4-4-2024

Non-Destructive Thickness Uniformity Measurement of Photosensitive Gelatin Film

Clayton Halper
Utah State University

Non-Destructive thickness uniformity measurement of photosensitive gelatin film

Clayton Halper

Physics 4900 Research
Department of Physics, Utah State University, Logan, Utah
Wasatch Photonics, Logan, Utah

Volume phase holographic gratings (VPHG's) depend on dichromate gelatin of which uniform thickness is vital. The photosensitive nature of the film makes current thin film measurement devices not viable for production means. This project attempts to create a non-destructive measurement of photosensitive gelatin film used in VPHG production. Application of thin film interference at chosen wavelengths enable analysis of uniformity by comparison between the thin film inference patterns at different wavelengths. An initial proof of concept was established and a path towards a production ready device is outlined.

Experiment date: August 2023 – April 2024

Report submitted: April 4th 2024

I. INTRODUCTION

Wasatch photonics is a company specializing in diffraction gratings and spectrometers. More specifically volume phase holographic gratings (VPHG's). This involves the utilization of dichromate gelatin which is photosensitive, allowing the use of holography optical tables to image interference patterns to create a diffraction grating. Given the elasticity and dynamical properties of gelatin, uniformity in film thickness is vital to producing evenly performing and high efficiency diffraction gratings. That necessity along with the production aspect of a company requires an effective method to measure said uniformity without destroying viable product.

The goal of this project is to develop a method by which the gelatin films could be measured without destroying it, either physically or by exposure to light in the low wavelength visible range and UV range. Current methods in film measurement typically rely on white light interferometry or some other method that would either be too costly for the given situation or would be harmful to the film used. Currently at Wasatch Photonics white light interferometry is used on a test piece of film which cannot be used for actual production parts. It is important to note that in holography very minor changes in film conditions can result in very large impacts into the final product. Thus a practical measurement process must not leave debris, physical changes, or create light exposure in the lower wavelength regions.

A device capable of measuring film thickness and uniformity would enable process improvements that would improve yield and enable the production of higher quality and higher efficiency diffraction gratings. More technical details will be discussed in the theory section of this paper. The general concept is to use interference patterns to analyze film thickness and uniformity as in white light interferometry.[1] In this case the light will be limited to greater than ~540 nm in wavelength and restricted across specific wavelengths below that value.

II. THEORY

Similar to current methods that use white light interferometry, the theory applied for this project is thin film interference. The method by which this happens is when light enters a film, at which point some light reflects at the interface with air and some transmits to then get reflected off at the interface with the substrate where the film is applied to. In this case the light that went through the film and reflected off the glass will have a longer path length than the light that simply reflected off the film surface. These two rays of light will be parallel to one another, meaning these rays can interfere with one another, either constructively or destructively [2]. This will be a function of the optical path difference between the two rays which will be a function of initial incident angle. index of refraction of the materials involved, and the thickness of the film layer. The optical path difference (OPD) can be defined as:

$$OPD = 2n_2 \, \mathrm{d}\cos(\theta_2) \tag{1}$$

where n_2 is the index of refraction of the film, d is the thickness of the film and θ_2 is the angle of refraction of the light through the film which is also the angle of the incident light onto the glass. This equation will be equated to an integer multiple of the wavelength. Due to a phase change upon reflection, when OPD is at ½ integer multiples of the wavelength the two rays will constructively interfere and when OPD is upon full integer wavelengths the light will destructively interfere [3]. An equation can be defined to relate the thickness of the film as an integer function of the wavelength, index of refraction, and refraction angle:

$$d = mD \tag{2}$$

where d is the thickness of the film, m is the integer or ½ integer multiple, and D is $\lambda/2n_2\cos(\theta_2)$.

In theory one wavelength will only be able to indicate uniformity with no reference to the absolute or relative thicknesses. This project will utilize more than one wavelength of light and in conjunction with one another will determine the relative and absolute thickness of the film across the glass substrate.

III. PROCEDURE

The goal of this project is to develop a device to measure film thickness and uniformity, and as such follows a more R&D style approach with trial and error to get the desired results. Before going through the process of designing a custom system, a general proof of concept was done using available resources. A green light source that is commonly used for inspection of glass for flatness was used as an initial test. This uses diffusion of a broadband light source and a color filter to pull out a specific spectral band. The device was put in front of a spectrometer to get a measure of the specific wavelength. The film coated glass was illuminated by this light source so thin film interference patterns could be observed and imaged by a camera. Points on the plate were marked and the theoretical values were compared to the measured values taken by a Filmetrics system designed to measure film thickness using white light. This image and the points marked on the plate can be seen in Fig. 1.

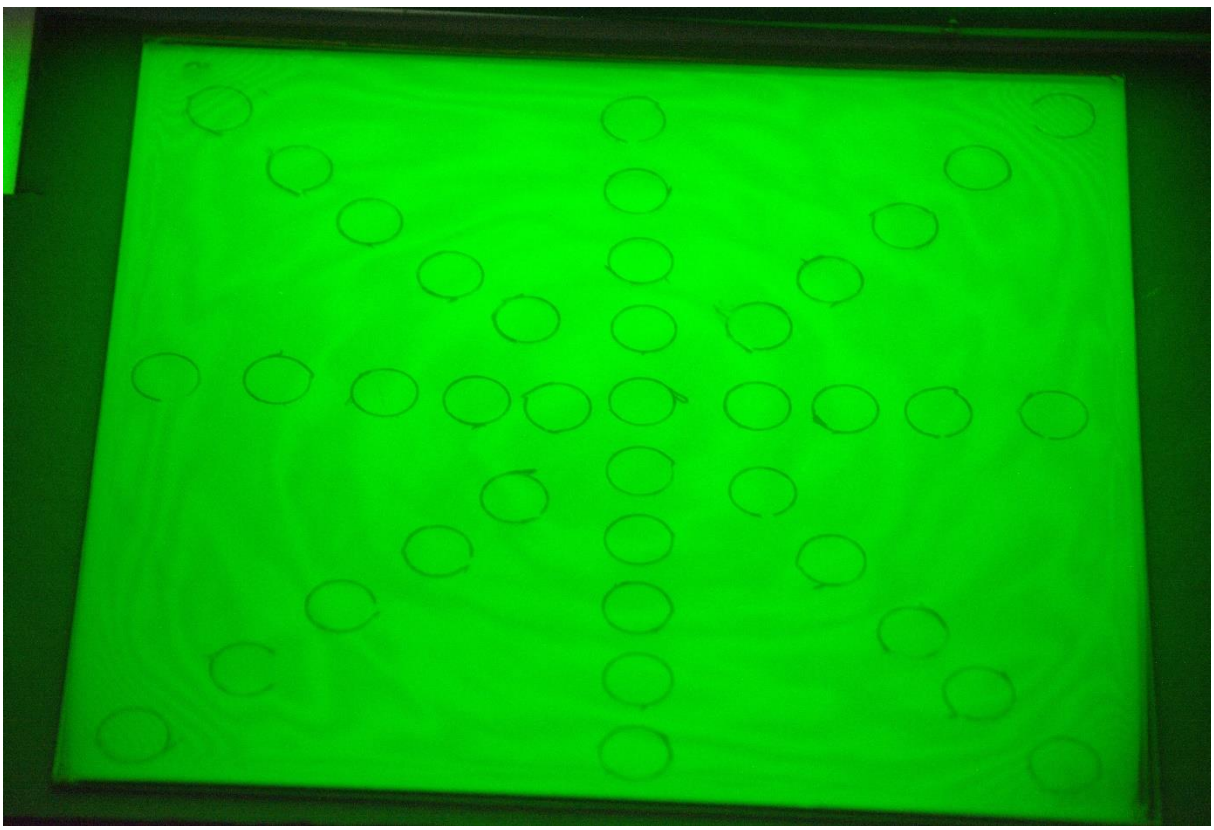

FIG. 1. Pictured is the initial proof of concept test film. The marked circles were reference points to make measurements on the Filmetrics device to compare to theoretical values given the imaged interference pattern.

The index of refraction of unexposed film was not a known value and since thin film interference depends on index of refraction, the next step in the process was attempting to find an index value for unexposed film. This was also crucial to account for potential phase changes upon reflection. Pieces of unexposed film were placed into a spectrophotometer and measured across a wavelength range of 10 nm in steps of .25 nm and across a range of 2 degrees in steps of .2 degrees. Many such trials were done with films ranging in film formula and age. These values were used to compute Brewsters angle for the unexposed film which in turn was able to provide a value of the index of refraction for the unexposed film.

After the initial trial proof of concept, and identification of an index for the film, the next step was to design a system by which thin film interference patterns could be imaged at a specific wavelength. The proposed system included a HeNe laser which was expanded through a ball lens and diffused through a diffusion film at which point it would illuminate the film where a camera could capture the interference pattern (Fig. 2).

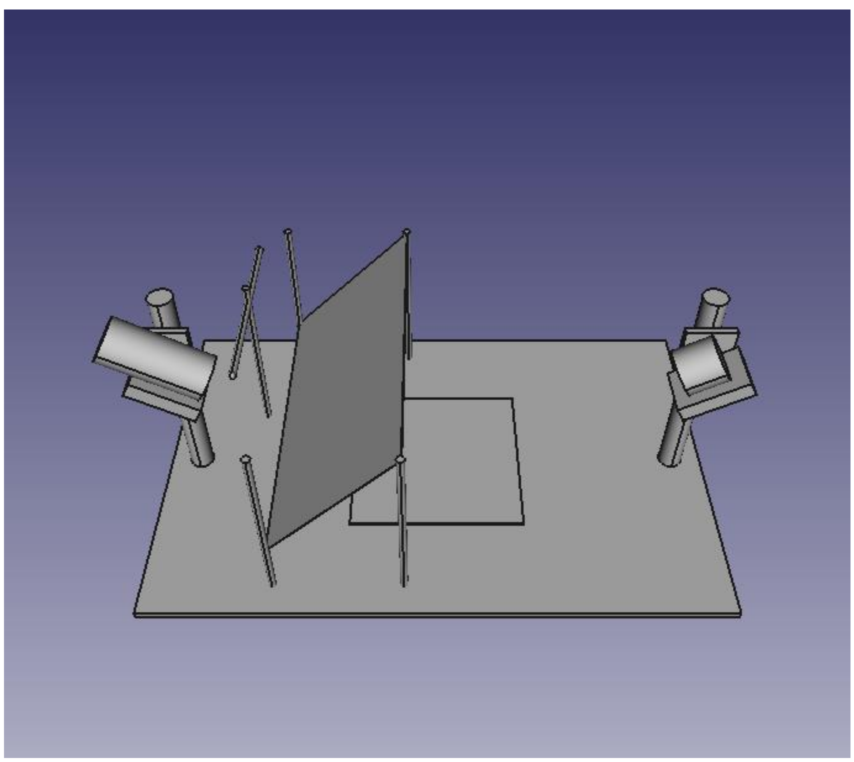

FIG. 2. Initial design setup. From left to right: HeNe laser, ball lens, diffusing film, gelatin film coated glass, and camera.

When this system was put together, there were some immediate problems that resulted in the system not functioning as intended. The first problem was the ball lens did not expand the beam quick enough. New methods had to be found to expand the beam, some methods that were looked at were reflection off a polished ball bearing, expansion through a microscope objective lens, and finally expansion through an engineered diffuser. The engineered diffuser expanded the beam quickly enough but left the light from the HeNe laser too dim to make any observations. At this point testing moved to a higher power laser to assess the different components of the system. Using the engineered diffuser in combination with the diffusing film still yielded no interference patterns, whereas reflective diffusion provided interference patterns. With concerns about the brightness of a laser in a reflective diffusion system, a new concept was designed based on the optical light source used in the initial proof of concept.

The next design process involved creating a broadband light source using two different elemental gas tubes. The wavelengths harmful to the film will be filtered out at the source of the light and the spectral bands of interest for analysis will be filtered out at the camera through use of narrowband bandpass filters. This will allow the light to illuminate the film non-destructively while making precise decisions on the wavelength of interest for thin film measurements.

IV. RESULTS

The first portion of this research involved doing a proof of concept using an optical flat inspection light source. The wavelength of this light source was found to be 546 nm. It was mentioned that the spots were measured using a Filmetrics system and those values were compared to the theoretical values. This is summarized in Table I.

TABLE I: Measurements for initial proof of concept

Point #	Filmetrics Thickness (µm)	Goodness of Fit	m	D	% error
1	5.7089	.95086	27.84	205.04	.57
2	5.6837	.95122	27.62	205.77	.44
3	5.9157	.95609	28.64	206.54	.49
4	6.1323	.95675	29.58	207.3	.27
17	5.9265	.94726	28.48	208.11	.07
18	5.8437	.95152	28.08	208.05	.29
19	5.8291	.94692	28.03	207.96	.11

In Table I the point # references the position on the plate of film, the second column is the measured thickness from the Filmetrics system, the third column is the system-reported goodness of fit value from the Filmetrics system for how well their theoretical values match observed values, the fourth and fifth columns are the values from equation (2) applied in this case, and the final column is the % difference between the "m" value obtained and the nearest integer or ½ integer that would be present for completely constructive or destructive interference. The positions on the plate were chosen based on the goodness of fit values. Analysis was only done for positions on the plate that reported a goodness of fit value greater than .94.

At this point it was apparent that a value was needed for the index of refraction of the film. For the initial proof of concept an estimate was used for the index of refraction based on previous and less sophisticated measurements. In the case of this project a spectrophotometer was used to measure reflection off the surface of the unexposed film. The angle of minimum reflection was used to calculate Brewsters angle for the film. Brewsters angle is related to the index of refraction of the material and thus knowledge of Brewsters angle enabled the calculation of the index of refraction for the unexposed film. This test was done over two wavelength ranges and with a variety of films. On each piece of film 3 trials were done, a curve was fit to the reflection data and the minimum was calculated, then the average angle across the 3 trials was recorded and used to calculate the associated index. The data for 545-555 is summarized in Table II and the data for 625-675 is summarized in Table III

Table II: The measured Brewsters Angle and Associated Index for unexposed film at 545-555 nm wavelength

Measured Brewsters Angle (degrees)	**Associated Index of Refraction**
57.31	1.558
57	1.54
56.975	1.538
57.22	1.552
57.3	1.558
57.24	1.554

Table III: The measured Brewsters Angle and associated index for unexposed film at 625-675 nm wavelength

Measured Brewsters Angle (degrees)	**Associated Index of Refraction**
56.97	1.538
56.85	1.531
57.07	1.544
56.95	1.537
57.11	1.546
57.105	1.546

As discussed in the procedural section, the initial design was put together and tested and multiple problems were encountered. The balls lens combined with the diffusing film did not expand the beam quickly enough to give uniform illumination of the film. Different expansion methods were tested including reflection off a ball bearing, expansion through an objective lens, and an engineered diffuser. Most of these had their own problems, primary of which being the diffusing film seemed to not properly diffuse light to allow the imaging of interference fringes. It was found reflective diffusion from a piece of paper restored fringes with any of the expansion methods but at different distances from the laser light source. The laser beam expanded by the engineered diffuser and reflected off the paper would show fringes almost immediately after beam expansion whereas the expansion through an objective lens and reflected from the paper did not show fringes until the beam was almost entirely expanded.

V. SUMMARY

This was an R&D project to design a system by which photosensitive gelatin film could be measured in a non-destructive and cost-effective manner. The use of an optical flat testing light source was able to prove the concept of applying thin film interference to thin film measurements of the gelatin involved in VPHG's produced by Wasatch Photonics. A spectrophotometer was able to give results for Brewsters angle which enabled the calculation of the index of refraction of unexposed gelatin film, a value that was previously not specifically known and only estimated. This value will be crucial for the continuation of this system design.

An initial system design was built and tested which enabled troubleshooting for a more accurate and thorough updated design. The initial system did not work largely due to the method of diffusion impacting the integrity of the light source such that interference fringes could not be observed. The conditions for interference involve the coherence of the light source as well as the Fresnel-Arago Laws [3]. It is not apparent how the diffusing film caused the laser light source to lose one of these properties but it was shown that interference fringes could be observed with reflective diffusion whereas those fringes could not be observed with the diffusion film used. The troubleshooting on this first system enabled a new system design that deviates significantly from the initial thought at the start of this project. The new system will be modeled after the light source that was originally used. This will require some custom fabrication but should provide the desired results for the overall purpose of this project.

The goal of this project was to produce a production ready device capable of non-destructively measuring film thickness and uniformity. The project began with a general idea but with little to no testing or proof of concept. A production ready device was not achieved within the scope of this project; however, a proof of concept was established and a route towards this final device is outlined.

ACKNOWLEDGEMENTS

A special thanks to Dr. Shen for mentorship during this project. I'd also like to thank to the people of Wasatch Photonics for providing the environment and funding for this project, primarily David Creasy, Elroy Pearson, and Andrew Clawson.

[1] Kim, J., Kim, K., & Pahk, H. J. (2017). Thickness measurement of a transparent thin film using phase change in White-Light Phase-Shift interferometry. *Current Optics and Photonics*, *1*(5), 505–513. https://doi.org/10.3807/copp.2017.1.5.505

[2] Saleh, B. E. A., & Teich, M. C. (2019). *Fundamentals of Photonics, 2 Volume Set*. Wiley.

[3] Hecht, E. (2017). *Optics*. Pearson.