM measurements [1].

This work proposes an extension of this technique to thin films made from photoresists. Thin films of various thicknesses have been fabricated, measured with the proposed method and verified with AFM technique. The results further confirmed the validity of the proposed technique, as the difference of the two methods was within the statistical error bounds. Further testing has been also performed in standard photolithography processes to investigate if the proposed technique affects the performance of photoresists. It was found that the influence in photoresist performance is negligible as they are sensitive only to the i-line of the spectrum from a mercury vapour lamp used for photolithography. The interferometer used here was operated with a low intensity green (550 nm wavelength) laser source.

2. Optical profilometry theory and thickness measurement methodology

This section starts with an overview of how light interference is implemented into a single-wavelength optical profilometer and it is used for the extraction of three dimensional (3D) topography images of substrates. Then it analytically describes the methodology of using the phenomenon of phase shift that appears in light propagated through transparent films, together with optical profilometry to extract the thickness of the film. Finally an overview of fringe order theory is given, which is useful in the determination of the maximum possible height the instrument is capable to measure.

2.1 Existing knowledge

In general, optical profilometry exploits the interference between a reference light beam and a measuring beam reflected from the sample surface in order to extract a 3D topographical image. If the travelled distances of the two beams differ by an even number of half-wavelengths then a bright fringe (constructive interference) is created, whilst a difference equal to an odd number of half-wavelengths creates a dark one (destructive interference). In intermediate cases, gray lines are created. The height of a particular point on the sample surface is determined by the type of interference, which in the resulting image of the surface is captured by the colour of the corresponding pixel. In this mode, the number of fringes defines the maximum possible measured height. It is generally recommended to use less than three fringes (which correspond to a height difference equal to three times the source wavelength), in order to avoid problems with the coherence length of the laser source. However, with some care slightly larger numbers of fringes can also be used.

Measurements of thicknesses and refractive indices of transparent thick polymer films based on light interference phenomena are well known and developed. They are based on the fact that fringes from the upper and lower boundaries of the film are well separated, and hence the film thickness or the refractive index can be calculated from the distance between the envelopes of the two fringe patterns. However, as the film thickness decreases, the envelopes of the two fringes come closer to each other, and for thicknesses less than about 1μm they overlap. This complicates significantly the calculation of the distance between fringes, and thus the film thickness. Recently, the helical conjugate field function (HCF) has been utilized in coherence correlation interferometry to circumvent this limitation [8, 9,10].

In many areas of engineering and physics, the above technique has been incorporated in methodologies of measuring physical properties and their response to the presence of external stimuli. In the field of mechanical engineering, this constituted the underlying principle in holographic interferometry and Moire inteferometry. The former concerns measuring the properties of transparent specimens [3], whereas the latter is used to measure small deformations of non-transparent solids due to external forces and thermal expansions, as for example is described in [4, 5]. Recently, a methodology which uses two sample probes for simultaneously measuring the refractive index and thickness of transparent specimens has been developed and demonstrated in [2]. Interferometry has also been used to measure parameters of transparent films made from nematic liquid crystals, such as birefringence, dielectric anisotropy, refractive index change in response to applied DC voltage [6] and thickness [1]. In this article, the methodology of [1] is extended on other transparent materials that are mainly used as photoresists in micro-fabrication.

2.2 Initial imaging of the borders of a transparent film

Generally, in optical profilometry (or interference profilometry) the measurement starts with the operator focusing to the point where the fringe pattern occurs. Then this pattern is subjected to appropriate processing by the software of the instrument in order to extract the 3D topographical image of the sample. In the case of a transparent film with thicknesses bigger than about one micron, two fringe patterns are generated (figure 1), from exactly the same area of the sample. The first is taken by focusing on the upper surface of the transparent film and the second by focusing on the substrate. Furthermore, for thicknesses less than a micron there is only one fringe pattern which may be attributed to the substrate, as its reflectivity is significantly higher than that of the upper boundary of the film.

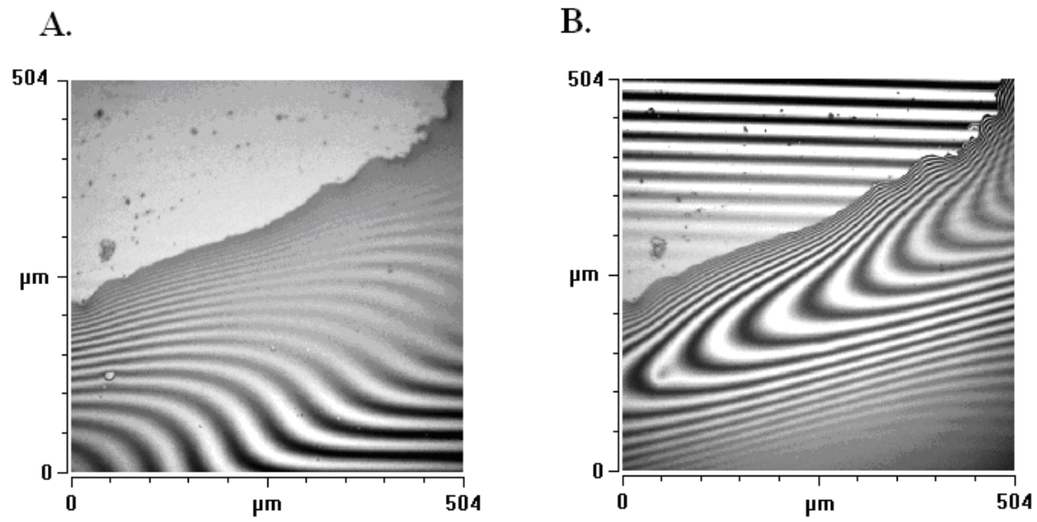


Fig. 1. A) First fringe pattern generated from the upper surface of the transparent film. B) fringe pattern generated from the substrate. The right image is distorted in the region of film because of the phase shift that light undergoes while it travels through the film.

In the case where the first fringe pattern is used, the topology of the upper boundary of the film is taken. However in the case where the second pattern is used, an image which corresponds to the substrate is obtained. Here the region that is coated by the transparent film appears to be at a lower height than the non-coated substrate. Such an image is shown in figure 8 in section 6. Definitely, this does not reflect the reality. This happens because to the instrument the existence of the transparent film is unaccounted for, and hence the phase difference registered by the instrument is taken to be due to height differences and not due to the delay of light propagation in the film. A method that considers the optical path changes in the film in order to correct the initially measured image follows.

2.3 Understanding the initial image

Here, the principle of operation of the instrument and the optical path of its light source are considered for the case of measuring the boundary of a transparent film. Figure 2 shows a

schematic presentation of an optical profilometer, operated both on a flat substrate and a transparent film. In both cases, monochromatic light from the light source impinges on the beam splitter where the beam splits into two halves. One half of the beam travels to the objective lens and then to the second beam splitter. At this point, the light beam splits into two identical beams. The one beam travels upwards to the reference mirror and reflected back towards the second beam splitter (optical path A′B′C′ in figure 2). The other travels downwards and reflected from the sample surface also back to the second beam splitter (optical path A′D′C′). The two beams interfere at point C′ and the interference pattern is captured by the camera. Further processing of the fringes pattern image from the instrument software, defines the type of interference in each pixel according to its intensity, and therefore its height. Now for the typical case of the non coated sample, the optical path difference of the two beams is given by A′B′C′ - A′D′C′. Since the distance A′B′C′ and the type of interferences are known, the distance A′C′D′ and therefore the height of point D′ can be extracted as follows:

$$Z_{D'} = 0.5(A'B'C' - A'D'C') \tag{1}$$

In the second case, where the substrate is coated by a transparent film, of refractive index n, the optical path difference of the two beams becomes ABC-(AD +nDEF +FC). In this situation the problem with the instrument is that it takes the delay of light travelling in the transparent film as a larger distance travelled in the air. However, the delay of light is due to the presence of a transparent film with a refractive index higher than that of air. As a result, in the final topographical image the region of the substrate that is coated with the thin transparent film, erroneously appears to be at a lower position than the non coated substrate. The height of point E in the initial image is calculated as:

$$Z_E = 0.5(ABC - (AD + nDEF + FC)) \tag{2}$$

Subtracting the two heights and taking that A′B′C′ = ABC and for the case of a non tilted substrate A′D′C′ = AEC, then:

$$Z_E - Z_{D'} = (n - 1)DE \tag{3}$$

2.4 Correcting the initial image

In subsection 2.3, the optical paths of instrument light during the measurement of both substrate and transparent film were explained and computed. In what follows, these optical paths are considered in a methodology that corrects the images presented in section 2.2 and thereby extracts the thickness of transparent films. This methodology can be used for instruments that use single wavelength optical (interference) profilometry.

The goal of this methodology is the correction of the z height values of pixels that correspond to the substrate area that is coated by a transparent film. Figure 3 depicts schematically the correction step. Figure 3 (A) represents the situation after the measurement but before the correction where the mean height of the film erroneously appears lower than the average height of non coated substrate, $Z_{\text{substrate average}}$. The correction equation 4 is obtained from the application of equation 3 to the general case of figure 3:

$$Z_{\text{film corrected}} = (n - 1)^{-1}(Z_{\text{substrate average}} - Z_{\text{film}}) \tag{4}$$

where $Z_{film\ corrected}$ is the actual height, z, value of the transparent film. This correction is implemented at the pixels corresponding to the film region. The correction step is depicted in figure 3 (B).

The benefit of this technique is that it does not need any instrumentation modification, but only a relatively simple image processing. Furthermore, only the lower fringe pattern is required. This overcomes the common interferometer issue where for very thin films only one fringe pattern appears, which is taken to be that of the lower boundary (substrate). The intensity of the fringe pattern due to the upper boundary (transparent film) is much weaker.

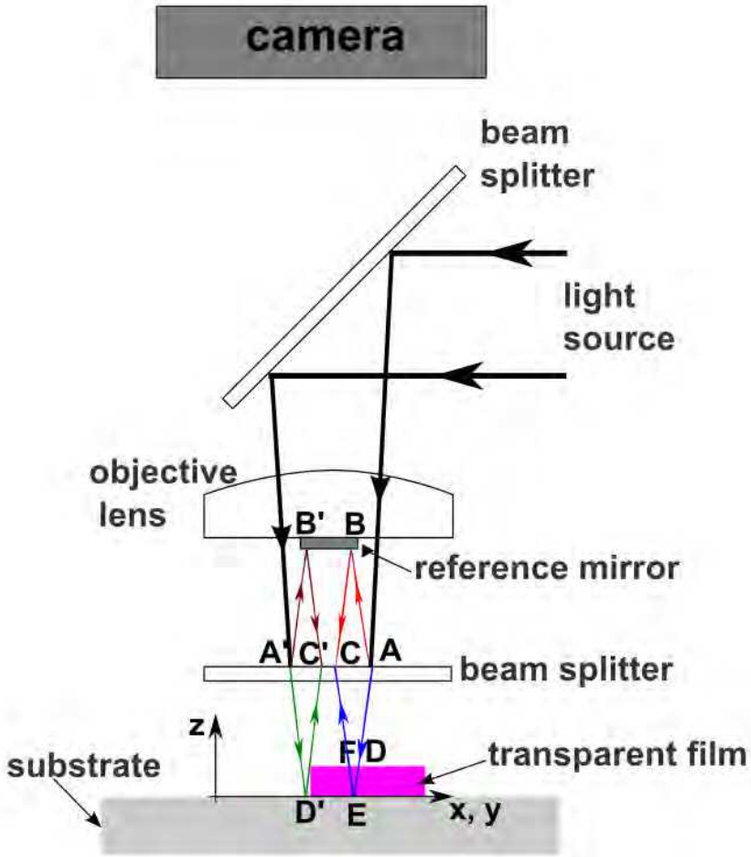


Fig. 2. Schematic representation of the principle of operation of the optical profilometry for both non coated substrate and coated with thin transparent film substrate.

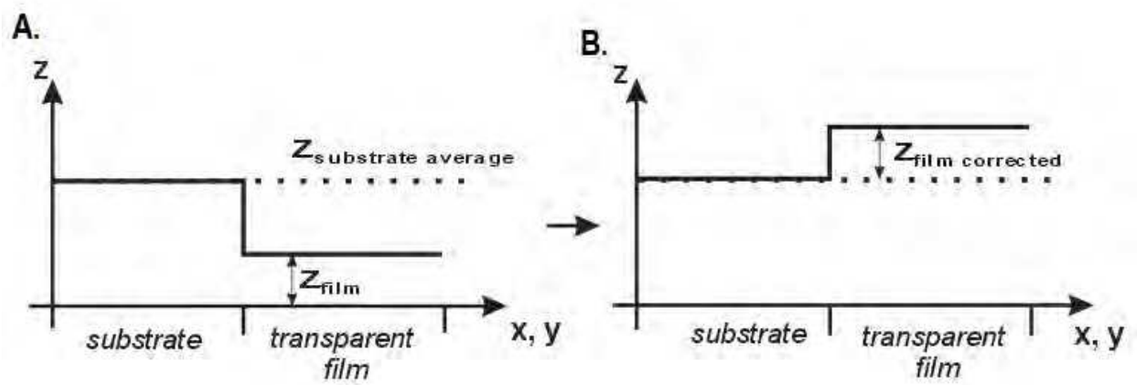


Fig. 3. 2D schematic presentation of (A) the initial image and of (B) the corrected image.

2.5 Fringe order

Let's consider figure 2, where for example the distances between the beam splitter and the reference mirror are taken as equal to the distance between the beam splitter and a sample point, while the profilometer is in focus range. In such a case, the optical path difference is equal to zero and constructive interference occurs, and therefore it is the zeroth order bright fringe. The zero order bright fringe appears with maximum intensity, compared to other fringes. Now consider an increase in the optical path equal to the wavelength, λ, due to sample moving a corresponding distance away from the beam splitter. Now, the first order bright fringe appears with lower brightness than the zeroth order fringe. The same will happen with the second, third and higher order fringes until moving out of the coherence length of the microscope where the intensity will drop to zero. A schematic curve that shows this variation in light intensity, I, with respect to the distance between the sample and lens, dx, is shown in figure 4. The optimum condition occurs when the instrument is focussed as near as possible to the zeroth order bright fringe. However as it is discussed in section 5, the confusion in fringe order for non smooth samples may lead to wrong measurements.

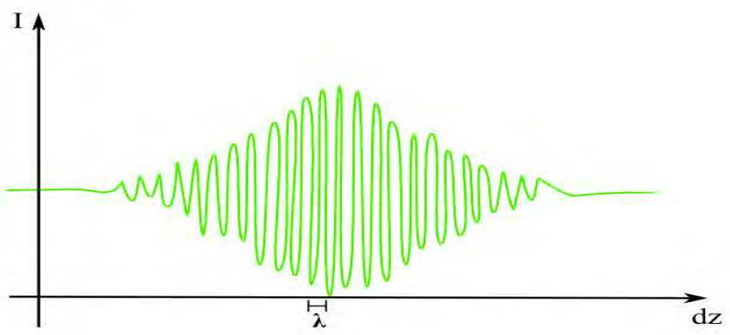


Fig. 4. Schematic representative of light intensity (I) with respect the distance between the sample and lens.

3. Experimental preparation

The optical profilometer used in thickness measurements was an Ambios Xi-100 with monochromatic light source. The light source was a green laser with wavelength equal to *550nm*. The atomic force microscope (AFM) used for the verification of film thickness measurements was a Quesant Qscope 350, operated in contact mode, whose the cantilever stiffness was *50N/m* and *10nm* tip diameter. The influence of the thickness measurement to photolithography performance was tested using a Suss MicroTech MA6 mask aligner. The spin coating machine (has been also supplied from Suss MicroTech). The negative AZ® nLOF 2070 supplied by MicroChemicals was used as photoresist. The value for the photoresist refractive index was taken from the supplier and was *n=1,64*. The photoresists were spin coated at 4000 rpms for 30sec. For the formation of thinner films the photoresist was thinned using AZ® EBR solvent. AZ® 826 MIF was finally used as a developer. The exact photolithography process started with soft bake at 100°C in a hot plate for 5min, exposure 15sec, post-exposure bake 115°C for 2sec and develop for 45sec.

4. Height difference limit

This section, based on the transparent film boundary and geometry, clarifies the rationale for selecting the correct operation mode and identifies the limitations associated with height measurements. This is very important step for correct measurement of thicknesses in general. Consider the maximum height difference that an optical profilometer may accurately measure in the *z* range. In the case where only one interference pattern is used for the extraction of the sample topography, the maximum *z* range that may be measured is different for the various cases described below:

1. Surfaces with high aspect ratio: Consider surfaces where the height of features increases (or decreases) abruptly, i.e. regions that are separated with only very few microns along any horizontal direction and their height difference is higher than half the wavelength. In such a case, the instrument confuses the fringe order and determines erroneous values for heights. The example in figure 5, represents a graded by strips silicon wafer where the edges of the strips are very sharp. The strips were fabricated on the surface by etching. The height of the graded steps is 1.2 microns and the sample was measured with an optical monochromatic profilometer of a light source generating light of 550 nm wavelength (figure 6). Therefore, by neglecting the sample tilt, the height difference at the edges of the strips is equal to about 4.18 times λ/2. The problem here is that the instrument can not determine if the optical path difference is 0.18 or 4.18 times λ/2, i.e. it is not possible to determine the fringe order with this mode. The instrument is programmed to consider smooth surfaces where all fringes are of the same order and therefore the step is taken as about 0.18 times λ/2. That is why the gradation in topographical images appears with wrong heights. As a result this mode is not suitable for surfaces with high aspect ratio.
2. Smooth surfaces: In this case, the features on the surface are separated by relative small inclinations. Therefore, the fringe order is always the same and the height of all features can be accurately measured. Here the *z* range may extend from few nanometers to several tens of micrometers without losing any precision. In figure 7, an example of measuring height differences of several microns on a smooth spherical sample is shown. The apex and base of a spherical surface are separated by a relatively large distance; however, the order of all fringes remains the same and this is reflected on the correct value of their intermediary distance.

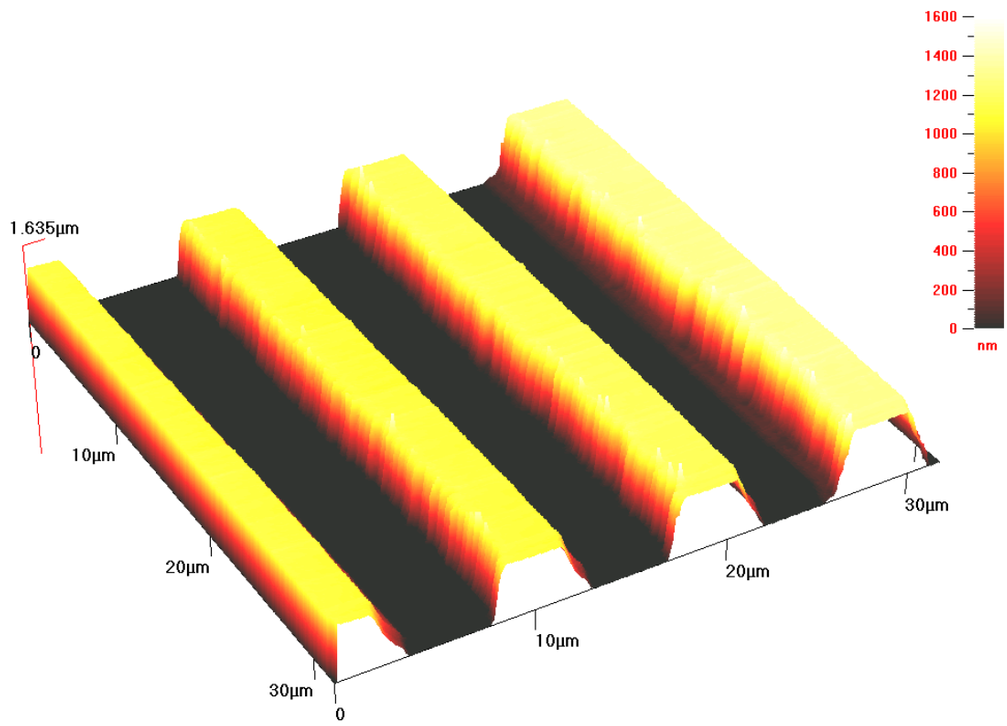


Fig. 5. 3D topography image of the sample with graded strips taken by atomic force microscopy. The steps are formed by sharp edges of 1.2µm height

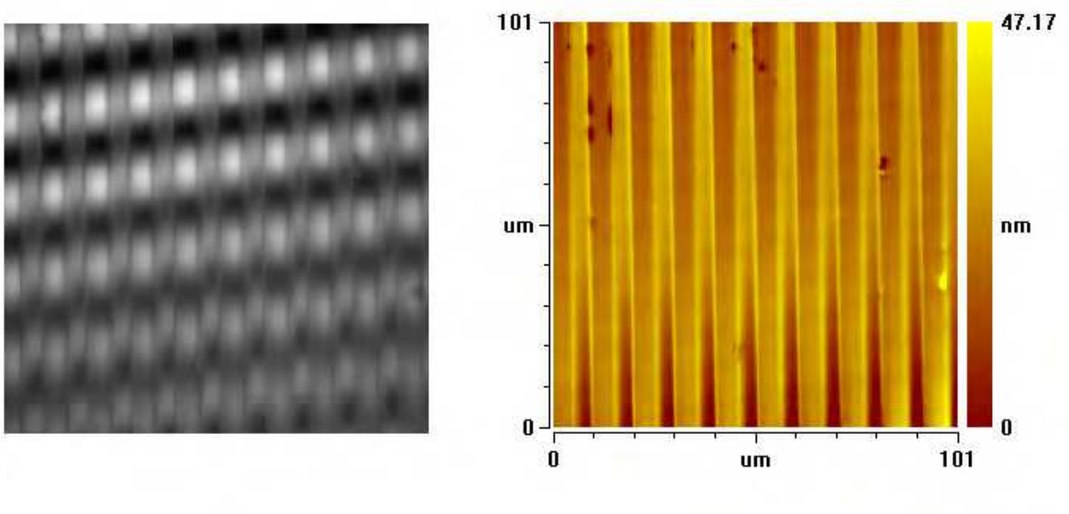


Fig. 6. Optical profilometer fringe pattern and topography images of the sample of figure 5. It is clear that the height dimensions are wrong because of the confusion of fringe order.

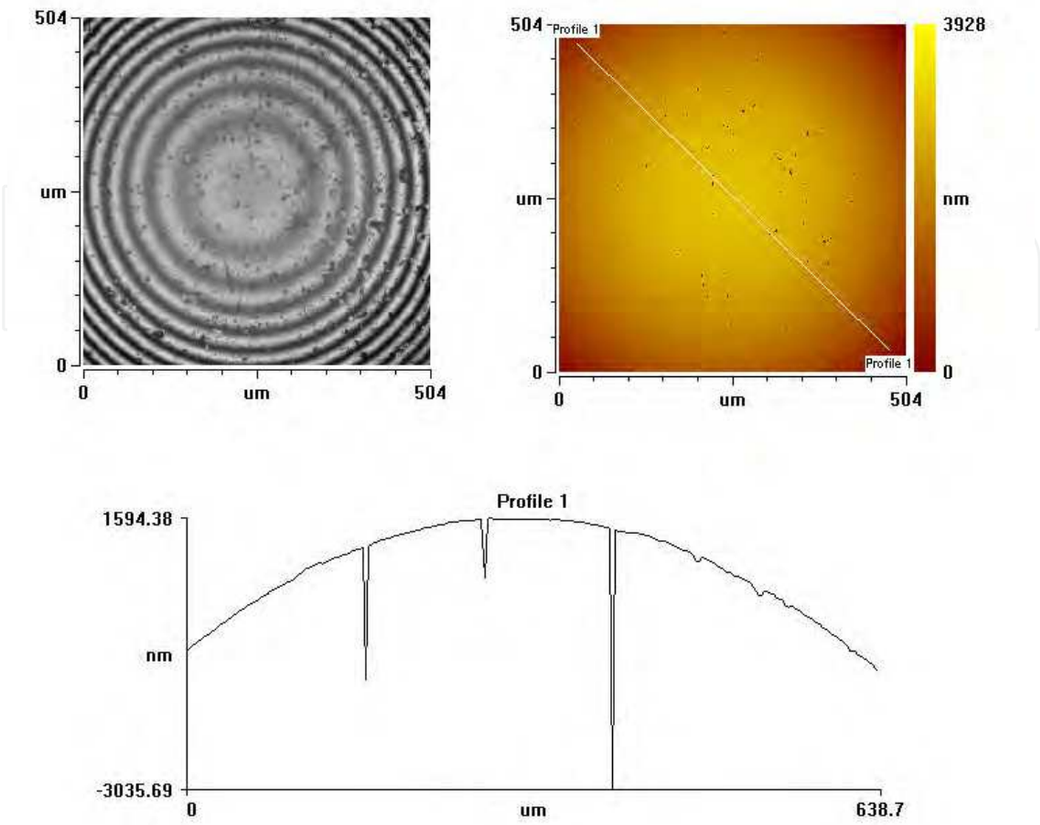


Fig. 7. The fringe pattern and the extracted topography image and profile of a smooth sphere with optical profilometry (diameter D= 10mm).

Finally, it is worth mentioning that further to the mode where only one fringe pattern is used, there is another mode usually called scanning mode. In this mode, a piezoelectric transducer accurately moves the beam splitter while the camera monitors the change in each pixel colour with respect to the travelled distance of the beam splitter. In this way, the instrument identifies the position of the zeroth order fringe (maximum intensity) for each pixel. The height of each pixel is defined by the position of the zeroth order fringe. The scanning mode z range is much higher and may be used up to several hundreds of microns.

The general conclusion of this section is that the technique for transparent thin film thickness measurement presented is applicable only in the smooth mode. Therefore, the technique can be applied only to films with thickness below $\lambda/2$, or to films of which the boundaries are smooth enough to avoid jumps on fringe order.

5. Method implementation to photoresist films

This section implements the technique described in section 2 by measuring the thickness of a photoresist thin film. It follows a detailed description of the steps required to implement this technique.

It starts by imaging a region at the boundaries of the transparent film of which the thickness has to be measured. In this step it is important to choose a region which: (a) is smooth enough to overcome the height limitation mentioned in section 5 and (b) includes both the substrate and the flat (thickness height equilibrium) area of the film. In this step, an image such as the one given in figure 8 is obtained. As it can be seen, the film appears to be lower than the substrate, as discussed in section 2.2. In order to circumvent this, the implementation of the height correction technique described in section 2.3 is applied.

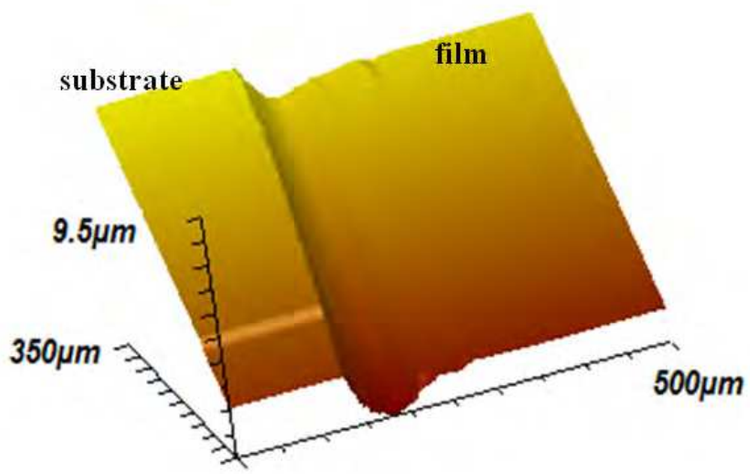


Fig. 8. Initial image taken by the optical profilometer at the boundaries of a photoresist thin film. Both the film and the substrate are also represented.

The second step includes the extraction of the profilometer image to a suitable format (.txt) for Matlab® processing. This removes the tilt that always appears in such images, due to the relative small (but significant for nano-scale measurements) natural tilt of the instrument base, or sometimes the sample apex with respect to the base surface. Tilt removal is performed with a code that fits image lines that are transverse to film boundaries with straight lines, but only to the region of the substrate. This fitting is demonstrated for a single profile in figure 9. The line extracted from this fitting is then subtracted from the corresponding image profile.

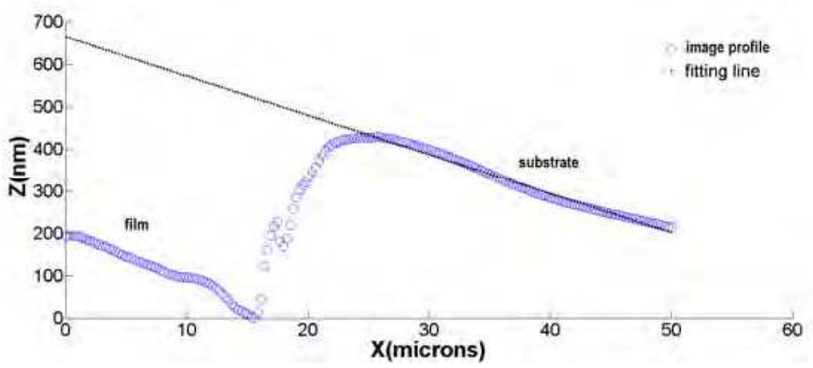


Fig. 9. Schematic representative of image fitting for tilt removal.

In the third step the boundaries of the transparent film are identified in the profilometer image using a code which scans across image lines that transverse the film boundaries and identifies where the tilt between three pixels of the image is higher than a threshold value. This value varies according to each image, and usually several values have to be tested to extract the optimal one. The image of figure 8 after tilt removal and film boundary definition are presented in figure 10.

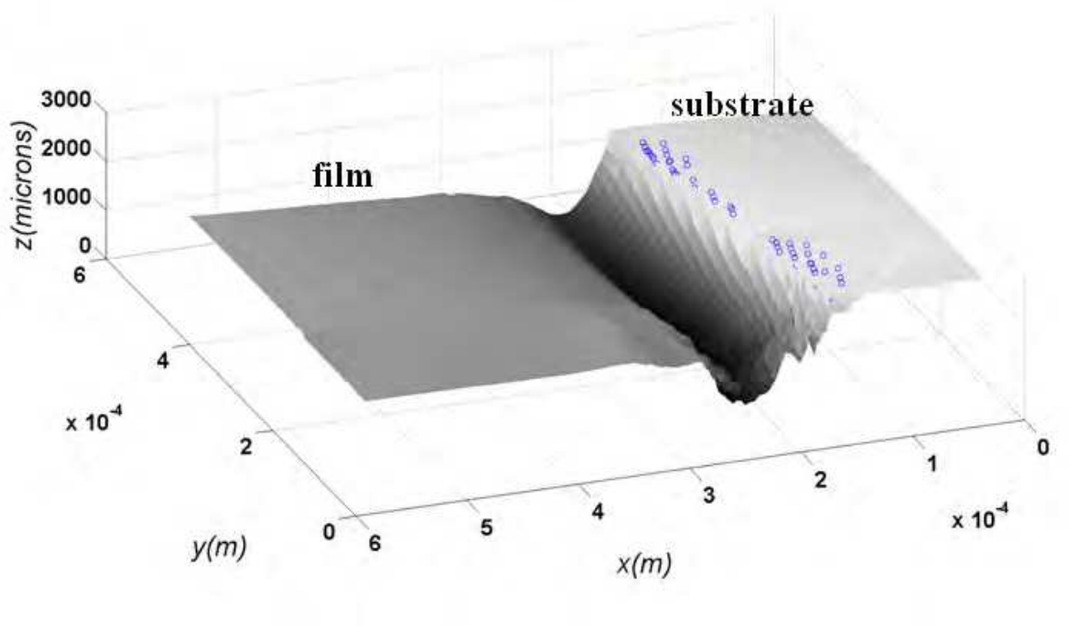


Fig. 10. The image of figure 8 with no tilt and film boundaries identified, with the curve consisting of small cycles.

Finally, the height of all pixels that belong to the transparent film region are subjected to the correction of equation (4) and demonstrated in figure 3. The result of this height correction code after implementation on the image in figure 8 is shown in figure 11. Furthermore the film thickness is calculated by subtracting the average height of an indicative film region from that of the substrate. Indicative examples of these regions are presented in figure 11 with red and blue colours respectively.

The image processing is not always necessary. In the general case where the exact topography of the film boundaries is not needed and only a thickness value is sought, the image processing may be avoided. In such a case the height difference of the substrate and film may be measured in the uncorrected optical profilometer image and then entered into equation (4) for the thickness measurement determination. This speeds up the process significantly as the image processing usually requires several hours while the alternative method needs only a few seconds. For thickness measurement both methods are equally accurate and therefore in the following section the image processing is avoided.

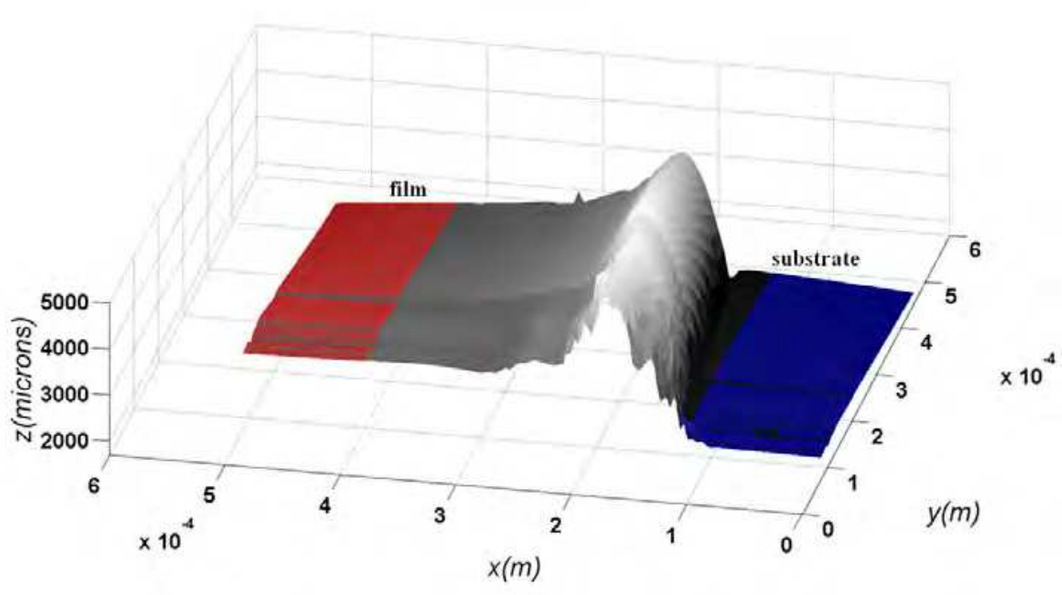


Fig. 11. Corrected image depicted by processing the image of figure 8. The regions used for thickness measurement are also highlighted.

6. Validation using atomic force microscopy

The technique for measuring transparent thin films such as photoresists, already described in section 6, has been validated here using atomic force microscopy (AFM). The atomic force microscope belongs to the scanning probe microscope (SPM) family of instruments. It mainly consists of a cantilever with a sharp tip (radius ~ 10nm), a scanner element and a laser with photo-diodes. The scanner consists of three piezoelectric elements and moves the tip in the x-y-z directions with sub nanometer precision. While the cantilever scans the sample surface, the laser light is reflected its light from the cantilever into a split photo-diode and thus the cantilever deflections at every sample point can be monitored. The whole procedure is controlled and monitored by dedicated software in a computer which reconstructs the three dimensional topographical image of the sample surface.

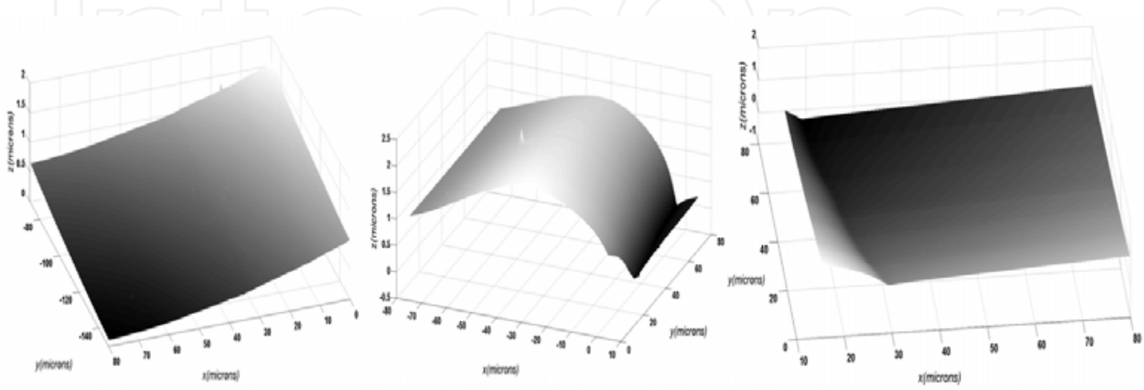


Fig. 12. Successive atomic force microscopy images of the boundaries of the photoresist film at the same region as in figure 11.

As an alternative method to measure the thickness of the photoresist film, AFM was used to scan the region in the boundary of the film with the substrate. A typical set of AFM images taken with this method is presented in figure 12. In this figures, it is obvious that due to the relatively small scanning area of the AFM image it is not possible to measure an area that completely contains both uncoated substrate and a film region with smooth and flat surface. It should be considered in this case that the film forms a hill at its borders as a result of the spin coating process. The width of this hill is typically more than 100μm, while the AFM maximum scanning area is 80μmx80μm. This limitation is overcome by scanning three adjacent areas of the sample using the motorized x-y translation stage of the AFM instrument. The three images were then subjected to the same tilt removal and tiled together using an image code developed in Matlab®. The image resulted from tiling of the three images of figure 12 is shown in figure 13. The thickness was calculated by subtraction of the substrate average height from the film average height using the same image. It was important to exclude the upward projected (hill) region of the film from the average film height calculation.

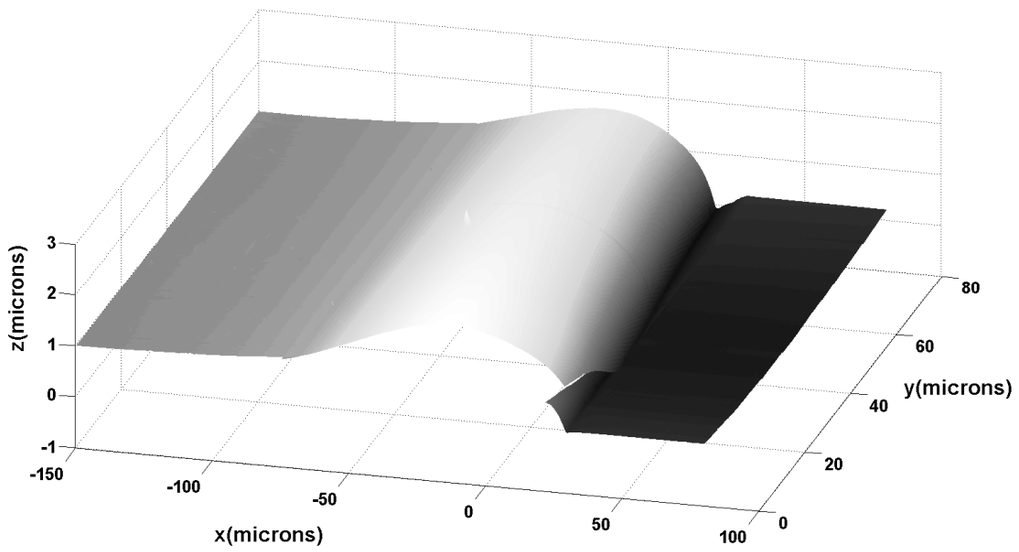


Fig. 13. Image resulted from the union of images in figure 12. It represents the borders of the same photoresist film as in figure 11.

Five measurements performed separately on two films using both AFM and the proposed technique based on optical profilometry. The one film has been made from the spin coating of a negative AZ® nLOF 2070 photoresist and the other from a solution of AZ® nLOF 2070 with AZ® EBR (volume concentration 1:1). As shown in table 1, the results were in good agreement and inside the statistical error. Further to the good agreement of the two techniques in the photoresist film thickness measurement, it is important to note further agreement of the profile and geometry of films boundaries such as the hill shape and its dimensions.

	Thickness obtained with optical Profilometer (nm)	Thickness obtained with AFM (nm)
AZ® nLOF 2070	2059±97	1966±90
Solution of AZ® nLOF 2070 and AZ® EBR (1:1)	531±87	551±39

Table 1. Average values of film thickness measured on the same film (n=5) with both AFM and optical profilometry.

The advantages and disadvantages of optical profilometry in comparison with AFM in film thickness measurement and in general are the following:

Pros:

- Optical profilometry has a much higher imaging area than AFM. With the appropriate lens of small magnification it can image areas with several millimetre width.
- It is a non destructive method as further shown in section 8. AFM is also non destructive for this application. However in other soft or wet transparent films like liquid crystals and elastomer polymers, the interaction and adhesion forces of AFM cantilever with the film may cause changes to the sample surface or even make imaging impossible.
- Optical profilometry is easier, simpler and faster.

Cons:

- The imaging of films with thickness of several hundreds of nanometers, according to section 5, is not possible by optical profilometry if the film edges are not sufficiently smooth.
- Optical profilometry in general requires surfaces with good reflectivity. Also if the sample surface consists of more than one material with different reflectivity this may result in poor imaging.
- The resolution of two techniques in film thickness measurement is practically the same. However in optical profilometry the usage of lenses with small magnification to increase the image area significantly decreases resolution.

7. Effect to photolithography performance

In the previous sections, the applicability and validity of a technique for measuring transparent films thickness has been proven based on optical profilometry on unexposed photoresists. As discussed in the introduction, photoresists are photo-sensitive polymers usually used in micro and nano patterning methods. Therefore it is possible that the exposure of photoresist films to the optical profilometer laser source for several minutes that measurements take, may influence the photolithography process. This section studies the influence of the thickness measurement technique in photolithography performance.

To investigate this, a plate substrate made from silicon wafer was spin coated with negative AZ® nLOF 2070 resist. The sample was subsequently exposed to the light of the optical profilometer. The exposure duration was 60sec in each point while the source scanned the sample at the successive points in lines to cover the area where the mask pattern was going

to be transferred, figure 14. Finally, the sample has been exposed to i-line (wavelength equal to 365nm) using a mask with a pattern consisted of black lines with 20μm width separated by 40μm spacing. The sample was then developed and imaged with optical microscopy, figure 14.

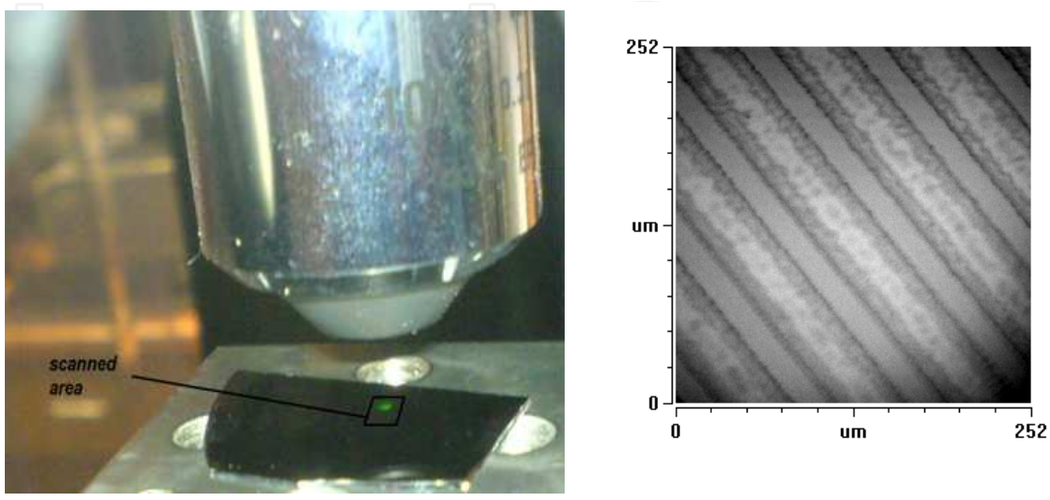


Fig. 14. Left image represents the light spot from the instrument and the sample area that was exposed to it. Right image shows an optical microscopy image of the transferred to the sample pattern after the exposure to instrument light.

As depicted from figure 14, the influence of optical profilometry was negligible. This is reasonable as in this case single wavelength optical profilometry was used with wavelength equals to 550nm while the photoresist used is sensitive to 365nm.

8. Conclusions

This work has further developed and tested on photoresist thin films a technique of measuring the thickness of LCs films [1]. It was shown that the thcikness of thin films of transparent films, like photoresists, can be measured accurately and the limitation of the the technique was also pointed out and analysed. This provides a cheap and quick non-destructive methdology of measuring photoresist thickness that could easily be adopted in the MEMS fabrication industry. In particular, the optical profilometry-based non-contact method introduced here for measurement of photoresist and other thin films is well suited not only to single and batch wafer fabrication, but also to continuous roll-to-roll (R2R) manufacturing on flexible substrates. Such R2R manufacturing, based on new deposition processes other than spin coating (e.g. doctor blading, transfer and jet printing etc). is becoming prevalent for flexible electronics and organic/hybrid photovoltaics production. The uncorrected (without image processing) version of the profilometric thickness measurement technique is fast enough for in-process monitoring of R2R production and quality control of the thin films. Such a real-time sensing tool therefore shows promise and could be valuable for feedback control of the photoresist deposition process, with considerable improvements in throughput and cost.

9. References

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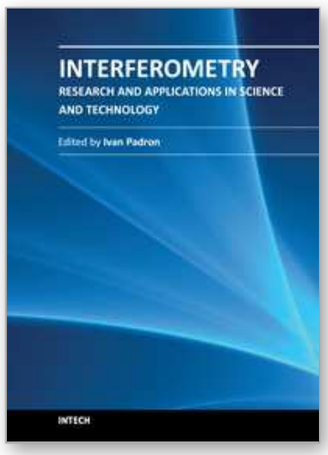
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