

## Large-Format Embossed Holograms

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### Abstract

Photoresist selection, recording, plating, and embossing methods used for mass-replication of large-format embossed holograms.

### Introduction

For the purposes of this paper "embossed holograms" are defined as "white light transmission holograms" replicated by heat and pressure, into plastic by means of a metal master containing a surface relief of the hologram interference pattern.

Only methods of embossing that are currently available in the commercial marketplace will be discussed.

"Large" is a term relative to what has been demonstrated in the marketplace and is particularly defined in this paper as holograms larger than 6"×6" or 10"×4" with three-dimensional imagery recorded in photoresist and replicated from a "single-piece metal embossing master."

### Basic steps for embossing

The basic steps for producing embossed holograms have been well covered in existing literature.<sup>2,3</sup>

1. Original recording or contact copying of a hologram into a positive photoresist.
2. Deposition of an electrically conductive layer onto the photoresist hologram.
3. Electroplating the conductive photoresist hologram to create a first-generation metal master and subsequent production of 2nd and 3rd generation metal masters.
4. Embossing of plastic by the metal masters.

### Embossing

With embossing it is best to begin at the end of the process.

Embossed holograms can be produced on PVC and other thermoplastics as well as on polyester and aluminized polyester. Holograms destined for hot-stamping foil use only aluminized polyester.

Companies engaged in embossing holograms claim that making equipment to emboss polyester, or aluminized polyester, in sizes larger than 6"×12" would be technically impractical because of the difficulty of maintaining sufficient pressure evenly across large rollers, and because of insufficient market demand.<sup>4,5</sup>

The largest equipment for embossing holograms onto aluminized polyester uses rollers only 10" long, capable of producing an image about 10"×4".<sup>6</sup> Other systems produce 6"×6" image areas using a combination roller/platen method.<sup>7</sup>

Embossed aluminized polyester (as opposed to vinyl) provides better adhesion of aluminization,<sup>8</sup> durability in handling, and dimensional stability.

### Large-format embossing

There are 2 methods of embossing larger holograms: roll embossing of thermoplastic, typically PVC, and flat-press embossing of thermoplastic.

Roll embossing. Roll embossing is the fastest method of production and is suitable for large quantities up to millions of square feet (Figure 1). Production rates over a hundred linear feet per minute have been achieved.<sup>9</sup> Because of the relatively low heat and pressure required to emboss vinyl, metal masters can last longer and rollers can be made with relatively wide diameters and long lengths, for example about 4" in diameter by 26" in length. A metal master in this case could be as large as 12"×24". Much larger thermoplastic roll embossing machinery exists but has not yet been used for embossing holograms.

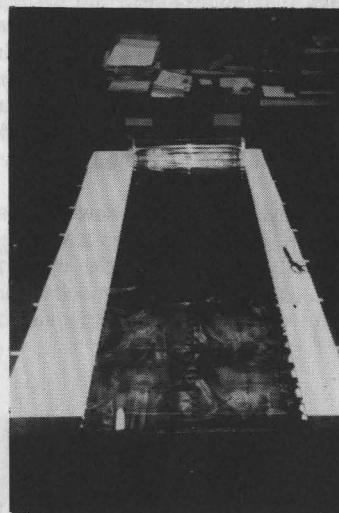


Figure 1: Roll-embossed Tiffen holograms (24" wide).

Roll embossing uses a multiple pressure-roller system. The metal master is attached to an internally heated roller and a roll of plastic is fed between this roller and an adjacent pressure roller.

In some systems, plastic in precalendered roll form is used while in others the plastic is extruded from pellets, calendered, and embossed in one continuous operation.

A dust free environment is desirable due to generation of large amounts of static electricity.

A new roller is used for each job since metal masters are permanently attached to the roller (Figure 2).<sup>10</sup>

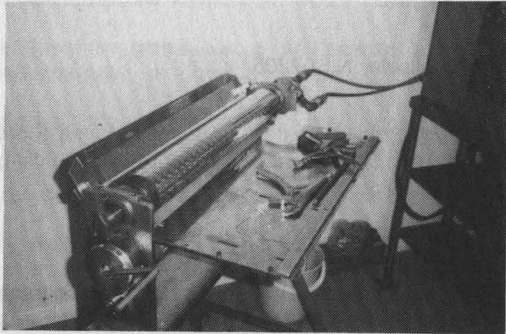


Figure 2: Roll-embossing roller with adhered master.

Optimum embossing is obtained by close control of heat, pressure, and roller speed. PVC with few impurities and plasticizers, no thickness variation of metal masters, and good quality rollers contribute to high quality embossing.

Typical finished embossed PVC thickness is 3 to 8 mils.

Variations in metal master thickness are to be avoided, with reasonable tolerances within an individual master and between multiple masters adhered to the same roller running to about  $\pm 1/20$ th mil. Greater variations tend to create dark areas, or image voids, in the embossed plastic where the metal master is thinner. The metal master must be flexible enough to conform exactly to the roller and should be about 2 mils thick.

The unembossed vinyl base is the viewing side because the embossed hologram surface is only a couple of thousand angstroms in depth and is vulnerable to degradation from scratching, finger oils, etc. Thus the embossed image must be backward, and the metal master image must be orthoscopic (Figure 3).



Figure 3: Tiffen hologram orthoscopic metal embossing master.

The embossed side is then aluminized to enhance image brightness. Mirrorization is also vulnerable to degradation and may be coated with a pressure-sensitive, self-adhesive backing with a plastic or paper peel-off covering called a "liner." Final purpose of holograms will determine specifications for aluminization and type of adhesive and liner.

**Flat press embossing.** The second method of producing large-format embossed holograms is by flat-press embossing of a thermoplastic, typically PVC (Figure 4).

<u>Large format embossing</u>	
<u>Roll embossing</u>	<u>Flat-press embossing</u>
- Equipment costs ~\$25K+.	- Equipment costs ~\$3K+.
- High production rate.	- Low production rate.
- Thin metal master.	- Thick metal master.
- Multiple rollers.	- Flat platens.
- Finished product is "floppy".	- Finished product can be rigid.

Figure 4

Two flat metal platens are each internally heated and then cooled while pressure is maintained on a metal master/plastic "sandwich." This is significantly slower than roll embossing but can produce a thicker, more rigid embossed hologram.

Unlike the roller method where a thin line of continuous pressure between two counter-rotating rollers does the embossing, a flat press embosses the entire hologram surface at once. An upper, heated platen is hydraulically pressed against a metal master/plastic sandwich resting on a heated lower platen. While pressure is maintained, the platens are chilled until the internal sandwich temperature is below the plastic flow point. Type and thickness of plastic, pressure, and heat affect dwell time.

Production rates are measured in seconds or minutes per impression. Rates can be increased by stacking layers of metal masters interleaved with plastic between platens and by using more than two platens in a single press." Plastic only a few mils thick can be roll-fed, but more rigid plastic must be sheet fed.

Metal masters for flat-press embossing should be rigid and very flat. Any nonflat surface contours of the metal master will be carried over into the embossed plastic and/or result in image voids. A thick master is desirable since it is subjected to alternate heating and cooling stresses and since the metal/plastic sandwich often must be separated by hand after embossing. Thickness variations in the master should be avoided.

Master thickness required for rigidity varies with hologram size from about 10 mils for a 2" x 2" hologram to 40 mils and up for holograms 12" x 12" and larger. Flat-press embossing can result in a higher quality replication than roll embossing, though much depends on plastic and metal master quality, and operator skill.

Most roll-embossing equipment has been custom-built by the companies using it, and its cost varies, generally from \$25,000.00 and up. Flat presses, or laminating presses, have been in use for decades by many industries and can be purchased for several thousand dollars and up.

### Metal masters

The process of creating a metal master is divided into three parts.

1. Putting an electrically conductive layer on the nonconductive photoresist.
2. Electroforming a thicker backup layer onto the 1st conductive layer.
3. Electroforming duplicate metal masters from the 1st electroformed metal master.

### First conductive layer

There are three different approaches for creating the initial conductive layer (Figure 5).

#### 1st conductive layer

##### Silver spray

- Inexpensive setup.
- High production rate.
- Alleged Q.C. problem.
- Could be most practical for large format.

##### Vacuum-deposited silver

- Expensive setup.
- Moderate production rate.
- Excellent & consistent quality.
- Not very practical for large format.

##### Electroless nickel

- Inexpensive setup.
- Moderate production rate.
- Quality depends on operator skill.
- Practical for large format.

Figure 5

1. Silver spray.
2. Vacuum deposited silver.
3. Electroless nickel.

Whichever process is used, the thickness of the initial layer need be no more than several hundred angstroms. Coverage of the photoresist must be complete, allowing no pinholes or cracks; the layer should be of even thickness; process temperatures must be lower than the photoresist flow temperature.

Silver spray. In the silver-spray approach, the photoresist hologram is sprayed from a 2 nozzle spray gun with a commercially available 2 part silver solution.<sup>12</sup> It can also be mixed in-house from formulas available in existing literature.<sup>13</sup>

The silver-spray approach is potentially the best for large holograms, though my company has not yet tried it. It allegedly offers high production rates, simplicity of operation, and low start-up costs, requiring only a spray-gun system, spray booth, and solutions. Black staining of metal masters has been reported by some workers using this process.<sup>14</sup>

Vacuum-deposited silver. Vacuum deposition of silver is used by some workers.<sup>15</sup> The photoresist is affixed at the top of a vacuum bell jar and a small quantity of pure silver ingot, powder, or wire is evaporated from a hot filament in the bottom of the jar onto the photoresist surface (Figure 6).<sup>16</sup> This process requires investment in vacuum metallization equipment and handling of liquid nitrogen, which is necessary for achieving the required vacuum. It is excellent for small-format holograms, but system costs increase rapidly with size. It requires little training or skill on the part of the operator.

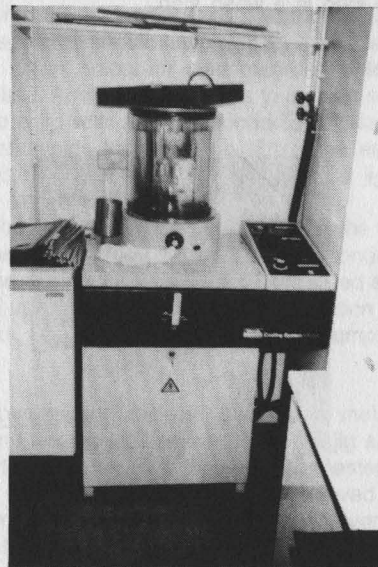


Figure 6: Vacuum-deposition system for silvering photoresist.

Electroless nickel. Electroless nickel deposition for holograms was pioneered by Dr. Nathan Feldstein at the David Sarnoff Research Center of RCA labs in the late 1960's for "Holotape", RCA's precursor to its videodisk.<sup>17</sup> My company has used this process extensively, notably for our work on the 14" x 14" initial conductive layer of the "Tiffen" photoresist (Figure 7).

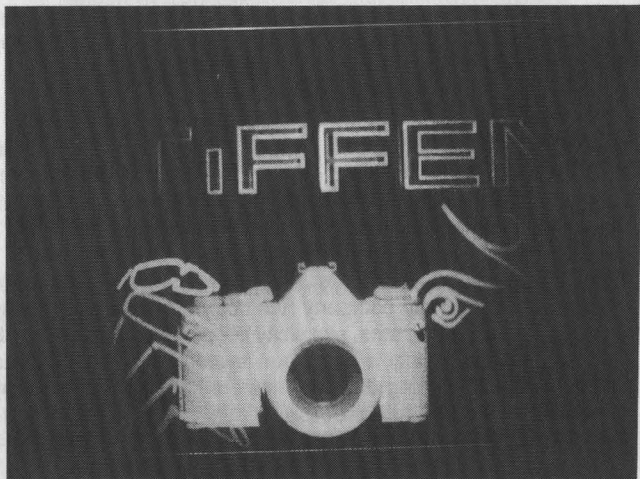


Figure 7: Tiffen hologram image.

It is a 3 step immersion process that is relatively inexpensive, requiring only dip tanks of appropriate size, 1 of which must be a heated tank, and solutions, which may be prepared in-house<sup>18</sup> or purchased commercially (Figure 8).<sup>19</sup> The first step is sensitization of the photoresist hologram with a stannous chloride solution followed by dipping in a palladium chloride solution. Electroless nickel deposition then takes place in a heated dip tank. Between each process step is a wash step.

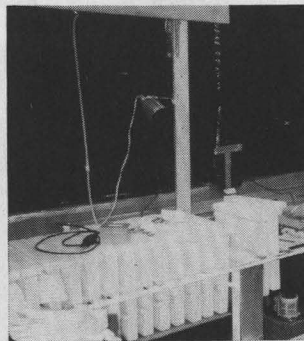


Figure 8: Electroless nickel dip-tank set-up.

This process requires solution maintenance and operator experience. Subtle differences in immersion time, agitation technique, and temperature of solutions make the difference in achieving a complete coating on the photoresist. Mistakes can be costly in

time and money by necessitating exposure of additional resist holograms, especially when working with large plates. The approach can be scaled up inexpensively and provides a nickel-to-nickel bond between the 1st conductive layer and subsequent electroforming.

#### Electroforming onto the 1st conductive layer

Nickel sulfamate is the universally used electroforming bath for making the 1st, 2nd, and 3rd generation metal masters. The solution is almost always purchased commercially and is relatively inexpensive.<sup>20</sup>

Size of plating tank and volume of solution are dependent on metal master size. The additional basic components of a plating system are:

1. DC rectifier.
2. Nickel anodes.
3. Filtration pump.
4. Solution heater.
5. Solution agitation system.

We use a 12 volt, 100 amp DC rectifier with less than 3% ripple. Our anodes are Inco nickel rounds contained in a titanium basket. We use a submersible filtration pump, 2 quartz immersion heaters, and rocker-arm agitation (Figure 9).

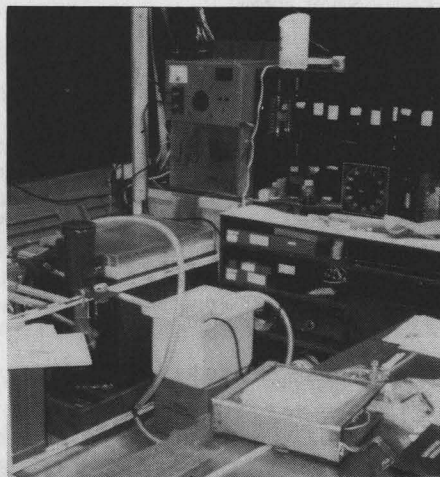


Figure 9: Electroforming system.

Interrelated factors<sup>21</sup> that must be tightly controlled to maintain plating bath efficiency are:

1. Current density.
2. Bath temperature.
3. Bath pH.
4. Bath specific gravity.

#### 5. Agitation quality.

#### 6. Bath cleanliness.

Current density initially must be kept low to avoid burning the first conductive layer off of the photoresist, and can be gradually increased to speed deposition rates.

Bath cleanliness requires constant attention. Organic materials introduced into the bath, such as plastic tubing, must be leached for at least 24 hours in a heated 10% sulphuric acid solution. Metallic materials should also be acid pickled to minimize superficial impurities. Continuous filtration of the bath minimizes contamination, helps keep the temperature constant throughout the solution volume, and can assist solution agitation. A continuous flow of fresh solution should pass evenly over the object being plated.

Additives are available to help control tensile stress of the nickel, but can degrade the quality of the deposit by causing increased grain size and a more brittle master.<sup>22</sup> Tensile stress is the tendency of the nickel deposit to curl away from the photoresist at its outside edges, i.e., a convex master, while compressive stress is the tendency to pull away at the center of the deposit, i.e., a concave master.<sup>23</sup>

#### Jigging

A conductive fixture, or "jig", holds the conductively coated photoresist for plating. Through the jig, the DC current from the rectifier reaches the conductive layer on the photoresist. The jig should make a complete circuit around the perimeter of the plate and not damage the initial conductive layer. Methods vary widely (Figure 10). Some are used once while others are reused (Figure 11). Jig fabrication is generally an in-house operation. The jig, together with the part to be electroformed, is referred to as the mandrel, which is the cathodic element of the plating system and is immersed in the solution during plating.

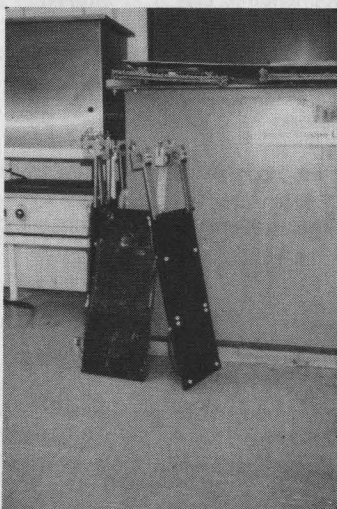


Figure 10: Reusable jig.



Figure 11: Disposable jig.

Avoiding variations in metal master thickness. Plating starts on the jig and works toward the center. This can result in a master that is thicker at the edges, the opposite of what a holographer with a well-centered image needs. Masters thicker around the edges can create voids in the central area of the embossed hologram.

Jig size larger than image size, correct placement of anodes, control of solution flow, and bath agitation minimize these problems.

The 1st generation jig should be at least 25% larger on each side than the desired finished embossed hologram area. An 8" x 10" hologram image area should have a jig area of at least 12" x 15". The 8" x 10" Tiffen hologram was recorded onto a 14" x 14" photoresist plate. Another approach is to insert a small photoresist plate into a jig with a larger conductive surface area.<sup>24</sup> An approach that avoids increased jig size uses empirically designed baffles located between the anode and jig.<sup>25</sup>

The thicker the plated deposit and the longer the plating cycle, the greater the probability of defects. If the 1st conductive layer is well done, and the plating bath is well maintained and operated, one can wind up with a perfectly flat 1st generation metal master without cracks, seepage, pinholes, blisters, bumps, or stains.<sup>26</sup>

Plate removal. Once the 1st generation metal master is finished plating, it must be removed from the jig and separated from the photoresist hologram. If the metal master is very thin, i.e., about 2 mils, it can sometimes be removed with a razor blade. Extreme care must be exercised to prevent dings from occurring in what is actually a piece of foil. If the metal master is thick and relatively rigid, it must be cut out of its jig with a more heavy-duty cutting tool. We use a "dremel" with a cut-off attachment for this purpose. Generally the metal master will separate easily from the photoresist plate (Figure 12). If the plate is thick, its edges should be deburred. After separation, the metal master is rinsed in a solvent to remove any resist that may adhere to it. At this point one learns if the entire process has been successful, or if a new photoresist hologram must be started.

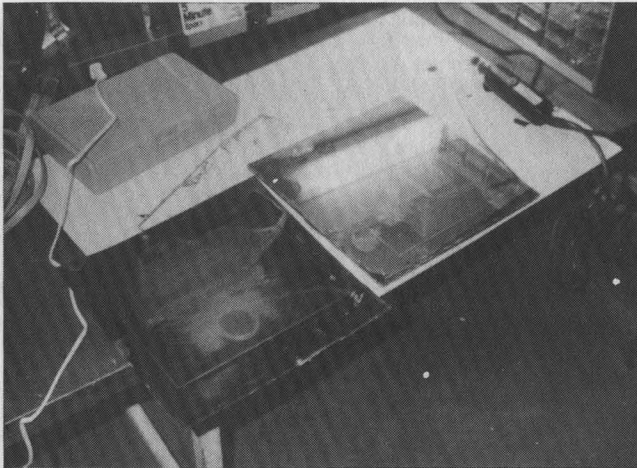


Figure 12: Tiffen metal master separated from photoresist.



Figure 13: Duplicate Tiffen metal masters.

### Duplicating metal masters

Production of 2nd and 3rd generation metal masters is similar to making a 1st generation.

The essential difference is that the mandrel to be electroformed is already completely conductive.

It is necessary to passivate the 1st generation metal master so that the duplicate electroform can subsequently be separated and will not permanently bond to the 1st nickel master. The master is immersed in a 2.5% potassium dichromate solution for 1-10 minutes and rinsed in distilled water prior to jiggling.<sup>27</sup>

If agitation is too violent during plating, the duplicate master can literally slip off the passivated mandrel. This is particularly true of larger surface area masters.

It is important to keep in mind that each generation reverses the hologram image of the previous generation and that each reduces the plate by .25-.5 inch on every side from jiggling losses.

Conventional wisdom in the LP plating business is that after 10 such passivation cycles a master's fine detail will start to degrade. This is also said to be true about taking the duping process beyond three generations. It should be possible to create 100 3rd generation metal masters from 1 photoresist hologram (Figure 13).

The plating equipment used for creating duplicates is the same as that for the initial electroform. An ideal operation would employ a separate system because the residual dichromate is difficult if not impossible to remove from the bath and inevitably results in contamination over the long haul.

### Photoresist recording

A photoresist plate with an orthoscopic hologram image will require 2 generations of metal masters, limiting duplicate metal master production to 10 units. This implies a photoresist hologram recorded as an H2 original from an H1, or, in the case of a contact-copied silver halide, reconstructing the virtual image.

A photoresist hologram with a reversed image will require 3 generations of metal masters, allowing for production of a full 100 metal masters. This implies a photoresist plate recorded as an H2 original with a mirror between H1 and H2 to revert the image, or in the case of a contact-copied silver halide, reconstructing the hologram as though for final display (Figure 14).

#### Resist image orientation

##### Orthoscopic

- Recorded as original H2.
- Contact copy of reconstructed H2 virtual image.
- 2nd generation metal is orthoscopic.

##### Reversed

- Recorded as original H2, w / mirror between H1-H2.
- Contact copy of reconstructed H2 projected slit.
- 1st & 3rd generation metal masters are orthoscopic.

Figure 14

Most holograms to be embossed are recorded in Shipley AZ1350J positive photoresist, spin-coated or dip-coated to a thickness of about 1 micron on a glass substrate, and are frequently exposed at a wavelength of 457.9nm using an argon laser.<sup>28</sup> This is the argon wavelength to which this resist is most sensitive. It is also the weakest argon wavelength. If an etalon is used, the available power is reduced by about 50%. An 18-watt argon with etalon can be expected to put out about 700mw of 457.9nm when new; a 5-watt argon with etalon will produce about 120mw of 457.9nm when new.

This same resist is about a factor of 10 more sensitive to the HeCd laser wavelength of 441.6nm.<sup>29</sup> HeCd lasers have not recently been readily available in models with more than 40mw in power. This is roughly equivalent to 400nm of the argon's 457.9nm. HeCd lasers suffer from poor coherence length relative to argon lasers equipped with etalons, and are generally suitable only for contact-copy systems.

Where an original H2 resist is to be recorded from an H1, an argon laser with etalon is preferred.

AZ1350J is very insensitive relative to most other hologram recording materials. It generally requires a total exposure energy varying between 250,000 ergs/cm<sup>2</sup> and 2,500,000 ergs/cm<sup>2</sup> at the 457.9nm wavelength, depending on length of exposure and thickness of resist coating. AZ1350J requires a threshold energy density to be effectively exposed. In practice, I have found this to be about 150 ergs/cm<sup>2</sup> per second, assuming a 30-minute exposure for a total exposure of 250,000 ergs/cm<sup>2</sup> and assuming system stability during the exposure. A more practical exposure time is about 10 minutes, to achieve the same total exposure. This is about 420 ergs/cm<sup>2</sup> per second.

An output of 100mw of 457.9nm from a 5-watt argon would limit an H2 original resist exposure to 6" x 6" from an H1 that is a single 16" long 3mm wide slit, even in the case where the H1 is bleached and laminated.

An 18-watt argon could be used to expose an 8" x 10" image onto resist from a maximum of 2 slits simultaneously (Figure 15).

#### Lasers for exposing resist

##### Argon 5WT

- 457.9nm = ~120mw w/etalon.
- Good for 1 slit H1-H2 6" x 6" resist.
- Large contact copy.

##### Argon 18WT

- 457.9nm = ~700mw w/etalon.
- Good for 2 slit H1-H2 8" x 10" resist.
- Very Large contact copy.

##### HeCd 40mw

- ~40mw of 441.6nm = ~400mw of 457.9nm
- Contact copy only.
- Large-very large contact copy.

Figure 15

For the Tiffen hologram, recorded as a 13 slit, 16" H1, H2 original resist exposure was not an option, especially with a 5-watt argon laser (Figure 16).

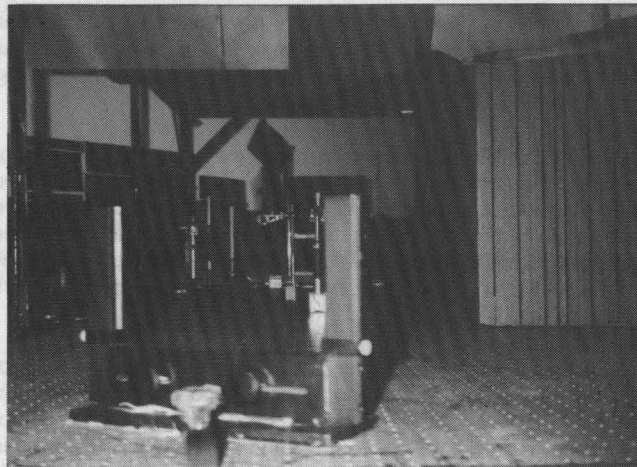


Figure 16: Multi-slit H1 master.

To take advantage of gravity, an overhead contact-copy camera configuration was selected (Figure 17). This also avoided the need for a plateholder and simplified placement of the 8" x 10" laminated silver halide H2 hologram in the center of the 14" x 14" resist plate. The original H2 silver halide was made in a single exposure from the H1, to optimize signal-to-noise ratio, and then laminated by Tiffen Manufacturing Company's proprietary process, to further enhance signal-to-noise ratio.

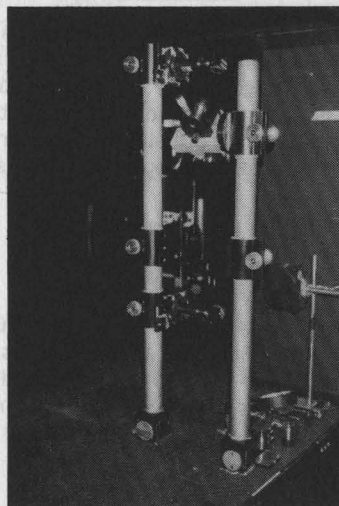


Figure 17: Overhead reversed-image contact copier.

Resist exposure intensity was about 1000 ergs/cm<sup>2</sup> per second, and exposure time was 4'38". Development was for 40" with tray agitation at a temperature of 53°F using Shipley 303A diluted 6:1 with distilled water.

### Photoresist selection

Some holographers do their own resist coating. We purchase ours commercially.

The substrate types we have tried have been plain soda lime glass, soda lime glass with an iron oxide layer between the glass and resist, and soda lime glass with a low reflection chrome layer between the glass and resist.

Plain glass creates antihalation problems unless the side opposite the resist is coated with matte black paint. We have achieved our finest resist imaging using resist on iron oxide, but have never succeeded in making a production metal master from one of these recordings. We have gotten excellent results with the LR chrome and have rarely failed to make production metal masters from these.

Spin-coated plates generally provide the most even coating of resist with dip-coating running a poor second due to ridging in commercially available plates. Ideal coatings should be mirror smooth, free of pinholes, comets, and striations when viewed in yellow safelight.

Resist plates of excellent quality up to 7"×7" are readily obtainable.<sup>30</sup> Larger plates are not so easy to find. One supplier will spin-coat large plates, but quality is fair to poor.<sup>31</sup> Another supplier will dip-coat large plates, but their quality is even worse.<sup>32</sup> This would not be a problem except that these plates cost from \$0.50 to \$1.00 per square inch.

The 14"×14" plate used for the Tiffen had AZ1350J resist spin-coated to a thickness of 1 micron on LR chrome. It had the best coating job on this size plate that I've seen and I would rate it only fair. One can see its spin-coating marks as raylike striations emanating from the center of the embossed Tiffen hologram.

### Conclusions

Large embossed holograms can be made as has been demonstrated by the Tiffen embossed hologram. The single biggest obstacle is obtaining high quality photoresist coating of large plates, and that is primarily a yield problem. Embossing equipment exists to emboss them; metal masters can be made for them; and exposure methods using existing lasers can accommodate the resist sizes.

### Acknowledgments

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