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PHOTOFABRICATION METHODS
with KTI Macro Photoresists

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INTRODUCTION

Photofabrication is a technique of working metals and nonmetals with the aid of photography and chemicals. At the heart of the method are the KTI photoresists — solutions of resins in organic solvents which become light-sensitive when applied in a coating to the chosen material, and dried. After exposure, the unwanted areas of the resist coating are removed by the developer, leaving a resist pattern which possesses extremely high chemical resistance.

Photofabrication has numerous applications, including the following:

Printed circuits
Thin-film circuits
Integrated circuits
Chemically milled metal parts
Nameplates
Reticles, grids, scales, and rulers
Decorative designs
Litho plates
Gravure printing cylinders
Letterpress plates
Weight reduction

Stencils produced using KTI photoresists are highly resistant to all commonly used etching solutions and will withstand electroplating and electroetching processes.

KTI Macro Photoresists are negative-working resists. In the *negative-working* system the *exposed* areas of the resist remain on the surface after development to form the stencil.

These photoresists are ideally suited to all types of photofabrication processes. Because they possess excellent chemical resistance plus an ability to hold exact detail, KTI Macro Photoresists can be used in such applications as weight reduction of parts for space vehicles down through the dimensional spectrum to the most precise, intricate, microelectronic device. In fact, the detail-holding characteristics of KTI Macro Photoresists have often made them the only reliable method of producing a part.

HOW KTI PHOTORESISTS WORK

When exposed to ultraviolet radiation through a photographic transparency, a negative-working resist "cross-links" or polymerizes in the areas that are struck by the ultraviolet radiation. The image formed by the ultraviolet radiation and defined by the clear areas of the transparency is insoluble in the developing bath. The opaque areas of the transparency act as a mask and allow those portions that have received no exposure to be dissolved in the developer. The soluble portions wash away and leave a tough, chemically resistant image in minute relief on the surface. This image acts as a clearly defined mask for etching, plating, or some other type of surface alteration.

CHEMICAL MILLING

Chemical milling is a method of, first, selectively masking portions of a metal surface, and then using an etchant to remove exposed portions of the metal *chemically.* KTI photoresists form a barrier to the penetration of etchants when they are suitably exposed and processed. Photographic techniques allow production of precise parts and provide methods for making each part the same as the last. Chemical milling is especially useful for working metals and foils where physical action might distort, set up strain in, or otherwise change the character of the metal. With this process, thin or brittle metals can be worked and the process yield will remain high.

PLATING AND ELECTROFORMING

In both plating and electroforming, a KTI photoresist is used to form a stencil through which metals can be deposited on a surface. The radiation-hardened and developed photoresist image forms a non-conducting pattern for the plating process. Plating may be used as a method for adding a design to a surface, as a preliminary step before etching, or to add metals with distinctive properties which are dissimilar to those of the base metal.

Electroforming is normally used to produce such highly detailed articles as fine-mesh screens and evaporation masks. In electroforming, the resist defines a pattern on a metallic mandrel, which serves as the electrical conductor for electroplating. After an article is electroformed on the mandrel, it can be stripped away and the original image is available for reuse. Because of the support given by the mandrel, very fine parts can be made with little damage.

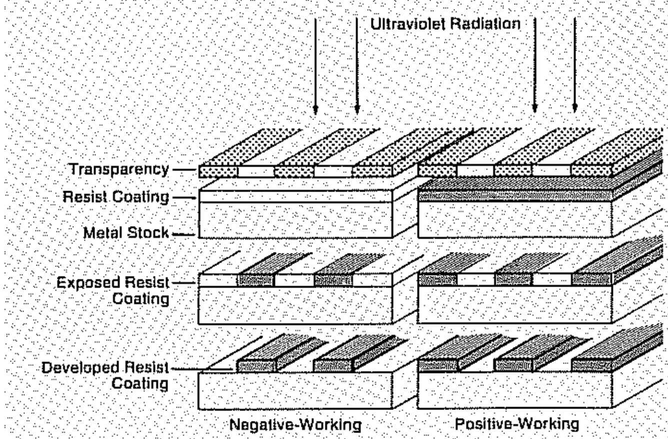

FIGURE I: These sequences show the effects of exposure and subsequent development on negative-working and positive-working resist coatings. (Cross-hatched areas show hardened resist.)

THE PHOTOFABRICATION PROCESS

All photofabrication processes have in common the use of a line transparency and a photoresist-coated surface. The photographic transparency defines the area of exposure on the photoresist coating. The resist—when exposed and developed—forms a stencil which confines further chemical operations to the unprotected portions of the surface. The typical photofabrication process proceeds with these steps:

1. Prepare the master drawing or artwork.
2. Produce a film transparency from the artwork for photoresist exposure control.
3. Prepare and coat a surface with photo resist.
4. Expose the photoresist surface, controlling the area of exposure with the transparency.
5. Process the resist to form a stencil.
6. Proceed with etching, plating, or electroforming.

Each of these steps is described in detail in the following pages.

A STEP-BY-STEP EXAMPLE

To illustrate the use of a photoresist, we'll use the photofabrication of a simple, one-sided printed-circuit board and follow it through the steps just discussed.

CLEANING THE SURFACE

1. Clean the copper-clad board with warm water and a small amount of abrasive copper cleaner.
2. After a thorough cleaning of the entire surface, rinse the board with running water to assure that all of the cleaner is removed.

   Note: From this point on, be sure to hold the board by the edges. Do not touch the surface because dirt or oil from your fingers can prevent future adhesion of the photoresist to the surface of the board.
3. Photoresists adhere better to surfaces which are acidic. To accomplish this, dip the board in a dilute bath of HCl (10 percent by volume) then rinse the board again under running water.
4. Place the board in a convection oven or in an infrared dryer until the board is thoroughly dried, then remove it from the oven and allow it to cool.

COATING THE RESIST

At this point, the board is ready for the application of the photoresist coating. Because photoresists are sensitive to ultraviolet radiation, steps must be taken to provide "safelight" conditions in which the resist can be handled. Safelight illumination is required during the coating, prebaking, exposing, and developing steps. Yellow, "anti-bug" incandescent bulbs can be used. The same precautions described later under "Safelights" should be reviewed.

Under safelight illumination, pour a small amount of resist into the middle of the board. The board is then tilted in various directions until the surface of the board is entirely covered. Allow the excess resist to drain from one corner and then stand the board in a near-vertical position until the resist has dried. This will take about 10 to 15 minutes.

Note: The resist coating will probably have a wedge shape caused by the vertical position of the board during drying. This shape can be overcome, if necessary, by repeating the coating and drying operation. This time, however, stand the board on its opposite edge during drying.

PREBAKING

1. Place the board in an oven and allow to stand for 10 minutes at a temperature of 80°C (176°F).
2. Remove the board from the oven and allow it to cool.

EXPOSING THE PHOTORESIST

The resist coating must be exposed using a light source rich in ultraviolet radiation. A carbon-arc light is excellent if one is available. If not, mercury-vapor lamps or ultraviolet fluorescent tubes can be used in a suitable exposing device such as a contact-printing box. A vacuum frame is a very useful device to use to maintain close contact between the photomask and the resist-coated board.

1. Place the negative or photomask, containing the circuit design, on the resist-coated board.

   Note: Make sure that the emulsion side of the photomask is in contact with the resist. Use pressure to maintain good contact between the photomask and the board. This will help provide good pattern resolution on the finished board.
2. Turn the exposing lamp on for the proper exposing time for your particular conditions.

   Note: The proper exposing time should be determined ahead of time, using the procedure described in the section "Determining the Proper Exposure," on page 17.

DEVELOPING THE IMAGE

1. Pour about ½ inch of a KTI Developer into a glass dish.
2. Lay the exposed board, image side up, in the developer and gently rock the dish back and forth.
3. Develop the board for 2 minutes and then remove it from the solution.
4. Rinse the board gently with a stream of warm water. You should now begin to see the circuit pattern on the board. Continue to rinse the board until all of the unwanted photoresist disappears.

   Note: If any of the unwanted resist won't rinse off, return the board to the developer for another ½ minute.

USE OF PHOTORESIST DYES

If inspection of the resist image is necessary, the image can be dyed with KTI Photo Resist Dye—Blue.

Photoresist images should be dyed immediately after development. The photoresist is most susceptible to maximum dye absorption at this point. If the resist is dried and allowed to stand for a long period (24 hours or more), it will not accept the dye as readily as it will in the just-developed state. Contact with the dye solution should be about 10 seconds or less. No water rinse is used between developing and drying.

Any dye step should be followed by a tempered-water rinse. This removes the residual dye solution and helps to prevent any possible scumming from the dye. Should scumming occur anyway, it can be removed by a 10-second dip in isopropyl alcohol, after the image has been dried.

POSTBAKING THE RESIST IMAGE

Place the board in the oven again and let it remain for 10 minutes at 80°C (176°F).

ETCHING

Use either ferric chloride, cupric chloride, or ammonium persulfate as an etchant. An important factor in good etching is constant agitation of the etching solution. This is necessary to carry away reaction products and provide fresh etchant to carry on the process.

Agitation can be provided by constantly moving the container in which etching is taking place, or by inserting air lines in the bottom of the container. The constant flow of air bubbles from a source of compressed air will provide the necessary agitation. Better uniformity and faster results can be obtained by heating the etchant to about 49°C (120°F).

1. Place the board in the etching container.
2. Occasionally remove the board and check on the progress of the copper removal. Continue the etching until all of the unwanted copper has been removed.
3. Rinse the board in running water to remove any excess etchant.

STRIPPING

The remaining photoresist must be removed before the board can be used. If this is not done, solder will not adhere to the remaining copper foil.

1. Thoroughly scrub the board with warm water and a small amount of pumice until the remaining resist is removed.
2. Rinse under running water and then allow the board to dry. It is then ready for use.

ARTWORK PREPARATION

Whether the end product is to be a printed circuit, a chemically milled part, an integrated circuit, or a nameplate, the result will be only as good as the initial planning and execution of the original artwork. This preplanning includes (1) the evaluation of the final tolerances which must be held on the piece, (2) the accuracy of artwork preparation (and subsequent reproduction), (3) the compensations in original artwork required by the etching process itself, and (4) the preferred methods for the subsequent photographic and photoresist operations (negative-working or positive-working).

ARTWORK SIZE

Note: *The word "transparency" is used in this and subsequent sections to denote either a negative or a positive photographic image on film or glass plates. This term was chosen to avoid the necessity of distinguishing image values each time.*

To produce a part by the photofabrication process, one needs a master or working transparency of the desired pattern. This is obtained by first making a master pattern and then photographically reducing this master artwork to the required size.

For most purposes, a piece of artwork is prepared at two to several hundred times the actual size of the finished part. This is done to make sure of dimensional accuracy on the photographic transparency, since any inaccuracy in the preparation technique is reflected in the accuracy of the finished part.

In artwork preparation and subsequent photographic reduction, the aim is to produce a master transparency with little or no departure from the dimensions (except for required compensations described below) stated on the original drawing. In this way, the stated dimensional tolerances can be taken up (as they most properly should be) in the relatively less controllable etching process. To achieve a dimensionally precise transparency, make the artwork several times larger than would normally be required to reduce the inaccuracies of artwork generation.

COMPENSATED ARTWORK

Since one of the basic processes for photofabrication—etching—is subtractive (material is removed), it may be necessary to make the artwork elements slightly oversized (for outside dimensions), or undersized (for inside dimensions, e.g., holes, slots), to compensate for lateral etch. When designing or specifying artwork, the "etch factor" (ratio of depth to sideways etch) and "etch allowance" must be considered. It is best to figure on the maximum amount of undercutting allowance, since tooling is costly and more metal can be removed; it cannot, however, be put back.

Lateral, or sideways, etch can be expressed as the amount of etching under a resist pattern when a surface is etched. Etch factor is defined as the depth of etch divided by the amount of sideways growth when a material is etched. It is calculated for one edge of an etched area only.

Thus, an etch factor of 2 means that the depth of etch in a material is twice as large as the undercutting of the resist edge. If a series of tests proves this to be a constant condition, changes can be made in the original artwork to compensate for this growth. The net result of artwork compensation is, of course, more accurate parts.

Artwork compensation can be the source of some confusion if one loses sight of the final width of the resist pattern. Consider for a moment, what happens at one edge of the resist pattern (Figure 2). As etching proceeds (Figure 3), the material is etched downward and to the side, undercutting the resist pattern. The etch factor is simply the downward etch (Y) divided by the sideways growth (X).

These figures have been drawn to show a broken "baseline" as the bottom of the material; this was done so that the example can be considered without regard to whether the material is to be etched from one side, or from both sides. For double-sided etching, simply imagine a mirror image of the diagram, below the broken baseline. This baseline would be the centerline of the material thickness.

"Breakthrough" of the material occurs when etching reaches the point shown in Figure 4. Occasionally with single-sided etching, and almost always with etching from both sides of the material, overetching is done to achieve a more nearly

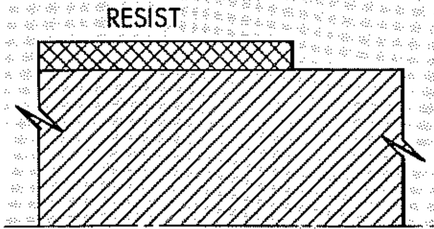

FIGURE 2

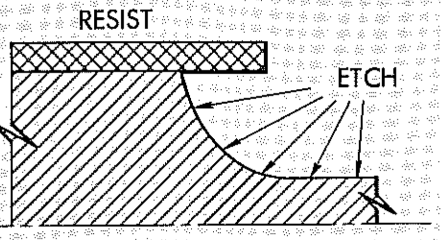

FIGURE 3

straight wall at the edge (Figure 5). This reduces the bevel (such as that shown by the part on the left in Figure 6), which would remain. The part on the right in this figure has been overetched to achieve a straight wall (a smooth, bevel-free edge).

The artwork must be designed to compensate for this undercutting of every resist edge. Exterior edges such as the perimeter of a part will grow inward, thus reducing the overall outside dimensions. Interior edges, also being undercut, will grow outward, increasing the dimensions of a hole or a slot. Obviously, outside dimensions must be made larger and inside dimensions made smaller on the artwork to compensate for undercutting.

Think of each line in the original drawing as an edge, and the appropriate artwork compensation will be obvious. For a practical illustration of the compensations required, compare the original drawing of a part shown in Figure 7 with the compensations shown by an overlay in Figure 8. When the compensations are made and etch bands added, the finished artwork will look like the cut-and-peel master image shown in Figure 9.

Etch factors must be determined empirically. If etching from both sides is to be done, artwork compensation must include, in addition to the basic etch factor, a "total etch allowance" to provide for overetching to a straight wall.

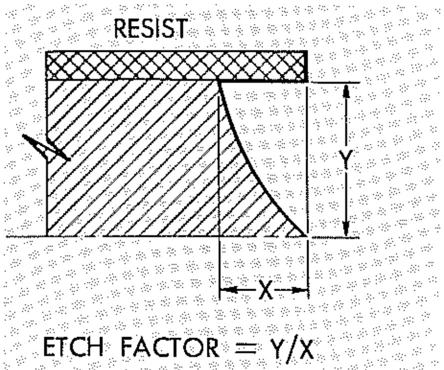

FIGURE 4

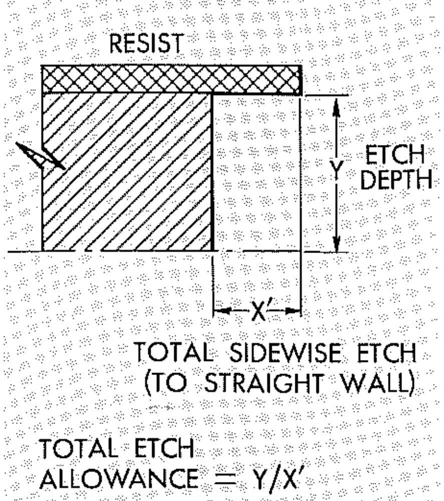

FIGURE 5

ETCH BANDS

One of the keys to consistent etching of high quality, chemically milled parts is the use of uniform "etch bands" as an outline for all part elements. In Figure 9, the artwork has been prepared with etch bands.

There are several reasons for using etch bands:

1. Etching rates can vary due to the width of the opening being produced. Use of a band of the same width then provides for consistent penetration of the material over the entire piece. Thus, when the etching machine provides a uniform pattern, breakthrough will occur everywhere at the same time. This makes maintenance of part tolerances easier.
2. Since most of the material is covered with resist, only a small portion is etched

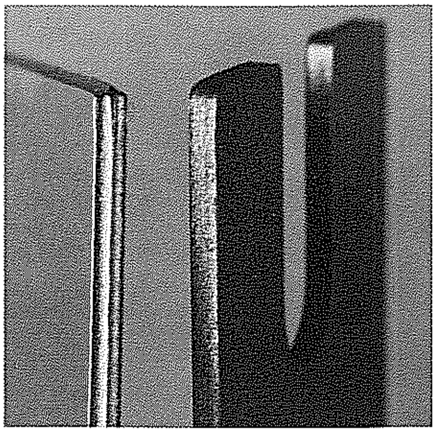

FIGURE 6: Piece on the left shows metal etched from both sides to breakthrough; material on the right was etched to straight wall.

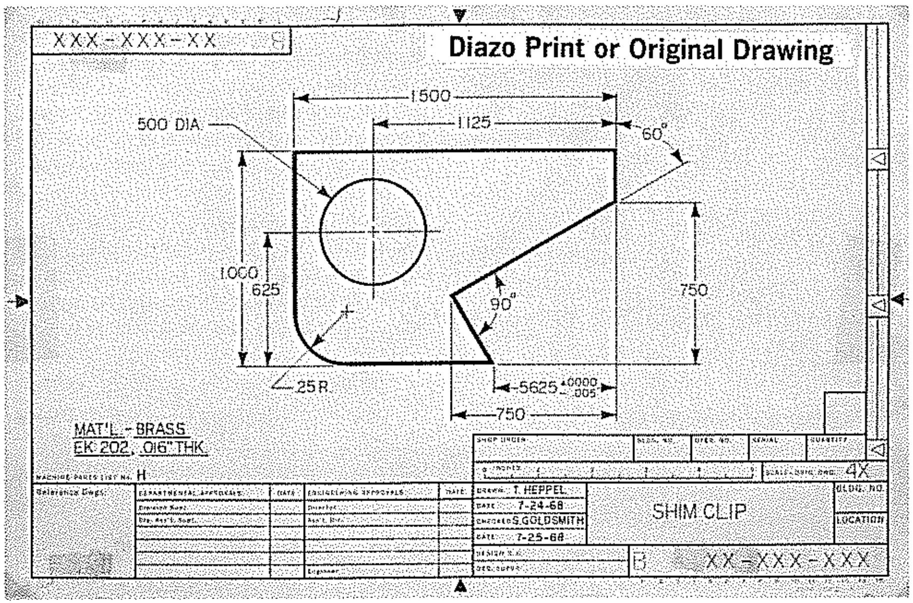

FIGURE 7: Finished parts dimensions appear on original drawing.

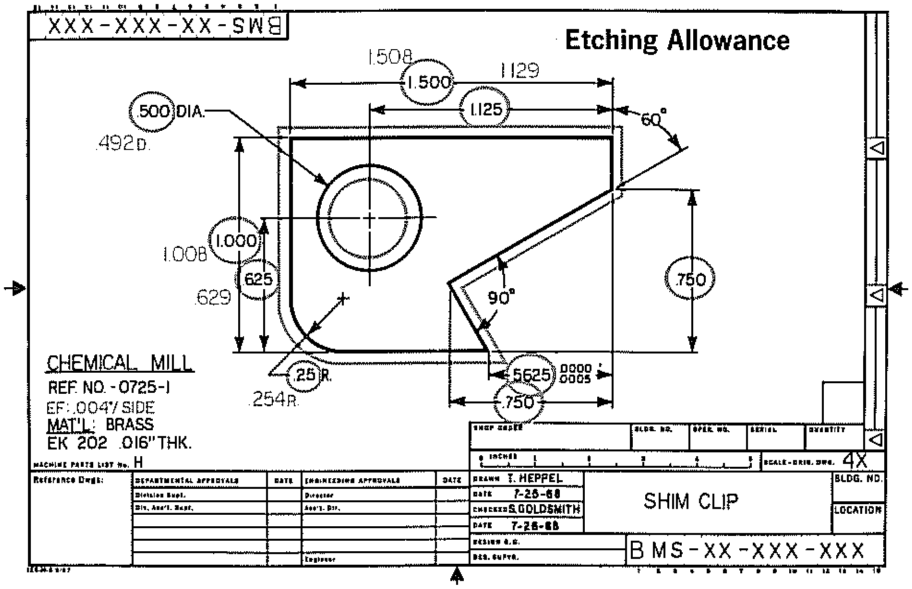

FIGURE 8: Etching allowances are computed and shown to assist in making artwork.

away. Etchant is conserved, and the downtime required to change etchant in the machine is reduced.

3. It is difficult to handle a sheet of parts when the material between has been nearly etched away leaving only a thin flexible web with sharp edges. With etch bands, the parts can be "tabbed" so that they will not fall from the sheet when the etching is completed. (See the artwork in Figure 9.)

Etch bands should be made uniform. Use 1/32-inch bands wherever possible. Geometry of parts may, at times, dictate a change from this, however. Tabs should be designed in a triangular shape to etch down to a point (0.005 inch) upon etch completion.

MATERIALS FOR ARTWORK PREPARATION

Consideration of possible size changes and allowable dimensional variations is most important in choosing material for artwork and in determining the artwork size. As the artwork is photographically reduced in size, the dimensional variations in it are correspondingly reduced. The size of the artwork and the reduction ratio depend on the allowable dimensional variations in the finished part, plus the possible size-changes which can occur in the artwork and in the photographic materials and processes.

When choosing a material for artwork, consider the following factors:

1. Ability to be photographed easily and to give results having suitable density and contrast.
2. Dimensional stability with respect to changes in temperature and relative humidity.
3. Toughness, resistance to tearing and abrasion.
4. Surface and acceptance of ink or of tape.

Polyester drafting film is used as base material for artwork. Matte-surface drafting film is generally used with ink or tapes; glossy film is used only with tape. A basic advantage of polyester film is its dimensional stability. Because the film can also be backlighted when photographed, contrast of the artwork and of the subsequent photographic transparency is increased. Backlighting also yields a corresponding reduction of pinholes in the transparency.

A cut-and-peel film consists of a photographically opaque layer laminated to a translucent or transparent polyester film base. This film is designed so that the desired pattern can be cut into the opaque layer. The unwanted portions of the opaque layer are then removed from the surface. Like the drafting films, cut-and-peel film can be backlighted.

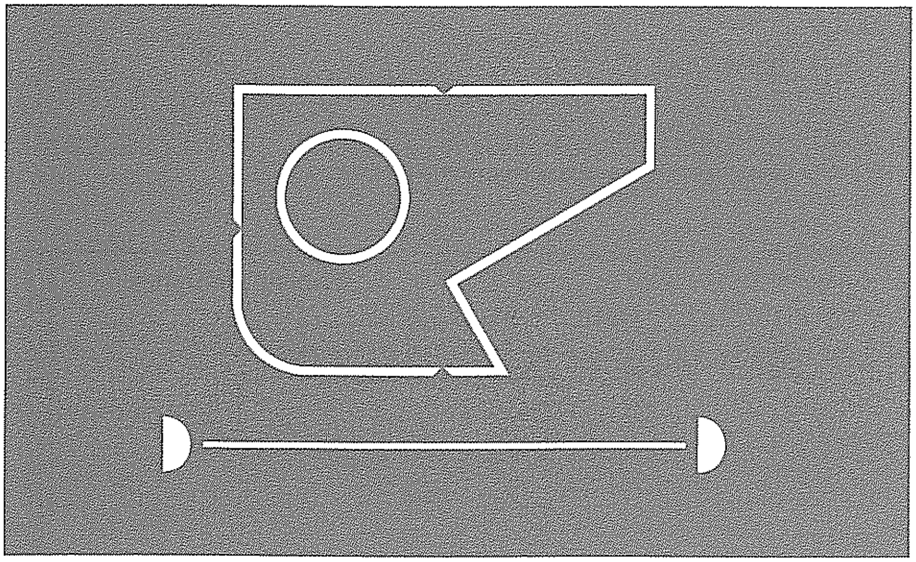

FIGURE 9: Artwork which will be photographically reduced

EQUIPMENT

The methods employed to produce a piece of artwork range from the simplest hand-drafting technique to automated or semi-automated coordinatographs. In terms of accuracy, the best dimensional control is offered by the coordinatograph. (In this respect, coordinatographs are customarily used to make master artwork for high-precision applications.) Conversely, hand-drafting techniques offer the least dimensional control, since their accuracy depends on the skill of the individual. Some types of tape- or computer-controlled equipment ("photoplotters") generate transparencies on film or glass plates thus bypassing the artwork stage.

REDUCTION SCALE

Measuring the accuracy of artwork is much easier if a reduction scale is provided on the artwork (see Figure 9). For example, if a piece of artwork is 10 times the final size, it is recommended that two parallel lines, 10 inches apart, be drawn on the artwork. When the artwork is reduced to final size, the line will be one inch. This method will afford a greater degree of control on pattern dimensions.

REGISTER

In some applications, it is necessary to register two or more patterns with respect to each other, or to locate a pattern accurately with respect to some mechanical characteristic. Two-sided printed-circuit boards, multiple steps to produce semiconductor electronic devices, and patterns located with respect to previously drilled holes are examples of applications with typical register problems. The best way to achieve register depends, to some extent, on the application.

1. Make register holes or mark a part of the artwork, and carry them through all steps of the photofabrication process.
2. Place transparencies in register and tape them together on one edge so that the material coated with photoresist can be sandwiched between the transparencies. This is good only for thin materials. This method is not recommended when the artwork is to be stored, since the tape may "creep," with resulting misregister of the transparencies.
3. Drill register holes (usually three) in the work before it is coated with photoresist. Matching holes in the transparencies fit over pins in the register holes.

PHOTOGRAPHY

To achieve the required sharp, dense, precise photographic transparency, make certain that all elements of the photographic system achieve maximum performance. Make the artwork original accurately and illuminate it properly. Choose a suitable lens and camera system. Make certain that the image produced by the optical system is accurately focused and properly sized. Correctly expose and properly process the photographic material. Finally, take similar care in further reproduction of this master image by contact printing, duplication, step-and-repeat exposure, or other operations.

ARTWORK ILLUMINATION

First step in the reproduction process is the mounting of artwork in the rear-illuminated copyboard of a precision camera. Rear illumination of the artwork is extremely important, since this method of lighting provides the highest contrast between the clear and opaque areas of the artwork. Frequently, the copyboard is illuminated by green fluorescent tubes. If this type of lighting is not available, introduce a green filter material between the lamps and the back of the artwork (do not insert a filter anywhere between the artwork and the film).

There are several reasons for using green light. Most "process" lenses are intended for color work and therefore have compromise corrections. Monochromatic light from the middle of the spectrum makes best use of these compromises. Most of the photographic materials to be used for camera exposure are orthochromatic (blue and green sensitive). Green light is scattered less than blue light by silver halide grains in a photographic emulsion, so that images are sharper. Finally, using green light for focusing and for exposure will make visual focusing of the image more reliable; the eye is most sensitive to green, and the monochromatic green light will show little or no shift of photographic focus with respect to visual focus.

CAMERAS FOR PHOTOFABRICATION

The rear-illuminated copyboard must be accurately positioned in relation to the lens axis and the film or plateholder of the camera. The graphic arts process camera provides most of the current requirements for photofabrication although camera manufacturers are recognizing the stringent requirements of this process and are offering sophisticated cameras specially engineered for photofabrication and precision reductions.

Alignment of the camera elements is extremely important. The copyboard and the film or plateholder must be parallel, and they must both be perpendicular to the axis of the lens. Most precise alignment (without actual optical bench conditions) is achieved using an autocollimator. A portable autocollimator can be set up and the alignment of camera elements can be made in a minimum of time. Most suppliers of precision cameras offer this service at the time of camera installation, when changes in facilities require movement of the camera, or as routine maintenance checks.

THE CAMERA LENS

Most process-camera lenses are designed to give proper focus and size with exposing light of different wavelengths, since this is required for color reproduction. This design necessarily entails some compromise in the total lens design. Use of monochromatic light (single wavelength) will help to reduce residual errors uncorrected in the original design. Only a lens specifically designed and corrected to work at one wavelength and at one reduction ratio can approach the theoretical, diffraction-limited resolution.

The lens used for photofabrication must have a flat field with no distortion (curvature of lines) and little residual aberration. In most process lenses, stopping the lens down (reducing the aperture) one or two stops will reduce the effects of field curvature or astigmatism. Because of diffraction effects, however, the image will become less sharp with a further decrease in aperture.

MAKING THE REDUCTION

Two procedures are most important in achieving a useful image. These are correct focus and precise image-sizing. While most cameras come with devices to show reduction ratios and the proper positioning of the lens, copyboard, and film holder, these mechanical devices may not give best focus for photofabrication images.

Visual focus can be obtained with the aid of a magnifier at the film plane. Usually such a magnifier is mounted so that the image can be focused on either a ground-glass or a clear-glass screen. First, focus the magnifier on the image plane; then focus the camera by observing the image through the magnifier. Focusing should be done at the working aperture. A standard test chart can be used as an aid in focusing and for ultimate evaluation of the photographed image. Mount the chart on the copyboard next to the functional pattern, along with a *KODAK* Control Scale, T-14, or a *KODAK* Photographic Step Tablet. (Use of these devices for control of exposure level and processing conditions will be described later.)

Because visual focus and photographic focus may not be exactly the same (although they are usually very close with green light illumination), a photographic test of focus should be made for correlation. Refer to the film data sheet for the approximate exposure; make an exposure series at the visual focus setting. Once the approximate exposure is determined, make a focus series of exposures, moving the lens on both sides of the best visual focus. Use the film which you would normally employ for making photofabrication transparencies, but develop it in a soft-working developer such as a developer for continuous-tone films (*KODAK* Developer *D-76)* or a dilute paper developer *(KODAK DEKTOL Developer).* Examine the results with a powerful magnifier or a microscope to determine best focus.

Next, determine the correct image size. It may be that the focus adjustments have caused an appreciable divergence from the original size setting. The correction can be done by resizing and refocusing, or by direct computation of the required lens movement.

The following equations will give the required values.

$$\Delta u = -RF\frac{\Delta x}{x}$$

$$\Delta v = \frac{F\,\Delta x}{R\,x}$$

Where: Δu = the required change in object-to-lens distance,

Δv = the required change in lens-to-image distance,

R = the reduction ratio, defined so that when the image is smaller than the object, $R > 1$,

F = the focal length of the lens

Δx = the change in image size required to go from the measured size to the desired size, and

x = the desired image size.

The minus sign in the first equation means that if the object-to-lens distance increases, the image size decreases. In other words, if the image is too large and must be made smaller, Δx is negative, Δu is positive, and the object-to-lens distance should be increased.

Provision of a reduction scale in the original artwork makes accurate measurement of the reduced image much easier.

From the master image shown in Figure 9, make the reduction as shown in Figure 11. Check the camera for proper alignment; then bring the image to accurate size. From previous tests, determine an approximate exposure level for *KODAK PRECISION LINE* Film LPF7.

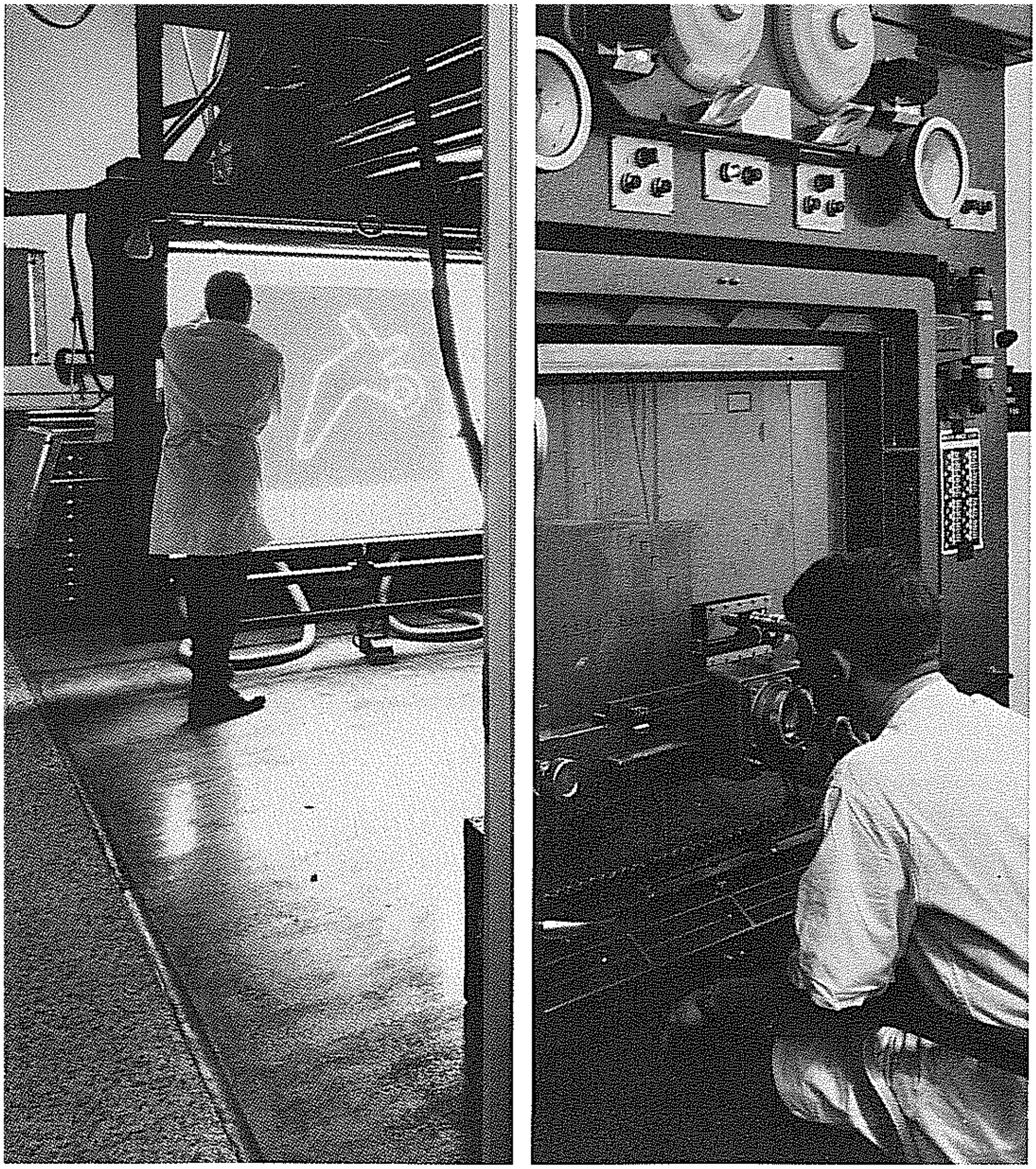

FIGURE 10: The artwork is placed on the backlit copyboard and the precision camera carefully focused.

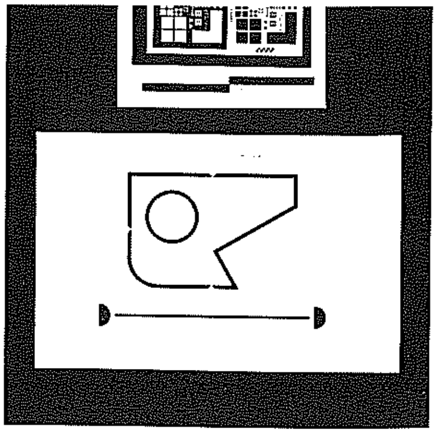

FIGURE 11: Reduced image made on *KODAK PRECISION LINE* Film LP7.

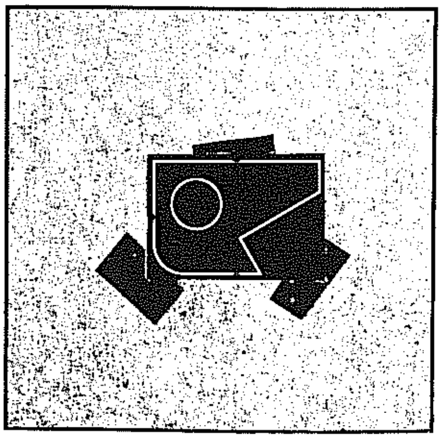

FIGURE 12: Contact print of camera reduction.

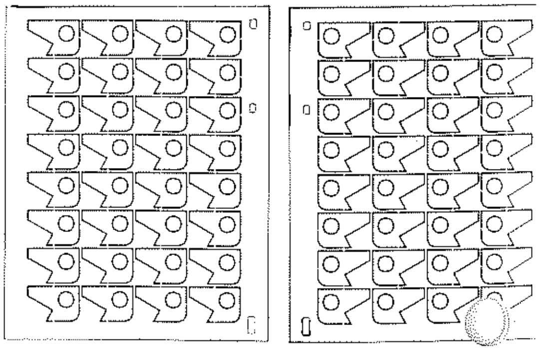

FIGURE 13: Reduced image was stepped-and-repeated; then a mirror-image duplicate was made in register.

Use an Image Test Chart and an opposed gray scale at the edge of the image area. These devices provide additional control of image reproduction. The image test chart helps to relate test exposures to the final reduction image shown here. It has various elements which provide a sensitive measure of image quality, image size, and exposure control.

An opposed gray scale provides a good visual control for exposure and development conditions. To construct such a scale, cut a *KODAK* Photographic Step Tablet, No. 2, in half lengthwise. Turn one piece and rejoin the strips so that the high-density end of one strip is opposite the clear area of the other. Use this opposed scale with all camera tests and determine how much separation or overlap occurs when film receives standard processing in fresh developer. To use this opposed scale guide in production jobs, move the strips so that "break" steps (where a sharp tone transition occurs on high-contrast films) adjoin. Direct-visual inspection will tell you immediately if exposure and development is correct. This is not meant to be a substitute for close control of all exposure and processing conditions but, rather, it is a constant check on them.

Use this camera negative directly for the first step-and-repeat procedure or use another piece of *KODAK PRECISION LINE* Film LPF7 in contact with this negative to make a transparency with image tone values like the original (as shown in Figure 12). The material used in the next steps will depend upon the image required for the working transparency. At any stage, a transparency can be duplicated, with the image remaining the same as the original, or with the tones reversed (a negative in the terms of conventional photography).

CONTACT PRINTING

Contact printing of the final working transparencies (shown in Figure 13) is not complicated, but it is here that careful attention to technique is important. Films must be in intimate registration. They must be properly exposed with a suitable light source.

Image orientation on the transparencies is important. For chemical milling from both sides of metal stock, matched, mirror-image transparencies are necessary. To make the matched transparencies, punch two pieces of negative-working film (such as KODAK PRECISION LINE Film LPF7), punch one with the emulsion up, and the other with the emulsion down.

Expose the first film to the step-and-repeat master (which has also been register-punched) with the emulsions of the films together. Expose the second film to the master (also aligned on pins with register punching) but with the emulsion of the master away from that of the film. Process the films identically, together if possible. The result is a well-matched set of transparencies with identical histories, made on the same film material.

An alternative method can be used to produce the matched transparencies. Expose a negative-working film emulsion-to-emulsion with the master. Process this film and use it for an emulsion-to-emulsion exposure with a positive-working film (such as *KODAK PRECISION LINE* Duplicating Film LPD7). Process this film and you have two mirror-image transparencies. This method avoids the out-of-contact step described in the previous method.

Expose films in a glass-topped or glassless vacuum frame. Make sure that the vacuum pump is operated long enough to allow complete and even contact between the unexposed film and the master transparency.

Although broad-source high-intensity lights minimize the recording of dust particles, there are advantages to using point-source lights. A point-source contact-printing light with a variable voltage transformer is suitable. Such a light will minimize image "undercutting" and give sharp, true-width lines whether film emulsions are in contact or whether exposures are made through the film base. Use the light at some distance from the vacuum frame to give a quasi-collimated beam which gives sufficient coverage and controllable exposure times (10 seconds or more).

Maintain register in this and subsequent operations by punching films with a suitable register punch and using register pins to hold films during exposure.

DIMENSIONAL STABILITY OF PHOTOGRAPHIC MATERIALS

All photographic materials vary dimensionally as a result of environmental temperature changes, and many also vary with changes in relative humidity. An increase or decrease in temperature causes a corresponding expansion or contraction of the material. Relative-humidity size changes are caused by the swelling or contraction resulting from absorption of moisture or from drying out. Since polyester materials have a very low moisture-absorption capability, the effect of relative humidity is much less on them than it is on paper, acetate, or acrylic plastic. Glass plates are not affected by changes in relative humidity.

Size changes in film or glass resulting from temperature changes can be counteracted simply by restoring the temperature condition that prevailed at the time of exposure. Size changes in film caused by relative humidity (glass is not affected by relative humidity) can to a great extent also be counteracted by restoring the original conditions. (Approximately 7 hours are required.) Size changes are best maintained by controlling temperature and relative humidity in the areas where such materials are used and stored.

PROCESSING TECHNIQUES

For optimum processing to be effective, the film or plate must have received the correct exposure. All photographic materials have some exposure latitude but, as the image requirements are increased, this latitude is decreased. One of the major attributes of *KODAK PRECISION LINE* Film LPF7 is that a considerable degree of exposure latitude is available.

Once the proper exposure level is achieved, standardized time/temperature controlled processing is most reliable. Attempts to rescue slightly overexposed or underexposed materials by processing variations can only lead to poor quality and incomplete process control.

Photographic materials for photofabrication are designed for optimum processing in one or two selected developers. Alternative recommendations are given to allow the user to choose developers which may offer convenience in mixing, a longer processing life, or higher capacity. For highest quality images, use the primary developer recommendation.

All processing steps from development to drying should be carefully controlled. The photographic image may be damaged by improper fixing, inadequate washing, or improper temperature control. Dimensional stability of films is related to the processing history of the film as well as to pre- and post-processing treatment.

The development techniques employed with each photographic material should be standardized, particularly in production situations. Use of the same developer for the same material, constant time and temperature of development, and consistent agitation procedures minimize development differences and aid considerably in producing uniform results. While variations in developer temperature can cause considerable variation in results, the most frequent cause of processing changes is improper and inconsistent agitation techniques during development. Only by proper agitation can uniform development and freedom from flow marks and pinholes (caused by bubbles) be provided.

Processing solutions, particularly developers, should not be used to complete exhaustion. For production installations, replace used solutions with freshly mixed solutions on a regular schedule, based both on the quantity of materials processed and on the time the solutions have been in use.

FILM PROCESSORS

Although the time and space saving and reduction of chemical costs are considerable, perhaps the chief advantage of mechanized processing is optimizing control over processing. Since the usual variables of time, temperature, developer strength or activity, and agitation are automatically maintained, the user can be sure of the delivery of a clean, dust-free negative.

Since the entire area of the film being processed is constantly flooded with fresh, active developer, dust particles are literally swept away. Line negatives emerge from the drying section with substantially fewer pinholes than with tray processing, and, thus, less opaquing is required.

Better, faster drying can improve size holding. Warm, forced air combined with squeegee rollers all but eliminates any possibility of streaks caused by uneven drying. The thermostatically controlled drying chamber yields uniformly dried film for dependable control of dimensional stability.

Mechanized processing is recommended for these photofabrication films:

KODAK PRECISION LINE Film LPF7.
KODAK PRECISION LINE Film LPD7.

METAL PREPARATION FOR RESIST COATING

It is very important to choose a metal that has the necessary physical properties for etching. Grain size and grain structure are important. Often, the poor etching quality of a metal can be traced to a differential in etching rate between adjacent grain boundaries. Another important factor to consider is the manner in which the metal was rolled or formed. Rolling can sometimes cause the surface of the metal to have a much greater resistance to etching than does the metal itself. Thus, the metal has much less solubility in the etchant, and control of etch factors may become troublesome.

For this and other reasons metal suppliers have introduced "etching grade" materials. These materials have been designed and manufactured solely for chemical milling. Since data on these materials are extensive, we suggest that you contact metal suppliers for further information on available types.

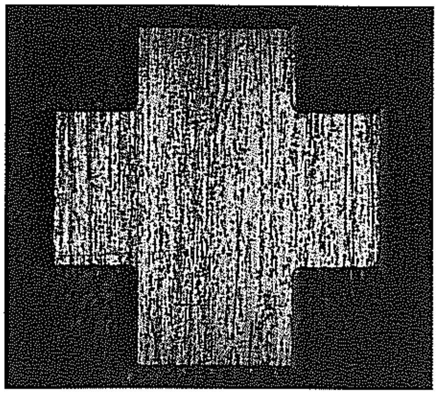

FIGURE 14: Scratched surfaces will produce an effect similar to the one shown here. Note that the surface is badly marred because of severe resist breakdown.

SURFACE PREPARATION

The condition of the metal surface that receives a coating of resist is as important as the physical properties of the metal itself. Avoid metal that has been badly marred or deeply scratched. Unidirectional scratches (caused by a belt sander, for example) are bothersome, especially when thin resist coatings are used. As a thin coating of resist dries, it tends to "bridge" the scratch. This leaves the bottom of the scratch unprotected, and a tiny capillary that can actually retain etching solution forms. As etching progresses, this capillary will open up even more and cause a loss in quality or a ruined part.

The cleaning cycles used prior to coating with a photoresist must be effective. Undercleaning a surface will often result in complete photoresist failure. Overcleaning will not usually result in resist failure, but may change the surface properties of the metal. Thus, the kinds of surface contamination must be analyzed and the most effective way of removing them must be found.

VAPOR DEGREASER—For most purposes, oil and grease can be removed from a metal surface in a vapor degreaser. For photofabrication processes, the degreaser should be charged with trichloroethylene. Trichloroethylene is both an efficient cleaning agent and a developer for exposed coatings of KTI KPR Resist, KTI KPR Resist, Type 3, and KTI KPR Resist, Type 4.

Vapor degreaser equipment is simple to use. Merely lower the oil- or grease-laden metal into a vapor phase of the degreaser and hold it there for a short period of time. Upon withdrawal the metal will, in most cases, be free from oil or grease.

EMULSIFIED SOLVENT CLEANERS—Sometimes it appears that vapor degreasing has failed to remove all contamination. This soil is a nonorganic type (such as mineral-bearing earth) which has been picked up by the metal during handling. The oil or grease is removed in the vapor degreaser but the insoluble contamination stays behind. It may, in fact, become more firmly attached to the surface after vapor degreasing. If you find this condition, use an emulsified-solvent cleaning solution. Give the solution a water rinse to remove the emulsified solvent and the soil it carries.

A spray rinse increases cleaning efficiency when applied to the metal after it is withdrawn from the cleaner. If cleaning efficiency is increased, better adhesion of the photoresist to the surface can be expected.

DETERGENT CLEANERS—Alkaline or detergent cleaning are also useful for oil and grease removal. Alkaline cleaners work best on light films or oily material on the surface. Use them to remove these films and as a regular part of a cleaning cycle.

ELECTRO-CLEANING—Another method of cleaning—and one that offers vigorous agitation—is electro-cleaning. A direct current flows between the metal to be cleaned and another electrode while both are immersed in the cleaning solution, generating large quantities of gas which impart a "scrubbing" action to the surface of the metal. This results in efficient cleaning.

After any chemical cleaning cycle (except vapor degreasing), the surface must be given a thorough rinsing. Either a double water rinse in rapidly changing water or, better still, a spray rinse should be considered the minimum.

Follow either rinsing technique with a weak-acid dip. Many alkaline and detergent cleaners leave trace amounts of the cleaner on the surface. If this is not removed from the surface, problems with resist adhesion can occur. The water rinse and acid dip also give an opportunity to check on the effectiveness of the cleaning cycle. An indication that a surface has been cleaned properly is its ability to hold an unbroken film of water over the entire surface without the aid of surface-active agents such as detergents. Once the surface is clean, subsequent processing steps should be carried out without delay.

MECHANICAL CLEANING—The surfaces of metal intended for photoetching or photomilling processes can also be cleaned by scrubbing with a bristle brush that has been charged with pumice. This method is acceptable for materials that have good mechanical strength, but it should be avoided when thin foils are being processed. Normally, thin foils do not have enough strength to survive the rigors of mechanical abrasion and may dimple or crumple even though they are handled carefully.

ACID CLEANING—Organic coatings adhere best to a surface that has been acidified prior to coating. Use acid cleaning for removing oxides and other inorganic matter which are part of the metal surface. Acid cleaning often removes surface-active residues carried over from alkaline cleaning methods.

Although it is extremely important to have a fresh, clean surface for photoresist coating, do not use excessively strong acidic solutions for cleaning. Dilute acid dips are more desirable, both for safety in handling and for the relatively slow rate with which they attack metals.

CONVERSION COATINGS

Some photoetching or photomilling processes for deep-etching applications (approximately 0.005 inch deep—or more) should include some sort of conversion-coating technique in the process line. Shallow etching often does not require a conversion coating. A conversion coating is a treatment that either oxidizes, reduces, renews, or applies a different substance to a metal surface for the purposes of increasing photoresist adhesion and decreasing lateral etching. Table 1 indicates some recommended conversion coatings and the metals to which they should be applied.

Conversion coatings give more favorable etch factors. Any etching process causes some undercutting of the pattern, but with a conversion coating present, undercutting is reduced. This greater control is the result of better resist adhesion and the *differential etching* rate introduced between the surface and the body of the metal; the surface etches more slowly than the metal itself.

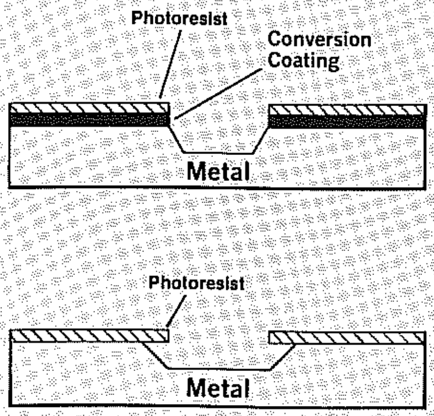

FIGURE 15: Differential etching of conversion coating and metal allows improved size-holding in patterns.

The effect of a conversion coating is illustrated in Figure 16. In each example the material is aluminum that has been etched in a 20 percent sodium hydroxide solution. Here, the advisability of having a conversion coating is obvious.

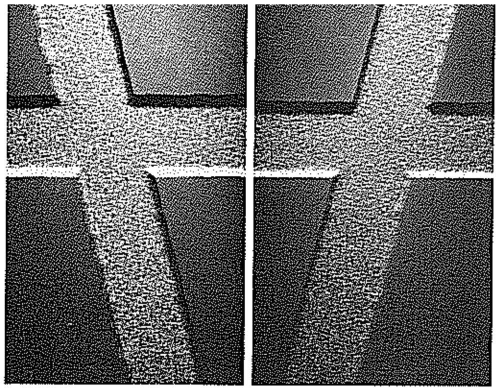

FIGURE 16: Without conversion coating (left) with conversion coating (right).

TABLE I
CONVERSION COATING

ALUMINUM[1]	Use a chromate conversion-coating bath.
COPPER AND COPPER ALLOYS[2]	For most purposes, the surface should be cleaned thoroughly and then given an acid dip. Heavy oxide buildups or chromate conversion coatings on copper are not normally needed, except when testing indicates their value for a particular application.
MAGNESIUM[2]	Use a 2 percent (by volume) concentrated phosphoric acid and water solution. Immerse for about 30 seconds. Use the solution at room temperature.
NICKEL	Use a solution consisting of 1 volume of concentrated phosphoric acid and 3 volumes of water. Immerse for 3 minutes. Use the solution at 75°C (167°F).
SPRING STEEL	The blued finish on many spring steels is essentially an oxide finish caused by tempering. It can be used as a conversion coating provided that either the metal thickness or the depth of etch does not exceed 10 mils (0.010 inch).
STAINLESS STEEL	A 20 percent (by volume) solution of nitric acid and water should be used. Immerse for 15 to 20 minutes. Use the solution at 66 to 71°C (151 to 160°F).
ZINC[2]	A 2 percent (by volume) solution of concentrated phosphoric acid and water should be used. Immerse for about 2 minutes. Use the solution at room temperature.

[1] Extremely heavy deposits of chromate conversion coatings should be avoided because problems in resist adhesion can occur. Coatings which exhibit a "powdery" appearance indicate that the conversion coating is not suitable for further processing.

[2] Conversion coatings may work for these metals. However, such coatings must be evaluated thoroughly with respect to process performance (photoresist adhesion and etch factors) before being used as an integral part of the process.

RECOMMENDED PRACTICES FOR USING KTI RESISTS

FINAL SURFACE TREATMENT BEFORE PHOTORESIST APPLICATION

To complete a chemical surface treatment satisfactorily, use suitable washing and drying methods. Wash the metal only to the point of removing the residues caused by the chemical-treatment step. Prolonged washing will often result in redepositing contaminants on the surface. Such an effect can be seen on a clean copper surface that has become oxidized as a result of lengthy soaking in water.

Use of compressed air is generally an effective drying technique, but the compressed air must be carefully filtered so that vaporized oil does not reach the surface. The oil would recontaminate the surface that had just been cleaned and treated.

Drying is normally done at room temperature so that reoxidation of the surface is minimized. However, residual amounts of moisture may be left on the surface, even though drying *appears* to be complete. Such moisture, if not removed, may cause difficulties with photoresist adhesion to the surface. Therefore, final drying should be in a film-drying cabinet, or similar device, set at a temperature of 49°C (120°F).

If the warm air drying step is used as part of the process, allow the surface to return to room temperature before the resist is applied. When a coating is applied to materials at elevated temperatures, solvent entrapment in the photoresist may occur. As with moisture retention, solvent entrapment can also spoil adhesion during subsequent processing steps.

Steel and several other metals adsorb quantities of gases (such as hydrogen) as a result of cleaning steps or chemical treatments. If allowed to remain in the metal, these gases can lift the resist off the surface during prebaking or etching cycles. Bake the metal prior to coating to drive these gases out of the surface. Use a time-temperature cycle of 120°C (248°F) for 10 minutes. A longer time may be necessary for materials in large pieces or greater thicknesses.

SAFETY AND HEALTH CONSIDERATIONS

Before handling or using KTI products, read and understand the Safety and Health Considerations section on page 32.

VENTILATION

With good general ventilation, solvent-vapor concentration will usually be within safe limits if photoresist materials are used only at brief intervals throughout the day. If a large volume of work is done, local exhaust may be needed in the vicinity of the developer and dye-bath tanks and in the resist-application area. Special or elevated heat treatment of the resist, as in extreme postbaking, may require local exhaust ventilation because of solvent-vapor loss from the coating.

VAPOR DEGREASERS

Techniques for volume developing that employ a spray and sump-type vapor degreaser and solvents such as trichloroethylene and trichloroethane are often used for photoresist development. The vapor degreaser must have the same controls for solvent vapor levels as any other standard vapor degreaser. The degreaser must not be located near a carbon-arc lamp, since phosgene gas, a deadly poison, may be generated.

TANKS

Loss of volatile solvents from resist, developer, and dye solutions can be minimized by using narrow, vertical stainless steel tanks which are kept tightly closed when not in use.

SPRAY COATING

Spray coating of KTI photoresists should be done in a well ventilated, paint-spray booth; in a "laminar-flow" spray booth designed for photoresist application; or in a chemical fume hood if processing is done on a laboratory scale.

DYE REMOVAL

If KPR Dye has been accidentally spilled on the hands, it can be removed by using one of the emulsifying-type or "waterless" hand cleaners. Try to remove the dye as soon as it is spilled. The longer it remains in contact with the skin, the more difficult it becomes to remove.

GENERAL WORKING-AREA CHARACTERISTICS

CLEANLINESS

Give careful attention to cleanliness in the general working area when using KTI photoresist products. Contamination of airborne dust and dirt can come from a variety of sources. Often, a manufacturing operation, such as cutting printed circuit stock, can cause a number of problems if the operation happens to be adjacent to the photofabrication work area. Dust from the clothing of personnel is another source of contamination. A very serious contaminant, and one that is extremely difficult to overcome, is silicone. Silicone release agents, occasionally used on printed circuit laminating and molding press plates, contaminate a surface so badly that photoresist adhesion is practically impossible. Finally, contamination may arise in a spray hood when particles of dried resist break free from inside the booth and fall on the freshly coated surface.

General working-area cleanliness therefore is of utmost importance. One type of contamination can lead to another until they build up to a point where photoresist processing is seriously affected. The method of control chosen, or the number of steps used, rests ultimately with the user and the environmental conditions of the particular area.

RESIST STORAGE

See Safety and Health Considerations section in rear of this publication. It is recommended that KTI photoresists be stored in their original containers. AISI Type 316 stainless steel is satisfactory and can be used for dip tanks, mixing vessels, and spray-gun components. *Do not* use copper, brass, or zinc-lined materials, since they may contaminate the photoresist.

SAFELIGHTS

When establishing a safelight procedure for a particular area, consider the following factors: level of illumination, distance from the working surface, and coating thickness of the photoresist. Each will affect the performance of the photoresist coating, and each should be evaluated with respect to process compatibility. Certain guidelines should be observed for all KTI photoresists.

The most commonly employed safelight for KTI photoresists is the gold fluorescent tube. Although light from these tubes will not detract from the capabilities of a photoresist, they should be used with caution. With gold fluorescent tubes the filter may lack efficiency around the ends of the tube. If this is not corrected, an inordinate amount of ultraviolet radiation will leak out into the room and nullify any attempt to make the area "safe." To overcome this, wrap the ends of the tube with opaque tape before installing the lamps in the fixtures.

For a more positive system of safelighting, use red fluorescent tubes. Although the working level of illumination with red tubes is not as high as with the gold, red tubes should be used if coated stock is to be left uncovered for an appreciable length of time. In some production areas, it is common to find red fluorescent tubes installed in the resist coating area, while the rest of the area is illuminated with the gold-colored tubes to provide a reasonable level of working illumination.

Other methods of safelighting are available. These include yellow, "anti-bug" incandescent bulbs.

Several methods are also available for filtering out ultraviolet radiation at windows. One method is to cover the windows with *KODAGRAPH* Sheeting, (orange). These measures, however, present the same difficulties as the gold fluorescent tubes and, in addition, the *KODAGRAPH* Sheeting, (orange), must be replaced periodically because of fading. A much better solution is to blank out the windows completely with an opaque material.

After choosing a safelight procedure, check it out by a simple light-fog test. To do this, place a line transparency in contact with a freshly coated resist surface. Then, expose the resist-transparency sandwich to the safelight illumination. Cover one half of the plate during this exposure, which should be longer than the amount of processing time between coating and development. Then expose a step tablet on both the "fogged" and protected halves of the plate, using a normal exposure for the resist under test. Fogging can then be determined by developing the images and comparing the two step-tablet images.

FILTRATION OF RESISTS

For many applications, KTI photoresists are adequately filtered as packaged. For the more critical applications, or with some types of coating equipment, it may be advisable to filter the resist before it is used.

If results indicate a need for additional filtering of the resists, first check carefully to determine the source of contamination. Housekeeping is very important. In many instances, more attention to the cleanliness of equipment and work areas will make additional filtration unnecessary.

When filtration is necessary, use one of the commonly accepted methods such as the gravity or the pressure system. For the gravity system, line a funnel with regular laboratory-type filter paper and place a piece of glass wool or similar material on top of the paper at the apex of the funnel. Pour a small quantity of the appropriate thinner through the funnel and discard. This will remove any foreign material from the filter paper and will make sure that no particles of filter paper will be caught up by the resist as it passes through the funnel. After rinsing the filter, proceed with filtration of the resist. The gravity method provides "coarse" filtration.

To provide pressure filtration of resist, apply compressed air to the container and force the resist through the filtering media. This system allows a finer degree of filtration than does the gravity system. When using equipment of this type, make sure that the compressed air supply is shut off before the last bit of resist goes through the filter. Failure to shut off the air supply generally results in recontamination.

RESIST VISCOSITY AND COATING THICKNESS

PHOTORESIST VISCOSITY

Coating thicknesses and coating procedures relate directly to the viscosity of the photoresist. Control of resist viscosity is important for uniform performance of the resist. Viscosity is influenced by the ambient temperature and by the degree of resist dilution.

The viscosity of KTI photoresists is controlled closely in manufacture. In use, the resist will lose solvent by evaporation and the resist viscosity will increase. To control the resist viscosity, add the appropriate thinner and mix the resist thoroughly. After adding thinner to the resist and mixing the solution, allow time for air bubbles to rise and dissipate.

Measurement of viscosity with a Cannon-Fenske pipette or Zahn cup viscometer is relatively simple. Manufacturers of each instrument supply instructions for proper use. Make all viscosity measurements at a fixed temperature as temperature variations will affect the viscosity. Absolute viscosity measurements may not be required; obtaining the same time-rate flow is frequently sufficient to indicate that the resist is at the starting point level and, hence, the coating thickness control will be maintained.

COATING THICKNESS AND RESOLUTION

In photoresist processing, it is impractical to predict a certain coating thickness for a specific application. Coating techniques and processing requirements vary, and the end result may require more or less than a specific coating thickness. Thus, for any given application, determine the best operating coating thickness by comparative testing.

A particular coating thickness will generally yield an image of a certain resolution. With KTI KTFR Resist, for example, a coating thickness of 0.4 micrometer (0.000016 inch) will give photographic resolution of lines and spaces down to 1 micrometer (0.00004 inch) in width. A coating thickness of 5 micrometers (0.0002 inch) provides photographic resolution of line and spaces down to 125 micrometers (0.005 inch) in width. Therefore, for images requiring very high photographic resolution, use thin coatings; for those requiring lesser resolution, a thicker coating is effective.

Exposing sources, developing techniques, and material surface conditions affect the resolution capabilities of a photoresist layer. Also, the type of resist used and the method of its application influence overall processing requirements. The selection of a particular coating thickness or a particular resist, therefore, often depends not on the resolution needed, but on the *overall processing procedure*. The selection of a coating thickness should represent the best balance between resolution, chemical performance, and process reliability for that application.

Coating thickness measurements can be made with nondestructive testing instruments which use beta radiation backscatter, ultraviolet reflectance, electromechanical profile, or light interference methods. Each of these is designed to measure a range of coating thicknesses.

VISCOSITY MEASUREMENT

TABLE II

Resist	*Viscosity Range (at 25°C) in centistokes*
KPR	11.6- 13.0
KPR Type 3	32 - 36
KPR Type 4	435 -515
KTFR	540 -618

TABLE III

Viscosity Range (centistokes)	*Cannon-Fenske Pipette*	*Zahn Cup*
3- 15	100	—
20-100	200	1 or 2
50-250	300	2 or 3
250-960	400	3 or 4

COATING METHODS

DIP COATING

Dip coating is the simplest method for coating thin metal foils. Simply immerse the cleaned foil in the resist, withdraw it, and hang the coated metal up to dry in a dust-free area. Equipment requirements are simple—a stainless steel tank with a narrow opening at the top is essentially all that is required.

Resist coatings produced by simple dipping are always wedge-shaped, with the "thin" end of the coating at the point where the work was hung up to dry. For some applications, this may be of no concern. For others, however, wedging may interfere with process reliability. If wedging causes any difficulty, it can be partially overcome by recoating the work a second time, but in the opposite direction. After coating, hang the work with the thicker end of the first coating uppermost.

FLOW COATING

Flow coating requires no coating equipment and minimum manual skill. Merely pour a pool of either diluted or undiluted resist on the coating surface while the plate is held in a horizontal position. Then tilt the plate variously to allow the resist to flow over the entire surface. Finally, prop the plate in a nearly vertical position and allow it to dry at room temperature.

The coatings obtained by this method are somewhat wedge-shaped. If wedging does not interfere with process requirements, flow coating is the simplest method for coating flat-surfaced plates. If wedging is undesirable, the surface may be flowed with a resist a second time and allowed to dry in a nearly vertical position with the side opposite the wedge facing down.

CONTROLLED WITHDRAWAL

A controlled withdrawal method is a refinement of the simple dip-coating process. In controlled withdrawal, the work is lowered into a tank of resist, held there for a few seconds, and then withdrawn at a constant rate of speed. To facilitate drying, some coating machines have an infrared heating station after the coating step. For most applications, controlled withdrawal methods offer better control of coating variables than simple dip coating.

Performance of controlled withdrawal coating methods is influenced by four major variables—withdrawal speed, resist viscosity, resist solids, and temperature.

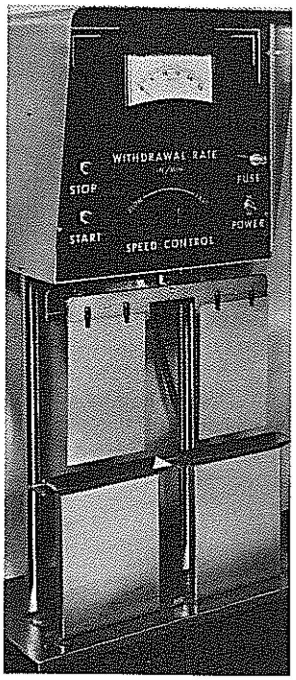

FIGURE 17: Controlled withdrawal is a refinement of dip coating.

Since the four factors are interrelated, all must be controlled to achieve a satisfactory resist coating. As any one variable changes, coating conditions change and adjustments must be made to regain the proper resist coating thickness. For instance, the viscosity of the resist tends to increase because of the natural evaporation of resist solvents in the coating process. Increased resist viscosity will lead to thicker coatings unless the resist is diluted or the withdrawal speed reduced.

Thin resist coatings are formed when the withdrawal rate is slow; thicker coatings are produced by increasing the withdrawal speed. Slow withdrawal speeds allow much of the resist to flow off the surface before the metal leaves the tank. Faster withdrawal minimizes this effect. Flow properties of the resist relate to viscosity and temperature as well as to surface tension and related forces.

Elevated temperatures will cause a decrease in viscosity until resist solvent evaporation begins. As the solvent evaporates, the resist viscosity will begin to increase. Because this temperature/viscosity relationship is complex, temperature control of the coating area is important if uniform coating conditions are to be maintained.

ROLLER COATING

Roller coating is another machine-assisted method of applying photoresists. In this method, the cleaned stock passes through a set of rollers which transfer photoresist to the stock. Coatings obtained from this method are extremely uniform, can be applied rapidly, and can be controlled easily.

A roller-coating method works well with rigid stock in such applications as the coating of panels for nameplates or coating printed circuit laminants. Thin foils can also be coated on these machines if the foil is backed up with a rigid plate or other stiff material. Accurate coating thickness can be applied to continuous webs of metal held under tension.

Several variables can affect roller coating. The most important are: resist viscosity, transport speed (the speed at which the stock passes under the coating roller), and transfer pressure (the amount of "squeeze" given to the stock as it passes through the roller system).

Typical roller coaters work basically like the machine shown in the schematic diagram in Figure 18. The drive roller moves the material to be coated beneath the applicator roller. The applicator roller functions as its name implies; it applies the resist to the work surface. Resist is held by the doctor blade and applied to the applicator roller. Control of the resist thickness is provided by mechanical pressure control and viscosity adjustment.

KTI KPR Resist, Type 4, is designed expressly for roller coating. It yields excellent results on copper and copper-base alloys. KTI KPR Resist, Type 4, is similar to KTI KPR Resist, Type 3, in handling characteristics.

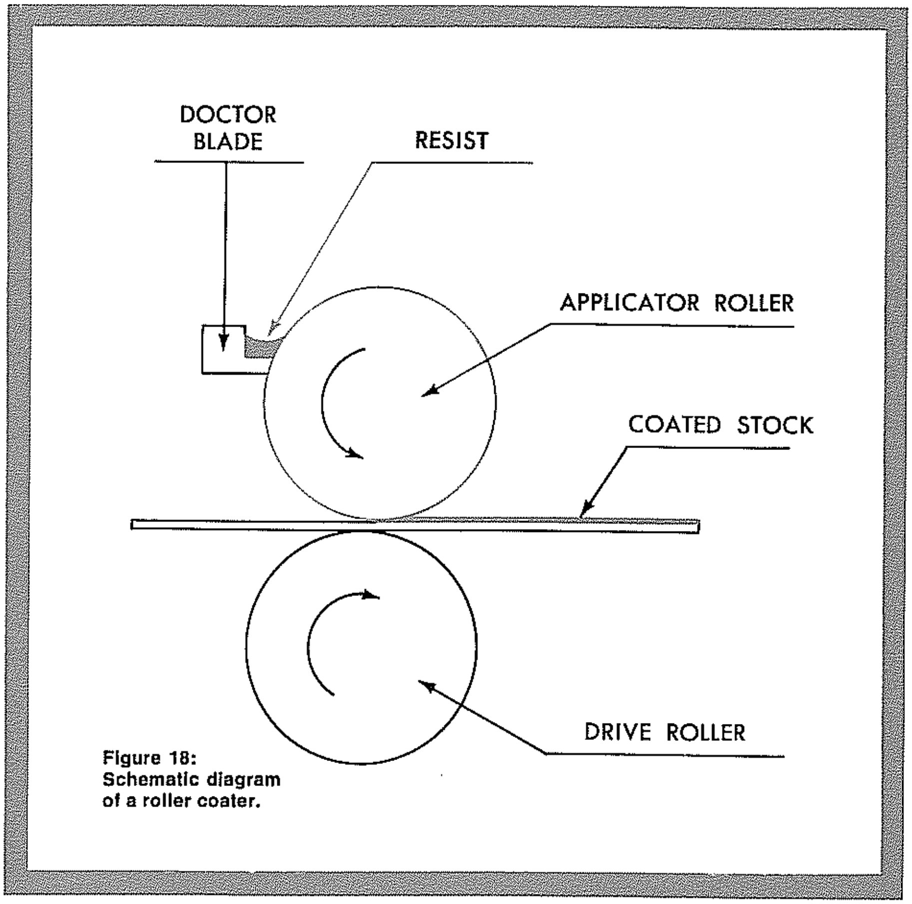

Figure 18: Schematic diagram of a roller coater.

SPRAY COATING

A typical spray-coating method includes a stainless steel spray gun and reservoir and a supply of clean oil- and water-free compressed air. Improper resist adhesion may occur when oil or moisture is deposited with the resist. Spray guns that operate without air atomization ("airless" systems) are more effective than conventional methods.

For spraying, dilute resists as necessary. The following table shows common dilutions of KTI resists for application by spray methods. These dilutions are typical and may have to be modified for a specific application.

Follow these steps to establish a spraying technique.

1. Dilute the resist to allow uniform spraying and buildup of coating thickness.
2. For air-driven systems, adjust the air pressure so that an operating pressure of 20 pounds per square inch is available at the spray gun. For the airless systems, follow the manufacturer's directions.
3. Operate the spray gun at a distance of 12 to 14 inches from the surface.
4. Use the spray gun in a well ventilated spray booth.
5. Use a nearly vertical easel as a holding device for the work. If the work does not have good mechanical strength, such as thin metal foils, fasten the material to a rigid backing plate.
6. Make certain that the surface of the work is at room temperature and free from dust.
7. To apply the resist, start at the bottom edge of the panel, move the gun back and forth horizontally at about 1½ feet per second, and progress to the top edge. Advance the spray upwards about 2 inches between each pass to cover the entire surface with a uniform, wet coat. If the coating appears thin, apply a second coating at right angles to the first. Immediately place the surface in a horizontal position to allow the coating to level off for 30 to 60 seconds; then dry the coating at room temperature until it is no longer tacky. For thicker images, give multiple spray applications, prebaking the coatings between applications.

"Cobwebs" or fine resist stringers in the spray hood indicate that the resist needs either initial or further dilution. Conversely, extremely thin or discontinuous coatings indicate that either the resist has been diluted too much or the speed with which it was applied was too great. If dilution adjustment does not help, check the spray gun for possible trouble. Refer to the manufacturer's recommendations on the spray equipment.

TABLE IV

TYPICAL RESIST DILUTIONS FOR SPRAYING

RESIST	DILUTION
KTI KTFR	4 parts resist 5 parts KTI KTFR Thinner
KTI KPR, Type 3	1 part resist 1 part KTI KPR Thinner, Type 3
KTI KPR	Full strength

PREBAKING THE PHOTORESIST

A prebake removes residual solvents in the photoresist layer before it is exposed. Retention of solvent in thick photoresist coatings can reduce photosensitivity. If the resist is not dry, the response of the material to ultraviolet radiation is low and adequate exposure cannot take place. The image will either be degraded or will slough off the surface when it is developed. A thin coating, however, may not require a prebake. Tests will determine prebaking requirements for thin resist coatings.

The following table lists prebaking times and temperatures. Note that the figures given are *typical*. Adjust the prebake times for large or thick materials.

TABLE V

TYPICAL PREBAKING TEMPERATURES FOR KTI PHOTORESISTS

KTI KPR Resist	10 minutes at 80°C (176°F).
KTI KPR Resist, Type 3	10 minutes at 80°C (176°F).
KTI KPR Resist, Type 4	10 minutes at 80°C (176°F).
KTI KTFR Resist	Not more than 20 minutes at 80°C (176°F). Temperatures over 104.5°C (220°F) may adversely affect adhesion.

EXPOSURE OF PHOTORESIST COATINGS

PHOTOGRAPHIC SPEED

Several factors influence the photographic speed of photoresists. The exposure required by the photoresist depends upon the coating thickness, the retention of solvents within the resist layer, the nature of the surface upon which the resist is coated, and the spectral output of the exposing source.

RESIST THICKNESS—Thicker resist coatings require more exposure. Complete exposure must take place from top to bottom of the resist coating. Since the radiant energy is absorbed as it penetrates the resist, more exposure must be supplied to reach the bottom of thick coatings.

SOLVENT RETENTION—Insufficient drying of the photoresist coating will allow retention of solvents which will inhibit exposure. In liquid form photoresists are relatively insensitive to radiation. As they dry, the resist sensitivity increases. Small amounts of residual solvent can affect the photographic speed appreciably. To avoid this condition, prebake the resists prior to exposure.

SURFACE COLORATION OR REFLECTIVITY—Reflectivity or color of the coated metal can influence exposing efficiency. Shiny surfaces will reflect radiation back into the coating, thus reducing exposure times. Dull surfaces will absorb much of the energy penetrating the resist coating and thus prolong exposure. Surface color, such as on dyed anodized aluminum, may have an unpredictable effect on exposure requirements.

EXPOSING SOURCES—The exposing source must provide energy at the wavelengths for which the resist is most sensitive. The peak sensitivity of these resists is in the near-ultraviolet region, between 280 and 550 nanometers. Typical exposing sources include carbon-arc lamps, mercury-vapor lamps, pulsed-xenon lamps, and unfiltered ultraviolet fluorescent tubes. In fact, any high-energy light source that is rich in the near-ultraviolet region of the spectrum may be used. Ultraviolet fluorescent tubes may be positioned a few inches from the resist-coated material. Other lamps should be positioned 0.6 to 0.9 meter (2 to 3 feet) away so that their heat is dissipated.

A factor not common to all resists, but one that may cause exposure difficulties, is the oxygen sensitivity of some resists. This effect is easily overcome by making exposures in a vacuum frame. Printing all photoresist images under vacuum is a good practice. In addition to excluding oxygen from the surface, the vacuum frame affords extremely close contact between the master transparency and the resist surface. Close contact of transparency and resist promotes improved image reproduction.

DETERMINING THE PROPER EXPOSURE

KTI photoresists are versatile materials and are used in a wide variety of applications. As a result, specific exposure recommendations are difficult to make. Since process techniques vary, exposures may range from a few seconds to several minutes at 1 meter (3 feet) from a mercury-vapor lamp. Thus, the optimum exposure in this range must be found with respect to the particular requirements of each process.

Optimum exposure is the exposure that gives the best reliability and quality in a process. It will be different for different procedures. If the exposure is not optimum, variations in the other steps become more critical.

Optimum exposure levels can be established as outlined in the following sections.

PREPARING AN EXPOSURE TEST PLATE

1. Prepare and coat a plate large enough to make a series of exposures, following the exact steps to be used in actual production. Do not vary these steps because the exposure values obtained depend on the entire processing procedure. When process changes are made, a new test plate should be processed to determine the effects of the change on best exposure conditions.
2. Use a test pattern that is meaningful to the process under consideration. The transparency should be large enough to cover the entire plate.
3. Make a series of exposures. Do this by exposing the resist-coated plate at increments through a test-pattern mask. Start with an arbitrarily established base figure such as 1 minute. Then, for each increment, double the exposure time.
4. Develop the plate.

INSPECT THE TEST PLATE

Check the test plate for the following results:

1. If there are a few satisfactory exposure steps, they then define the useful exposure range for the particular procedure used. Pick the middle of this range for the best exposure.
2. If there is only one satisfactory exposure step, it can be used as the best exposure. However, any slight variation in processing conditions would present difficulties.
3. If no satisfactory exposure steps are found, make additional test plates, using a range of exposures above and below those used on the first plate. If this still fails to produce the desired results, examine the other steps in the process, particularly the surface preparation, coating, and prebake steps.
4. The object of all of this is to be able to spot a processing or exposure difficulty as soon as it occurs. If, on a series of plates apparently handled identically, the best exposure and the exposure range vary from plate to plate, then one or more of the steps in the procedure lack sufficient control or the exposure is nonuniform.

THE USE OF A STEP TABLET AS AN EXPOSING GUIDE

A *KODAK* Control Scale T-14, (Figure 19), is a very useful tool to use to establish exposure levels. The step tablet is a piece of film that has been exposed to yield steps of increasing density. Each step in the tablet is numbered.

The steps range from a density of 0.04 (Step 1) to a density of 2.05 (Step 14), with each step increasing in density by 0.15.

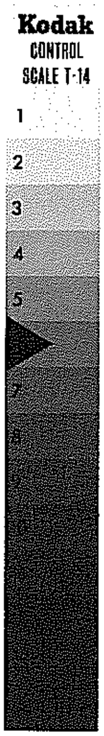

FIGURE 19: A *KODAK* Control Scale T-14, is a reliable control device for use in establishing and maintaining standard conditions of exposure.

Step-tablet control will show subtle changes in the exposing conditions when used in the following manner:

1. Use the step tablet with each series of test exposures made on a plate.
2. Make the exposures as directed in the preceding section, "Preparing an Exposure Test Plate," and develop the plate.
3. Note the number of the last visible step that corresponds to the most satisfactory exposure.

You now have an arbitrary number that defines the critical, or best, exposure step in an exposure area. This number can then be used to control the best exposure, irrespective of changes in the light source, exposure time, or other variables. Best exposure is duplicated by maintaining exposures at the proper step, regardless of conditions. A break at the sixth step of the *KODAK* Control Scale, T-14, usually yields useful images.

For most applications, the exposure range for a given process is rather large. When images decrease in size, however, the exposure latitude decreases.

Example: Assume that a series of test exposures were made and the best exposure turned out to be 15 seconds. At this exposure, the last complete step on the step tablet was Step 6. This number would then become the control number for that set of exposures. If a check a few days later, with the processing conditions unchanged, showed that the step-tablet image was now "breaking" at Step 4, it would indicate that the resist coating needed additional exposure to bring it back up to Step 6.

Since each step in the tablet corresponds to 0.15 density, a change of two steps would correspond to 0.30 density, or 1 stop. In this case, it would mean that the exposure would have to be doubled. If the test-plate image was breaking at Step 8, then it would be necessary to cut the exposure in half.

DEVELOPMENT OF EXPOSED PHOTORESIST COATINGS

As an exposed resist coating is subject to the action of a developer, portions of the resist coating dissolve. This selective dissolution of the coating depends upon the internal and surface bonding set up by the resist exposure. If the resist coating is properly exposed, the development process will completely remove resist in the desired areas without excessively swelling the remainder of the resist coating.

To avoid excessive swelling of the resist image, develop only long enough to remove the resist completely from the soluble areas. Do not develop any resist image more than three minutes. If the soluble areas of resist are not completely removed by the developer in this time, improper exposure of the resist or fogging of the coating may have occurred and no amount of overdevelopment will promote dissolution of the resist.

Use the developer designed to work with each resist. Since resists are formulated with different solvents, the wrong developer may have no effect at all, or it may lift the entire resist layer off the surface. The possible exception to these development recommendations is the volume processing of resist images in the vapors of a boiling solvent. This is usually done in the vapor phase of a boiling-sump vapor degreaser. Such a vapor degreaser should also be equipped with spray bars or a spray wand to complement the vapor development. Normally, a chlorinated solvent such as 1, 1, 1-trichloroethane serves as the developing solvent. This solvent, however, is not an effective developer when used at room temperature.

TANK OR TRAY DEVELOPMENT

All resists will develop satisfactorily upon immersion in the appropriate resist developer. For most reliable development, use a two-step process with two separate containers of developer. Divide the processing time between the first and second containers. As the developer in the first container becomes charged with dissolved resist and is exhausted, discard it and move the second developer container and place it in the first position. This technique results in reliable development, since the dissolved resist is not allowed to redeposit on the surface. In the tank and tray development method, agitate the material for the full development time in each container. Allow excess developer to drain back into the first container before transferring the material to the second container.

SPRAY DEVELOPMENT

An alternative to tank or tray development with the recommended resist developer is spray development with that developer. Spray development provides high agitation at the material surface and provides a continuous rinsing of the resist from the developed areas. This development method is especially useful for thick photoresist coatings. A spray rinse of clean developer is also useful after tank development. The spray tends to remove any residual resist that may be on the surface after initial development.

VAPOR DEGREASER DEVELOPMENT

Lower the exposed, resist-coated stock into the solvent vapors. Turn on the spraying equipment and hold the stock in the spray for 15 seconds. Turn off the spray and allow the stock to come up to the temperature of the solvent. This normally takes another 15 seconds, but may vary, depending on the mass of work involved. Withdraw the work from the degreaser. (*Do not* withdraw the work from the degreaser while the spraying equipment is operating.) If the image does not appear to be fully developed, repeat the process cycle exactly as before.

POSTBAKING THE RESIST IMAGE

A postbake after development often increases the durability of a processed photoresist image during etching or plating. This is especially true for relatively thick photoresist images. Here, solvent retention (from the developing step) can promote premature failure of the coating during etching or plating.

The need for a postbake must be determined by experiment and by examination of the process procedures. For example, a process requiring an extremely strong etchant, such as aqua regia, needs a resist postbake step. Conversely, a process using a relatively weak etchant, such as ferric chloride, may not need a postbake step.

Postbaking can make the resist more difficult to remove. This can be a problem with delicate objects or very small parts. Use no more postbaking than is necessary for the process application. Some experimentation will be required to determine the exact requirement for postbake in critical applications.

TABLE VI
POSTBAKING TIME/TEMPERATURE CYCLES

RESIST	TIME/TEMPERATURE
KTI KPR	10 minutes at 120°C (248°F). Temperatures up to 260°C (500°F) may be used; do not exceed 5 minutes at this temperature.
KTI KPR, Type 3	10 minutes at 120°C (248°F). Temperatures up to 260°C (500°F) may be used; do not exceed 5 minutes at this temperature.
KTI KPR, Type 4	10 minutes at 120°C (248°F). Temperatures up to 260°C (500°F) may be used; do not exceed 5 minutes at this temperature.
KTI KTFR	Postbake KTFR images at 120°C (248°F) for 10 minutes. Temperatures above 148°C (298°F) are to be avoided.

ETCHING SYSTEMS

The subject of etching various metals is too broad to be covered in a few paragraphs. Here, the discussion centers on etching methods. In many cases, specific recommendations for specific metals must be made; these recommendations will be found in other data sheets on particular applications. The more commonly encountered metals and the etchants that apply to them appear in Table VII.

STILL ETCHING

Use still etching only where shallow, surface etching of a metal is desired. It is not recommended for photoetching parts from metal because of its relative slowness and the attendant increase in etch factor.

AGITATED ETCHING

Agitation of the etchant helps to remove reaction products, and to improve etch factors. Provide agitation by stirring the etching bath, by bubbling air through the solution, or by spraying the etchant on the surface.

In spray etching, a high-velocity spray of etchant impinges on the surface of the material to be etched. The spray sweeps away any residual etching by-products from the surface. This provides a continually fresh surface for the etchant to act upon and reduces etching time and severe undercutting of the image.

ELECTROETCHING

Electroetching is the removal of metal from a surface through the action of an electrically conductive solution and direct current. A typical electroetching system consists of a source of low-voltage direct current, an anode (the work), a cathode (used to complete the electrical circuit within the solution), and a high-conductivity electrode. (See Figure 23.)

When current is applied to the electrodes, positive ions flow toward the negative electrode, and negative ions flow toward the positive electrode. The net effect is exactly the reverse of electroplating, i.e., metal is removed from the work, not placed on it.

Electroetching has its greatest application in surface etching of metals. It is not practical for the manufacture of small parts because electrical conductivity must be maintained to each part for etching to take place. Electroetching can be used prior to conventional etching if the conventional etchants lack enough activity to penetrate the surface film found on some metals.

The electrolytes for electroetching are mineral acids diluted to a given concentration, chemical salt solutions, or combinations of the two. Specific recommendations on electroetching (where they apply) will be found in the data sheets in the back of this book.

TABLE VIII

METAL, ETCHING SOLUTION, AND RESIST COMBINATIONS USED IN PHOTOFABRICATION

Metal	Resist(s) Used	Etchant
Copper	KPR, KPR Type 3, KPR Type 4,	Ferric Chloride (42 deg. Baumé), Ammonium Persulfate, Cupric Chloride
Brass	KPR, KPR Type 3, KPR Type 4,	Ferric Chloride (as above)
Beryllium Copper	KPR, KPR Type 3, KPR Type 4,	Ferric Chloride (as above)
Aluminum	KTFR	Sodium Hydroxide, Ferric Chloride (infrequent)
Anodized Aluminum	KPR, KPR Type 3,	Sodium Hydroxide (for anodized layer)
Stainless Steel	KTFR	Ferric Chloride (as above)

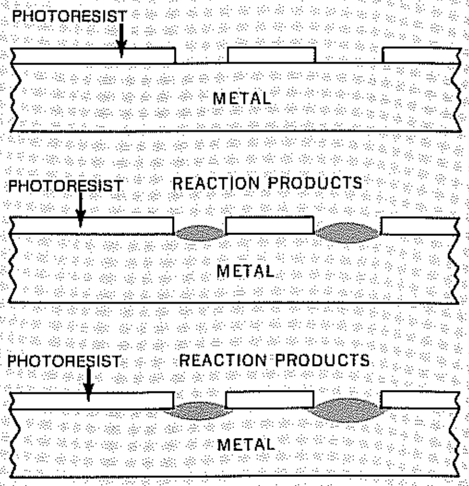

FIGURE 21: Still etching often produces etching by-products that can interfere with the etching efficiency of the solution.

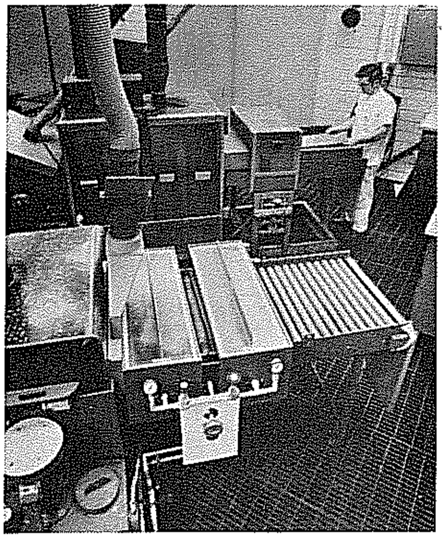

FIGURE 22: Power spray etchers provide speed, high capacity, and increased etch factors.

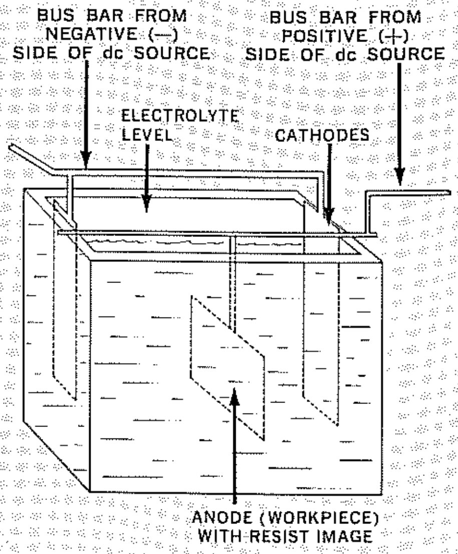

FIGURE 23: Electroetching, with the workpiece the anode, is often useful for surface etching. This technique can also be used as a preliminary etch prior to conventional etching.

PLATING

To use the resist coating as a plating stencil, form the resist image on a surface by the same methods described earlier for etching. Remember that the plating process is additive, i.e., material is added to the surface, not taken away as in etching.

When using KTI photoresists in plating, observe these rules:

1. Be sure that the work is clean. If the photoresist is not *fully* developed from the pattern area, poor plating or no plating will result. Images of KTI KPR Resist, KTI KPR Resist, Type 3, and KTI KPR Resist, Type 4, can be dyed after development to determine the extent of development. Generally, the work should be treated in a weak solution of an etchant immediately after development and dyeing of the resist image. This dip etches the material very slightly, and helps remove any further monomolecular films of contamination in the pattern area.
2. Provide an acid dip after pre-etching. In most cases, it is desirable to provide a weak acid dip identical to the type of acid contained in the plating bath prior to actual plating of the work. This will minimize contamination of the plating bath by drag-in of dissimilar chemical components. While initially insignificant, contamination of this sort can become troublesome over a period of time.
3. Be sure that the plating bath is at the recommended concentration and temperature, and that agitation is being used where required.
4. Be sure that the current density in the tank is correct for the solution and that the total amount of exposed metal on the circuit has been determined in terms of its effect on current consumption. Otherwise, cracked, burned, or peeling deposits will result.

   (For example, assume that the bath requires an operating current density of 40 amperes per square foot. Then 1 square inch equals 40/144 or 0.3 amperes per square inch [rounded off to the nearest tenth of an inch]. Therefore, if the pattern area totals 20 square inches, 6 amperes will be needed to obtain maximum plating efficiency from the bath.)
5. Be sure that the plating solution is clean. Poor housekeeping and poor plating practices in general can lead to rapid contamination of a plating bath. For example, oil or grease from a low-pressure blower used for bath agitation can cause severe peeling of the deposit in particular, and rapid deterioration of the deposit in general.
6. Strip resist in a proprietary stripper or in an organic solvent. Be sure that the proprietary stripper does not attack or affect the plating. Some mechanical abrasion may be necessary.
7. As a check on plating quality and adhesion, make the following test: Firmly apply a strip of tape to the board, making sure that the tape is at right angles to the direction of the pattern. Leave one end of the tape free. Pull the tape from the surface with a jerking motion, making sure that the tape is pulled off at right angles to the surface of the board. No plating on the tape indicates that the adhesion of the plating to the surface is satisfactory.

PLATING SOLUTIONS

Nearly any plating bath of a neutral or acidic composition (pH 7 or below) can be used with KTI photoresists. Avoid baths containing cyanides since the normal anode-cathode reaction produces a large amount of hydrogen gas during electrolysis. The gas forms as a constant film of bubbles on the cathode and will literally "scrub" the resist off the work.

In the following sections, some of the more commonly used plating baths that are compatible with KTI photoresists are reviewed.

COPPER-PLATING BATHS

There are three types of copper-plating baths in common use: acid copper, copper pyrophosphate, and copper fluoborate.

ACID COPPER BATHS—Acid copper baths are satisfactory for plating printed circuits and in electroforming if their deposits are modified through the addition of chemical agents. Such addition agents control grain size and smoothness and, in some instances, increase the throwing power* of the bath.

COPPER PYROPHOSPHATE BATHS—Pyrophosphate copper solutions are used extensively in plated printed-circuit work. Because of its near-neutral composition, it does not attack resists. Thick deposits can be built up without severe grain difficulties. Copper pyrophosphate baths are sold as proprietary solutions. For best results with this bath, follow the manufacturer's instructions carefully.

COPPER FLUOBORATE BATHS—Deposits from copper fluoborate solutions are similar to those obtained from copper pyrophosphate baths. Copper fluoborate baths can operate over a wide range of concentrations, temperatures, and current densities. Because of this flexibility, deposits from this bath can be modified to meet a particular application requirement.

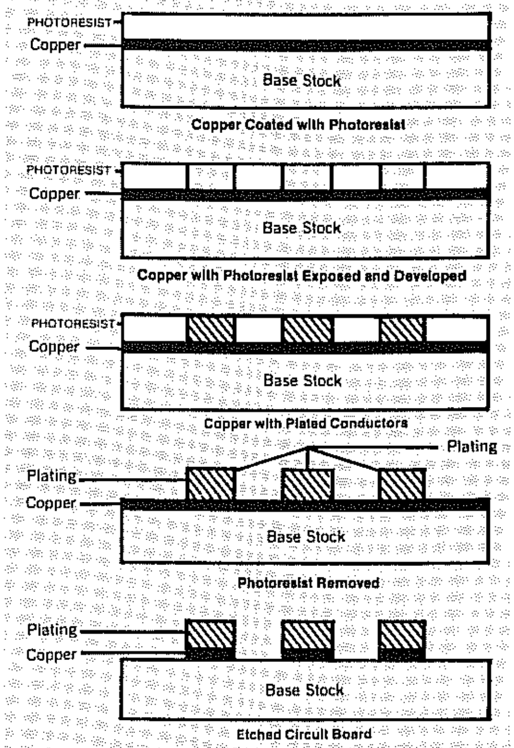

FIGURE 24: Plating and etching techniques are combined in making one type of printed circuit board. A resist stencil serves as the pattern for electroplating of a copper substrate.

NICKEL-PLATING BATHS

NICKEL SULFAMATE—Deposits from nickel sulfamate solutions show low internal stress. In addition to low stress, sulfamate nickel solutions produce deposits that are hard and have high tensile strength and good ductility. The throwing power of these baths is excellent.

WATTS NICKEL baths are among the most widely used solutions in electroplating. These baths can be used in printed circuit processing and in electroforming, but their control is somewhat more difficult than with the sulfamate bath.

NICKEL FLUOBORATE—Nickel fluoborate deposits are suitable for printed-circuit work because they have good ductility and low internal stress. Tensile strength and hardness of fluoborate deposits compare favorably with those obtained from the Watts bath. The deposits adhere well to copper. Like copper fluoborate solutions, nickel fluoborate solutions are proprietary plating baths. Therefore, follow the manufacturer's recommendations carefully.

**Throwing power* can be defined as the ability to plate a relatively uniform, thick deposit on an irregularly shaped surface or object.

SOLDER-PLATING BATHS

LEAD-TIN ALLOY BATH—Generally, lead-tin alloy deposits are obtained from a fluoborate bath. The alloy composition of the deposit depends on three characteristics: the metal-salts ratio in the bath, the alloy content of the anodes, and the current density.

For most work, the 60-40 tin-lead alloy is satisfactory. This particular composition approaches the eutectic point of most common lead-tin solders.

TIN-NICKEL ALLOY BATH—The tin-nickel bath is operated at metal concentrations of about 65 percent tin and 35 percent nickel. Small variations in alloy ratio (±2 or 3 percent) appear to have no appreciable effect on the deposit. Separate anodes in the ratio of 1 tin to 2 nickel are recommended.

The tin-nickel bath has excellent throwing power, with anode and cathode efficiencies at nearly 100 percent.

GOLD-PLATING BATHS

Although expensive, gold is widely used in printed circuit applications. Gold has outstanding corrosion-resisting properties. Always deposit gold from acid-type or near-neutral baths when using KTI photoresists. Most of these baths are proprietary formulations. Gold deposits are used to improve solderability, increase electrical resistivity, or increase the corrosion resistance of a printed circuit.

RHODIUM-PLATING BATHS

Rhodium, like gold, is used frequently in printed circuit manufacturing. Rhodium is an extremely hard metal. When combined with a nickel undercoat, it is used as a "wiping surface" for movable contacts.

Rhodium is deposited from acid-type solutions, which have limited throwing power. For most purposes, only a thin rhodium electroplate is required. If necessary, thick coatings can be plated from solutions containing a stress reducer.

ELECTROLESS PLATING

Electroless plating solutions operate on an autocatalytic principle, the reduction of a metal ion in solution to a metal on the surface of a catalyzed substrate.

Because electroless baths operate on the autocatalytic principle, they build up metal thicknesses superior to those obtainable from older precipitation methods. The mechanics of the electroless deposition are quite complex; no attempt will be made to explain them here. However, successful metallization depends on extreme cleanliness of the substrate. Any compromise in cleanliness leads to a compromise in electroless-plating quality.

ELECTROFORMING

Many precise, highly detailed articles such as fine-mesh screens and evaporation masks are produced by the electroforming process. Electroplated deposits of metal can be separated from certain surfaces quite easily. Such surfaces include stainless steel, electroplated nickel, and chromium deposits on other metals such as steel, copper, and brass. Electroforming is aided by the inherently high dielectric properties of KTI photoresists. These materials act as very effective "stop-offs" and can prevent plating in areas predetermined by the photographic pattern used to expose the resist. The combination of these factors results in production of ultraprecise, perforated articles.

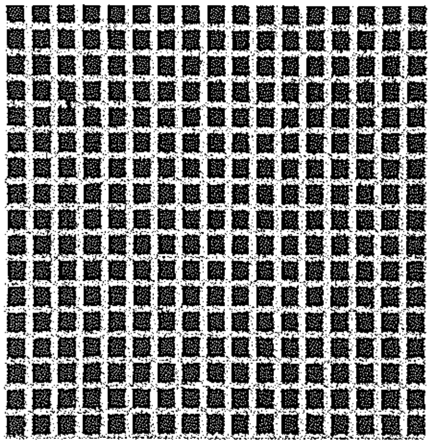

FIGURE 25: Advantage can be taken of the image-detail characteristics of KTI photoresists to produce fine electroformed items such as this 500-line/inch nickel mesh (magnification X125).

PROCESS STEPS

Basically a simple process, electroforming has several preparation steps that are very important. To understand the technique, examine each of the following steps in detail. A summary of the various processes appears at the end of this section.

STEP 1. CHOOSE A SURFACE. To electroformers, the surface to receive a deposit that will subsequently be stripped is known as a "mandrel." Mandrel materials may be stainless steel, Invar, Kovar, or electroplated nickel or chromium on other metals such as steel, copper, and brass. All of these are "permanent mandrels." This means that they can be used repeatedly. Whether or not the resist coated mandrel can be used repeatedly to photofabricate parts depends on the condition of the resist image after the first part is produced.

STEP 2. CLEAN THE SURFACE. As for any photofabrication process, clean the surface of the metal. This allows the photoresist to adhere properly to the surface. Use chemical cleaning techniques prior to coating with resist in order to retain the surface finish of the mandrel material. Usually a simple alkaline soak, followed by thorough rinsing and an acid dip, will be enough. Stainless steel is an excellent mandrel material because of its inherent corrosion resistance and its ready acceptance of passivating coating.

STEP 3. PASSIVATE THE SURFACE. Electroplated deposits can be easily separated from "passive" surfaces because they allow an electrodeposit to be built up *on* them. Stainless steel, nickel, and chromium are inherently passive. However, the passivity of these surfaces may not be uniform. For this reason, use an additional passivation step even though the surface already may be in a passive state.

Before passivation, clean the surface of the mandrel to remove any contamination left as a result of the photoresist processing step, and to remove any natural passivity. Use standard, concentrated ferric chloride baths, but do not prolong the cleaning step. Usually a slight color change in the pattern areas will indicate that cleaning is complete. Proceed immediately with the passivation step.

Passivate stainless steel and chromium surfaces in a nitric acid bath. Treat nickel mandrels in a chromate solution to form a passive film. Such a solution consists of about ½ ounce of sodium dichromate dissolved in a gallon of water. Immerse the nickel surface for 30 to 60 seconds.

STEP 4. COAT THE SURFACE WITH PHOTORESIST. Coat the mandrel by any of the recommended coating methods. Use the KTI photoresist recommended for the mandrel surface.

STEPS 5 AND 6. EXPOSE AND DEVELOP THE RESIST. Follow the processing procedures previously described. Be sure that development is complete. Residual resist on the surface will interfere with subsequent processing steps.

STEP 7. ELECTROPLATE THE DESIRED METAL. A number of plating baths are useful for electroforming. It is important that the deposits from the plating baths be free from tensile stress. If stress in the deposit is severe, the plating will separate from the mandrel prematurely. Even if this does not happen during processing, the deposit, after stripping, will be badly curled and may be useless. Several commercially available electroplating baths yield deposits in which tensile stress is not a factor. In fact, some formulations are available that will yield *compressive* stress —stress that causes the deposit to adhere more tightly to the mandrel.

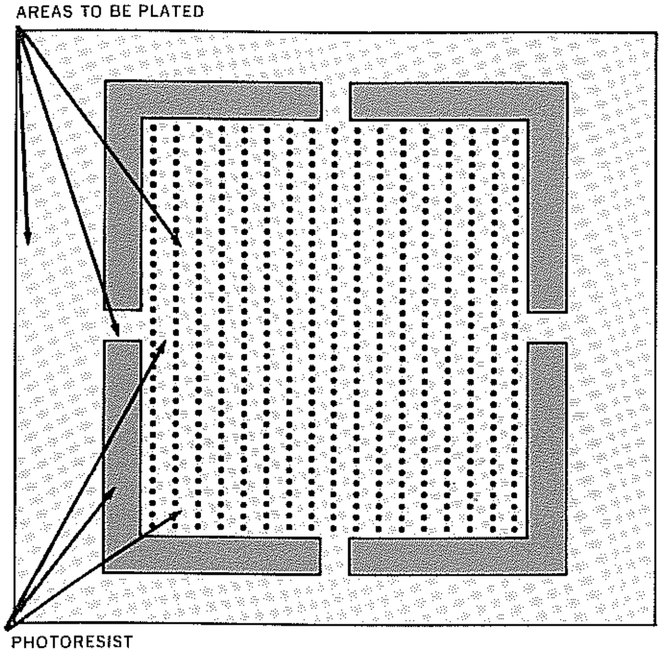

FIGURE 26: Providing a border around a mandrel intended for electroforming is one way of controlling the current distribution across a plated part.

Copper plating. Either copper sulfate or copper fluoborate plating baths are useful for electroforming processes. These baths, when operated at the proper levels, will yield low stress deposits. Consult plating references for specific plating-bath formulations.

Nickel Plating. Three nickel-plating baths are adaptable to electroforming processes. These are the nickel sulfamate bath, the Watts nickel bath, and the nickel fluoborate bath. Nickel sulfamate solutions produce deposits that have low tensile stress. With addition agents, deposits from the Watts nickel bath can assume stress levels comparable to those from nickel sulfamate solutions. Nickel fluoborate solutions give deposits that have moderate tensile stress. Because of this, flat, thin articles such as fine mesh may be difficult to produce with this solution.

Before placing the mandrel in the bath, examine it to make certain that the areas which are to receive the plating will hold an unbroken film of water. If contamination is present on the surface of these areas, a poor deposit, or no deposit will result. If waterbreaks occur, use a mild alkaline cleaner, followed by the acid cleaning and passivating steps to correct this condition.

Be sure that the total current used in the bath does not exceed the calculated amount of exposed metal on the mandrel.

Take care to obtain an even current distribution over the entire surface of the mandrel. A convenient way to do this is to provide a border around the part. Such a border should be about ¼ to ½ inch in width, and isolated from the part itself by a narrow band of photoresist. See Figure 26.

A border is recommended for plating because flat surfaces tend to concentrate current around the edges. In electroforming, this tendency may prevent production of thin, perforated parts. Obviously, if the edges of an electrodeposit build up at a rate faster than the center, nonuniform pattern dimensions result.

Plating times depend on current density in the tank, on temperature and concentration of the electrolyte, and, to a lesser extent, on agitation. These factors should be kept constant to standardize plating times. Plating times are predictable for specific plating thicknesses. Plating textbooks give nomographs which have plating thickness as a function of time.

STEP 8. REMOVE THE ELECTROFORM FROM THE MANDREL. With proper step-by-step operation, the electrodeposit, or electroform, will strip away from the surface easily. If the deposit sticks and subsequent removal is difficult, improper passivation is usually at fault.

Methods for stripping the electroform are varied. If the mandrel will flex without being permanently deformed, use this method. However, if the mandrel is soft and deforms easily, use a sharp object such as a razor blade to lift the plating from the surface. If the mandrel is thick, try thermal shock or heating.

Stripping the electroform always involves some danger of damage to the resist image when a simple mandrel is used. The physical forces of stripping the electrodeposit cause edge degradation, thus making the resist-image mandrel good for about one (and at most, two) electroforms.

ETCHED MANDRELS

A second type of mandrel for this process is the etched mandrel. Prepare a mandrel exactly as outlined previously, but introduce an additional etching step just prior to plating. This provides more relief to the image, but a slight loss of dimensional accuracy. After etching, passivate the mandrel, and proceed with electroplating without delay.

Do not carry etching too far or dimensional accuracy will suffer. In addition, separation of the electrodeposit from the mandrel may be more difficult because the deposit is "locking in" against the etched sidewall of the image.

Determine the actual etching depth by trial. Fine patterns will require less etching, while wide patterns can tolerate more.

PLANOGRAPHIC MANDRELS

The planographic mandrel is designed for high-production electroforming techniques. "Planographic" means that the image and the dielectric material are in one plane. There is essentially no surface relief.

With the planographic mandrel the dielectric is locked into place by etched depressions in the surface. Thus, it is possible to use this mandrel over and over again without image damage or loss of dimensional accuracy.

The essential steps in making a planographic mandrel consist of (1) forming the image, (2) etching depressions into the surface, (3) removing the resist, (4) filling the etched depressions with a dielectric filler, and (5) removing the excess filler so that a flush surface results.

THE BIMETAL MANDREL

The bimetal mandrel is a refinement of the basic etch-and-fill mandrel technique. To form a bimetal mandrel, plate a metal on the surface of the mandrel base. Form a photoresist image, and etch the plating away in the desired areas. Then, change the etchant and etch the mandrel base to the desired depth. Thereafter, use normal filling and finishing techniques to complete the process.

THE TRIMETAL MANDREL

Another refinement of the etch-and-fill mandrel technique is the trimetal mandrel. To form this mandrel give a thick plate of steel an electroplate of copper (about 15 to 20 mils), and a thin electroplate of another, dissimilar metal (such as nickel or silver). Use the same processing techniques as for both the etch-and-fill and the bimetal mandrel process, but *do not* etch the copper completely through to the steel.

Make trimetal mandrels for processes where the mandrels must be used over and over again. Trimetal mandrels are exceptionally durable and require little maintenance after they have been made.

REMOVING THE RESIST AFTER ETCHING OR PLATING

Efficient removal of the resist image depends on many factors. These include the thickness of the coating, the degree of postbaking, and compatibility of the metal surface with the particular stripping solution. As with all resist strippers, these materials work best if the resist coating is not overbaked. Where complete, clean stripping is needed, resist postbake should be limited to that actually needed to make sure of resist adherence during etching. Use a forceful water-spray rinse after soaking a resist-coated surface in any of the strippers. Avoid contaminating the stripper with quantities of water. Use the strippers in a polypropylene, glass, or ceramic-lined tank. Complete information can be obtained from manufacturers of the stripping mixtures.

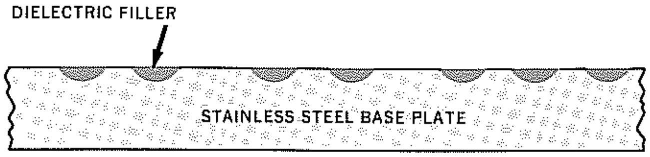

FIGURE 27: This is a cross-sectional diagram (greatly exaggerated) of the planographic mandrel. Note that the depressions are filled with a dielectric material to prevent plating in those areas.

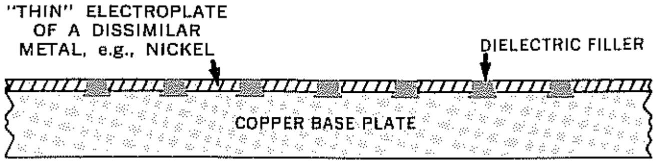

FIGURE 28: The bimetal mandrel (and the trimetal mandrel diagrammed in Figure 29) is made by combining photoresist techniques with etching and plating systems.

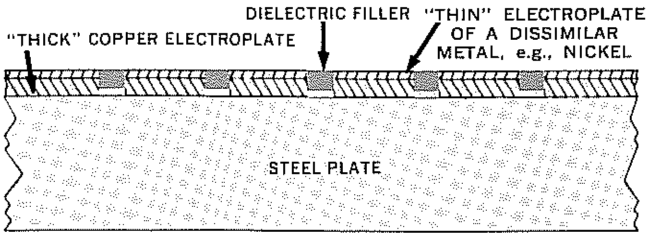

FIGURE 29: The trimetal mandrel diagrammed in cross section.

PROCESS DATA SHEETS

The data in this section are intended to give supplementary information on the use of KTI photoresists with various materials. Where departures from the normal procedures may be desirable, these are noted. Otherwise, the normal process methods described in the body of this book are applicable.

Caution: Many of the acids, acid solutions, salts, etching solutions, and solvents recommended for use in this Data Book are toxic and corrosive, and require special handling. Strong acids such as sulfuric, nitric, and hydrofluoric can cause burns of the skin, eyes, and mucous membranes. In addition, burns from hydrofluoric acid may not be detected until the acid has been in contact with the skin for some time. The vapors of hydrofluoric acid are extremely irritating to the respiratory tract. Therefore, this acid should be handled only under adequate ventilation control. Any accidental contact with these acids requires immediate flushing with plenty of water, and if the eyes are involved, prompt medical attention should be obtained.

Cyanide solutions must be handled with extreme caution, because they are deadly poisons. Do not mix cyanides (either dry or in solutions) with acids; the resulting neutralization reaction yields hydrogen cyanide, a deadly poisonous gas.

Mercury must be handled with great care. Avoid skin contact with the metal and its soluble compounds and solutions. Local exhaust ventilation is needed where mercury is being handled in quantity or on a regular basis. Spills of mercury should be quickly and completely cleaned up or the residual metal can present a hazard for vapor inhalation.

Organic solvents such as xylene, acetone, and trichloroethylene should be handled in such a way as to prevent direct skin contact with solvents and inhalation of the vapors.

ALUMINUM

Recommended Resist: *KTFR.*

Surface Treatment: Degrease in a vapor degreaser or by other suitable means. Pumice or otherwise mechanically clean the surface when necessary. Rinse in running water. Chemically clean the surface by using one of the following methods:

- Immerse in a hot solution of non-etch detergent — such as Ridoline 53 (Amchem Products Co., Brookside Ave., Ambler, Pennsylvania 19002).
- Immerse in a satinizing, alkaline, pre-etch bath—such as a 3 to 6 percent solution of caustic soda (4 to 8 ounces of sodium hydroxide pellets per gallon of water), an 18 percent nitric acid solution (one volume of 70 percent nitric acid—specific gravity 1.42—added slowly to one volume of water, with constant stirring), or proprietary deoxidizing solutions. The proprietary cleaner suggested above provides a suitable surface for many etching applications. The following chromate conversion-coating materials are generally required to provide satisfactory etch factors when deep etching (photomilling) is done with alkaline solutions. Conversion coatings of these materials are also suitable for acid etching solutions.
    a. Iridite 14-2 (supplied by Allied-Kelite Products, Division of the Richardson Co., 81 Industrial Rd., Berkeley Heights, New Jersey 07922)
    b. Alodine 1200 S (supplied by Amchem Products Co., Brookside Ave., Ambler, Pennsylvania 19002)

    An immersion time of 3 minutes at room temperature in solutions containing 2.0 to 7.5 grams of Alodine 1200 S, or 8.5 grams of Iridite 14-2, and about 9 mL of concentrated nitric acid per liter of solution (to obtain a pH of about 1.35) is preferred. Thick conversion coatings generally give poor process performance.* After the conversion-coating treatment, rinse the surface in running water. Remove water with clean, oil-free compressed air. Heat may be used to complete drying.

Etching: Use one of the following methods.

- Alkaline Etching

    Immerse the surface in a 20 percent sodium or potassium hydroxide solution at 60 to 90.5°C (140 to 195°F) or in any other alkaline etchant desired. Heat is generated during etching. Lower temperatures and concentrations can be used if preferred.

- Acid Etching

    Immerse the surface in a solution of hydrochloric acid made by slowly adding 1 volume of concentrated hydrochloric acid to 4 volumes of water.

    An alternate etching bath consists of: 1 gallon of photoengraver's grade ferric chloride (42-degree Baumé) and 12 ounces of 38 percent (concentrated) hydrochloric acid. These baths are rapid-acting and evolve considerable heat. For best results, the baths should be kept below 43.5°C (110°F) by cooling.

Etching-Smut Removal: A dip in 35 percent (by volume) nitric acid and water, at room temperature, is usually sufficient. Commercially available deoxidizers can also be used if preferred.

ANODIZED ALUMINUM

Recommended Resists: *KPR* or *KPR Type 3.* Do not use *KTFR* on clear, anodized aluminum where a yellowish cast would be objectionable.

Surface Treatment: Generally, the surface is clean except for dust. If fingerprints or oils are on the surface, degrease but avoid alkaline materials which will etch the anodized layer. Polish with a polishing-grade buff powder, if necessary. Do not use harsh abrasives. Rinse in running water. Remove with oil-free compressed air. Heat may be used to complete drying.

Etching: For applications in which only the anodized layer is to be etched, use Solution 1 or 2. For deep etching of aluminum, first remove the anodic layer, using Solution 1 or 2; then follow with a deep-etching solution, such as Solution 3 or 4.

1. A 20 percent solution of sodium hydroxide at room temperature. *Care must be exercised to prevent overetching and attack upon the aluminum.*
2. Proprietary anodizing strippers or deoxidizers—such as Turco 2897 (Turco Products/Division of Purex Corp., P.O. Box 6200, Carson, California 90749).
3. When deep etching is required for chemical milling or nameplates, use an acid solution, such as:

    One gallon of photoengraver's grade ferric chloride (42 degrees Baumé) combined with 12 ounces of 38 percent (concentrated) hydrochloric acid. This bath is a very rapid etchant and evolves considerable heat. The bath should be kept below 43.5°C (110°F) by cooling. The rate of etching may be controlled by varying the amount of hydrochloric acid used.
4. A solution of hydrochloric acid alone may also be used. Such a solution can be made by combining 1 volume of 38 percent (concentrated) hydrochloric acid with 4 volumes of water. This bath evolves heat and may require cooling. Very high etch rates are obtained at about 65.5°C (150°F). Since the anodized layer is not etched by the HCl solution, deep etching may be accomplished with relatively thin resist coatings. The deoxidizers mentioned in No. 2 can also be used to remove smut and impart adhesion to fillers when etched-and-filled nameplates are being made.

Resist Removal: Generally not necessary, as the resist acts as a protective lacquer.

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*Coatings which exhibit a "powdery" appearance indicate that the conversion coating is not suitable for further processing.

CHROMIUM

Recommended Resists: For vacuum-deposited chromium, use *KTFR*.

Surface Treatment: Generally, the surface should be clean, except for dust. Degrease, if necessary, in a vapor degreaser or by other suitable means. Clean the surface in a mild detergent if needed. Rinse in running water. Remove water with oil-free compressed air. Heat may be used to complete drying.

Etching: *Electrolytically deposited chromium.* Use Solution 1 or 2.

1. Use a solution containing 3 volumes of concentrated nitric acid and 7 volumes of water at about 79.5°C (175°F).
2. Use a solution containing 2 volumes of 42-degree Baumé ferric chloride and 1 volume of concentrated hydrochloric acid at about 79.5°C (175°F).

Vacuum-deposited chromium. Prepare as follows.

Stock Solution A	Metric	Avoirdupois
Distilled water	1000 mL	34 fluid ounces
Sodium Hydroxide Granular	500 grams	17½ ounces

Stock Solution B	Metric	Avoirdupois
Distilled water	3000 mL	102 fluid ounces
Potassium Ferricyanide (Anhydrous)	1000 grams	35 ounces

Prepare a working solution of 1 part of Stock Solution A, and 3 parts of Stock Solution B. Use the working solution at room temperature.

COPPER AND COPPER ALLOYS

Recommended Resists: *KPR*, *KPR Type 3*, and *KPR Type 4*.

Surface Treatment: Degrease in a vapor degreaser or by other suitable means. Pumice scrubbing may be needed for heavy oxides; otherwise, a chemical cleaning should be sufficient. Immerse the surface in a solution of 8 percent (by volume) hydrochloric acid for about 30 seconds at room temperature. Rinse well in running water at room temperature. Remove the water with oil-free compressed air. Heat may be used to complete drying as long as it does not reoxidize the surface. As an alternative cleaning method, a dry buffing system can be used. Rotary-type bristle brushes are generally acceptable if they do not introduce other types of soil onto the surface.

Etching: For chemical etching, use any of the following solutions.

1. Solutions of ferric chloride.
2. Solutions of ammonium persulfate and mercuric chloride.
3. Electrolytic etching can be done in solutions of ammonium chloride saturated with sodium chloride.
4. Solutions of cupric chloride.

Plating: Thorough image development is most essential for quality plating. Standard plating solutions and techniques can be used, except that severe precleaning cycles must be avoided. Instead, use one of the following methods:

- Immerse the surface in a weak solution of ferric chloride for 10 to 15 seconds. Immediately follow this with a dip in a 10 percent (by volume) solution of hydrochloric acid and water. The acid dip will prevent hydrolysis of the ferric chloride to ferric hydroxide. Rinse well in running water.
- Use a 10 percent solution of ammonium persulfate made by dissolving approximately ¾ pound of ammonium persulfate in a gallon of water. A 10- to 15-second dip should be sufficient. Rinse well in running water.

Note: Use these dips just prior to the plating cycle.

GLASS, FINE-LINE ETCHING

Recommended Resists: *KPR*, *KPR Type 3* and *KPR Type 4*.

Surface Preparation: This process depends on a thin metallic interlayer between the photoresist and the glass. This kind of metallic film can be deposited by sputtering, vacuum, or chemical precipitation methods. Of the various metallizing methods available for this process, silver precipitation from chemical solutions appears to be the most popular. Silver is deposited on the glass in much the same way as it is deposited in mirror-making. Many formulas exist for this purpose. Some are commercially available, while others can be formulated by the individual. In any case, however, remember that the film must be completely opaque in order to perform adequately.

Coating: The metallic film should be coated with photoresist as soon as possible after deposition. Do not attempt to clean the film, since it is soft and can be damaged easily. Whirling, dipping, or spraying techniques can be used to apply the resist.

Dry: Generally not necessary; however, the resist must be free from solvent.

Dye: The image should be dyed in order to facilitate inspection prior to etching.

Postbake: For 5 minutes at 65.5°C (150°F).

Etching: Prepare the following solutions.

Stock Solution A	**Metric**	**Avoirdupois**
Potassium Ferricyanide (Anhydrous)	37.5 grams	1¼ ounces
Water to make	500 mL	16 ounces

Stock Solution B	**Metric**	**Avoirdupois**
Sodium Thiosulfate (Hypo)	480.0 grams	16 ounces
Water to make	2.0 liters	64 ounces

For use, take 1 ounce (30 cc) of Stock Solution A, 4 ounces (120 cc) of Stock Solution B, and water to make 32 ounces. Add Solution A to Solution B, then add the water, and pour the mixed solutions at once over the part to be etched. In order to watch the etching action, it is advisable to place the part to be etched in a clear receptacle that has been placed over an illuminator. This makes sure that the etching can be stopped as soon as the silver has been removed from the desired areas. When all the silver has been etched, rinse the part immediately with running water. Dry thoroughly. Spot any pinholes with asphaltum or some other type of acid-resistant material, and coat the back of the glass as well. Dry the acid-resistant material thoroughly. The glass is now ready for etching in hydrofluoric acid.

Immerse the plate in 48 percent hydrofluoric acid. *Observe caution with this material.* Generally, a 15- to 30-second immersion is sufficient for an average line width of 3 mils. Larger or smaller line widths will require an adjustment of etching time. After etching, rinse immediately in running water. If preferred, the etching action of the hydrofluoric acid can be stopped immediately by dipping the plate into a dilute solution of ammonium hydroxide.

Resist and Silver Removal: The photoresist and resist coating on the back of the glass can be removed from the surface by immersing them in a proprietary stripper or organic solvent. The silver can be removed by immersing the plate in a solution of nitric acid and water.

GOLD

Recommended Resist: *KTFR*.

Surface Treatment: Degrease in a vapor degreaser or by other suitable means. Clean chemically, using a detergent or a solution containing 1 part by weight of ammonium persulfate and 10 parts by weight of water. Immerse for 5 minutes at room temperature. Rinse in running water. Remove the water with oil-free compressed air. Heat may be used to complete drying.

> **Note:** Vacuum-deposited gold films should be clean as received. If grease, oil or fingerprints are present, remove them by solvent cleaning only. Do not use harsh mechanical- or chemical-cleaning methods.

Etching: For thin films of gold use either Solution 1 or 2.

1. *Aqua Regia*
   3 parts of hydrochloric acid
   1 part of nitric acid
   Mix these acids with constant stirring, in a fume hood.
2. Solution of sodium cyanide containing hydrogen peroxide. Use caution in handling both Solution 1 and Solution 2.

For electrolytic etching, use alkaline cyanide solutions with a steel cathode at 6 volts.

MAGNESIUM

Recommended Resists: *KPR*, *KTFR*, *KPR Type 3*, and *KPR Type 4*.

Surface Treatment: Degrease in a vapor degreaser or by other suitable means. Pumice or otherwise mechanically clean the surface. Rinse in running water.

Conversion Coating: Immerse in a solution of 2 percent (by volume) concentrated phosphoric acid and water for about 30 seconds at room temperature. Rinse in running water. Remove the water with oil-free compressed air. Heat may be used to complete drying.

Postbake: Required whenever the Dow Powderless Etch Process is used. Otherwise, postbaking is optional except for *KTFR* images.

Etching: Use solutions containing 5 to 10 percent by weight of nitric acid.

MOLYBDENUM

Recommended Resists: *KTFR* is preferred for deep etching. *KPR, KPR Type 3,* or *KPR Type 4* can be used when depths of 2 mils or less are required.

Surface Treatment: Degrease in a vapor degreaser or by other suitable means. Pumice or otherwise mechanically clean the surface when necessary. Chemically clean by using one of the following solutions.

1. Immerse the surface for about 3 minutes in a 76.5°C (170°F) solution containing 20 percent (by volume) concentrated sulfuric acid and 5 percent (by volume) potassium tartrate.
2. Acid-peroxide etch solutions.
3. Electropolishing in phosphoric-sulfuric acid solutions.

Conversion Coating: Treat the surface for about 3 minutes in a 76.5°C (170°F) solution containing 20 percent sodium dichromate. Rinse in running water. Remove water with oil-free compressed air. Heat may be used to complete drying.

Etching: For chemical etching, use one of the following solutions:

1. 1 volume of concentrated nitric acid
   1 volume of concentrated sulfuric acid
   3 volumes of water
2. 200 g/L of potassium ferricyanide
   20-25 g/L of sodium hydroxide
   3-3.5 g/L of sodium oxalate

For electrolytic etching, use a 10 to 20 percent solution of sodium hydroxide which contains a small percentage of sodium oxalate.

NICKEL AND NICKEL-BASE MAGNETIC ALLOYS

Recommended Resists: Use *KTFR* for deep etching. *KPR, KPR Type 3,* or *KPR Type 4* may be used for shallow etching only.

Surface Treatment: Degrease in a vapor degreaser or by other suitable means. Pumice or otherwise mechanically clean the surface, if needed. Rinse in running water.

Conversion Coating: Immerse the surface for about 3 minutes in a solution containing 1 volume of concentrated phosphoric acid and 3 volumes of water, at 76.5°C (170°F). Rinse in running water. Remove the water with oil-free compressed air. Heat may be used to complete drying.

Etching: Use solutions of ferric chloride or a solution such as 1 volume of concentrated nitric acid, 1 volume of hydrochloric acid, and 3 volumes of water. Spray or splash etching is recommended.

PLATINUM

Recommended Resist: *KTFR.*

Surface Treatment: Degrease in a vapor degreaser or by other suitable means. Chemically clean the surface by immersing it for 30 seconds in a 48 percent solution of hydrofluoric acid. Rinse in running water. Remove water with oil-free compressed air. Heat may be used to complete drying.

Caution: Hydrofluoric acid is extremely corrosive to the skin and mucous membranes. Follow the safe-handling procedures given on the acid bottle labels.

Etching: Repeat the hydrofluoric acid dip as recommended above. Follow this with a water rinse; then etch the platinum in a solution of aqua regia at room temperature.

PIGMENTED IMAGES

Recommended Resist: Pigmented *KPR.* This resist will yield images that are free of ash after firing. Pigmenting and processing procedures are described below.

Surface Treatment: Degrease by using an 82°C (180°F) degreasing solvent, such as a solution of trisodium phosphate. Other degreasing solutions can be used. Rinse in running water. Chemically clean the surface by a dip in the appropriate dilute-acid bath at room temperature; for example, a 2 percent solution of hydrofluoric acid and water at room temperature for glass. Rinse in running water. Dry thoroughly.

Resist Preparation: Filter and weigh an amount of *KPR* into a clean, dry, ball-mill jar. Add a weighed amount of the desired pigment to the jar. Up to 40 percent (undiluted *KPR* weight) of ceramic pigment can usually be tolerated without impairing *KPR* binding properties. Likewise, up to 8 percent of a color pigment of a TV phosphor can be used, depending on how finely it is to be milled. The optimum pigment concentration and milling time will depend on the particular pigment used and the size of the pigment particles desired in the end product.

Remove Excess Pigment: Excess pigment may remain in the nonimage area unless the image is spray-developed. The excess pigment can be removed by cotton swabbing with isopropyl alcohol after the developed image has *completely dried.* Denatured ethyl alcohols should be avoided when working with glass or ceramic supports, since the water they contain tends to lift the exposed image.

Coat Additional Layers (optional): Multiple coatings of pigmented *KPR* can be superimposed on the same support by repeating the coating process (up to 3 coatings). Remove excess pigment after each coating.

Fire the Image (optional): Images of ceramic or phosphor pigments can be bonded to ceramic or glass supports by conventional firing techniques. At 399 to 499°C (750 to 930°F) *KPR* fires off completely, with practically no residual ash. Ceramic fluxes and glazes may be included in the *KPR* mixture (if they are dry) or may be applied to the support either before or after formation of the pigmented *KPR* image.

Causes of Image Stripping:

- Ratio of pigment to *KPR* binder is too high.
- Pigmented *KPR* coating is too thick.
- Underexposure or overexposure.
- Image "fog," or halation. The back surface of transparent supports should be coated with a suitable ultraviolet-absorbing layer to prevent exposure by reflected light returning through the support.
- Water in the alcohol used in removing excess pigment.

SILVER

Recommended Resist: *KTFR.*

Surface Treatment: Degrease in a vapor degreaser or by other suitable means. Pumicing is recommended for surfaces where finishes are not critical. If surface finish is important, use a polishing-grade buff powder such as BPA No. 1 Fine, made by Carborundum Co., Buffalo Ave. at 88th St., Niagara Falls, New York 14302. Where appropriate, a slight etch with dilute nitric acid is sometimes used as a chemical cleaning method. Rinse in running water. Remove the water with oil-free compressed air. Heat may be used to complete drying, as long as it does not re-oxidize the surface.

Etching: Use one of the following solutions:

1. A 55 percent weight by volume solution of ferric nitrate used at 43.5 to 49°C (110 to 120°F). This solution is made by adding 7½ pounds of ferric nitrate crystals to 1 gallon of water.
2. A matte surface can be produced by using a solution containing:

Chromium Trioxide	40 grams
Sulfuric Acid (sp gr = 1.84)	20 mL
Water	2000 mL

3. For electrolytic etching, use a 15 percent (by volume) solution of concentrated nitric acid and water and a stainless steel cathode at about 2 volts.
4. Use solutions described in etching glass section on page 27. These solutions are effective only on thin films of silver.

SPRING STEEL

Recommended Resist: *KTFR.*

Surface Treatment: If the blued finish found on many spring steels is to be retained, follow the procedure outlined below. If this finish is unimportant, follow the suggested surface treatment outlined under "Steel."

Degrease in a vapor degreaser or by other suitable means. Chemically clean by soaking the surface in a solution of 10 percent (by volume) sodium hydroxide and water at room temperature. Detergent-type cleaning solutions may be used if preferred. Rinse in running water. Remove excess water with oil-free compressed air. Warm air may be used to complete drying.

Etching: Use 36- to 42-degree Baumé ferric chloride at 52 to 54.5°C (125 to 130°F) in a spray- or splash-type etching machine. Be sure that the etchant does not contain any dissolved copper. *Do not etch by immersion techniques.*

Resist Removal: Xylene, toluene, trichloroethylene, or a proprietary stripper (if compatible with the blued finish) can be used as a resist stripper.

STAINLESS STEEL

Recommended Resist: *KTFR.*

Surface Treatment: Degrease in a vapor degreaser or by other suitable means. Pumice the surface, if necessary. If surface conditions are such that pumice cannot be used, buff the surface with a slurry of fine abrasive and water. If scrubbing is impractical, an alkaline soak or electrolytic cleaning may be used. Any of these steps should be followed with an acid rinse consisting of 10 percent (by volume) nitric acid and water at room temperature. Rinse in running water. Remove water with oil-free compressed air. Heat may be used to complete drying.

Conversion Coating: Use one of the following methods:

- Immerse the surface for 15 to 20 minutes in a 20 percent solution of nitric acid and water (concentrated nitric acid, 20 percent by volume; water, 80 percent by volume) at 65.5 to 71°C (150 to 160°F). Rinse in running water. Remove the water with oil-free compressed air. Heat may be used to complete drying.
- Clean as described under "Surface Treatment" above. Then heat the surface to 260°C (500°F) for about 5 minutes, cool, and coat with resist.

Etching: Use one of the following solutions:

1. Solutions of ferric chloride (photoengraver's grade) 36- to 42-degrees Baumé, in spray-etching equipment.
2. Solutions of ferric chloride and hydrochloric acid.
3. A solution consisting of:

   1 volume of concentrated hydrochloric acid (37 percent)
   1 volume of concentrated nitric acid (70 percent)
   3 volumes of water

4. Electrolytic etching techniques can also be used to obtain rapid metal removal. A typical bath consists of:

   1 volume of concentrated hydrochloric acid (37 percent)
   3 volumes of water

   Use the bath at 6 volts with a stainless steel cathode.

   Note: When ferric chloride etching systems are used, be sure that dissimilar ions, especially copper, are not present in the etching bath. Such ions will cause poor etch factors and generally unsatisfactory results.

STEEL (includes Silicon Steel)

Recommended Resist: *KTFR.*

Surface Treatment: Degrease in a vapor degreaser or by other suitable means. If necessary, pumice or otherwise mechanically clean the surface. Rinse with running water.

Conversion Coating: Some proprietary conversion-coating materials are available. However, they should be evaluated on their performance with respect to photoresist adhesion and to etch factors for the particular process used.

Etching: Chemical etching may be done in one of the following solutions:

Solution 1. A solution of ferric chloride 36- to 42-degree Baumé.*

Solution 2. A solution of ferric chloride and nitric acid.*

Solutions 3 and 4. The approximate etch rates for tool steel (SAE 0-1) with Solutions 3 and 4 are given below. Solution 4 has a tendency to stop etching below 79.5°C (175°F). Greater etch rates are obtained when spray- or splash-type etching machines are used. (This statement also applies to Solutions 1 and 2.)

Temperature	Etch Rates (mil/min)
25°C (77°F)	0.18
50°C (122°F)	1.7
79.5°C (175°F)	10.0

Solution 3	Solution 4
300 mL of concentrated nitric acid	300 mL of concentrated nitric acid
700 mL of water	35 grams of silver nitrate
	700 mL of water

Solutions 5 and 6. Solutions such as these are frequently used for making steel engravings and embossing dies.

Solution 5
"Spencer's Acid"

Solution A		Solution B	
Concentrated Nitric Acid C.P.	5 parts	Concentrated Nitric Acid C.P.	1 part
Silver Metal	1 part	Mercury	5 parts
Distilled Water	5 parts	Distilled Water	5 parts

Dissolve metals as indicated. Combine Solutions A and B, and dilute 1:1 with distilled water.

Use as follows:

1. Pour on steel surface.
2. Start the etch by dipping a strip of zinc in the solution and touching the zinc to the steel. The reaction will continue when the zinc is removed.

   Note: Solutions containing high concentrations of hydrochloric, nitric, or hydrofluoric acid promote resist failure when used for deep etching.

Solution 6. Cronite Etching Acid for Steel.

TITANIUM

Recommended Resist: *KTFR.*

Surface Treatment: Degrease in a vapor degreaser or by other suitable means. Pumice or otherwise mechanically clean the surface. Descale in a hot aqueous solution of 10 percent nitric acid and 2 percent hydrofluoric acid. An alternative treatment, which produces a smoother and more uniform titanium surface, is described in U.S. Patent 2,827,402 (General Electric Company). Use of this procedure may require a license. After cleaning, rinse in running water. Remove the water with oil-free compressed air. Heat may be used to complete drying.

Etching: Titanium can be etched in a solution of 48 percent (concentrated) hydrofluoric acid and water, diluted 9:1, at the rate of ½ mil per minute at 29.5 to 32°C (85 to 90°F). A variation of this solution consists of water, 70 percent; hydrofluoric acid, 10 percent; and nitric acid, 20 percent. This yields an etch rate of ¾ mil per minute if used at 32°C (90°F). U.S. Patent 2,942,954 (A.B. Thomas to General Motors Corporation) describes a persulfate-fluoride solution for etching titanium.

ZINC

Recommended Resists: *KPR, KTFR, KPR Type 3,* and *KPR Type 4.*

Surface Treatment: Degrease in a vapor degreaser or by other suitable means. Pumice or otherwise mechanically clean the surface. Rinse in running water.

Conversion Coating: Immerse the surface in a solution of 2 percent (by volume) concentrated phosphoric acid and water at room temperature for about 2 minutes. Agitation will improve the phosphatized surface. (Note that proprietary conversion coatings should be evaluated on their performance with respect to photoresist adhesion and to etch factors for the particular process used.) Rinse with running water. Remove the water with oil-free compressed air. Heat may be used to complete drying.

Etching: Use a nitric acid solution of the type used by photoengravers. Etching is usually done in a conventional etching machine of the spray or splash type.

***Note:** When ferric-chloride etching systems are used, be sure that dissimilar ions, especially copper, are not present in the etching bath. Such ions will cause poor etch factors and generally unsatisfactory results.

FAHRENHEIT-CELSIUS CONVERSION TABLE

Degrees Fahrenheit	Degrees Celsius	Degrees Fahrenheit	Degrees Celsius
70	21	194	90
74	23.5	198	92
78	25.5	202	94.5
82	28	206	96.5
86	30	210	99
90	32	214	101
94	34.5	218	103.5
102	39	222	105.5
106	41	226	108
110	43.5	230	110
114	45.5	234	112
118	48	238	114.5
122	50	242	116.5
126	52	246	119
130	54.5	250	121
134	56.5	254	123
138	59	258	125.5
142	61	262	127.5
146	63.5	266	130
150	65.5	270	132
154	68	274	134
158	70	278	136.5
162	72	282	138.5
166	74.5	286	142
170	76.5	290	143
174	79	294	145
178	81	298	147.5
182	83.5	302	150.5
186	85.5	306	152
190	88	310	154

Finding Intermediate Temperature Conversions—If, for example, the Celsius value for 156°F is desired, simply add the Celsius value for 154°F and the Celsius value for 158°F and divide their sum by 2.

68°C
70°C
2 | 138
69°C

If odd-numbered values are desired, such as 157°F, or if values higher than those in the table are needed, apply the formula

$$°C = \frac{5}{9}(°F - 32).$$

SAFETY & HEALTH CONSIDERATIONS

Before handling or using, read and understand the current KTI Chemicals Material Safety Data Sheet for these products.

Recommended Storage: Store in original container in a dry area. Do not store in direct sunlight. Refer to product label for recommended storage temperatures for maximum shelf life.

Handling: Handle with care. Many KTI photoresist materials are flammable or combustible, and may be harmful if swallowed, inhaled or absorbed through skin. Refer to current MSDS for further details on specific products. Use with adequate ventilation to keep vapors below recommended exposure limits. Loosen closure cautiously before opening. Keep away from heat, sparks, and flame. Keep container closed. Do not breathe vapor. Photoresists are light sensitive. Open containers only under safelight conditions.

Photoresists cause skin and eye irritation. Do not get in eyes, on skin, or on clothing. Wear chemical goggles, resistant gloves, and protective clothing to prevent contact. Accidental contact should be washed away immediately.

KTI KPR Resist contains 2-methoxyethyl acetate.

KTI KPR Resist Type 3 contains monochlorobenzene, 2-ethoxyethanol, cyclohexanone, and hydroquinone.

KTI KPR Resist Type 4 contains 2-ethoxyethyl acetate and 4-butyrolactone.

KTI Resist 930 contains 2-ethoxyethyl acetate and 4-butyrolactone.

KTI KTFR Negative Photoresist contains xylene and 2-methoxyethanol.

KTI KPR Developer contains xylene and 2-methoxyethyl acetate.

KTI KPR Developer Type 3/Type 4 contains xylene and cyclohexanone.

KTI KTFR Developer contains Stoddard solvent.

KTI Thinner Type 3 contains monochlorobenzene, cyclohexanone, and 2-ethoxyethanol.

KTI Thinner Type 4 contains 2-ethoxyethyl acetate and 4-butyrolactone.

KTI KPR Thinner for Photoengraving contains 2-methoxyethyl acetate.

KTI KPR Thinner for Photolithography contains 2-methoxyethyl acetate and triethanolamine.

KTI KTFR Thinner contains xylene.

KTI KPR Dye Blue contains xylene, benzaldehyde, and 2-ethoxyethanol.

WARNING

In laboratory animal studies, birth defects and adverse effects on pregnancy have been observed with 2-ethoxyethanol, 2-ethoxyethyl acetate, and 2-methoxyethanol, which is closely related to 2-methoxyethyl acetate.

In laboratory animals, prolonged and repeated exposure to 2-methoxyethanol has caused damage to reproductive organs.

For 2-ethoxyethyl acetate and 2-ethoxyethanol:

Maximum personal exposure limit in air: 5 ppm (ACGH 1987-88), 8 hour time-weighted average. Avoid all skin contact.

For 2-methoxyethanol and 2-methoxyethyl acetate:

For women of childbearing potential,

- Maximum personal exposure limit in air: 2 ppm (8 hour time-weighted average).
- Maximum 15 minute excursion level: 5 ppm (no more than four such excursions daily with a minimum separation of one hour).
- Avoid all skin contact.

For persons other than women of childbearing potential,

- Maximum personal exposure limit in air: 5 ppm (ACGIH 1987-88), 8 hour time-weighted average.
- Avoid all skin contact.

FIRST AID: In case of contact, immediately flush eyes with plenty of water and continue flushing for at least 15 minutes. Seek medical attention. Remove contaminated clothing and wash skin with soap and water. Wash clothing before reuse. Discard contaminated shoes.

If inhaled, remove to fresh air. If breathing has stopped, give artificial respiration. If breathing is difficult, qualified personnel may give oxygen. Call a physician.

For **KPR Resist, KPR Resist Type 3, KPR Resist Type 4, Resist 930, KPR Thinner Type 4, KPR Thinner for Photoengraving, and KPR Thinner for Photolithography:** If swallowed, give two glasses of water and induce vomiting. Do not make an unconscious person vomit or take anything by mouth. Call a physician.

For **KPR Thinner Type 3:** If swallowed, give two glasses of milk or water. **DO NOT** induce vomiting. Obtain medical attention without delay.

For **KTFR Negative Photoresist, KTFR Developer, KPR Developer Type 3/Type 4, KPR Developer, KTFR Thinner and KPR Dye Blue:** If swallowed, **DO NOT** induce vomiting. **DO NOT** give anything to drink. Obtain medical attention without delay.

FIRE: Use water spray, carbon dioxide, dry chemical, or "alcohol-type" foam.

SPILL OR LEAK: Cover with absorbent or flush with water.

Prevent runoff. Collect and dispose. Observe government regulations. Extinguish and do not turn on any ignition source until the area is determined to be free from explosion or fire hazards.

ATTENTION: Emptied product container retains vapor and product residues. Do not cut or weld on or near product containers. Obey all label warnings. Refer to all federal, state, and local regulations prior to reuse or disposal of containers. Never attempt to clean or reuse photoresist containers. The contents may react violently with the cleaning materials used.

For Industry Use Only

Emergency: In emergencies such as spills, leaks, fire or exposure involving KTI Chemicals Inc. products, call Union Carbide Corporation H.E.L.P. day or night at **1-(800) UCC-HELP**. For any chemical emergency, call CHEMTREC **1-(800) 424-9300**.

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