In Situ Color Control
for Reflection Holography

by

Julie L. Walker

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Signature of the Author

Julie L. Walker
Department of Architecture
January 16, 1987

Certified by

Stephen A. Benton
Associate Professor of Media Technology
Thesis Supervisor

Accepted by

Nicholas Negroponte
Chairman
Departmental Committee on Graduate Students
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Abstract

Multi-color reflection holography requires control of the emulsion thickness prior to exposure. Previous methods have involved the removal of the film or plate from the holographic setup between exposures to facilitate swelling, which becomes increasingly difficult when using large format film. This thesis investigates systems that cause the emulsion to come to a swollen equilibrium while remaining registered in the exposure setup.

Thesis Supervisor: Stephen A. Benton
Title: Associate Professor of Media Technology

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Chapter 1

Introduction

The MIT Spatial Imaging Group is currently developing a reflection hologram utilizing an alcove format. This imaging system involves a large amount of pre-compensation in computer image rendering, and sophisticated optics in the holographic printing setup [52] [24]. To date, the alcove format has been tested only with laser transmission reconstruction in one color.

The next step in this project is to approach the question of full color imagery in reflection format. Because of the lack of a suitable pan-chromatic film, and the difficulties involved in operating and aligning several lasers simultaneously, a method of color control that involves one laser (one wavelength) and one piece of film for all exposures is desirable. For such a system, it will be necessary to pre-swell the emulsion before two of the three color exposures (999 separate frames each) that create the 3-dimensional image. Registration of the film is critical as the width of each frame is only one millimeter (a more detailed description of this alcove format stereogram
is given in the previously cited theses \cite{52} \cite{24}). This makes it difficult to remove the film from the setup between exposures to use existing methods of pre-swelling. These techniques require a physical application of a tri-ethanolamine (TEA) solution followed by a careful drying of the emulsion. A new technique must be developed that will swell the emulsion while it remains in the setup, so that the registration of the large piece of film is preserved.

With the TEA method for color control, the emulsion is effectively bulk-loaded with large molecules, preventing a drydown to its original thickness. There are several other ways to control the thickness of the gelatin binder that surrounds the silver bromide crystals, which comprise the emulsion. A balancing of internal and external forces can be observed in the changing color of reflection holograms when exposed to changes in relative humidity, due to the absorption of water by the gelatin. This suggests that one solution to the color control problem is to survey available solvents that are miscible with water to see if they could be used as effective water carriers into the gelatin layer. With such a method, the emulsion should balance internal and external forces and come to some swollen equilibrium just as it does with the moisture in the air. The percentage of water in the particular solvent should determine the amount of swelling.

Another way to affect emulsion thickness is with temperature. Increasing the temperature of the emulsion forces excess water out, causing the emulsion to shrink. It is also possible to cause the emulsion to absorb water from the ambient humidity of the air by cooling the emulsion.

The hardness of the emulsion and the degree of ionization of the solvent
will effect how much the emulsion will swell (or shrink) in a given environment. This thesis looks at some of these factors in detail, describing experimental procedures and results.

First, a short history of color control is presented, intending to show where this thesis fits in with past research on methods for obtaining full color reflection holograms. The second chapter gives background on the physical properties of the emulsion, its structure, and some of the factors that affect its thickness. Because a majority of this research concentrated on the immersion method for color control, a description of these experiments is presented, followed by an analysis of the data. Many other approaches to the problem of emulsion thickness control became apparent as this work progressed. Some of these are described in chapter 6. Finally, a description of the proposed method using a 2-propanol immersion system is presented, along with a brief analysis of the results, and some speculations on the future possibilities for in situ color control.
Chapter 2

History of Color Control for Reflection Holography

The technique of reflection holography was established by Yuri Denisyuk in 1962 [11]. Shortly thereafter, the possibilities of multicolor reflection holography were investigated. Leith and Upatnieks, in 1964 [34], were the first to note the ability to record separate diffraction patterns within a single emulsion using multicolor lasers and hence, reconstruct a multicolor image by presenting the plate with the original reference sources. This idea was extended to the white light viewable Denisyuk reflection hologram by Lin, Pennington, Stroke and Labeyrie [35], and by Upatnieks, Marks and Fedorowicz in 1966 [53]. Both these groups found problems with emulsion shrinkage, resulting in reconstruction at shorter wavelengths. Both corrected shrinkage by omitting the fixing step. (This avoids taking out the undeveloped silver halide, which is what causes shrinkage).

This was not an altogether satisfactory solution to the problem of shrinkage during processing, as the residual silver halide generated a fair amount
of scatter noise, especially in the blue end of the spectrum. In 1967, Lin and LoBianco discussed the use of triethanolamine, referred to here as TEA, to post-swell the emulsion in order to regain the desired red component of the image. In this way, they eliminated the problems of noise generated by the residual silver halide from the fixerless process, allowing the emulsion to shrink, and bringing it back to size by leaving the TEA to dry in the emulsion [36]. This solution was not without its own problems, however. A discussion of the effects of grain size on noise and diffraction efficiency is given by Benton [1].

It is interesting to note that the use of TEA was originated by Denisyuk in 1963 [12] as a sensitizing agent for his emulsion. At that time he discovered that all of the TEA was washed out during normal processing, thereby distorting the recorded fringe pattern. He had to reswell with the same percentage TEA solution in order to regain the recording wavelength.

Nishida [38] continued the work of Lin and LoBianco with a systematic look at the long term effects of TEA post-swelling and the effects of optical density and humidity on the diffraction efficiency. The efficiency was measured by reconstructing the “corrected” (post-swelled) emulsion with the original laser reference beam and by measuring the intensity of the output. Nishida found that as the humidity changed, so did the diffraction efficiency.

At this time, white light color holography was limited to multiple wavelength exposures on one piece of film, and diffraction efficiency was a major problem. The only panchromatic emulsion was (and still is) Kodak 649F. This emulsion was not developed specifically for holography, and has some
drawbacks for that application. The sensitivity is low, and its scattering is relatively high due to large grain size [23], a high concentration of silver halide [36], and a relatively thick emulsion (approximately 12 microns) [19]. This made multicolor holography using several wavelengths less than satisfactory.

Researchers tried to overcome the problems with Kodak 649F by searching for other emulsion possibilities. A group in Czechoslovakia successfully extended the sensitivity of Agfa 8E56 into the red in 1974 (Muzik and Ruzek as cited by Hariharan [23]). Others worked with dichromated gelatin to extend its sensitivity into the red with methylene blue. Graube was the first to suggest this in 1973 [15], and his work was followed up by Kubota, Ose, Sasaki, and Honda in 1976 [31], and Kubota and Ose in 1979 [32].

Hariharan in 1980 [20] used a sandwich technique for a multi-color image, using the thinner 8E56 and 8E75 emulsions to record separate diffraction patterns. The red exposure was recorded on the 8E75 emulsion using a HeNe laser and the green and blue exposures were recorded on the 8E56 emulsion using the two suitable lines from an Argon laser. The two emulsions were sandwiched together for the final full color image. In this way the problems inherent in the Kodak 649F emulsion, and the reduced diffraction efficiency due to successive incoherent exposures on the same plate were avoided. Hariharan also suggested the use of D-sorbitol as a post-swelling agent to minimize the progressive darkening or "print out" effect so evident with TEA.

Kubota in 1981 [33] did similar work with the combination of emulsions in a sandwich format. His work involved a combination of silver halide
emulsion for the red exposure and a dichromated gel emulsion for the green and blue exposures.

Another aspect of research aimed at solving problems of shrinkage and low diffraction efficiency was a concentrated investigation of new processing techniques to gain a better understanding of the theoretical and physical properties of the available emulsions. Hariharan 1971 [16] discussed the suppression of printout by converting the remaining silver halide into silver iodide, which had been found to be the most light-stable of the halides. In 1972 he published a paper on the advantages of reversal bleaching enabling higher diffraction efficiencies and less noise, but provided no solution to the shrinkage problem [17].

In 1974, Benton developed the IEDT process [2]. This process made possible the development of efficient red reflection holograms without the need to post-swell the emulsion. In this process, fixer is not used to dissolve the undeveloped silver halide. Instead, the silver halide is made to migrate to the already developed sites where its reduction to metallic silver occurs. A rehalogenating bleach then converts these enlarged silver deposits back to silver halide, gaining a higher diffraction efficiency. There is no loss of silver halide in this process, hence no shrinkage of the emulsion. The details of the IEDT process were published in Holosphere 1978 [4].

Research continued in the area of low noise, high efficiency bleaches. Joly and Vanhorebeek [27], and van Renesse [54] in 1980, made important contributions in the theory and understanding of the mechanisms involved. Also in 1980, Nick Phillips developed the PBQ bleach chemistry [40], giving the holographer an efficient, low noise process, at the expense of high
toxicity of the components. In 1981 Walter Spierings reported the use of "pyrochrome" processing developed by van Renesse. This yielded high efficiency and hardening to obtain "results...comparable with some recent holograms made by NJ Phillips" [42]. Pyrochrome proved to be simple and quick, without the critical controls required by the IEDT or PBQ processes.

After the advent of a chemical procedure which enabled the playback of the original recording wavelength without the need for post-swelling, came the idea that color could be controlled by pre-swelling the emulsion. It was not until 1979 that this concept of pre-swelling was reported in a short paper by Jeff Blyth [5] and by Hariharan in 1980 [22], 16 years after its unrecognized discovery by Denisyuk [12]. An interesting note is that Kubota and Ose in their 1979 paper on dichromated gels also described the use of TEA and other possible swelling solutions to be baked into the gelatin as a pre-swelling agent [32]. Blyth demonstrated his technique using TEA as the pre-swelling agent in conjunction with an IEDT-like processing that would yield close to the original recording wavelength upon reconstruction with white light.

The IEDT process was reported to be difficult to use. The combination of the pre-swelling technique with pyrochrome processing gave a system for color reflection holography that was easy and convenient, using the standard HeNe laser and readily available chemistry.

Hariharan combined the sandwich of two emulsions with the pre-swell method. This system required only one laser and the pre-swelling of only the plate that would record the green and the blue exposures [22]. The first to popularize the TEA method by making it accessible to the growing com-
munity of art holographers were Lon Moore [37] and John Kaufman [29]. Both were able to refine this technique, and present it at the First International Symposium on Display Holography at Lake Forest in 1982. Smith and Cvetkovich [41] later quantified the process in more detail, presenting some starting points for calibration and color work.

Since its inception in 1979, the technique of pre-swelling has not received much research attention. Few swelling agents other than TEA have been investigated, although the use of ethylene glycol and glycerin were mentioned in the paper on dichromated gels by Kubota and Ose in 1979 [32], and D-sorbitol was mentioned by Hariharan as a post-swelling agent [20].

It is time to address the problem of color control with a concern for convenience. Holograms are becoming larger and their geometries more complex, limiting the ability to physically smooth the surface of the emulsion in order to facilitate an even drying, which is necessary with the TEA process. A new type of emulsion control that will enable the holographer to control thickness while the emulsion remains registered on the table is necessary. This control, in conjunction with new advances in computer generated imagery, increased ability to compute predistortions, and to generate simple color separations of a scene or object will define the look of a new class of holographic technology.
Chapter 3

Gelatin

3.1 Background

Two factors are involved in obtaining multi-color holograms using one recording wavelength. Appropriate processing is necessary for the hologram to replay the same color with which it was recorded. And, a system to swell the emulsion before exposure is needed to obtain shorter reconstructed wavelengths.

TEA has been used as a pre-swelling agent for the color control of reflection holograms since 1979 [5] [22]. This involves taking the film or plate off the table between exposures to soak in a TEA/water solution, then spinning, or "squeegeeing" the emulsion to facilitate an even coating, and letting the emulsion dry. It is also necessary to clean the back of the substrate before exposure to obtain uniform color. At best, this is an arduous task, and the plate holder must have the ability to accurately re-register the holographic emulsion.

A change in humidity can change the color of a hologram. Water ab-
sorbed from the atmosphere affects the balance of internal forces in the gelatin network, causing it to come to a new equilibrium thickness. It may be possible that the moisture content in some substance other than air would also allow these forces to re-equilibrate. Immersing the emulsion in a solution with differing concentrations of water might allow the gelatin to swell to a stable equilibrium. There are many other factors which affect how much the gelatin will swell in a particular environment. Temperature, pH, salt concentration, and the hardness of the emulsion all affect how the gelatin will react to moisture. To understand how these factors affect the emulsion, one must examine the chemical and physical properties of gelatins.

3.2 Structure

The gelatin emulsion binder can be described as a mass of tangled protein chains, with crosslinking bonds that run between them. The silver halide crystals are embedded in this mass, the ends of some protein chains adsorbed into the surface of the silver halide grain. This network incorporates a moisture content, typically ten to fifteen percent [13]. These water molecules keep the gelatin structure from collapsing, while the protein network keeps the moisture trapped inside [48].

Gelatin is made from processed collagen. The primary structure of the protein molecule incorporates eighteen different amino acids. The amino acid composition and molecular weight (20,000 to 200,000) are dependent on the source of the collagen and the method employed in its conversion.
to gelatin [9]. Glycine, proline, alanine and hydroxyproline constitute approximately sixty-five percent of the collagen amino acids [7]. These amino acids are covalently linked in a large number of semi-random sequences (some sequences are found more often than others).

The osmotic pressure of a gel, which determines the gelatin equilibrium, has three components: the polymer-polymer affinity, the rubber elasticity, and the hydrogen ion pressure [48]. Although these forces have been described primarily with respect to a non-ionic polyacrylamide gel, there are similarities with collagen gelatin.

The polymer-polymer affinity is comparable to the attraction of the peptide chain to bond to itself (by hydrogen bonding), or to bond to other peptide chains in its vicinity. This force acts to limit the swelling of the gelatin.

The elasticity of the gelatin is the ability of the peptide chain to stretch or to compress. The acrylamide gel can be represented by short crosslinks with single flexible chains between them. The random movements of these short segments hold the emulsion at its desired thickness. As the gel swells away from equilibrium, these movements pull the peptide chain together. As the gel shrinks, these movements push the peptide chains apart. The crosslinks in (collagen) gelatin, however, involve multiple interactions between chains forming “fibrils” [30]. This reduces the effect predicted by elasticity as compared to the polyacrylamide gel.

The hydrogen ion pressure is produced by the random movements of the hydrogen ions in the gelatin network. These movements are much like those of a contained gas, presenting an outward force to the boundary of
the gelatin.

In addition to these three forces described for a non-ionic gel, there are forces in the collagen gelatin due to its highly ionized nature. There is an equilibrium point at which the attractive and repulsive electrical forces balance with the molecules at some distance from each other.

### 3.3 Swelling

A gelatin swells in solutions in which it is soluble. Crosslinks within the gelatin structure prevent the gelatin from going into solution in cold water. Further crosslinking is necessary to prevent the gelatin from dissolving at higher temperatures.

A gelatin, in its dry state, has its polypeptide chains folded upon one another, linked by hydrogen bonds. These bonds are released in the presence of water in favor of hydration, causing the protein molecules to unfold and the gelatin to swell. The water molecules attach to the backbone of the polypeptide structure.

The gelatin swells until the swelling pressure is in balance with the internal forces of the network. This is not the swelling equilibrium point, however, as there is an additional osmotic effect.

The osmotic pressure is a function of the difference in ionic character between the solution and the interior of the gelatin. The surface of the gelatin acts as a semi-permeable membrane, through which the gelatin molecules cannot diffuse. Hydrogen ions from the ionized groups on the protein chain cannot diffuse out without disruption of the local electrical
neutrality. Hence, water will continue to be drawn into the gelatin, producing a further extension of the network. This osmotic swelling is known as the Donnan equilibrium [30].

3.4 Factors that Affect Swelling

The degree of ionization of the immersion solution, and the amount of bonding within the gelatin (increased by “hardeners”) will both affect the gelatin’s swelling characteristics. The ionization of the solution can be changed either by adjusting the pH or by the addition of a salt.

The pH affects swelling of the gelatin in several ways. The amount of folding of the amino-acid chain depends on how close the pH of the solution is to the isoionic pH of the gelatin [9]. The maximum amount of folding occurs at the isoionic point due to the attraction of even numbers of opposite charges. Swelling increases with the solution pH on either side of the isoionic point. The effects of pH at extreme acidity or alkalinity are more complex. When an acid or base is added to a solution, excess ions are added too. The effect of these excess “counter” ions is to reduce the forces between charges causing a reduction in the amount of swelling [43].

The Donnan equilibrium is also dependent on the pH of the solution, since it depends on the net charge on the polypeptide molecule. Swelling will increase on either side of the isoelectric point, until the ion concentration gets too high at extreme pH values, where swelling will again decrease [30].

The addition of salt to the solution can either increase or decrease the
amount of emulsion swelling. At the isoelectric point, where there is no
Donnan effect, the addition of salt reduces the attraction between oppo-
sitely charged groups on the gelatin chain, thus swelling the gelatin. Away
from the isoelectric pH, the salt will reduce the osmotic effect caused by
the pH imbalance, thereby causing the gelatin to swell less.

The gelatin consists of a three dimensional network of polypeptide
molecules where each molecule is linked to those in close proximity by (rela-
tively weak) hydrogen bonds at sites along its backbone. The structure can
be strengthened by the addition of a hardener which increases the amount
of cross-link bonding between these proteins. This will limit the amount
of possible swelling of the gelatin, and may even impede the diffusion of
molecules with molecular weights between two hundred and three hundred
[9].

There are a multitude of possible reagents that act as crosslinkers with
the peptide protein. Aldehydes and ketones are included in the long list of
covalent hardeners. Formaldehyde is one of the most widely used crosslink-
ing agents for protein [7]. Its reactions take place at the amino groups on
the peptide chain. Sulfate esters have been used as photographic hardeners,
possibly forming ionic bonds with the protein. Chromium, one of the earli-
est tanning agents, reacts by the formation of a bond between a chromium
ion and the carboxyl groups of two gelatin molecules, along with encourag-
ing some degree of hydrogen bonding between peptide molecules [9]. These
factors, the ionization of the solution and the degree of hardening in the
gelatin, can be used to fine tune the proposed system.
Chapter 4

Immersion Experiments

4.1 Overview - Theory and Equipment

The technique of swelling the emulsion during exposure as a means of color control was developed in 1979 [5] [22]. Since then, the standard swelling method has been to soak the emulsion in an aqueous solution of triethanolamine and dry it before exposure. The gelatin structure traps the large TEA molecules, preventing the full collapse of the gelatin structure. After exposure, the TEA is released from the emulsion in the developer, and the emulsion is allowed to dry to its normal thickness. A search of the literature to date revealed no detailed research into other methods of pre-swelling. This chapter describes an investigation into the possibilities of bringing the emulsion to a swollen equilibrium by balancing the internal and external forces on the gelatin.

The methodology of these experiments considered the change in the wavelength reflected by a simple holographic grating to be an indication of a change in thickness of the emulsion. If the wavelength shift could be
controlled for an existing grating, then perhaps the same forces (techniques) could be applied to an emulsion before exposure, facilitating a shift in the final color of the hologram.

A system was developed to observe a grating in a given environment, while various forces were applied in an attempt to change its thickness. This involved measuring and recording the response of a holographic planar grating (i.e., one with fringes parallel to the surface) with a spectroradiometer and an xy-plotter.

The setup consisted of an immersion plateholder and the monochrometer/photomultiplier tube positioned on an optical rail. An immersed grating was illuminated by a tungsten source (microscope illuminator). The light entering the radiometer consisted of light reflecting from the surface of the glass, plus the diffracted light from the grating (figure 4.1).

An International Light Spectroradiometer Model 700A equipped with a holographic grating with a blaze of 500 nm was used in conjunction with an IL photomultiplier tube model 760. The output of the photomultiplier was connected to the y axis of the xy-recorder, and the x axis driven by an electrical link to the position of the grating in the monochromometer. All equipment, including the illumination light, received power from a voltage and frequency stabilized, noise filtered power source, as fluctuations from the direct power line were apparent.
Figure 4.1: Diagram of radiometer setup
4.2 Preparation of Test Gratings

The gratings used were exposed on Agfa 8E75 HD emulsions (all from batch number 598802-N) with a helium-neon laser. The emulsion was index-matched to a front surface mirror and exposed with a collimated beam at zero degrees to the normal of the plate. They were then processed without hardening, fixed, and rehalogenated in bromine water in an attempt to simulate an unused emulsion. The peak wavelength varied with the exposure, as it was dependent on the collapse of the emulsion due to the removal of the undeveloped silver by the fixer. Most of the gratings had an initial peak wavelength between 500nm and 550nm.

4.3 Solvent Immersion

The theory behind the solvent immersion technique considers moisture in the air and moisture dissolved within a solvent to be equivalent in the manner in which they affect the gelatin. Thus, as the gelatin thickness reaches an equilibrium with the ambient humidity, so should the emulsion reach an equilibrium with the moisture dissolved within a solvent.

4.3.1 Initial Testing

Most of the solvents were initially tested for their effect on the emulsion by observation with a spot test. In this test, a drop of solvent was placed on the emulsion and a prism wedge placed on top. The index-matched prism enabled observations of the color of the grating separately from the
reflected light source. The suitable solvents (those that did not themselves swell the emulsion and were miscible with water) were investigated further using the immersion/radiometer setup.

4.3.2 Immersion Procedure

The immersion plate holder, light source, monochrometer and photomultiplier tube were placed in a fume hood to avoid noxious vapors from the various solvents. The profile of a dry grating was measured, keeping record of the ambient humidity and temperature.

The grating was immersed in 100 percent solvent. The starting time and wavelength setting of the monochrometer were recorded so that the time of the peak output could be derived. This was necessary because the scan moved at a rate of 100 nm/min, relatively slowly compared with some of the wavelength shifts of the emulsion. The motor was later changed to increase the scan rate to 300 nm/min to gain a higher resolution of the change in peak wavelength as a function of time. The grating was monitored from the time of immersion to the time of equilibrium with no agitation of the solution. Equilibrium was determined to be the point at which there was no change in the profile for several traces (eight to ten minutes), indicating little movement of the fringe structure within the emulsion.

Once equilibrium was obtained for the 100 percent solution, the grating was removed and allowed to dry. A 7.5 percent (by volume) water in solvent solution was mixed, by adding the appropriate amount of water to the previously used solvent. The same procedure was followed as for the
100 percent solution, immersing the plate into solution and repeating the scan until equilibrium was obtained.

The grating was dried between solutions because uneven swelling occurred if the solution was poured over the grating while in the plateholder, or if additional water to change the concentration was added slowly with agitation. If the 7.5 percent solution equilibrated in less than 25 minutes, the grating was again dried, immersed in a 15 percent water/solvent solution, and the changing spectral reflectance profile recorded.

4.4 Observations

4.4.1 Solvent Solutions

A grating with its initial reflectance peak in the blue region of the spectrum was immersed into 100 percent methanol. It was found that methanol alone swelled the immersed grating, making it unsuitable as a simple water carrier.

Another grating was immersed in 100 percent ethanol, producing a slight amount of shrinkage. The sequential profiles of the grating in a 7.5 percent solution showed the swelling response of the emulsion over time. In this particular case, the peak moved a small amount, then split into two peaks, the first losing efficiency while the second gained efficiency. This continued until the peak had completely moved to a new location.

A solution of 1-propanol and water acted very similarly to the ethanol and water solution by first losing efficiency, then increasing bandwidth and
very slowly coming to a new swollen equilibrium wavelength. Neither 1-propanol or ethanol were tested at higher water concentrations.

A solution of 2-propanol, on the other hand, moved the peak comparatively quickly, settling down within 10 minutes of initial immersion. The peak for the 7.5 percent 2-propanol solution simply shifted, without losing efficiency. In the 15 percent solution, however, the grating lost efficiency at the initial peak, while it gained efficiency at a higher wavelength (figure 4.2). Again, the time to final equilibrium was approximately 10 minutes.

A 7.5 percent solution of water in 1-butanol swelled the grating very quickly initially but did not reach an equilibrium point in less than 25 minutes. 2-butanol had a very fast equilibrium for the 7.5 percent water/alcohol solution, less than 10 minutes, but the higher concentration took much longer. In addition, it was observed that the 2-butanol solution swelled the grating farther than the same concentration solution with 2-propanol.

The 3-Pentanol mixture needed to be vigorously agitated to mix the alcohol and water. The 7.5 percent solution swelled the grating beyond the range of the monochrometer system (800nm), so only smaller percentages were tracked. In a 4 percent solution, the grating swelled quickly at first, then took an extended period of time to come to equilibrium.

A few other hydrogen bonded solvents were investigated. These included glycerol, acetone, and ethylene glycol. The acetone solution showed a relatively slow moving profile.

Ethylene glycol, in a spot test (section 4.3.1), reticulated the grating
Figure 4.2: Change in grating profile for 15 percent water/2-propanol solution in order of increasing wavelengths.
while shrinking it. When exposed to air, it had a strong hygroscopic action causing the grating to swell past the red. Ethylene glycol was not studied further.

Glycerin shrunk the grating in a spot test and seemed to come to a stable thickness in a reasonable amount of time. Theoretically there should be a concentration of water and glycerin that would hold the grating at a stable equilibrium, with the ability to swell with higher concentrations of water. The disadvantages of glycerin include its very high viscosity and its slow drying.

4.4.2 Addition of Heat to the Solvent Solution

The temperature of the solvent solution is another way to control the amount of moisture in the gelatin and hence to control its thickness. Swelling was demonstrated with increasing temperature of the solvent bath. This is contrary to effects of increasing the temperature of an emulsion in air. A plate immersed in 7.5 percent water/2-propanol was taken from room temperature to the boiling point of the mixture (approximately 50\(^\circ\) C). This was accomplished by heating (and stirring) the solution with an immersion heater. The wavelength shifted toward red with increased temperature. However, the amount of shift was not sufficient to swell the grating enough to allow a blue output with the intended process. Also, the swelling did not seem to be reversible as the grating did not shrink to its original thickness when the solution cooled.
4.4.3 Changes in Ionization of the Solvent Solution

The ionization of the solvent mix also effects how the emulsion reacts to changing amounts of moisture. A grating was placed in a 2-propanol solution mixed with 7.5 percent water and several drops of hydrobromic acid (the pH of the water was 1.5). The grating that was put in a low pH solvent solution swelled slightly more than in the corresponding neutral solvent solution. In addition, this grating showed a memory of the increased swelling, which was seen in a subsequent run of neutral solvent mixes. The effect of the pH change was not enough, by itself, to swell the grating within the necessary range for this application. No further investigations of pH effects were undertaken.

There may be an optimum pH for the solvent solutions that minimizes the time to equilibrium. At low pH, it was observed that the 7.5 percent water/2-propanol solution came to equilibrium faster than with the neutral water solution. However, the 15 percent solution was affected by low pH in the opposite way. It took much longer for the emulsion to stop swelling.

Another way to affect the ionization of the grating is to add a salt. The pH test was done using HBr instead of a chlorine compound because chlorine has a tendency to attack the silver bromide, damaging the emulsion. For the same reason, NaBr was chosen for the ionization test instead of NaCl. A saturated salt (NaBr) solution was used in place of pure water in a 7.5 percent isopropyl solution. The grating swelled much further with the salt solution, however, this process extended the time to equilibrium.

It is necessary to take a closer look at the data to analyze which of these
methods hold the most promise of practicality for the process involved in actually making full color holograms. The data produced by solvent immersion tests is reviewed in chapter 5.
Chapter 5

Data Analysis for Solvent Immersion

The spectral profile of light reflected by a grating was obtained using the radiometer setup described in chapter 4. The initial thickness of the emulsion was considered to be its thickness when soaking in 100 percent solvent. This 100 percent reference point was used as a means of minimizing the variable swelling effects of ambient humidity. The equilibrium wavelength (the peak wavelength after the emulsion had stabilized in the solvent mix) was taken to be a measure of the change in thickness of the emulsion, as related to the initial wavelength (thickness) of the grating. The percentage of swelling was calculated from the difference between the initial wavelength and the equilibrium wavelength. The grating profiles and peak wavelengths were supplied to a computer via a digitizing tablet and pen so that the data could be normalized to the light source and graphed.
5.1 Solvent Characteristics

The interactions of the gelatin with its surrounding environment are complex. Figure 5.1 shows the molecular configurations of the solvents that were investigated. Each of these solvents produced characteristic changes in grating profile, time of equilibrium, and amounts of swelling from various percentages of dissolved water. These observed variations may be dependent on the solvent’s affinity for water versus the gelatin’s affinity for water. This attraction is probably dependent on the structure and molecular weight of a solvent. Therefore, the amount of swelling and time to equilibrium were analyzed first with respect to each solvent’s molecular weight and structure. Hydrogen-bonded solvents were considered because of their high miscibility with water.

The amount of swelling of the emulsion in a given percentage of water varies with both molecular weight and molecular configuration of the solvent. The comparison of the percentage swell of an emulsion in 1-propanol and 2-propanol, and 1-butanol and 2-butanol solutions (figure 5.2), shows the differences in the amount of water absorbed due to the change in structure of molecules with identical molecular weights. The data indicates that the emulsion swells more if the solvent carrier has an hydroxyl group on the end of the molecule, rather than in the center of the molecule where it is surrounded by hydrogen atoms.

The dependence of the percent of swell on the molecular weight of the solvent is more ambiguous. Methanol, the lightest of the alcohols, caused the emulsion to swell without the addition of water, whereas the other alco-
METHANOL

H
\[
| \]
H--C--OH
\[
| \]
H

1-PROPANOL

H H H
\[
| \]
H--C--C--OH
\[
| \]
H H H

1-BUTANOL

H H H H
\[
| \]
H--C--C--C--OH
\[
| \]
H H H H

3-PENTANOL

H H OH H H
\[
| \]
H--C--C--C--C--OH
\[
| \]
H H H H H

ETHANOL

H H
\[
| \]
H--C--C--OH
\[
| \]
H H

2-PROPANOL

H OH H
\[
| \]
H--C--C--C--H
\[
| \]
H H H

2-BUTANOL

H H OH H
\[
| \]
H--C--C--C--C--H
\[
| \]
H H H H

3-PENTANOL

H OH H H H
\[
| \]
H--C--C--C--C--C--H
\[
| \]
H H H H H

ACETONE

H OH
\[
| \]
H--C--C--H
\[
| \]
H H H

Figure 5.1: Molecular structure of investigated solvents.
hols investigated shrank the emulsion with no water present. The amount of swelling with a given amount of dissolved water initially decreased with increasing molecular weight reaching a low with 2-propanol, then increased with solutions of increasing molecular weight (figure 5.3).

From this data alone, a mixture might be chosen to allow the greatest amount of swelling with the least amount of water. However, some of these solutions allowed the emulsion to reach its equilibrium thickness only after extended periods of time.

The time of equilibrium is charted in figures 5.4 and 5.5. Most of the alcohol solutions reached an equilibrium point at times greater than thirty minutes. 2-propanol and 2-butanol, however, swelled relatively quickly, stabilizing in less than ten minutes with the 7.5 percent solutions. As can be seen from the comparison of these two solvents at a higher concentration (figure 5.6), 2-propanol is the more suitable swelling agent for this application.

5.2 Experimental Results

In order to obtain final wavelengths of 620nm, 520nm, and 470nm, it is necessary to pre-swell the emulsion by 4 percent, 24 percent, and 38 percent respectively (assuming the chemistry is properly chosen to replay the original recording wavelength when exposing an untreated emulsion). A 38 percent swell corresponded to a 15 percent solution of water in 2-propanol
Figure 5.2: The amount of swelling due to various solvent carriers.
Figure 5.3: The amount of swelling as compared to the molecular weight of various solvent carriers.
Percent Swell vs. Time for Various Solvents

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Solvent</th>
</tr>
</thead>
<tbody>
<tr>
<td>▼</td>
<td>7.5% Water/Ethanol</td>
</tr>
<tr>
<td>□</td>
<td>7.5% Water/2-Propanol/NaBr</td>
</tr>
<tr>
<td>*</td>
<td>7.5% Water/1-Propanol</td>
</tr>
<tr>
<td>+</td>
<td>7.5% Water/1-Butanol</td>
</tr>
<tr>
<td>----</td>
<td>Estimated from off range data point</td>
</tr>
</tbody>
</table>

Figure 5.4: Comparison of swelling times for various solvents.
Figure 5.5: Comparison of swelling times for various solvents.
Figure 5.6: Comparison of swelling times for 15 percent solutions with 2-propanol and 2-butanol.
(figure 5.6) when measured from a processed grating. Initially, this processed, rehalogenated emulsion was considered to simulate an unused emulsion. A discrepancy was found, however, when comparing the calculated percent swell with the final wavelength of the emulsion pre-swelled before exposure. Experimental results showed the percent of swelling to be less for emulsions swollen prior to processing than for processed gratings swollen in the same percentage water/2-propanol solution. A higher percentage of water was necessary to obtain the 38 percent pre-swell than was determined from the processed grating. Figure 5.7 shows the experimental values for the final wavelengths for pre-swelled exposures in various solutions.

To obtain a wavelength of 470nm (ie. a 38 percent swell) by pre-swelling an unprocessed emulsion, it was necessary to use a solution of 21.5 percent water in 2-propanol. There are several reasons for the discrepancy between this and the corresponding percentage solution for the processed grating (15 percent water for a 38 percent swell). In order to compare various solvents, using different gratings on days with differing relative humidity, it was necessary to normalize the data to a consistent reference point. This reference point was chosen to be the output of the grating while in a 100 percent solvent solution, as this was independent of the environment. The pre-swelled gratings, however, dry down to a thickness that is larger (approximately 5 percent) depending on the relative humidity. Another reason for the discrepancy is that the gelatin is altered by its immersion in water and by exposure to differing pH solutions during development. When a gelatin dries from its “sol” state, hydrogen-bonds are formed. Some of
Final wavelength vs. Percent Water in 2-Propanol Pre-Swell

Figure 5.7: Final wavelength as a function of 2-propanol percentage solutions with and without pre-soaking the emulsion.
these bonds are easily broken when immersed in water or other softening agents, and do not reform upon drying.

In addition to the discrepancy in the amount of swelling in various concentrations of water in 2-propanol, there appeared a swelling anomaly for the 15 through 17 percent solutions. For these solutions, the emulsion partially swelled (or swelled much more slowly), causing two gratings of different fringe separations to be present in the processed emulsion (figure 5.7). This was partially corrected by a 3 minute soak in water, followed by a graded drying with methanol (2 minute soaks in 50:50, 75:25 and 100 percent methanol/water solutions) before the immersion in the swelling solution. Increasing the time of the pre-wet, or the temperature, or pH of the water may be more effective in further reducing this anomaly. Other emulsion softening agents may also be effective. A 3 minute soak time was sufficient for this project to obtain the calculated final wavelengths of 620nm, 520nm and 470nm (corresponding to 7.5 percent, 15 percent and 21.5 percent water/2-propanol solutions).

5.3 Remarks

The effects of changing the emulsion’s immediate environment was investigated. It was found that the emulsion responded by reaching a swollen equilibrium within the necessary range for obtaining blue holograms from a red laser. The effects on the emulsion by immersion in various water miscible solvents were considered. Most of the water solutions tested produced a swelling equilibrium. However, 2-propanol seemed to be the most practi-
cal of the solvents in that it had an equilibrium time of approximately ten minutes with good repeatability. Using this data, it is possible to predict the final wavelengths of a multicolor exposure.
Chapter 6

Other Possibilities for Emulsion Thickness Control

This chapter will discuss some of the possibilities that became apparent during the investigation of the solvent immersion system. The most promising of these ideas seems to be the use of heat to control the amount of moisture in a dry grating.

6.1 Conducted Heat

6.1.1 Heatpump - Heating and Cooling

While the effect of solvent temperature on an immersed grating showed little promise as a method for controlling thickness, the effect of temperature on a dry grating was dramatic. Using a thermoelectric heat pump, a grating with output wavelength in the middle of the spectrum was observed. Cooling the grating caused the emulsion to swell past the visible red, while heating removed the moisture and shrunk the emulsion into the
blue. This process was completely reversible as long as the melting temperature of the emulsion (60°C) was not exceeded. Excessive temperature broke the H-bonds in the gelatin, softening the emulsion. The effect of this was an emulsion that collapsed to a shrunken equilibrium upon returning to room temperature. This was observed with a section of one of the gratings that was allowed to heat for an extended period of time. The majority of the grating reflected a green wavelength upon cooling to room temperature. However, the section of the grating that was heated to excessive temperatures reflected a deep blue wavelength.

The prospect of simply cooling to swell the emulsion is very attractive, however, the amount of swelling with change in temperature may depend on the ambient humidity. In addition, cooling the emulsion may cause condensation to form on its surface. These factors could make this method difficult to use without also controlling the humidity.

6.1.2 Temperature Controlled Plate

The temperature of a resistive plate (heated with an AC current) was controlled by a YSI model 72 temperature controller. The substrate of a dry grating was index matched to the heating plate with mineral oil. The temperature of the emulsion was measured with a small surface thermocouple probe, while the changes in reflected wavelength were monitored (see section 4.1 for a description of the monitoring system).

The temperature of the emulsion was held constant to within half a degree. The emulsion shrank with each incremental increase in temperature
to a new stable thickness. It may be possible to use this technique to obtain a blue wavelength from a red laser by first pre-swelling the emulsion past the thickness necessary for the blue, and shrinking the emulsion to the proper thickness for each exposure with increasing temperatures.

This procedure was repeated with a TEA pre-swollen grating with similar results. However the range of shrinking is limited by the physical properties of the bulk-loaded emulsion. A hygroscopic agent is needed that will hold moisture from the ambient humidity in the emulsion while at room temperature, and release it at increasing temperatures. This would cause the emulsion to shrink to the correct "swollen" thickness to obtain the shorter wavelengths. Using a transparent resistive coating on glass, and a temperature controller, the temperature of the emulsion could be kept constant during exposure. There are many possibilities for hygroscopic agents that might be suitable for this application. Salts are possible agents as they display a wide range of effects on the emulsion [30].

This method seems promising, although it may be necessary to search for a swelling agent that would release moisture at high temperatures and subsequently retain a thickness dependent on the maximum temperature achieved before cooling. In this way thermal instabilities may be avoided as exposure could take place at room temperature.
6.2 Other Possibilities

6.2.1 Electric Field

The method described in section 6.1.2, is attractive in that it would enable the control of emulsion thickness by turning a dial, without the need to change solvent baths etc.. Another possibility that would involve similar interaction is to subject the emulsion to a variable electric field which would mobilize the ionized carboxyl groups. This has been shown to be very effective in bringing about a “volume phase transition” (a discrete change in volume) in polyacrylamide gels [49]. This method was investigated in a preliminary way. However, problems arose due to the non-conductive nature of the emulsion substrate.

6.2.2 Hardeners

The possibility of using a hardener bath that would limit the amount of swelling to a specific thickness while immersed was briefly considered. It was found that the hardener in solution did not limit the amount of swelling within the necessary range. Most of the crosslinking of the gelatin takes place during drying of the emulsion. For example, a sixteen percent (by weight) solution of chromium trioxide was found to swell a grating past the visible upon immersion (too far for this application), and substantially shrink the emulsion upon drying. Hardeners may be useful in affecting the characteristics of the swelling in other agents, but was not found to be useful as a means of controlling the emulsion thickness by itself.
6.2.3 Humidity

The prospect of simply controlling the humidity of the emulsion’s immediate environment was also considered, though not pursued experimentally. It was observed that gratings responded very quickly to gross changes in humidity, by displacing the air within a Ziploc™ bag, with dry gas (freon 21). This method of emulsion control warrants more investigation. Different gases could increase the range and speed of swelling for particular relative humidities. However, the control of humidity is a substantial task as the temperature must also be controlled, and condensation must be avoided.

6.3 Discussion

The use of a temperature controlled element showed promise in controlling the thickness of the emulsion as the observed response to increasing temperature was fast, and stable. However, the stability of the optical setup may be a problem with a heating element on the table. Changes in humidity also produced a fast response from the emulsion, but the technology needed for such a system would be complex, as temperature in addition to moisture content of the ambient air would need to be controlled. Simply cooling the emulsion has the disadvantage of possibly creating condensation on its surface.

At present, the immersion method shows the most promise as a practical solution to the problem of in situ color control for reflection holography. This method requires a minimum of equipment making it technically...
pragmatic and economically feasible for most labs. A description of the developed immersion technique, using the data from chapter 5, is presented in chapter 7.
Chapter 7

Proposed Method Using 2-Propanol

7.1 Preparation

A multicolor reflection hologram must be made in 3 separate exposures, each of a different color separation of the image. A chart was designed to show overlap in colors of successive exposures. Solid red, green, and blue areas were included, as well as intermediate overlapping colors and a greyscale (figure 7.1). The color separations of the chart were exposed onto Ektachrome slide film. Master holograms were made of these separations projected onto a rear projection screen using the 647nm emission of a krypton ion laser.

In another setup, these masters were focused into a specially designed and fabricated immersion plateholder. The plateholder had a sloping bottom with a drain spout to facilitate quick removal of the solution. The internal width of the cell was just wide enough to load the plate, keeping
Figure 7.1: Color separations for test chart.
the volume to a minimum (300ml). The plate was held by a 3 point system, consisting of two "V" shaped wedges, to hold the bottom of the plate, and a "V" shaped wedge on a hinged arm that stabilized the top of the plate. Loading the plate involved lowering it into the cell, then moving the hinged arm into place. Along one vertical edge of the plateholder was a separate filling channel, open to the main cell at the bottom. This was necessary so that the solution did not pour over the plate in an uneven manner, causing streaks (figure 7.2).

According to the results of the experiments described in chapter 5, it was necessary to pre-soak the emulsion to gain similar responses to the various percentages of water/2-propanol solutions as were obtained from already processed gratings observed with the radiometer. Several gratings were soaked for approximately 3 minutes, then dried in sequential steps of increasing concentrations of methanol and water (50/50, 75/25, and 100 percent alcohol). These plates were stored for about an hour before use.

One of the pre-wet plates was loaded into the immersion plateholder. A solution of 7.5 percent water in 2-propanol was poured into the filling channel of the plateholder using a removable funnel. The plate was then allowed to settle in this solution for 10 minutes at which point an exposure was made. (Better results were obtained if the solution was mixed in advance, and let to sit for a short period of time. A freshly mixed solution caused small bubbles to form on the surface of the emulsion).

For the green exposure, the 7.5 percent solution was allowed to flow rapidly out of the plateholder. The next solution (15 percent) was im-
Figure 7.2: Diagram of immersion plateholder.
mediately poured in, and allowed to settle 10 minutes before exposure. This procedure differed from the tests using the existing gratings in the radiometer setup, where the plate was pulled out of immersion to facilitate the change in solution concentration. It was necessary to let the emulsion dry evenly to obtain uniform swelling. With this new plateholder setup, the plate does not dry without some airflow through the cell. Therefore, the solutions were changed quickly to prevent the partial drying of the emulsion. This procedure was repeated for the blue exposure using a 21.5 percent water/2-propanol solution.

A total exposure of 1000 ergs, corresponding to 2.8 seconds, was given to the plate. The red and green exposures received .8 seconds, while the blue exposure received 1.2 seconds. This increase in time for the last exposure was done for two reasons: first, to take into account the reciprocity failure of sequential exposures [26]; second, there is a possibility that the alcohol slowly removes the sensitizing dye from the plate, causing the later exposures to need a little more light.

7.2 Chemistry

Two types of developing chemistry were tested (PAA and Ilford pyrogallol developer) using EDTA bleach [25]. PAA was used for most of the pre-swell data, as it gave brighter results than the Ilford developer. However, it was found for the triple exposure plate that although the PAA developer was brighter overall, the Ilford developer had better contrast with a narrower bandwidth, giving cleaner results and more saturated colors. The
Ilford developer incorporates a hardener in its composition, causing the final wavelength to be slightly longer than those obtained with the PAA developer.

The plate was developed (3 minutes with the Ilford developer, and 4 minutes for the PAA developer), stopped, then placed directly in the EDTA bleach till clear. After washing, the plate was dried in increasing sequential concentrations of methanol.

### 7.3 Results

Several plates were exposed three times using the calculated solutions (7.5 percent, 15 percent and 21.5 percent water/2-propanol) to obtain final wavelengths of 620nm, 520nm and 470nm. One of these plates was developed with PAA developer, and the other with Ilford developer.

The three color plate developed with PAA produced final wavelengths of 614nm, 522nm and 480nm. The Ilford developer gave a slight shift to the wavelengths compared to those calculated with results from the PAA developer. The final wavelength of the color plate processed with Ilford developer were 629nm, 537nm, and 487nm.

These full color plates, with the red, green and blue primaries as well as mixed colors approaching magenta, yellow and cyan were obtained without moving them from their registered position in the holographic setup. The areas where all three exposures had the same density on the color chart were clearly achromatic.
Chapter 8

Conclusions

The observation of a processed grating was used to study the reaction of the emulsion to various environments. Initially, the processed emulsion was thought to be equivalent to a raw, unused emulsion, because it was developed without hardening, and rehalogenated after fixing. While investigating the immersion technique, a discrepancy was observed between the amount of swelling from the processed grating in a 2-propanol solution, and the amount of swelling from a raw emulsion in the same solution. In addition, a swelling anomaly for the unused emulsion was found to exist wherein only part of the emulsion swelled, causing two separate gratings to be recorded at differing depths in the emulsion. This anomaly was partially eliminated by a short pre-wetting of the emulsion in water followed by drying with graded alcohol soaks. The emulsion could then be stored for an undetermined amount of time before exposure.

The method of swelling in an immersion bath offers the advantage of eliminating the effect of ambient humidity during exposure. The immersion technique was used with solutions of water in 2-propanol to obtain full color
results of a test pattern showing the 3 primaries and their overlapping colors, including an achromatic greyscale.

This immersion system could be applied to different sets of circumstances. Using 2-propanol solutions, a calibration chart could be obtained for different Agfa emulsion batch numbers, and different developing techniques. A sequence of pre-swell concentration tests would reposition the existing curves (figure 5.7) to enable reliable exposure of full color holograms. It may be possible to refine this procedure to expose larger plates, or, perhaps, film that is to be contact copied [10]. Emulsions other than Agfa 8E75 HD may need to be observed using the described technique with a developed grating and radiometer setup to determine the time to equilibrium. This is because the conditions in which emulsions are coated (the temperature and humidity of setting etc.) will affect its ability to swell.

There are several areas of this swelling technique that need more investigation. A short pre-wet was enough to reduce the swelling anomaly to obtain wavelengths in the desired primaries. However, there are wavelengths that are unobtainable with the present technique. Longer water soaks at higher temperatures, or differing pH values and other emulsion softeners, should be investigated in an attempt to eliminate this anomaly. Other solvents should be considered that may provide a more rapid swelling equilibrium, perhaps mixed with a softener so that the pre-wet step is no longer necessary.

One of the current research goals of the Spatial Imaging Group is the development of a large scale, one step, multicolor reflection stereogram in alcove format. It should be possible to scale this technique to expose
large scale (1x3 meter) film. A system can be imagined that would roll a
large piece of film through an immersion bath to an exposure point. After
all the frames have been exposed for one color separation, the film might
be rewound and the solution concentration changed for the next set of
overlapping exposures. In this way, the film would stay registered in the
setup.

Another possibility would be to pre-treat the emulsion with a hygro-
scopic agent so that it swells past the thickness necessary for a blue wave-
length. Then roll the film onto a heated strip which corresponds to the size
of each exposure. The emulsion would release increasing amounts of water
when subjected to higher temperatures, shrinking to the proper thickness
for the blue, green and red exposures. With this method it may be possible
to locally control the thickness of the emulsion so as to expose the blue,
green, then the red color-separated images sequentially. This would elimi-
nate the need to roll the film back to its original starting position for the
next color separation.

There are many possibilities for in situ control of emulsion thickness.
This thesis has investigated only a few of these, but has proven that the
concept of a swollen equilibrium for the emulsion is plausible. With further
refinement, these techniques could be applied to the production of large
scale full color reflection holograms.
Bibliography


