

Grade: 1,0

Protocol for the experiment

Optical Lithography

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1 Introduction

The last 50 years have seen rapid advances in computer technology. One reason for this has been optical lithography and its constant further development. It offers the possibility to apply small structures on a chip and thus to produce space-saving, cost-effective and material-saving high-performance electronic components. As a result, it has developed into the most widely used lithography method in the manufacture of electronic nanodevices [1].

In the protocol, the influence of different exposure times and associated exposure doses on the pattern, which was deposited on substrates by optical lithography, will be analysed. For this purpose, a substrate was prepared, coated with a resist and the pattern of a mask was transferred onto the coated substrate with different exposure times using the contact printing method. Finally, the resulting pattern was examined under a microscope after the development process and images of the substrates were taken. Based on the images, the resulting patterns is evaluated and compared, the width of the stripes of the pattern is calculated and the resist thickness is determined [2].

2 Technical background

Basic principle of optical lithography

Lithography is a method to transfer small structures of a mask onto a substrate. There are different techniques where a sample is exposed either to electrons (electron beam lithography), x-rays (x-ray lithography), ions (ion beam lithography) or monochromatic light (optical lithography).

In optical lithography, a light source generates monochromatic light that passes through a mask with a pattern of translucent and opaque areas. In the beam path behind the mask the substrate is located, which has a thin layer of resist on its surface. The light rays reach only the areas of the substrate that are behind the light-transmissive areas of the mask. Chemical reactions occur with the exposed resist regions on the substrate, changing their solubility in a solvent called developer. Either the irradiated area of the resist becomes less soluble in the developer than the non-irradiated areas (negative type resist), or the solubility of the irradiated area increases (positive-type resist). By panning the substrate in the corresponding developer after exposure, the soluble areas of the resist are removed and thus the pattern of the mask in the resist is visible. The resulting pattern of the resist can be transferred to the underlying substrate by utilising substances or etching agents [3].

Types and properties of photoresists

There are different types of resists, that can be applied to the substrate. They differ in their properties such as sensitivity, i.e. the minimum light dose at which the desired amount of resist can be removed, and resolution, which is reflected in the print size of the pattern. In addition, the robustness of the resist to other processes, the uniformity of application, the adhesion to a substrate, and the ease with which the resist can be removed after the transfer process all play a role.

Furthermore, resists differ in their chemical modification when irradiated with light. On one hand, there are the positive type resists, which are removed from the substrate after exposure due to their solubility in a developer.

One example of this group of resists is the resist whose polymer chains are split by cleavage, resulting in shorter chain fragments. These are more easily soluble than the original longer polymer chains and can thus be removed by means of a developer.

Another positive type resist is the novolak polymer-based resist, to which a photoactive compound (PAC) is added. When UV light hits this resist, the PAC converts to an acid when water is added, which significantly increases the solubility of novolak in a developer such as a sodium hydroxide solution.

Similar characteristics are shown by chemically amplified resists, which also contain a polymer and a PAC. In addition, these resists contain a converter. After irradiation of the resist, during which acid molecules are formed from PAC, the resist is heated, whereby the converters, catalysed by the acid molecules, also convert to acids, thus increasing the solubility of the polymer in a developer. This type of resist includes positive as well as negative type resists.

In the case of negative type resists, the areas that were not irradiated are removed after the exposure process, since the solubility in a specific developer is higher than that of the exposed regions. Resists in which cross-linking of polymer chains occurs belong to this group of resists. In this case, additional bonds are formed between the polymer chains after exposure. The solubility of the chains is reduced by the new bonds, so the exposed regions remain on the substrate after the development process [2].

Contrast und sensitivity of resists

The two properties contrast and sensitivity of a resist are determined by a so-called contrast curve (see figure 1).

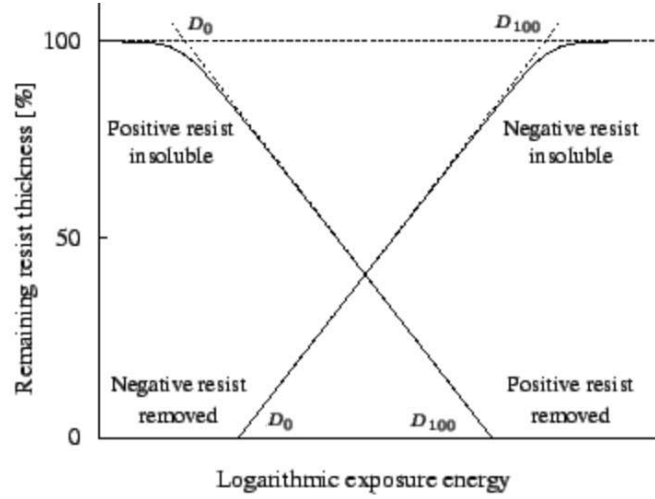


Figure 1: Contrast curve of a positive and negative type resist [2]

This is a graph in which the remaining resist thicknesses, which represent the ratio of the remaining resist thickness to the original resist thickness, are plotted for different exposure times. This can be done for both positive type resists and negative type resists. In the case of positive type resists, the resist thickness in the exposed areas is 100% at the beginning and decreases linearly with increasing dose from a threshold dose until it reaches the dose from which the thickness remains constant. For negative type resists, the resist thickness increases linearly from a threshold value with increasing exposure energy until it also remains constant.

The contrast γ of the contrast curves results from the slope of the linear range and is calculated as follows (see formula 1) [4]:

$$\gamma = \frac{1}{\log_{10} \frac{D_{100}}{D_0}} \quad (1)$$

Where D_0 is the dose at which a change in resist thickness occurs and D_{100} is the sensitivity at which the dose is reached where the resist thickness remains constant.

In addition to the contrast curve described above, there are also more detailed models in which the effect of development time on the residual thickness is studied. The longer the irradiated resist is in the developer, the more resist is dissolved [2] .

Exposure methods

In optical lithography, there are three different exposure methods: contact printing, proximity printing and projection printing. The main difference between the three methods is the distance between the mask and the substrate.

In contact printing, the mask lies directly on the substrate. On one hand, this minimizes refraction effects and thus maximizes the resolution of the pattern. On the other hand, however, damage to the mask and resist can occur due to mechanical contact.

A gap in the micrometre range exists between the mask and substrate surface in proximity printing. This reduces the risk of damage to the two components. Due to the larger distance between the mask and the substrate, refraction effects occur more frequently, which reduce the resolution of the pattern.

With projection printing, in contrast to the first two methods, there is a greater distance of several millimetre between the mask and the substrate. For this reason, the light is focused onto the substrate surface using an optical system. The resolution of this method depends on the refractions implied by the lenses of the optical system [3].

Resolution Limits

The three methods of contact printing, proximity printing and projection printing have limitations in terms of resolution. The reason for this is that refraction effects of the light occur at the edges of the mask during the irradiation process, which impairs the clarity of the pattern transferred to the resist.

For contact and proximity printing, the Fresnel approximation is used to describe the refraction effects in the near field. With this, the intensity distribution at the edges of the mask can be determined and, by means of this, the smallest representable line width b_{min} of the pattern can be calculated (see formula 2):

$$b_{min} = 1.5\sqrt{\lambda G} \quad (2)$$

λ is the wavelength of the irradiated light and G the distance between the mask and the center of the resist.

Since there is a larger gap between the mask and the sample in projection-printing, the Fraunhofer approximation is used here to describe the refraction effects in the far field. Two different types of illumination of the mask are distinguished: incoherent illumination and coherent illumination. In the former, the mask is illuminated with many different angles. Each point of the mask is imaged on the substrate as an intensity distribution, the so-called airy distribution. The individual intensity distributions of the points superimpose. By means of the minimum distance at which the points can still be discriminated individually, one can ultimately determine the minimum printable line width b_{min} (see formula 3):

$$b_{min} \approx \frac{0.3\lambda}{NA} \quad (3)$$

Where λ is the wavelength of the used light and NA is the numerical aperture, which is proportionally related to the aperture angle of the optical system.

In coherent illumination, the mask is illuminated vertically with a plane wave. Due to refraction effects, different interfering diffraction orders occur in the far field, which are limited by the aperture angle. This ultimately results in a minimum printable line width of (see formula 4):

$$b_{min} = \frac{\lambda}{2NA} \quad (4)$$

Again, the line width b_{min} depends on the numerical aperture NA and on the wavelength of the irradiated light λ .

Besides the occurring refraction effects, the resolution is influenced by further factors. One factor is the depth of field. Since the light is incident on the mask in a cone shape, each pixel of the mask has a certain width due to the refraction effects. The depth of field is the distance range in which the pixels are bundled and a sharp image is created. It is important that the depth of field is greater than the thickness of the resist to ensure a good resolution.

Another factor is standing waves. These occur when incident waves are reflected at the resist-substrate interface with a phase jump of 180° and interfere with the incident waves. The superimposed waves can be reflected again at the resist-air interface, creating a standing wave in the resist.

Depending on the resist thickness, constructive or destructive interference occurs. Due to an uneven resist surface and inaccuracies in the resist thickness, different intensity distributions of the coupled light can occur [2].

Measures to improve resolution

To improve the resolution, it is primarily suitable to use a large numerical apparatus, i.e. a large aperture angle of the optical system, and a wavelength as small as possible. However, since the aperture angle of the apparatus cannot be infinitely large and the wavelength cannot be chosen indefinitely smaller, because it can then no longer be refracted with optical instruments, additional possibilities are used to improve the resolution [2].

One is immersion, in which a liquid such as water is introduced between the lens of the optical system and the specimen. As the liquid has a higher refractive index n than air, the wavelength λ is reduced in it according to the form $\lambda' = \lambda/n$ and due to this the resolution is increased [5].

Another method is to use a phase mask. In this case, an alternating phase is introduced into some translucent areas of the mask so that a phase shift of 180° occurs when a light wave passes through. This prevents areas behind opaque regions of the mask from being illuminated by refraction effects, as the light waves from two adjacent bright regions destructively overlap in the dark region. The third method to improve resolution is off-axis illumination, where the mask is illuminated obliquely. The zeroth order of refraction is shifted to the edge of the apparatus, resulting in refraction of light at twice the angle. This allows smaller periods and structures to be transmitted from the mask to the substrate [5].

3 Execution of the experiment

The experiment was mainly divided into four steps: preparation and cleaning of the substrate, application of the resist on the substrate, exposure and developing of the coated substrate and investigation of the generated pattern on the exposed substrate under a light microscope.

For the first step, a thin round slice of the silicon substrate with a diameter of 76 mm was quartered by first cutting it parallel and perpendicular to the crystal axis with a diamond stylus and then breaking it along the cuts. The quarters were then cleaned to ensure that no contamination was present on the substrate pieces when the resist was applied. To do this, small remaining slivers of silicon and dirt were first blown off the pieces with nitrogen and each was rinsed in turn with

acetone, isopropanol and water. After cleaning, they were blown off again with nitrogen to dry and placed on a hotplate so that the substrate was dehydrated. This should lead to better adhesion of the resist to the substrate in the later steps.

After preparing the four samples, they were coated with the resist using the spin coating method. First, a platform was placed in the centre of a rotating fixture in the spin coater and the power supply to a pump was switched on, which sucks in the substrate by negative pressure during coating and thus fixes it. In addition, the normal light was turned off and instead yellow light was turned on because the resist was sensitive to room light. The respective substrate was removed from the hotplate, cooled with nitrogen, placed on the platform of the spin coater. First, the promoter hexamethyldisilazane (HMDS) was added to the sample, which was supposed to provide better adhesion between the substrate and the resist. After a reaction time of 30 s, the substrate was aspirated by the pump and the spin coater was switched on so that the platform rotated. The substrate with the HMDS was rotated on the platform at 2000 rps for 20 s and then at 4500 rps for 15 s. After the process was completed, the substrate was coated with the resist ma-P 1215. For this purpose, the resist liquid was applied to the sample so that the surface was almost completely covered, air bubbles were burst with a blower and resist drops that had run onto the platform were removed with a cloth. Subsequently, the respective sample was fixed again with the pump and rotated at 2000 rps for 20 s and at 4500 rps for 45 s. In this way, a thin and relatively homogeneously distributed resist film was generated on the specimen. To dry the resist, the sample was again placed on the hotplate until the solvent evaporated. No moisture was allowed to remain, since the exposure of the sample was performed using the contact printing method, in which the sample is in direct physical contact with the mask. There is a risk that if the resist is wet, the mask would become contaminated and possibly stick to the sample.

In the third step, the coated substrate quarters were irradiated with UV light, which transferred the pattern of a mask onto the resist. For this purpose, the contact printing method was used, in which the mask MJB 3 is brought into contact with the sample so that there is no gap between the mask and the sample. The exposure of the four samples was performed with six different exposure times of 7, 10, 14, 20, 30 and 40 seconds.

To apply the pattern, the mask was placed in the upper stage of the apparatus and the substrate was placed in the second lower stage. The position of the mask was adjusted using the apparatus with built-in microscope and the area of the substrate to be exposed was positioned underneath using a yellow light circle. Care was taken to ensure that the area to be irradiated was as far as possible away from the edge of the substrate, as the thickness of the photoresist was uneven in this region. The substrate and mask were then fixed in place and brought into contact by means of two buttons

on the apperature. The respective irradiation time was entered into the device and the substrate was then irradiated with the UV light. In order not to damage the eye, it was not allowed to look into the light. For developing, the sample was swivelled into the developer maD 331 for 20 s after irradiation, then cleaned in water and blown off with nitrogen to dry.

The resulting patterns of resists of all exposure times were then examined under a light microscope with a magnification of 3.2 and images were taken for later analysis with the LAS EZ program. Of the pattern with the longest irradiation time of 40 s, additional images were taken at 50 and 60x objective magnification using the LAS EZ program, and a scaling grid was also taken at these magnifications for the later scaling in the analysis.

Finally, data of the height profile of the widest line were recorded with a profiler in order to be able to determine the resist thickness later on [2].

4 Results

4.1 Description of the fabricated pattern and the effect of different exposure doses

For the qualitative study of the patterns of different exposure times, the specimens were examined with a lens magnification of 3.2. The pattern of the mask had the shape of the capital letter E, which consisted of different rectangular segments. These segments, in turn, are composed of 14 lines of different thicknesses, with the line thickness decreasing in one direction of the rectangle. Figure 2 shows images of the rectangular segments with the different exposure times.

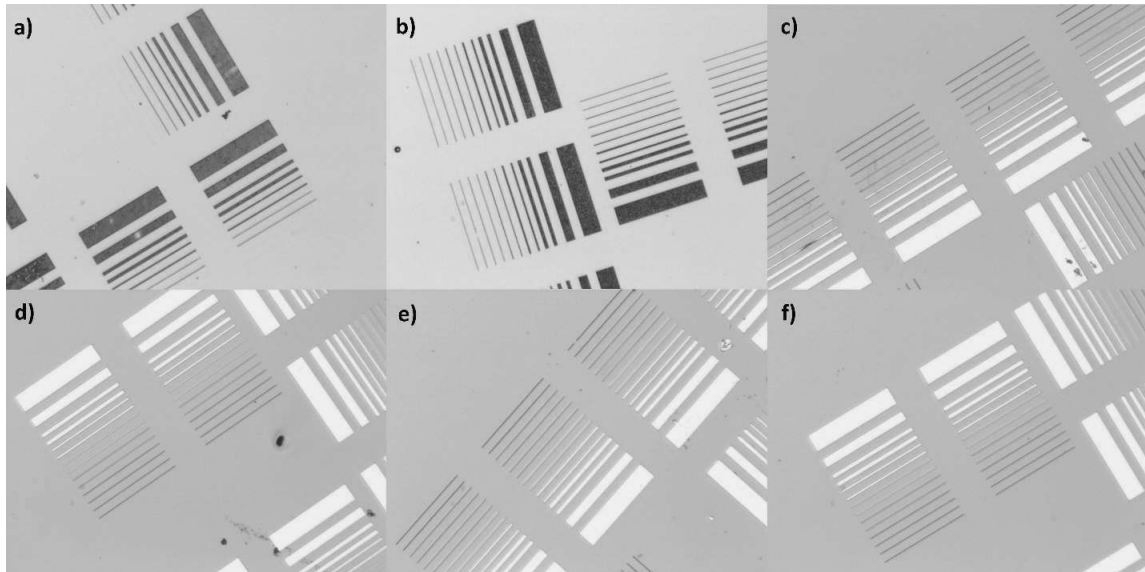


Figure 2: Resist pattern at an objective magnification of 3.2 and different exposure times **a)** Pattern with the exposure time of 5 s **b)** Pattern with the exposure time of 10 s **c)** Pattern with the exposure time of 14 s **d)** Pattern with the exposure time of 20 s **e)** Pattern with the exposure time of 30 s **f)** Pattern with the exposure time of 40 s

It can be seen that in a) where the resist was irradiated for 7 s, only 8 lines are fully visible. These stand out darkly from the background, in contrast to the images from c) to f), where the stripes are light. The edges of the 6 wider lines are sharp, whereas the edges of the thinner ones appear blurred. In addition to the 8 lines, two other narrower lines can be seen, but they can only be guessed.

At the next higher exposure time of 10 s, 10 complete stripes can be seen in the pattern (see figure 2.b)). In this image, the background appears brighter than in the other images and the stripes are dark as it was the case in a). The 4 narrowest stripes have blurred edges, whereas the thicker ones have relatively sharp edges.

Figure c) shows the stripes that can be seen with an irradiation time of 14 s. As in b), 12 stripes can be seen, but they differ in their brightness. The 6 widest stripes stand out brightly from the background and have sharp dark edges. The narrowest stripes appear dark against the background, as was the case for all stripes in the images of a) and b). Thereby the 2 narrowest stripes have sharp edges, the others of the thin stripes have blurred edges.

The result of the irradiation time of 20 s, 30 s and 40 s are shown in the images d), e) and f), in which the patterns look almost identical. In them all 14 stripes of the pattern can be seen, which have different brightness levels. The 6 widest stripes stand out brightly from the background and have a dark sharp edge. The next two thinner stripes are barely visible, as they have a similar colour to the background. A thin, blurred and dark border can be observed on them. The thinnest stripes appear completely dark, becoming sharper with decreasing thickness.

4.2 Quantitative report of the line widths of the pattern

To determine the line width of the 14 stripes, the pattern with the highest exposure dose of 40 s was examined under a microscope with the objective magnification of 50 and 80. To obtain the width in micrometres, images of a scaling grid were taken under the microscope at 50x and 80x magnification. The following describes the calculation of the conversion factor.

At 50x objective magnification, a scaling grid was imaged with a factor of $16,67 \frac{\text{Stripes}}{\text{mm}}$ (see figure 3). By calculating the reciprocal, the distance between the bottom edges of two strokes is determined which is 0.06 mm. Using Fiji, the pixel count of this distance is measured with the "Straight" tool, which is 923 pixels [7]. For the calculation of the scaling factor, the tool "SetScale" is used in Fiji, in which the determined number of pixels and the corresponding length in μm were entered. From this the conversion factor of $15,3883 \frac{\text{Pixel}}{\mu\text{m}}$ for an objective magnification of 50 is determined by dividing the distance in pixels by the distance in micrometres.

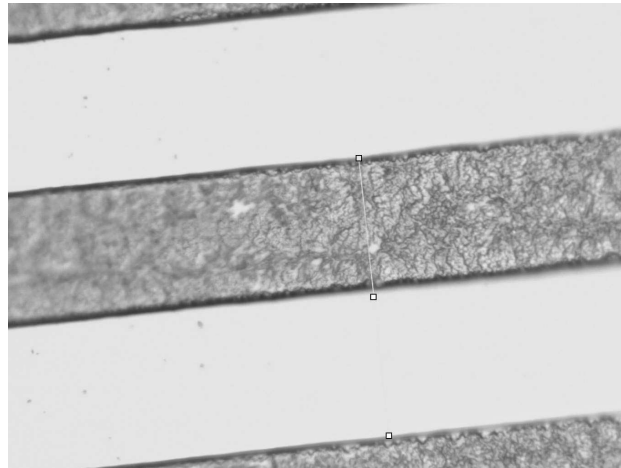


Figure 3: Scaling grid with 16.67 stripes per millimetre, taken with an objective magnification of 50

The conversion factor from pixels to micrometres is calculated in the same way for 80x magnification (see figure 4). Here, an image of a grid with a factor of $25,00 \frac{\text{Stripes}}{\text{mm}}$ is taken and the reciprocal, which represents the distance between the lower edges of two stripes, of 0.04 mm is calculated from it. The determined number of pixels of the corresponding distance in Fiji is 998 pixels, which resulted in a conversion factor from pixel to micrometre of $24,9500 \frac{\text{Pixel}}{\mu\text{m}}$ for the objective magnification 80 using the tool "SetScale".

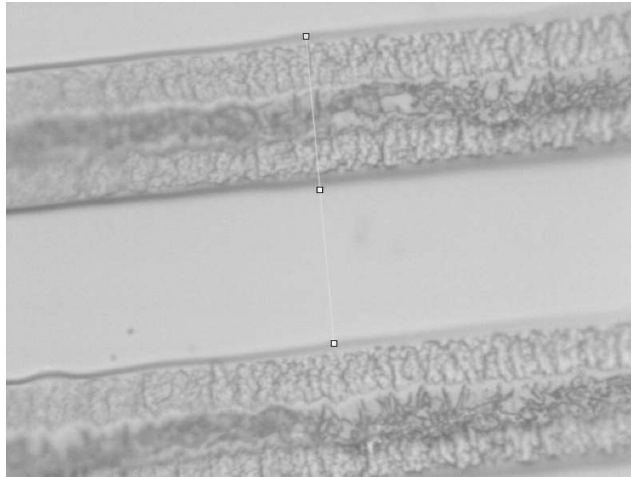


Figure 4: Scaling grid with 25 stripes per millimetre, taken with an objective magnification of 80

After the scaling values are determined, the width of the stripes is determined using Fiji by drawing a line along the width of each stripe with the "Straight" tool. The stripes are evaluated in decreasing order of thickness and numbered accordingly.

The first and widest stripe was recorded under an objective magnification of 50, since it could no longer be completely displayed under the microscope at a magnification of 80 (see figure 5). With the scaling factor for the objective magnification of 50, Fiji automatically calculates the length of the line and thus the width in micrometres.

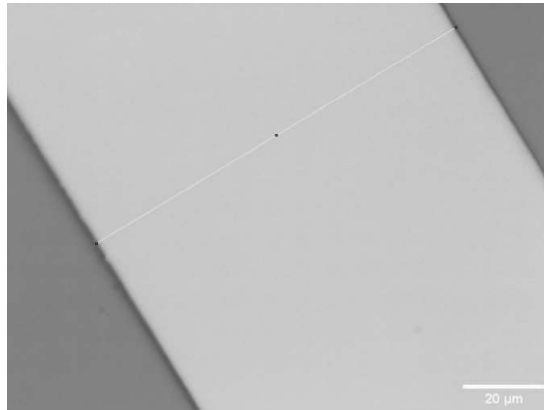


Figure 5: Widest stripe no. 1 of the pattern at the longest exposure time of 40, taken with an objective magnification of 50

The other 13 strips were taken at an objective magnification of 80 and are also automatically evaluated the width in micrometres of Fiji (see figure 6).

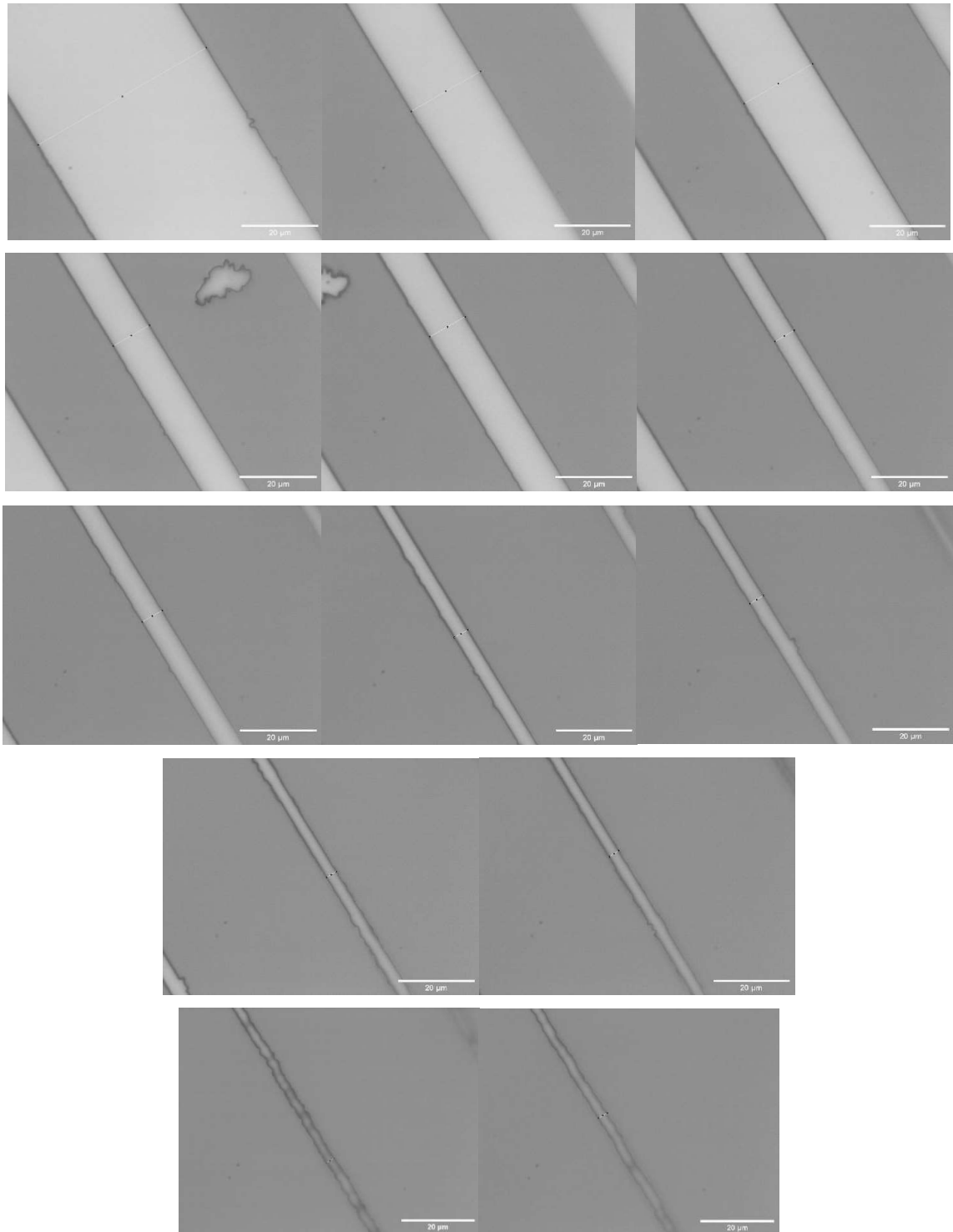


Figure 6: From wide to thin sorted stripes no. 2-14 of the pattern at the longest exposure time of 40, taken with an objective magnification of 80

The results of measuring all strips are plotted in Table 1. In the case of the two narrowest stripes, uneven edges in relation to the width occur (see figure 6), which is why the thickness of these varies depending on the location of the measurement.

Table 1: Thickness of strips sorted from wide to narrow

	Objective Magnification	Width [μm]
Stripe 1	50	102.007
Stripe 2	80	50.261
Stripe 3	80	20.539
Stripe 4	80	20.522
Stripe 5	80	10.579
Stripe 6	80	10.675
Stripe 7	80	5.838
Stripe 8	80	5.926
Stripe 9	80	3.898
Stripe 10	80	3.963
Stripe 11	80	3.033
Stripe 12	80	2.936
Stripe 13	80	2.429
Stripe 14	80	2.750

4.3 Measurement of the remaining resist thickness

The remaining resist thickness was determined from a height profile of the substrate surface taken with a profiler. The height profile is shown in Figure 7.

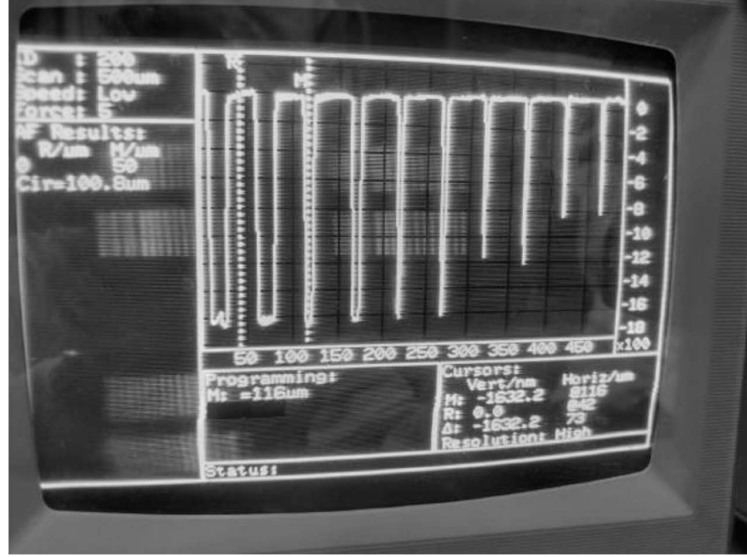


Figure 7: Height profile of the resist coated substrate surface recorded with a profiler

There are 10 stripes of the pattern, which can be recognized by the downward oriented peaks. The widest stripe can be seen as the widest peak on the right. Along the x-axis, the width of the peaks decreases and thus, correlatively, the width of the measured individual stripes. This corresponds exactly to the stripe course of the original pattern.

It can also be seen that 6 left stripes have peaks of the same length, whereas those of the 4 right stripes are shorter and decrease along the x-axis. For the measurement of resist thickness, it is assumed that the 6 left stripes are fully developed stripes due to their uniformity in length, making them suitable for measuring the resist thickness originally applied to the substrate. For this purpose, markers R and M were set during the measurement. Marker R was set at a location where no peak was present, so no resist was removed from the substrate at this location. Marker M was set at the third peak from the left, which is assumed to be one of the strips that is fully developed. The height values of the two marks can be read on the display of the instrument and the resist thickness z can be calculated from them as follows (see formula 4):

$$\begin{aligned} z &= R - M \\ &= 0.0 \text{ nm} - (-1632.2) \text{ nm} = 1632.2 \text{ nm} \end{aligned} \quad (5)$$

Thus, the resist thickness applied during coating with the resist is approximately $1.6322 \mu\text{m}$.

5 Discussion

The results from chapter 4.1. showed that with increasing exposure time and thus increasing radiation dose, more lines of the pattern were transferred to the resist. Therefore, it can be assumed that more resist was removed with increasing exposure time during development. This is consistent with the positive type resist contrast curve (see figure 1, chapter 2), where above a threshold value there is a linear decrease in resist thickness with increasing exposure dose. The coloration of the pattern also changes the longer it was irradiated. Stripes that appear dark at shorter exposure times appear increasingly lighter, the longer they are exposed. Therefore, based on the positive type resist contrast curve, it can be assumed that the dark areas are not fully developed areas on the substrate where resist has remained. In contrast, the bright areas are likely to be fully developed areas where the light of the microscope incident from below must not pass through the dark resist material before reaching the eye. Intuitively, one would assume that the incompletely developed areas would also appear brighter than the non-irradiated areas, since there is less resist thickness there. It is possible, however, that destructive interferential effects, such as standing waves, occur there due to the certain emerged resist thickness, so that no or fewer light waves reach the eye from these areas.

Another observation is that the edges of the stripes and also thin lines that are narrower than about $3\text{ }\mu\text{m}$ appear darker even at the highest exposure time of 40s. It can be assumed that the boundaries of the stripes are not fully developed because refraction effects occurred on the mask edges during the exposure process, leading to a reduction of resolution of the pattern. The thin stripes have a small inner area in relation to the edge, which makes them appear completely dark as a whole at a resolution of 3.2 (see chapter 2).

In addition, from an exposure time of 30 s, no significant change in the pattern occurs despite increasing exposure dose. It is possible that from this time on, the limit dose is reached at which no change in the resulting resist thickness of the exposed areas occurs (see figure 1, chapter 2).

6 Conclusion

In this experiment, optical lithography proved to be a good method for transferring the smallest structures of up to $2.5\text{ }\mu\text{m}$ onto a substrate. In doing this, it was shown that with increasing irradiation times up to 20 s, increasingly finer structures became visible and the irradiated areas also increasingly showed complete development. This corresponds to the course of the theoretical contrast curve. However, it was found that edges and narrow strips with a width of less than $3\text{ }\mu\text{m}$, which have a high proportion of edge to central area, could not be fully developed even at higher

exposure doses. The reason for this is assumed to be refraction effects at the mask, whereby a lower proportion of waves reached the areas behind the transparent edge areas, thus reducing the resolution.

It should be mentioned that inaccuracies occurred during the experiment, which had an influence on the result. For example, errors happened during the development of the individual samples, as it was difficult to remove the substrate quarters from the developer liquid with tweezers. Therefore, some samples remained in the developer longer than the prescribed 20 s, possibly causing more resist to be dissolved from the exposed areas on these samples and thus removed.

In addition, the substrate was not uniformly coated with resist during spin coating, resulting in uneven resist thicknesses, especially at the edges of the substrate. A solution for this could be, to use a different type of resist, that is easier to apply more uniformly to the substrate.

Minor inaccuracies may also have occurred during irradiation of the sample. However, the irradiation time was controlled by the instrument, so it is assumed that the irradiation time was relatively accurate.

Additionally, one of the mentioned measures to improve resolution could be used to increase the sharpness of the edges of the stripes.

By correcting the above-mentioned errors as well as using the methods to increase the resolution, the experiment could be carried out in a more reproducible manner and the effects of different irradiation times on the individual samples could be compared more accurately.

7 References

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