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Abstract
Attempts were made to manufacture a thin parallel-plate gradient-index converging lens by modifying techniques recently used by Mark Zilm to fabricate GRIN prisms. A metal washer, of 1/2” inner diameter, is placed on a glass microscope slide. It is then filled with a modified mixture of resin, one that allows the mixture up to 3 weeks to harden. Each washer has a small crystal, of 20 various designated salts, placed directly in the center of the washer, in hopes that it will diffuse across the resin before it sets and hardens, the desired index being highest near the center of the lens and gradually decreasing away from the center. It was discovered that none of the 20 salts is sufficiently soluble to be used successfully in manufacturing a GRIN converging lens.

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ATTEMPTS TO MANUFACTURE
A CONVERGING
GRADIENT INDEX LENS

A THESIS
SUBMITTED IN PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR THE DEGREE OF
DOCTOR OF OPTOMETRY
PACIFIC UNIVERSITY COLLEGE OF OPTOMETRY

BY
PAK-HO LEONG

MAY 11, 1983

ADVISOR
Dr. Jurgen R. Meyer-Arendt
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ABSTRACT

Attempts were made to manufacture a thin parallel-plate gradient-index converging lens by modifying techniques recently used by Mark Zilm to fabricate GRIN prisms. A metal washer, of 1/2" inner diameter, is placed on a glass microscope slide. It is then filled with a modified mixture of resin, one that allows the mixture up to 3 weeks to harden. Each washer has a small crystal, of 20 various designated salts, placed directly in the center of the washer, in hopes that it will diffuse across the resin before it sets and hardens, the desired index being highest near the center of the lens and gradually decreasing away from the center.

It was discovered that none of the 20 salts is sufficiently soluble to be used successfully in manufacturing a GRIN converging lens.
ATTEMPTS TO MANUFACTURE A CONVERGING GRADIENT INDEX LENS

Present day ophthalmic lenses are made of homogeneous transparent materials. The basic light bending property of these lenses came from varying their curves, thickness, and refractive index. These parameters also lead to one of the largest drawbacks for high power lenses, they are very thick and heavy and cosmetically unattractive. There may now be a solution to this problem: GRIN, or gradient-index, optics. A GRIN lens receives its refractive properties from its refractive index which varies continuously within the material. GRIN lenses theoretically can be made thinner and lighter than conventional lenses. They may also be used to replace aspheric lenses and multi-lens systems.

Gradient index materials have been known for a long time, in fact many occur in nature. Mirages are caused by a density gradient in air created by the temperature difference. The lens in the human eye is inhomogeneous, the nucleus being denser than the surrounding cortex.

Maxwell in 1854 described a lens, known as Maxwell’s fisheye lens, which used GRIN optics. However, his lens is all but impossible to make and serves little useful purpose.

Robert W. Wood in 1905, was the first to experiment with GRIN optics. He made a cylinder of gelatin which he then sliced in to small sections and soaked in water which
diffused into the gelatin and changed its refractive index. Gelatin lenses, however, are not permanent.\(^2\)

Luneburg in 1964 discovered a lens that incorporated a variable index material. Unfortunately, this lens can't be used with visible light. Its best use is with microwaves.\(^1\)

Schott prepared a cylinder of molten glass and subjected it to rapid cooling which induced an index gradient through tension. This produced a concave lens effect.

Green reported in 1978 that he did an experiment and succeeded in focusing light by a vertical temperature gradient in aerated water.\(^3\)

Much of the major work being done at present is in GRIN fiber optics. The main use is in telecommunications.

Some of the techniques for manufacturing gradients in glass and plastic are:

1. Neutron irradiation, in which a boron-rich material such as BK7 is irradiated by neutrons to produce an index change.\(^4\)

2. Chemical Vapor Deposition (CVD). Several layers of different index materials are deposited layer by layer on a tube.\(^4\)

3. Polymerization technique where a monomer of plastic is changed into a polymer by exposure to UV light or laser radiation.\(^4\)

4. Ion exchange. The glass is placed into a salt bath and the salt ions replace ions in the glass.\(^4\)

5. Ion stuffing. A special glass that phase-
separates when heated, is heated. It is then placed in an acid that dissolves one of the phases. The glass is then placed in a bath in which the ions diffuse into the glass. After a short time the glass is pulled out and recondensed by heating.

6. Crystal growing. The glass is placed in a bath of sodium chloride and silver chloride. The sodium chloride is "pulled" into the glass and after the concentration is high enough the silver chloride is also pulled into the glass.

It was my intent to manufacture a GRIN lens by another method in which I was using many different types of chemicals, allowing them to diffuse into a plastic resin which is slowly allowed to harden.
CHEMICALS

The chemicals used in my thesis are:

1. Ammonium Bromide (NH₄Br)
2. Barium Chloride, dihydrate (BaCl₂ - 2H₂O)
3. Calcium Iodide (CaI₂)
4. Cupric Chloride, dihydrate (CuCl₂ - 2H₂O)
5. Cupric Sulfate, pentahydrate (CuSO₄ - 5H₂O)
6. Glucose (C₆H₁₂O₆)
7. Lead Nitrate (Pb(NO₃)₂)
8. Lithium Chloride (LiCl)
9. Lithium Sulfate (Li₂SO₄)
10. Nickel (II) Ammonium Sulfate, hexahydrate (Ni(NH₄)₂(SO₄)₂ - 6H₂O)
11. Potassium Iodide (KI)
12. Potassium Nitrate (KNO₃)
13. Potassium Permanganate (KMnO₄)
14. Silver Iodide (AgI)
15. Silver Nitrate (AgNO₃)
16. Silver Sulfate (Ag₂SO₄)
17. Sodium Bicarbonate (NaHCO₃)
18. Sodium Chloride (NaCl)
19. Sodium Chloride mixed with Sodium Iodide (NaCl + NaI)
20. Strontium Chloride, hexahydrate (SrCl₂ - 6H₂O)
METHODOLOGY for series #1 GRIN lenses

Note: The following method for manufacture and testing of a GRIN lens has been supplied mainly by Dr. Jurgen R. Meyer-Arendt and Mark Zilm.5

1. Thoroughly clean and dry all the 1"x3" glass microscope slides so as to insure against any contamination and provide for a better optical surface. Slides with any imperfections in the glass must be discarded so as not to interfere with any possible lens effects. Mark the slides with the name of each chemical.

2. Place the metal washers, with 1/2" inner diameters, upon the slides.

3. Measure the "Envirotex Polymer Coating" in a ratio of 4 parts resin to 1 part hardener and mix thoroughly with a glass stirring rod.

4. Carefully pour the mixture into the center of each of the washers.

5. Carefully select a small crystal, less than 2x2x2mm., of each chemical and place it precisely in the center of each washer. Wait to allow the crystal to settle down and also wait for any remaining bubbles to disappear.
6. Carefully place another slide on top of the washer to cover the entire "lens". Allow at least 3 weeks for the resin to harden.
DATA AND RESULTS for series #1 GRIN lenses

Initially I had assumed that, since the lenses were so small, I wouldn’t have to mix much resin and that I could mix it in a small container, i.e. a test tube. Both assumptions proved out to be false. First, when mixing small amounts of the resin there is less of a probability that the mixture will be thoroughly and completely mixed. Secondly, the mixing process itself creates many bubbles in the resin and if you are using a tall slender test tube you don’t have the surface area you need to aid in allowing the bubbles to escape.

It was soon painfully discovered that the pouring procedure itself aided in creating more bubbles in the resin. This was not the desired result.

A crystal of each of the chemicals was carefully selected, preferably of a size of approximately 2x2x2mm. The difficulty arose in attempting to place each crystal precisely in the center of each of the lenses. Many of the crystals did not settle into the resin readily, presumably because of surface tension. Many bubbles were still in the resin so I allowed them to set for a few hours.

In my earlier experiments I discovered that within a few hours much of the resin had leaked out of the washers. I found that part of the problem was due to small burrs on the washers that kept the washer and slide from making a complete seal.
METHODOLOGY for series #2 GRIN lenses

1. Same as before, thoroughly clean slides.

2. Carefully file down and smooth all surfaces of the washers, so as to provide a nice even surface to make complete contact with the slide. Place the washers on slides. Find a large, flat, level surface and spread a sheet of aluminum foil over it. Place the slide-washer combinations on this sheet of aluminum foil. (If any leakage occurs again, the foil provides an easy surface to "peel" the slide-washer combinations off. The foil also helps protect the table top from any permanent damage.)

3. Measure larger quantities of Envirotex, 40ml. of resin to 10ml. of hardener, and mix thoroughly, in a large cup, with a glass stirring rod. Allow the resin to set for 15 to 30 minutes to let bubbles escape.

4. Carefully take the glass stirring rod and slowly dip it into the resin, as to not create any more bubbles. Slowly withdraw the stirring rod, now covered with resin, and fill the washer with the resin, a few drops at a time. This procedure is to be repeated until the washer is filled and has a slight convex meniscus. (This slight bit of extra resin will be flattened and squeezed out later with the cover slide. The excess will also help seal the slide-washer combination.)
5. This time use a smaller crystal, 1x1x1mm., of each of the chemicals and carefully place it in the center of the washer. Use a toothpick, or similar tool, to aid in centering the crystals and to help break the surface tension so the crystals can settle down below the surface of the resin.

6. Allow bubbles time to escape. Then carefully cover the washer with another slide.
DATA AND RESULTS from series #2 GRIN lenses

This time the use of larger quantities of Envirotex combined with a larger mixing container aided greatly in decreasing the amount of bubbles. The larger container allowed for a shallower depth and a greater surface area with which the bubbles could escape. Also, it was discovered that allowing the mixture to set for 15 to 30 minutes was an adequate amount of time for most of the bubbles to escape, and yet was not too long as for the resin to become too unmanageable to fill the washers.

The technique, of using the stirring rod to slowly fill the washers with resin, was a tremendous help in decreasing the amount of bubbles formed during the pouring process.

The toothpick was a great aid in helping to center the crystal in the washer and at the same time helping to submerge the crystal in the resin.

The most difficult problem that remained was to cover the washer with a cover slide and not get any bubbles. I tried numerous techniques such as: various speeds, from a very quick to a very slow, all combined with placing the slide on at various different angles. No matter what technique I used or how carefully I tried, I was unable to avoid creating any new air bubbles when placing the cover slide in position. I did discover though, that allowing the resin to set for 1 to 2 hours after pouring aided in decreasing bubble formation. I tried experimenting with longer times but found that the surface started to get tacky and that placing a slide on
top of this surface created an irregular optical surface. Also, precaution must be taken to protect against dust.

Upon observation the next day, I discovered that the resin had again leaked out of most of the washers.

After 3 weeks it was observed that none of the crystals had completely dissolved and that only 1 showed any perceivable results. That crystal was silver nitrate, AgNO₃. This crystal turned brown and also had a diffuse brown ring surrounding it.

I have also observed that even those lenses that originally had just a few bubbles now showed more of them. The only conclusion I can draw from this is that many unobserved very tiny bubbles, that were dissolved in the resin, over time, grouped together to form these small bubbles.

I also discovered another interesting piece of information. After originally filling all the washers with resin, I left the remaining resin in the container to harden. After 3 weeks, although the resin was hard, the surface was still very tacky! (I checked again 8 weeks later and it still was!) This means that if this method will ever be used, the lenses will have to be laminated between something that is much more stable, i.e. glass.
METHODOLOGY for series #3 GRIN lenses

1. Same as Methodology for series #2, step 1, clean slides thoroughly.

2. File and smooth all burrs on the washers. To help prevent any leakage, seal the washer to the slide. (This is done by first preparing a normal 1:1 mixture of the Envirotex.) Mark the slides, place a sheet of foil on a nice level surface and then arrange the slides on this. Place a very thin coating of the mixture on the bottom of each washer. Carefully place each washer onto a slide. Rotate the washer a few degrees in both directions to insure a proper seal. Allow these slide-washer combinations to harden for 2 days, then check for proper seal.

3. Same as Methodology for series #2 lenses, step 3, measure and mix the Envirotex and allow bubbles to escape.

4. Same as Methodology for series #2 lenses, step 4, carefully fill each washer with mixture via the glass stirring rod technique.

5. Select an even smaller crystal of each chemical. The size to be approximately 0.1x0.1x0.1mm. The rest is the same as Methodology for series #2, step 5, center and submerge crystal with a toothpick.
6. Allow bubbles to escape, about 1 to 2 hours, and carefully place a cover slide on each lens.
DATA AND RESULTS for series #3 GRIN lenses

The techniques discovered in earlier experiments greatly decreased the number of bubbles in the resin, prior to placing the cover slide on. However, I still wasn’t able to prevent the introduction of more air bubbles when placing the cover slide on. The only thing I could try was to rotate the slide to shift bubbles away from the crystal.

This time, upon inspection the next day, it was discovered that the washers didn’t leak. The washers were all sealed properly with this added procedure.

Again, the lenses were observed. As before, none of the crystals had completely dissolved and only the AgNO₃ and the Ag₂SO₄ showed any perceivable changes. They both turned brown. This time, however, no diffuse brown ring was observable in either. These lenses also showed an increase in the number of small air bubbles.
CONCLUSIONS

Although no miraculous discoveries were made in this thesis, it has been worthwhile to discover "how not to do it." Mark Zilm's GRIN prism technique has a few advantages that my experimental technique for GRIN lenses doesn't have. First, both the quantity of the chemical available for diffusion and the surface area is greater. In Mark's prism he placed a fairly large quantity of potassium nitrate, KNO₃, on the bottom of his mold. I could only use very small crystals, 0.1x0.1x0.1mm, in size. Whatever chemicals in the bottom of Mark's prism didn't dissolve he could grind off or even leave because it would not affect the optics of the prism. However, in a plus lens, if the crystal doesn't diffuse completely, the lens is worthless.

The second problem was the physical structure of the lens and the problem with bubbles. Zilm's prism is upright and thin and open at the top. This allows the bubbles more time to rise to the top and escape. Once the bubbles are at the top they would not affect the optics of the prism. My lenses are flat and thin and must be covered. No bubbles must be present at all and the process of placing the cover slide over the washer actually adds more bubbles to the lenses. (If no cover slides were used, the bubbles would escape; however, the surface would not be plano and a lens effect would be created by this surface. I tried this.)

In closing, I have found that Mark Zilm's technique for making a GRIN prism cannot be directly applied when attempting to manufacture a positive GRIN lens.
Upon conclusion of the experimental portion of my thesis, the thought occurred to me that (any) future work to be done with my method of attempting to manufacture a converging GRIN lens must include more extensive investigation (research) into finding either a different type of resin which would be a better solvent for salts. Or, finding a chemical which will dissolve in Envirotex.

The chemicals that I have listed on page 5 of my thesis are mostly inorganic salts; these are most appropriate for different types of glasses. Since glass and plastics are so chemically different it follows that materials that work for glass are definitely different than those that work for plastics. To change the index of refraction of plastics one needs totally different materials. An exhaustive listing of such organic modifiers has been published, as I found out after completion of my work, in the patent by Moore, 1973.6
BIBLIOGRAPHY


