Solder Fusing with Heated Liquids

Heat transfer liquids offer advantages over other heating methods but liquid selection requires careful checkout

BY D. SCHÖNTHALER

ABSTRACT. Multilayer printed circuits used in new-generation electronic systems are large, complex and costly. Their manufacture requires processes that are easily controlled and provide a high yield. The solder fusing process is used for removing solder slivers and for the batch fabrication of soldered interconnections. Without good process control, multilayer circuits can be easily damaged during the application of heat.

Forced convection heat transfer with heated liquids offers several advantages for solder fusing. As the most distinct advantage, this method of heating limits the maximum component temperature to that of the heated liquid, thus avoiding several process control problems associated with the commonly used infrared or hot air methods. This paper discusses the process control requirements for fusing, the capability of commonly used heating methods, the application of the liquid immersion fusing process to circuit manufacturing, and the selection of heat transfer liquids.

Introduction

Solder fusing is commonly used for two basic reasons in the manufacture of electronic equipment systems. First, as a measure to assure product reliability, fusing is required to remove solder slivers which occur during the generation of printed circuitry. Slivers result from undercutting of the electrodeposited solder used as the etch resist during copper removal. Secondly, solder is fused in joining applications where predeposited solder is melted to form electrical interconnections between various components. In these reflow soldering processes solder is usually applied by either electroplating or the placement of individual preformed solder doughnuts.

A high process yield is necessary because fusing is performed near the end of either the circuit or assembly fabrication at which point the circuit or assembly is at its peak cost. The high yield requirement is becoming more significant with the increasing use of large costly multilayer circuits, which contain as many as 10 or more copper circuit planes sandwiched between layers of dielectric such as epoxy-glass. In providing a high yield, the fusing process must be capable of processing circuit configurations and materials which are heat sensitive and therefore are easily damaged by excessive heating.

Process Requirements

The use of various heating methods for solder fusing can be best described by considering the properties of typical new-generation circuitry. Figure 1 illustrates a multilayer circuit assembly consisting of an epoxy-glass multilayer circuit board.
soldered to 40 connectors containing 80 terminals each. The connector terminals, introduced through the multilayer board from the underside, must remain free of solder because additional interconnections are made to the terminals by wire-wrap or pressure contacts. As a result considerable clearance is provided between the plated-thru holes of the multilayer board and the terminal. Solder of sufficient quantity to provide fillets on both the circuit board and the barrel of the plated-thru hole is added by solder preforms. Additional terminals may be staked in at various locations. With staked or press-fit terminals it is often not necessary to add solder, since the solder electropolished on the multilayer board is usually sufficient to form joints.

The soldering process selected for fabricating the interconnections on this assembly must heat the circuit board and terminals to temperatures near 410°F for good flow of liquid solder. The process should be tolerant of the variations in heating requirements across the surface of the circuit. The central area of the assembly, without connector terminals, requires less heat than the connector areas because of the reduced heat capacity. The circuit and terminals should be at identical temperatures for good wetting of both surfaces by the solder and the formation of idealized fillet shapes as predicted by T. Y. Chu (Ref. 1). Overheating of the terminals, for example, may produce soldered climb as a result of an increase in wetting rate with temperature. Uniform heating is difficult because of the variations in contact area between the terminal and circuit and variations in heat capacity between short, stacked-in terminals and long, connector terminals.

The soldering process must also be capable of heating circuits which are heat sensitive. Copper to epoxy-glass laminates commonly used for single and double sided circuits can withstand solder temperatures of 500°F for 20 seconds without exhibiting evidence of thermal degradation (Ref. 2). More severe thermal cycles cause thermal degradation.

One form of such degradation results in "measles", which are discrete locations where the glass fibers separate from other fibers or from the epoxy resin. Their rate of formation is both time and temperature dependent. Compared to the basic laminate, it is more difficult to prevent thermal degradation in multilayer circuits because of the longer heating cycles involved. The long cycles are due to their large size and high volumetric specific heat, typically 29 Btu/ft²deg F.* The high value results from the use of very thin individual laminates which provide an increased amount of copper compared to epoxy-glass.

Since the heat capacity increases faster than the surface area, multilayer boards cannot dissipate heat at a rate sufficient to permit exposure to the above 500°F, 20 second heating cycle. For example, a large 18×24 in. multilayer circuit (without connectors) weighing 3 lb would require 246 Btu to elevate its temperature from 70°F to 410°F. Nearly 10 minutes is required to return to room temperature. The same circuit, assembled to connectors would require more than 1500 Btu and require significantly longer cooling times. Multilayer circuit construction, therefore, place constraints on the thermal endurance of commonly used epoxy-glass laminates. In order to avoid thermal damage to the circuit board, the fusion process should minimize the length of the heating cycle without thermally shocking the circuit at a rate sufficient to cause mechanical failure.

**Characteristics of Fusing Processes**

Solder fusing of large circuits and assemblies is accomplished with a variety of heating methods. Each method has its unique characteristics which determine its applicability to specific manufacturing requirements. The heating methods currently used are described below. The need for better process control, as provided by forced convection heating with heated liquids, is clearly indicated.

**Radiant Heating**

Radiant heating has been successfully applied to the fabrication of backplane interconnectors similar to those illustrated in Fig. 1 (Refs. 3, 4). In addition this method of heating is widely used for solder silver removal (Ref. 5). In both applications high process yield results from close engineering supervision and careful optimization of the operating parameters.

Equipment used for fusing large circuits typically consists of a workpiece conveyor for transporting the circuit, at a controlled speed, through a heat zone of infrared energy. The heat zone is either a narrow, intense region of high heat flux (30 to 140 W/cm²) generated by a focusing reflector (Ref. 3) or a broad area of relatively low heat flux (3 to 15 W/cm²) generated by a diffuse reflector (Ref. 4). In some equipment diffuse radiant energy is obtained by using the focused system with the workpiece placed in a plane which does not pass through the focal point, i.e., where the energy is defocused.

The net heat transfer by radiation from the source reflector system to the circuit being fused is given by

$$q_r = \sigma A_2 F_{12} \left( T_1^4 - T_2^4 \right) \text{ Btu/h}$$

where

$$A_2 = \text{Area of the circuit in square feet}$$

$$F_{12} = \text{A factor which accounts for the emissivity of the source, the absorptivity of the circuit and the geometrical shape of both the circuit and source}$$

$$T_1 = \text{The circuit temperature in degrees Rankine}$$

$$T_2 = \text{The source temperature in degrees Rankine}$$

$$\sigma = \text{The Stefan Boltzmann constant, 1.797×10^{-8} Btu/ft^2 h deg R}$$

The radiant energy source commonly used is an incandescent-filament, infrared lamp usually operated at filament temperature between 2300 and 4000°F. Higher temperatures are achieved with halogen cycle lamps. The filament temperature is usually held constant by using the optimum lamp voltage setting and the circuit temperature is increased to approximately 410°F for good solder flow. Because of the fourth power relationship the heat radiated by the workpiece has little effect on the net heat transfer to the circuit for high source temperatures (i.e., high levels of incident radiation).

The radiant heat transfer rate $q_r$ can also be computed using the radiant heat transfer coefficient $h_r$ in Btu/h ft² deg F, as follows:

$$q_r = h_r A_2 (T_1 - T_2)$$

The other factors remain as described above and $A_2$, the workpiece area is the area upon which $F_{12}$ is based. Therefore, as presented by Kreith (Ref. 6) the heat transfer coefficient $h_r$ is determined from equations (1) and (2) as being

$$h_r = \frac{q_r}{A_2 (T_1 - T_2)} = F_{12} \frac{\sigma \left( T_1^4 - T_2^4 \right)}{T_1 - T_2}$$

The heat rate of multilayer circuits exposed to radiant energy can be
Thermal response of multilayer circuits for various convective heating methods calculated as in Ref. 7.
forms to a predetermined (programmed) time-temperature cycle (Ref. 4). The circuit temperature is measured and compared to the programmed value with the temperature difference being used to drive the power controller for regulating the lamp voltage and heat flux. Good process control is achieved but the heat shield design, temperature cycle, and temperature-measuring detector location continue to be empirically determined.

Convective Heating

Solder fusing by the transfer of heat from high temperature fluid in contact with the circuit is governed by the relation.

\[ q_c = \frac{h_c A (T_c - T_f)}{L} \text{ Btu/h} \]  

where

- \( q_c \) = The rate of heat transfer from the fluid to the circuit.
- \( A \) = The circuit area in square feet.
- \( T_c \) = The circuit temperature in deg F.
- \( T_f \) = The fluid temperature in deg F at a distance remote from the circuit.

The convective heat-transfer coefficient, \( h_c \), in Btu/h ft° deg F, will range from 1 to 5 for natural air convection, 5 to 50 for forced hot-air convection, and 10 to 300 for forced convection with hot oils as reported by Kreith (Ref. 6). With the exception of the high \( h_c \) values for the hot oils, the heat transfer rates are similar to those reported previously for radiant heating.

The convection heat transfer coefficient, \( h_c \), is a function of the geometry of the circuit, the fluid dynamics and the fluid properties including thermal conductivity, velocity, density, specific heat and viscosity. Once \( h_c \) is determined for each side of the circuit, the values are used in equations (4), (5) and (6) of Mollendorf’s modified transient analysis to calculate the heating rate for fusing. Again, if additional modes of heating occur concurrently, their transfer coefficients should be included in the analysis. As a result of the numerous parameters to be considered, the rate of heat transfer is significantly affected by the choice of fluid, its temperature and flow characteristics.

Forced hot air convection heating has been used for joining applications as reported by Weltha (Ref. 8). The equipment reported on by Weltha heats circuit assemblies with 572-617 F hot air striking the circuit at a velocity of 1300 to 1600 ft/min. With the counter flow equipment reported, the circuit is initially preheated by the downstream air. Mollendorf (Ref. 7) considered a circuit similar to that of Fig. 1 for the case of forced hot air heating at 1000 F with an exceptionally high velocity of 3000 ft/min. Under these conditions he found the heat transfer coefficient \( h_c \) to be 7.77 Btu/h ft° deg F and a time of nearly 50 seconds required to reach the solders flow temperature of 410 F. The convection coefficient and heating time is slightly less than the radiant coefficient, \( h_r \), of 9.4 Btu/h ft° deg F for an incident flux of 3 W/cm², and heating times of 50 seconds from Fig. 2. As a result, the same heating effects are expected on the circuit board where for low heating rates thermal damage was observed before solder flow. The parameters used by Weltha would result in even lower heating rates which would be unacceptable for the massive circuit studied; however, they may be well suited for conventional circuitry. Weltha also reports that excessive air velocity will force solder to one side of the terminal and higher temperatures result in solder wicking up the terminals excessively.

Forced hot air heating may have the advantage of heating terminals more directly than radiant heating, but this advantage appears to be outweighed by the disadvantage of longer heating times. In each of the central portions of the assembly (Fig. 1) would again be overheated while the connector areas are coming up to temperature at a lower heating rate. It is therefore difficult to heat complex assemblies quickly and uniformly with forced hot air without again developing unique baffles (shields) to protect low mass areas, as with radiant heating.

Let us now consider increasing the convective heat transfer coefficient in order to shorten the heating time. Liquid solder is commonly used in wave generators for soldering printed circuits with the solder temperature range of 480 to 540 F being used. This mode of heating has been analyzed and the heat transfer coefficient was computed to be 3025 Btu/h ft° deg F for solder wave at 480 F with a solder velocity of 42 ft/min (Ref. 7). The heating time was found to be approximately 4 seconds for the board to reach the 480 F solder temperature. The rapid heating rate, relatively high temperature and the exceptionally long cooling period lead to thermal damage of the circuit. Controlling the heating rate to reduce the maximum temperature would be nearly impossible for the large assemblies typical of Fig. 1. Again the central area would be overheated compared to the connector region. In addition to thermal degradation, solder would plug the plated-thru holes if wave soldering techniques were used for eliminating solder slivers, and solder would be deposited on the connector terminals in joining applications. Solder coated terminals are not permitted where high quality wire wrap connections are subsequently added.

Overheating can be eliminated by reducing the temperature of the fluids used in convective heating to a temperature considered “safe” for the circuit during heating times significantly longer than fusing requirements. By using a hot fluid at a selected “safe” temperature the circuit or circuit assembly can be heated without specially designed fixtures, shields or baffles being required to prevent overheating. No point on the circuit will exceed the fluid temperature regardless of fluid properties as illustrated in Fig. 4. For a required 410 F solder flow temperature, a 420 F fluid temperature was used in the subsequent investigation. Mollendorf considered polyethylene glycol as a liquid for heat transfer and found the heat transfer coefficient to be 80.9 Btu/h ft° deg F for a fluid velocity of 60 ft/min (Ref. 7). The circuit approaches the 420 F glycol temperature in approximately 35 seconds, a heating rate comparable to the acceptable radiant heating conditions of Fig. 2 which did not result in thermal degradation of the circuit. However, for massive multilayer circuits, hot air at 420 F with a velocity of 1500 ft/min requires nearly 10 min to reach the 410 F solder flow temperature and 4 min to cause solder to melt at 361 F. Thermal degradation occurs with these conditions. A solder wave at this unusually low temperature is required to reach 420 F, but solder cannot be considered for joining wire wrap terminals to circuits as discussed earlier.

Fusing for silver removal would not be required if the hot solder coating process developed by Allen and Sylvester (Ref. 9) could be utilized. In their process, solder is applied by dipping and air knives blow excess solder out of the holes. However, the conventional techniques can remove excess solder from the plated-thru holes on large (18 x 24 in. x 0.1 to 0.15 in. thick) circuits with up to 25,000 holes requires study. The solder thickness variations are greater than desired for this method of solder coating. In addition the thermal shock of exceptionally high heat transfer rates may be detrimental to the reliability of the thru holes of multilayer structures.

The following studies are evidence of the desirable characteristics of
cress, and its properties will be subsequently discussed with regard to its applicability for solder fusing. One major restriction of the condensation process is the limited choice of processing temperature. This temperature corresponds to the boiling point of liquids available, unless the operating pressure differs from atmospheric.

**Heat Transfer Liquids**

A major task in the development of the liquid immiscible solder fusing process is the selection of a suitable heat transfer liquid. Several authors have reported on the successful application of heated liquids for solder sliver removal (Refs. 11, 12, 13). Liquids considered in these studies include peanut oil, polyethylene glycol, and proprietary liquids derived from polyalkylene glycol. Polyethylene glycol was also reported to be used in joining applications for manufacturing assemblies with a multiplicity of circuit boards stacked on a common array of terminals (Ref. 14).

**Properties Studied**

In selecting a heat transfer liquid a considerable number of properties must be studied. The liquid properties of interest may be classified in two general categories: (1) intrinsic thermal, physical and chemical properties and (2) extrinsic properties related to the product or processing equipment. An excellent discussion of many of these properties and a survey of specific fluid characteristics is available (Refs. 15, 16).

The intrinsic thermal properties which determine the convection heat transfer coefficient were previously discussed. Over the temperature range usually considered for soldering (300 to 480 F), the liquid should be physically stable. However, in the case of condensation heating a change of phase from liquid to vapor is necessary in the above temperature range. Many liquids used for heat transfer are blends of different chemical compounds mixed in a manner which optimizes desirable characteristics. The liquid selected must be chemically stable such that its processing performance will be consistent. For example, additives are often mixed with polyglycols to control their pH level but their effective life is not without limit when the liquid is placed into service.

Thermal stability is of interest since thermal decomposition usually results in a degradation of the physical and chemical properties. The rate of decomposition increases with increases in operating temperature. Physiological properties must be examined for the effect of the fluid on operating personnel and the environment for both room and fusing temperatures. Surface tension is indicative of the ability of the liquid to wet surfaces and may be of significance in designing handling equipment. Liquids having low surface tension are prone to leaking through mechanical seals. Flammability must be considered for reasons of safety and taken into account in subsequent equipment design. Corrosivity should be low at both room and fusing temperatures for reasons of operator safety, equipment durability, and product reliability. Last, but certainly not of least importance, the intrinsic property of cost must be ascertained.

The "liquid's solderability" is an important extrinsic property because the fusing process is intended for applications in both solder sliver removal and metals joining. As studied, the "liquid's solderability" is a measure of its ability to permit solder to wet metals used in circuit assemblies. An equally important property is the effect of the liquid on the insulation properties of circuit materials. Related to this consideration is the ease of liquid removal from the cir-
cuit. Although the effect of the liquid on the circuit is of prime interest, the behavior of the fluid on the processing equipment must be studied. Manufacturing equipment must perform its function well with a minimum of shop maintenance for long periods of time. At operating temperature, some liquids, especially oils, tend to leave carbon-like deposits on equipment surfaces in relatively short times. Unless they can be removed easily, equipment maintenance becomes exceedingly difficult. Finally, the economic justifications for using the selected liquid will depend on the intrinsic cost of the liquid, the value of the product, and the yield or reliability of the fusing process.

Liquid Evaluation Program

More than thirty liquids were considered for use in fusing and eighteen were tested. The remainder were ruled out because of extremely low flash points, high toxicity, or rapid decomposition when exposed to the atmosphere at fusing temperatures. The liquids tested are classified into seven categories: (1) trihydric alcohol, (2) polyglycol, (3) fluorinated polyoxypropylene, (4) polychlorotrifluoroethylene, (5) polyphenol ether, (6) mineral oil, and (7) vegetable oil. Typical values of important physical properties of the liquids are listed in Table 1. Liquids from these categories were evaluated for their solderability, performance in fusing, and ease of cleaning from both the workpiece and equipment. Subsequent to this evaluation selected liquids were tested for their influence on the electrical properties of circuit materials.

The "liquid's solderability" was determined by evaluating the ability of solder to wet selected materials when fusing was attempted at temperatures typical of those expected to be used for manufacturing. A solder spread test similar to that developed by Pessel (Ref. 17) for use with electrical lead wires, was used. Figures 5 through 10 illustrate the results of these tests which use wire-wrap terminals available from a Western Electric manufacturing plant. The copper-nickel-tin (Cu-Ni-Sn) alloy terminals have 0.050 by 0.040 in. cross section in the wire wrap area. Two surface conditions were used: unplated and gold (Au) plated to a thickness of 25 X 10^-4 in. Prior to testing, all terminals were degreased and the unplated terminals were cleaned with diluted HCl and washed well to remove surface oxides. Properly prepared terminals were stored in a dry box with a desiccant and nitrogen purge for preserving their solderable surfaces.

Solder for the spread test was applied with a preformed doughnut 0.10 in. OD by 0.050 ID by 0.015 in. thick. Since the terminal diagonal is 0.059 in., intimate contact with the preform is assured. The preforms were coated with a dry, unactivated, water-white rosin flux (type R of specification MIL-14256C) as supplied by the manufacturer and stored in a dry box prior to use. Unactivated rosin was selected in order to accentuate the variations in solderability between liquids. The clean Cu-Ni-Sn alloy was known to have good solderability with mildly activated flux (type

<table>
<thead>
<tr>
<th>LIQUID A</th>
<th>BATCH I: 460°F-10 SEC.</th>
<th>BATCH II: 440°F-20 SEC.</th>
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<tbody>
<tr>
<td>AU PLATED</td>
<td>LIQUID A</td>
<td>430°F-20 SEC.</td>
</tr>
<tr>
<td>2,120,380 -HR-</td>
<td>1,50,105 -HR-</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 6 — Solderability test results for polyglycols

<table>
<thead>
<tr>
<th>LIQUID A</th>
<th>LIQUID B</th>
<th>460°F-10 SEC.</th>
</tr>
</thead>
<tbody>
<tr>
<td>24,311,504 -HR-</td>
<td>1,49,90 -HOURS-</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 7 — Solderability test results for fluorinated polyoxypropylene

<table>
<thead>
<tr>
<th>LIQUID A</th>
<th>LIQUID B</th>
<th>460°F-10 SEC.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,49,90 -HOURS-</td>
<td>1,49,90 -HOURS-</td>
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</table>

Fig. 8 — Solderability test results for polychlorotrifluoroethylene
RMA of MIL-14256C), and thought to be marginally solderable with type R flux. The preforms were applied over the terminals and moved down until they contacted the shoulder.

The test was made by immersing the terminal into the various liquids for the time, temperature and liquid age as shown in the figures. The “liquid’s solderability” is assessed from the degree of solder spread and the contact angle between the solder and terminal. If the included angle is acute, solderability is considered good with zero degrees being excellent. The initial series of evaluations was conducted with the liquids being aged at 460 °F. This was considered the maximum temperature which would be required for processing and would result in the most rapid thermal degradation. It was subsequently determined that 440 °F was more realistic and thus a second series of evaluation was based on this temperature.

The performance of the liquids in fusing for solder removal was determined by observing the characteristics of electroplated solder after immersion in selected liquids heated to 440 °F. The circuit coupons used were 2 in. square, single-sided, epoxy-glass laminates having a copper comb pattern plated with a 60% tin, lead-tin alloy to a thickness of 0.001 in. Half of the circuits were processed unflaxed and half were fluxed with a mildly-activated rosin-base flux, type RMA-10% solids content, and dried before immersion. The comb patterns were heated at times from 5 to 40 seconds. After immersion the samples were rated and designated (1) for no evidence of solder melting, (2) for marginal evidence of melting but having little or no movement of the oxide film covering the solder plate and (3) for excellent fusing where a bright, reflective, oxide-free convex surface is apparent. The samples were immersed in selected liquids aged at 440 °F for approximately 1, 50, and 100 h. The results of this test are provided in Tables 2 and 3 for 1 h and 100 h liquid age respectively. While processing these samples, judgements were made of the ease of liquid removal during washing in the appropriate solvent.

The equipment used for aging the liquids under test is shown in Fig. 11 where, for one particular liquid, a carbon like deposit is clearly shown. Deposits were usually observed on the stainless-steel beaker, chrome plated stirrer, and glass temperature sensor/controller. With some liquids a surface scum was observed as illustrated in Fig. 11. The stainless steel was supported in an electric heating mantel which uniformly heated the liquid and the temperature was controlled to ±5 °F. The liquids were stirred during the heating cycles to prevent overheating at the walls of the container. Periodically the condition of the vessel was observed and both the degree of contamination and ease of removal were recorded.

Evaluation Results

The liquid categories are discussed independently in the following evaluation of the liquids. The conclusions drawn for these categories are based on tests with particular liquids. The author wishes to emphasize the danger in assuming that all liquids in a category behave in a similar manner. Liquids must be independently evaluated according to the user’s requirements.

Trishydric Alcohol

Glycerol is the trihydric alcohol evaluated and a complete description of its properties is provided by
Physically, glycerol is colorless, hygroscopic and highly water soluble. Physiologically it is nontoxic, easily digested and is normally present in the human body. It is not irritating to the skin or mucous excepting in high concentration when it has a dehydrating effect (Ref. 21).

Glycerol is reported to be a constituent of some soldering fluxes and improves the wetting of the metals since it remains fluid on the hot metal (Ref. 21). It has been used as a major constituent in water-soluble soldering fluxes and masks for protecting plated-thru holes during wave soldering (Ref. 22).

Glycerol's low flash point, 350 °F, and high vapor pressure, see Table 1, require special consideration in designing equipment for reasons of safety and reducing liquid consumption. Glycerol when strongly heated dehydrates into acrolein which has a highly penetrating odor. The maximum limit for eight hours exposure to acrolein is 0.5 parts per million of air; however, “the irritating effect of acrolein at very low concentrations usually gives adequate warning before exposure to toxic quantities can occur” (Ref. 23). Glycerol can be obtained for less than $10/gallon.

The solderability of glycerol is shown in Fig. 5 to be excellent for both gold plated and unplated Cu-Ni- Sn terminals.

Glycerol was easily washed from both the circuits and heating equipment with warm water; no scum or residues were observed. Glycerol, even when aged 572 h exhibited good to excellent solubility in warm water. From Tables 2 and 3 it is evident that glycerol performs excellently for fusing electroplated solder both with and without flux. It also fuses the solder more rapidly than the other liquids, as expected from its high heat transfer coefficient. The insulation resistance typically decreased by 50% compared to the control samples during environmental testing; and glycerol passed the copper mirror test for corrosivity (Ref. 24).

### Polyglycols

This category includes the polyethylene glycols and polyalkylene glycols reported previously. Seven proprietary, commercially-available liquids were tested along with two pure polyethylene glycols. The liquids can be purchased for less than

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**Table 2 — Results of Fusing Electroplated Solder on Comb Patterns with New Liquids**

<table>
<thead>
<tr>
<th>Liquid</th>
<th>Ratings (see footnote) for fusing times of 5 to 40 seconds</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>5</td>
</tr>
<tr>
<td>Trityric alcohol</td>
<td>1/1</td>
</tr>
<tr>
<td>Polyglycol</td>
<td>A</td>
</tr>
<tr>
<td></td>
<td>B</td>
</tr>
<tr>
<td></td>
<td>C</td>
</tr>
<tr>
<td>Fluorinated poly-</td>
<td>A</td>
</tr>
<tr>
<td>oxypropylene</td>
<td>B</td>
</tr>
<tr>
<td>Polychlorotrfluoroethylene</td>
<td>A</td>
</tr>
<tr>
<td>MINERAL OIL</td>
<td>A</td>
</tr>
<tr>
<td>Vegetable oil</td>
<td>—</td>
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</tbody>
</table>

Rating criteria: 1 = no solder flow; 3 = marginal; 5 = excellent.
Conditions: unfluxed/fluxed; Liquid age: New.

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**Table 3 — Results of Fusing Electroplated Solder on Comb Patterns with Liquids Aged 100 Hours**

<table>
<thead>
<tr>
<th>Liquid</th>
<th>Ratings (see footnote) for fusing times of 5 to 40 seconds</th>
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<tbody>
<tr>
<td></td>
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<tr>
<td>Trityric alcohol</td>
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<td>Polyglycol</td>
<td>A</td>
</tr>
<tr>
<td></td>
<td>B</td>
</tr>
<tr>
<td></td>
<td>C</td>
</tr>
<tr>
<td>Fluorinated poly-</td>
<td>A</td>
</tr>
<tr>
<td>oxypropylene</td>
<td>B</td>
</tr>
<tr>
<td>Polychlorotrfluoroethylene</td>
<td>A</td>
</tr>
<tr>
<td>MINERAL OIL</td>
<td>A</td>
</tr>
<tr>
<td>Vegetable oil</td>
<td>—</td>
</tr>
</tbody>
</table>

Rating criteria: 1 = no solder flow; 3 = marginal; 5 = excellent.
Conditions: unfluxed/fluxed; Liquid age: 100 h.
the surface. The deposits could not be removed with common detergents or solvents. The polyglycols investigated had flash points above 500 F, with the exception of a low molecular weight pure glycol, and all were reported to be water soluble. A heat transfer coefficient lower than glycerol results from the lower values of thermal conductivity, specific heat, and density combined with the slightly higher viscosity as listed in Table 1. An analysis of two proprietary liquids showed them to be similar in composition but with different ratios of the ethylene oxide to propylene oxide monomers; being 1:1 for one and 3:1 for the other (Ref. 24). At high temperatures and in the presence of air the polyglycols undergo decomposition. For this reason their stability is usually increased by the use of antioxidants such as hydroquinone (Ref. 12). The decomposition products are soluble in the liquid or leave the liquid as a vapor. Since these products are toxic, ample ventilation and exhaust must be provided.

Toxicological studies on several proprietary liquids have indicated that they do not present unusual handling hazards. The glycols are more toxic than glycerol but are considered sewerage-disposable.

The results of the solderability study are shown in Fig. 6 for two polyglycol liquids. Liquid A exhibited the same behavior for two different batches tested one year apart. For both batches the solderability to gold plated terminals was good for liquid ages extending to 380 h, but the area of solder spread was less than for glycerol. Wetting of Cu-Ni-Sn was rated poor to nonexistent for liquid age less than 120 h. At 380 h a slight improvement was noted, indicating a possible decrease in pH. Liquid B behaved in a similar manner with the plated terminals and was aged to 504 h. A slight improvement is observed for the Cu-Ni-Sn surfaces; however the contact angle was large. There was no apparent improvement in solderability to Cu-Ni-Sn surfaces with age.

The performance in fusing electroplated solder was unsatisfactory with new liquid for the three polyglycols tested, as illustrated in Table 2. From Table 3 it is apparent that A and B polyglycols, when aged 100 h, gave excellent performance for fusing times of 30 seconds or more, when flux was used. Without flux, B is slightly better than A. After 50 h of heating deposits similar to that of Fig. 11 were observed on the heating equipment; however, no scum was observed on the surfaces. The deposits could not be removed with common detergents or solvents. One liquid, reported to be completely water soluble, discolored the samples with a solvent soluble substance. Insulation resistance tests of one liquid resulted in no apparent reduction after four days of conditioning and four liquids have passed the corrosion test (Ref. 24). Mulholland reported a reduction of electrical properties by a factor of 1.7 for a polyalkylene glycol (Ref. 13).

**Fluorinated Polyoxypropylene**

The fluorinated polyoxypropylene liquids are nonflammable, extremely low in toxicity and stable both chemically and thermally to temperatures over 500 F. The liquids are available with many different molecular weights each having a sharp, well-defined boiling point. In addition, their dielectric strength and resistivity is high and they are compatible with plastics, elastomers and coatings. The fluorinated polyoxypropylene liquids are costly — at more than $300/gallon.

The two liquids tested had boiling points of 435.6 F and 554 F for Liquid A and Liquid B of Fig. 7, respectively. Table 1 lists a vapor pressure typical of Liquid B, since the low volumetric specific heat and low thermal conductivity indicate a low heat transfer coefficient, but this is somewhat offset by the low viscosity. These fluorinated liquids have the lowest surface tension of the liquids listed in Table 1. Both Liquid A and Liquid B exhibited good wetting of gold plated terminals when new, having reasonably good solder spread and acceptable contact angle. As the liquids were aged to 100 h the contact angle increased, approaching an unacceptable 75 deg. Poor wetting with contact angles greater than 90 deg were observed with both liquids for the Cu-Ni-Sn surfaces.

Tables 2 and 3 show both liquids to perform well, when new, for fusing electroplated solder, providing the mildly activated rosin flux is used. No deposits were observed on the heating equipment and the fluorinated polyoxypropylene was easily cleaned from the samples with common fluorinated solvents. An order of magnitude increase in the insulation resistance was recorded during environmental testing (Refs. 7,24). This may be due to the entrainment of high dielectric liquid within the sample laminate. Since these liquids are chemically inert the copper mirror test was not performed.

**Polyphenol Ether**

Polyphenol ether, as developed for high temperature applications, has an exceptionally good resistance to heat with a decomposition temperature of 847 F (Ref. 15). It has a flash point of 550 F and a very low vapor pressure at fusing temperatures, from Table 1. Its low thermal conductivity and volumetric specific heat suggest a low convective heat transfer coefficient. Physiologically it is inert and requires no special handling or precautions.

Polyphenol ether exhibited fair solderability to gold plated terminals when new and nonwetting to Cu-Ni-Sn surfaces, as illustrated in Fig. 9. The liquid is costly at more than $300/gallon. Because of a limited quantity available, further testing was discontinued.

**Mineral Oil**

Hydrocarbon oils, specially refined for heat transfer applications, are.
readily available from the petroleum industry at low cost; usually less than $10/gallon. They have been widely used in wave soldering processes for covering molten solder to reduce dross formation. Some users claim improved soldering yield because of a reduction of surface tension during soldering which tends to eliminate "icicling" and "bridging."

At high temperatures, mineral oils decompose by two different mechanisms (Ref. 15): cracking and oxidation. During cracking the oil breaks down to form volatile substances, which lower the flash point, and heavy insoluble substances, which deposit on equipment surfaces and reduce heat transfer coefficients. Heat transfer efficiency is also reduced by the formation of insoluble carbon-like materials resulting from oxidation. Oils developed for high temperature soldering applications are blended with fatty acids and oxidation inhibitors. The fatty acids tend to increase the oil's "solderability" (Ref. 24). As listed in Table 1, the oils tested have flash points greater than 500 F.

Two oils were tested for solderability, as shown in Fig. 10. Oil A exhibited excellent solder spread and low contact angles with gold plated terminals; even when aged to 548 h. Initially, Oil A permitted good wetting of Cu-Ni-Sn, however the contact angle increased to an unacceptable level by 356 h. At 548 h, nonwetting existed. Oil B showed good solderability to plated terminals from the new condition to 105 h. When new, the wetting to Cu-Ni-Sn was unacceptable and nonwetting was evident at 50 h. Both oils performed well in fusing solder plate with both fluxed and unfluxed samples after a 30 second immersion time as shown in Table 2. Table 3 indicates the need for the mildly activated flux after Oil B has aged 100 h, while Oil A has not changed in fusing capability.

The carbon-like deposit and surface scum shown in Fig. 11 resulted from Oil B after 50 h of heating. These deposits could not be removed with chlorinated solvents, however, the samples were easily cleaned. Similar deposits resulted with Oil A. Insulation resistance measurements indicated no significant reduction in resistance values during environmental testing; and the corrosion test showed the oils to be corrosive to copper when new but not after high temperature aging (Ref. 24).

Vegetable Oil

The one oil commonly used as a basis of comparison for fusing solder is a vegetable oil, namely peanut oil (Refs. 11,13). Many oils in this category are used in cooking, are inexpensive at less than $5/gallon, and are described in Ref. 25. They are generally made up of a molecule of glycerol joined to three fatty acid molecules. As the oils are heated a smoke point is reached before the flash point. This smoke temperature is an indicator of the oil's thermal stability and as decomposition occurs it becomes lower. Although decomposition products are toxic and should be properly exhausted, the amount of such vapors at 400 F should be minimal because of their low vapor pressure. The physical properties listed in Table 1 are comparable to the polyglycols and the heat transfer coefficient should be similar.

Two vegetable oils have been investigated: peanut oil and safflower oil. Peanut oil has a 440 F smoke point (Ref. 25) and a 540 F flash point (Ref. 24). Safflower oil has a very high smoke point of 535 F and a flash point exceeding 609 F (Ref. 25). Figure 9 shows the vegetable oil to have fair solderability for gold plated terminals, having a high contact angle which increases to an unacceptable angle at 105 h. Nonwetting occurred for Cu-Ni-Sn surfaces. Good performance in melting electroplated solder was observed for long heating times (30 seconds), aged oil (100 h) and with samples coated with the mildly activated flux (see Tables 2 and 3).

The samples were easily cleaned with chlorinated solvents; but the heating equipment was impossible to clean, even after 50 h, since the deposits were thicker than shown in Fig. 11. Peanut oil is reported to increase the electrical properties by a factor greater than 2 (Ref. 13) and it has passed the corrosion test (Ref. 24).

Liquid Selection

The choice of a suitable liquid for production processing is difficult because compromises must be made on some of the many characteristics considered for evaluation. In review, these characteristics included solderability, flammability, heat transfer coefficient, ease of cleaning, equip-
Fusing Applications with Glycerol

Glycerol was used to fuse the assembly of Fig. 1, whereby high quality solder joints were obtained between the connector terminals and the circuit's plated-thru holes. Solder joining is accomplished by placing fluxed solder preforms, one per terminal, over the terminals of each connector prior to assembly. These preforms contact the underside of the circuit after the connector terminals are inserted into their respective plated-thru holes. Although not required, a mildly activated flux is applied to the circuit to assure a high yield. With the glycerol maintained at a "safe" temperature of 440°F, the circuit is immersed, terminal side down, until the liquid covers the circuit. The connector bodies are not immersed.

Joints of excellent quality, as shown in Fig. 12, were formed with a defect rate of less than 0.1% with laboratory equipment. During heating the circuit is heated at a rate which is in good agreement with the theoretical rate predicted by equation (4). The results of numerous temperature measurements show that the first area to reach 410°F required 9 seconds and the immersion cycle was terminated when the last area reached 410°F at 27 seconds. No thermal degradation occurred to the multilayer circuit.

Because of the complex construction of the assembly, including connectors assembled of many individual piece parts, thousands of capillaries and crevices occur where heat transfer fluids could be trapped. It was determined that there could be no assurance of complete removal of the liquid during subsequent cleaning steps. For this reason it is not recommended that a poor dielectric liquid, such as glycerol be used for similar complex assemblies. Soldering of simple wire-wrap terminals to printed circuits should present no problems, since adequate cleaning is possible. Liquid immersion heat transfer with glycerol is being used as a manufacturing process for solder sliver removal. Figure 13 illustrates a concentration of solder slivers on a multilayer circuit with 0.125 in. center to center hole spacings. These solder overhangs were broken away from the bulk solder plate with ultrasonic agitation. Without fusing, a potential for short circuiting from pad layer to pad layer exists. On a production basis, circuits are first baked at a temperature in excess of 300°F to remove entrapped moisture. The circuits are typically fused at a 430°F glycerol temperature for 25 seconds. After several washing steps, circuits are again baked at a temperature above 300°F for drying. Time and temperature requirements are dependent on product size and equipment capability.

Production Equipment

Caution must be exercised in developing production equipment for use with glycerol. The equipment must handle combustible liquids in a safe manner since ordinary dip tanks cannot be used (Ref. 26). Figure 14 illustrates the production facility designed for fusing circuits up to 30 by 38 in. in size. In operation, the facility processes the circuits through a fusing cycle to melt the solder, a dwell period to permit both liquid drain off and cooling of the circuit, and a water rinse to remove drag-out heat transfer liquid. Circuits are supported horizontally and moved very slowly in a smooth manner to minimize disturbances to the molten solder. Liquid waves of high velocity were considered unacceptable due to potential disturbances of the solder.

Special emphasis has been placed on instrumentation of the machine functions and the corresponding recording of such data for process...
evaluation cycle can be controlled manually, by cycle time or by circuit temperature. An electrostatic precipitator is used to clean the exhaust air and the bulk of the trapped vapor is filtered for recycling. Finally the liquid is covered at all times except during the immersion cycle and an automatically-actuated, carbon dioxide, fire extinguishing system is included.

Conclusion

The complex thermal considerations involved with fusing new generation electronic circuits and assemblies have been discussed. It was illustrated that it is often necessary to heat various components at a nearly uniform rate without causing thermal damage to the circuit and surrounding materials. This can be accomplished without unique protective heat shields, usually required with radiant or hot air methods, by using liquid convective heating processes.

Choosing a suitable heat transfer liquid is not a simple task. Trade-offs must be made between several significant properties. Glycerol ranks first, of the liquids tested, for having excellent fusing characteristics providing electrical requirements can be relaxed. Fluorinated polyoxypropylene yields the best electrical properties but its use must be economically justified.

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References