Optical characterization of an amorphous-hydrogenated carbon film and its application in phase modulated diffractive optical elements

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Abstract
A four level, phase only modulated, diffractive optical element was designed, manufactured and characterized. The active part of the device was made of a well defined topology in a hydrogenated amorphous thin film. In order to develop the correct thin film etching process, it was necessary to determine the refractive index of the film. The refractive index of the glass substrate and the absorption of the film at the 633 nm wavelength were also measured. The device was manufactured using standard optical lithography and an oxygen plasma etching process. The resulting RMS roughness in the etched carbon film was less than 5 nm. In order to perform the optical characterization of the device, the power in the zero order diffracted beam and in the desired light pattern of a ring shaped hologram, specifically designed for this purpose, was measured. The speckle was extremely low and the (total) optical efficiency is as high as 95%, what is suitable for most real life applications.

1. Introduction
Diffractive Optical Elements (DOE’s) are very useful devices with a wide range of applications in the fields of robotics, artificial vision, holographic filters for pattern recognition, optical interconnects, correction of chromatic aberrations of standard optical systems, etc. [1,2]. For phase modulated devices, it is necessary to create a well defined topology in a transparent film or substrate. In this work, this topology was created in an amorphous hydrogenated carbon (a:C-H) thin film, often called a Diamond Like Carbon (DLC) film.

2. Deposition of the DLC film
A three inch diameter, high transparency, optical quality glass substrate with a refractive index of approximately 1.52 (B270 - Schott) serves mainly as a mechanical support for the active parts of the device [7]. On this substrate, films of amorphous hydrogenated carbon (a:C-H), were reactive magnetron sputter deposited in a home made system. A methane plasma was created to sputter a graphite target and deposited a hydrogenated carbon film on the glass substrate. Distance between graphite target and glass substrate was 10 cm, what resulted in a deposition rate of approximately 16.4 nm/min. Varying the processing times up to 90 minutes yielded films with different thicknesses up to 1500 nm. The deposition rate proved to be constant in time. AFM measurements determined that the RMS roughness of the deposited film was 2.5 nm over an area of 15 µm by 15 µm. The deposition process and mechanical-electrical-chemical characteristics of the films are described in detail in [8].

3. Determination of the refractive index of the DLC film
For several thin films, ellipsometry is an easy and very reliable method to determine their refractive index, n. For these tests, a Rudolph Auto EL NIR 3 ellipsometer was used. It has the possibility to fix the thickness or the refractive index of the film, and measure the other parameter, or measure both parameters at the same time. The main drawbacks of this technique are: 1) the method looses a lot of precision and accuracy when the substrate is transparent (as in our case with a glass substrate) and 2) the measurement procedure gives the same results if the film is exactly 1 period thicker (for the used equipment: 1 period = (λ/2n²) . [n² - sin²(70°)]¹/² , so
one needs to know in advance the approximate thickness of the film (typically with an accuracy of ± 50 nm).

Therefore a special sample was prepared, only for a first determination of the refractive index of the film: approximately 200 nm of DLC was deposited on a 3 inch diameter, (100) Si wafer, whose optical characteristics are very well known. For this sample it was possible to use a program which determines both local thickness and refractive index of the film. The sample was measured 5 times and the result was an average refractive index of the DLC film, \( n_{DLC} = 1.617 \) with a standard deviation of 0.0045.

The DLC was deposited by magnetron sputtering. This is an additive process and therefore the resulting film suffers very little influence of the substrate material. Hence, the characteristics of a film deposited on a glass substrate should be approximately the same as for a film deposited on silicon. Even so, it is necessary to determine the refractive index of the DLC film deposited on the glass substrate.

The index of refraction of the DLC deposited on a B270 glass substrate was determined by the UV-VIS-NIR spectrometric technique. The equipment used for these experiments was a Perkin-Elmer, model Lambda 9. The technique consists in measuring the intensity of the transmitted light as a function of wavelength. Because of (constructive and destructive) interference of light reflected at the air-film and film-substrate interfaces, more or less light is reflected back from the device and as a consequence less or more light passes through the sample. By determining the maxima (or minima) of the modulation of the transmitted light as a function of wavelength, it is possible to determine the refractive index [9]. The UV-VIS-NIR spectrometric technique has a drawback that, for these films, it yields only reliable results for wavelengths longer than approximately 700 nm, because there is some absorption at lower wavelengths, as shown in figure 1.

It is necessary to know the refractive index of the substrate. For this purpose, the light transmission of a blank substrate was measured. A transmittance of 0.9175, for wavelengths longer than 330 nm, was obtained. (Below this wavelength, the absorption of light by the substrate increases very rapidly, as can be seen from the dotted lines in figure 1.) Assuming zero absorption, it is possible to calculate the refractive index of the substrate, which was in this case : \( n = 1.516 \).

Figures 1a to 1d show the results for samples with approximate DLC thicknesses of 100 nm, 500 nm, 1000 nm and 1500 nm, whose transmittances were measured at the centre, border and halfway centre-border of the sample. The modulation for the 100 nm sample is insufficient to obtain any result. For the 500 nm thick film, the second maximum was too broad to obtain a precise result, but for the thicker samples of 1000 and 1500 nm, the modulation allowed a precise determination of the refractive index. This thickness range is
complementary to the one for which the ellipsometry technique yields good results.

<table>
<thead>
<tr>
<th>Thickness [nm]</th>
<th>sample local</th>
<th>Wavelength [nm]</th>
<th>Refractive index</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000</td>
<td>C</td>
<td>768.5</td>
<td>1.590</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1056.5</td>
<td>1.605</td>
</tr>
<tr>
<td></td>
<td>C/B</td>
<td>748.5</td>
<td>1.598</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1016.0</td>
<td>1.612</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>760.5</td>
<td>1.597</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1037.5</td>
<td>1.611</td>
</tr>
<tr>
<td>1500</td>
<td>C</td>
<td>788.5</td>
<td>1.597</td>
</tr>
<tr>
<td></td>
<td></td>
<td>951.0</td>
<td>1.593</td>
</tr>
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<td>1197.0</td>
<td>1.596</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1654.0</td>
<td>1.602</td>
</tr>
<tr>
<td></td>
<td>C/B</td>
<td>790.0</td>
<td>1.593</td>
</tr>
<tr>
<td></td>
<td></td>
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<td>1.611</td>
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<tr>
<td></td>
<td>B</td>
<td>791.5</td>
<td>1.597</td>
</tr>
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<td>1651.0</td>
<td>1.611</td>
</tr>
</tbody>
</table>

Table 1 – Refractive index results for film thickness d=1000 nm and d=1500 nm as measured at different locations of the sample: centre C, border B and half way from centre to border, C/B.

Table 1 shows the results for the 1000 nm and 1500 nm thick film samples. This table shows that it is possible to determine the refractive index for different wavelengths for these thicker films. For the wavelengths in the 700 nm - 800 nm range an average refractive index $n_{ave} = 1.596$ with a standard deviation of 0.0032 was obtained. One may conclude that this result is compatible with the result obtained by Ellipsometry ($\lambda = 633$ nm, $n_{ave} = 1.617$) for a DLC film on a silicon substrate. Hence, it is our hypothesis that the refractive index for the films deposited on the glass substrates are the same as for the silicon substrate, and even extrapolate that the films themselves are the same for both substrates, as could be expected.

The percentage transmitted light for the 633 nm wavelength is also shown in figures 1.a-1.d. As we know that the substrate is responsible for the loss of approximately 8% of the intensity of the light we may e.g. conclude that for a 1500 nm thick film, we loose an extra 6.6% of the incoming power.

4. Fabrication of a phase only modulated DOE with four levels

The process used for the manufacturing of these devices can only fabricate discrete steps in a film or a substrate and not films with a continuously (and well controlled) varying thickness. Hence, one needs to perform a quantization of the allowed values of the phase [10]. In this paper we used four possible levels of the diffractive element phase function. This implied two lithographic masks in the fabrication steps, as can be seen in figure 2.

With four levels it is possible to impose phase delays of the wavefront by $0\lambda, \lambda/4, \lambda/2, 3\lambda/4$. These delays are caused by the fact that the light travels slower in a medium with high refractive index (here in the DLC film) than in a medium with low refractive index (here air, with $n=1$). In order to obtain the desired phase delays, the step heights in the DLC film are determined by the formula (for the device immersed in air) [11]:

$$d = \frac{\lambda}{k(n_{DLC} - 1)}$$

with $d =$ step height; $\lambda =$ wavelength employed; $k =$ factor dependent on phase delay : in this case for $\pi/4$ phase delay $\rightarrow k = 4$, for $\pi/2 \rightarrow k = 2$ and for $3\pi/4 \rightarrow k = 4/3$.

In the former section we determined that for $\lambda = 633$ nm, $n_{DLC} = 1.617$. In order to obtain the different phase delays, the step heights in the DLC film, i.e. the depths of the features etched in the DLC films have to be : for $\lambda/4 \rightarrow d=256$ nm, for $\lambda/2 \rightarrow d=513$ nm, for $3\lambda/4 \rightarrow d=769$ nm.

In order to fabricate the pixels with these four different optical film thicknesses, it is necessary to perform two lithography + etching sequences: the first one which removes 256 nm ($\lambda/4$ phase delay) and the second one which removes 513 nm ($\lambda/2$ phase delay).

Figure 2 shows schematically the complete fabrication sequence of the four level phase-only diffractive optical element (DOE).

![Figure 2 - Schematic view of the complete fabrication sequence of the four level phase-only diffractive optical element (DOE).](image)
Table 2: Etch depth in the amorphous carbon film as a function of the masks (see also figure 2).

<table>
<thead>
<tr>
<th>Mask 1 / Mask 2</th>
<th>Dark / Dark</th>
<th>Light / Light</th>
<th>Dark / Light</th>
<th>Light / Light</th>
</tr>
</thead>
<tbody>
<tr>
<td>First etching</td>
<td>0</td>
<td>256 nm</td>
<td>0</td>
<td>256 nm</td>
</tr>
<tr>
<td>Second etching</td>
<td>0</td>
<td>0</td>
<td>513 nm</td>
<td>513 nm</td>
</tr>
<tr>
<td>Total removal</td>
<td>0</td>
<td>256 nm</td>
<td>513 nm</td>
<td>769 nm</td>
</tr>
</tbody>
</table>

sequence of the four level DOE.

A 1.2 µm thick, positive photoresist was spun on the wafers, exposed by UV light, developed and baked. Then the amorphous carbon layer was etched in an oxygen plasma, in a single wafer Reactive Ion Etching (RIE) system [12]. The remaining photoresist was then stripped away in an acetone bath, which didn’t affect the amorphous carbon layer. The lithography, etching and resist stripping steps were then repeated, using a second lithography mask and an etching time of twice the time of the first etching. By combining the two lithography masks, the desired pixel depth can be obtained, as shown in table 2.

After some preliminary tests, the following etching process was established: 20 sccm of O₂, 100 mTorr pressure and 50 W RF power, what resulted in a DC self-bias voltage of approximately -515 V.

With this process, four 3 inch diameter substrates were etched, using a light field mask. The average DLC etch rate was 296 nm/min with a standard deviation of 1.1 %. The resist etch rate was 274 nm/min. This is low enough to permit the etching of the DLC film without any resist problem (Selectivity is 1.08).

The resulting step heights, as measured with a step height meter, were within 2% of the desired values.

An SEM image of a detail of the holographic device is shown in figure 3. The RMS roughness, as measured by the AFM, was only 4.7 nm (λ / 130), in areas where 769 nm of DLC had been etched. This is very important in order to minimise the speckle noise in the diffracted pattern (reconstructed image) which appears when the roughness levels are high.

5. Optical characterization of the DOEs

With the used mask set, 12 different diffractive elements were implemented. Some are based on the Fresnel diffraction theory (Fresnel-type element) and some other are based on the Fraunhofer diffraction theory (Fourier-type element). The optical characterization of a diffractive element is in general a qualitative assessment. In order to perform a more quantitative characterization of the “quality” of the hologram, a special device was designed. It is a Fraunhofer type of a diffractive element that simply consists of a ring, which is shown in figure 4. This element consists only of two phase-levels in the DLC, but that is enough to characterize the etching process.

![Figure 3](image1.png)

**Figure 3** – SEM image of a detail of the holographic device.

![Figure 4](image2.png)

**Figure 4** - (a) computer-simulated reconstructed image (256 by 256 pixels) of the designed two phase-level diffractive optical element; (b) CCD-camera image reconstruction obtained from the fabricated diffractive element; this image is magnified by a factor of 2.4 with respect to image (a), (c) a diametric cross section of the light distribution of (b). For this image about 5% of the total optical power is concentrated in the central spot, the efficiency is therefore approximately η = 95%. 

![Figure 5](image3.png)
A Fraunhofer type of device has the characteristic that if the phase modulation is not well performed - in our case, if the height of the pixels is not exactly correct - the zero order diffraction (dc peak) appears in the centre of the device. If the device is perfectly manufactured then the zero order will contain no optical power. Beside the wrong dimension of the pixels, their roughness induces a second undesired effect: a haze all over the image: the so-called speckle. Both these effects remove optical power from the desired feature. Therefore a quantitative way to characterize the optical quality of the device is to compare the power in the zero order beam to the power in the desired image. With this ring shaped device, it is easily possible to measure power in the zero order beam and in the diffracted part of the image (i.e. the ring).

Figure 4.a shows a simulated image of the designed diffractive optical element; figure 4.b shows the obtained image reconstructed by the fabricated diffractive element. It is possible to note that there is minimal speckle noise in the reconstructed image, but it presents the mentioned dc-peak at the centre of the image.

The light intensity of a diametric cross section of the light distribution of figure 4.b, as measured by a CCD imager and quantified by an imaging processing software (NIH) [13], is presented in figure 4.c. The diffraction efficiency, $\eta$, can be defined as:

$$\eta = \frac{\int \text{image points}}{\int \text{total points}} .$$

For this device, we calculated the efficiency as:

$$\eta = \frac{\int \text{power in the ring}}{\int \text{power in the ring} + \text{power in the zero order beam}} .$$

For the presented image this efficiency is approximately 95%. This value is satisfactory for most applications. With this process sequence it is possible to manufacture whatever four level modulated optical diffractive device. Figure 5 shows another reconstructed image of a four-level phase-only diffractive optical element. It is possible to see the zeroth diffracted order superimposed on the designed image. Here again, we estimate that less than 5% of the total optical power is concentrated in this central beam spot.

**6. Conclusions**

In this work, a hydrogenated amorphous carbon thin film was used to implement the four levels of a phase only modulated device. The refractive indexes of the carbon film and of the glass substrate were determined, and also the absorption of a 1500 nm thick carbon film, for the 633 nm wavelength.

The device was manufactured and the optical characterization showed an extremely low speckle level, because of the low roughness level of the processed carbon film.

The optical efficiency was as high as 95%. This process can be used for other designs of four level, phase only modulated diffractive optical devices.

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**References**


