Pollution Control and Hazardous Materials Minimization in a Printed Wiring Board Shop: A Case Study

by

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FOREWORD

This report discusses the environmental improvement efforts performed by the Electronics Prototype Section (EPS) of the Engineering Department from 1988 through 1991. Contained, herein, are the past practices of EPS in processing printed wiring boards, improvements implemented in the processes, problems encountered, and future improvements.

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Environmental protection issues are making headlines daily. Some concerns are well founded while others are based on myth and/or fear. As inhabitants of this planet everyone must do their best to be wise stewards or our environment. The Electronics Prototype Section, Naval Air Warfare Center Weapons Division (NAWCWPNS), China Lake, California, have taken the responsibility of environmental stewardship seriously. This case study details their efforts to protect themselves and the environment from the hazardous materials used to fabricate printed wiring boards. This report discusses previous practices and the solutions developed to ensure a cleaner environment. Additionally, this report contains encountered problems and future plans.
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INTRODUCTION

Fabricating printed wiring boards (PWB) involves a variety of different processes that are divided into two categories: dry and wet fabrication. Dry fabrication is composed of drilling, routing, and imaging; whereas wet fabrication is composed of scrubbing, developing, plating, and etching. This study focuses only on the wet processes and the associated problems relative to environmental stewardship.

This shop had much to learn about environmental responsibility and natural resource conservation. The past processes of the shop were not a deliberate "nose-thumbing" attitude toward the environment and natural resources; but rather it was brought about by lack of knowledge and awareness.

All of the chemicals involved in wet processing are hazardous to some degree. These hazards revolve around the properties of the chemicals. For example, strong mineral acids are extremely corrosive and the vapors readily degrade most metals on contact. Inhalation of these vapors can cause a range of problems such as slight respiratory irritation, pulmonary edema, and death. All these hazards can be reduced or even eliminated from the workplace by engineering and administrative controls. Engineering controls include proper ventilation and material substitution. Administrative controls include Standard Operating Procedures (SOP) and training. Personal protective equipment (PPE) is used when the hazard hasn't been eliminated from the workplace.

PAST OPERATION PRACTICES

PROCESSING

The processes of environmental concern were electroless copper deposition, electrolytic copper plating, and electrolytic tin/lead or solder plating. All of the plating shop rinses consisted of tap water overflow that were discharged directly into an Industrial Sewer System (ISS). The initial processing flow rates were approximately 2,000 gallons/hour. Contained within this substantial rinse discharge were regulated materials (i.e., copper, tin, lead, and formaldehyde).

Scrubbing and deburring equipment used to process PWB panels was plumbed to discharge directly into the ISS. This discharge was approximately 12 gallons/minute and contained copper particles along with fibrous material from the scrubber wheels.

The photoresist developer and stripper used in the imaging process were also plumbed to discharge directly into the ISS with approximately 6 gallons/minute of outflow. Alkaline effluent in the imaging process contained residual organic material in solution and suspended particles.
Conveyorized spray etching equipment used to remove unwanted copper was plumbed to discharge directly into the ISS with an outflow of approximately 12 gallons/minute. This effluent contained acidic and alkaline waste solutions, copper, hexavalent chromium, and ammonium ion.

The clean/neutralization line used in support of the Hot Air Solder Leveling (HASL) process was plumbed to discharge directly to the ISS with an outflow of approximately 6 gallons/minute. The effluent contained residual organic compounds as well as tin/lead solder particles.

**PROCESS CONTROLS**

Control over the parameters that govern the effectiveness and efficiency of a process was important to the quality of the product being fabricated. The extent to which a process should be controlled was determined by various factors with cost being the most critical factor. Too little or too much money invested in process control resulted in higher operating costs. Too little money invested resulted in production of marginal and scrap products; while too much money resulted in no return to the extra investment. A proper balance between these two extremes had to be achieved to maximize benefits while minimizing operational costs.

Control of the plating process was accomplished by titration and Hull Cell analysis. There were problems associated with these methods. An example of one such problem variable was the presence of an entity that interfered with the endpoint determination of the titration. A Hull Cell is simply a small electroplating tank that was designed specifically to analyze electroplating baths. This apparatus enabled the analysis of all relevant current density areas on a single-cell panel. The appearance of the panel indicated the condition of the bath and its components. However, this analysis did not produce any quantitative data on which to base corrective action.

Application of these process controls for plating necessitated frequent dumping (every 6 to 8 weeks) of the various process baths. This was done to prevent the "crash" of the process resulting from the build-up of constituents that may have had deleterious effects on the product. Continual renewal of the plating chemistry required the procurement and storage of a large amount of chemicals plus associated labor costs. It became apparent that these process control techniques were costly—both monetarily and environmentally.

Control of the photoresist developing process was accomplished by adjusting the process time to compensate for the deterioration and dilution of the developer chemistry. This developing process chemically "washed" unexposed imaging photopolymer from a panel by using an alkaline rinse. As the developer chemistry became saturated with unexposed photopolymer, the pH of the solution began to decrease. This decrease caused a concomitant reduction in the effectiveness of the rinse solution. When PWBs were observed to have residues after processing, the processing speed was reduced until the residues disappeared. When the performance of the process was determined to be unacceptable, either an addition was made to rejuvenate the solution or the developer chemistry was discarded. Control of the photoresist stripping process was accomplished in the same manner.
Temperature control was a critical parameter in many of the steps involved in fabricating PWBs. Maintaining predetermined temperature parameters was accomplished with tap water circulation. All of the cooling water was discharged into the ISS. There were no recycling steps practiced. The etchers, developer, lamination press, and vapor degreasers required tap water cooling to control the temperature. During normal operations, approximately 8,000 gallons of noncontact cooling water was used weekly. (Water usage fluctuated depending on the temperature of the ground water.)

HAZARDOUS MATERIALS

Materials used in the wet fabrication processes are hazardous. The chemicals employed can be categorized into three groups: oxidizers, corrosives, and toxins.

1. Oxidizers are materials that readily oxidize substances with which they come into contact. An example is chlorine gas, which was used to regenerate an etching system. The etchant was chemically reduced as copper was removed from the PWB panel. Chlorine gas was used to reoxidize the etchant and restore etching ability.

2. Corrosive materials include acids, bases, and halogens. Each of these materials could cause corrosion on substances with which they come into contact. (An example of this is the effect that strong mineral acid vapors have on metals and respiratory systems.)

3. Toxins include toxic materials, irritants, carcinogens, and asphyxiants. These materials are known to have an adverse effect on humans. Most of the chemicals used in the fabrication of PWBs possessed one or more of these undesirable properties. Since there was a great concern for protecting the personnel exposed to these substances, it was extremely important that the PPE be properly used.

The solution employed to etch copper without destroying the tin/lead plated pattern was chrome trioxide and sulfuric acid. The etching solution was heated to 120°F in a conveyorized spray module and was used until spent. Waste products were then transferred into drums, labeled, manifested, and sent to waste disposal. (The main component, chrome trioxide, is a known carcinogen and exposure to this compound has been documented to cause lung cancer.)

The solvent used to remove dryfilm photoresist was methylene chloride, a suspected carcinogen. Removal was accomplished by placing a large solvent-resistant tray in an open sink and adding solvent. A number of panels were then placed in the solution and allowed to soak for several minutes. During this dwell time, the photoresist blistered and floated free of the panel surface. The panels were then withdrawn from the tray and held under running water to rinse the residual solvent into the Domestic Sewer System (DSS).

A liquid photoimagable resist was used to fabricate fine line circuitry. This resist was a xylene-based material formulated with photosensitive compounds. The resist was applied by pumping the compound onto a set of rollers which, in turn, was deposited onto the panels being processed. After each use, the equipment had to be cleaned by wiping the soiled areas with xylene. (Xylene is a chemical suspected of damaging bone marrow, thus causing anemia.)
A solution of ammonium *persulfate* and sulfuric acid was used as a microetchant to remove oxides and prepare the PWB panel surfaces prior to plating. This same solution was used to strip the electroless copper deposit from the processing baskets. (Exposure to the fugitive emissions from this solution caused severe respiratory irritation that resulted in a gasping cough and nose bleed.)

The organic compound 1,1,1-Trichloroethane was used as a cleaning solvent and developer in a drum vapor degreaser. It was very effective in removing rosin flux residue after tin/lead deposit reflow and was the only compound capable of developing an image on photosensitive aluminum. As with methylene chloride, this compound is an asphyxiant. Additionally, it has the potential to destroy the stratospheric ozone. (Destruction of the ozone layer is thought to increase the incidences of a number of health problems including skin cancer and cataracts.)

Sn 63 tin/lead solder was used in the HASL process, as well as to thermally stress quality control coupons. Prior to each operation of the leveler, it was necessary to scrape the solder dross from the surface of the molten solder. Dross was put into cans for future disposal. Periodically, the solder had to be replaced due to the build-up of various contaminants. Spent solder pot dumpings were also put into cans for future disposal. Disposal consisted of either sending the material to a landfill or to salvage. (Long-term exposure to lead solder can cause chronic lead poisoning; kidney, liver, and nervous system damage; and digestive disorders.)

**ERA OF AWARENESS**

**PERSONNEL**

Slowly and subtly a culture change occurred in this PWB shop. Interest developed in the materials being used and questions arose as to what effect these materials were having. This culture change redefined the way that personnel viewed health, safety, and responsibility.

**ENVIRONMENT**

This environmental awareness gave rise to questions and concerns about what was leaving the PWB shop and the subsequent effects on the environment. Direct observation illustrated that products from processing (e.g., acids, bases, metals, and organics) were being disposed into lined evaporative ponds. Bath dumps accounted for approximately 2400 gallons/year of chemistry that were being sent to waste disposal. From this PWB shop, hazardous waste was transported to the on-station Hazardous Materials Holding Site and then transported to San Diego where it was treated and the resultant sludge incinerated. Transportation and treatment of our waste carried a heavy price tag both financially and environmentally. 1,1,1-Trichloroethane, a volatile organic compound, emitted vapors during processing that were exhausted directly into the atmosphere. (As previously stated, this compound has the potential to destroy the stratospheric ozone.)

None of these processes were tailored around natural resource conservation. The waste of water was especially perplexing when considering that this facility is located in the
California desert and that California was experiencing a 7-year drought. A hard look was taken into operation practices.

ENVIRONMENTAL RESPONSIBILITY

PERSONNEL

The most important responsibility was to protect personnel from the hazardous materials. This was accomplished by education, substitution, and procedure development. Training sessions were held to familiarize personnel with the Material Safety Data Sheet (MSDS) format. Each MSDS was thoroughly covered in the training sessions and personnel learned the hazardous ingredients, health hazard data, first-aid procedures, special protection requirements, and special precautions regarding each material used in fabricating circuit boards. As the awareness of hazardous materials increased, it was arranged for a physician to observe the procedures in action. Afterward, the physician and personnel discussed industrial illness and health concerns.

All materials were closely examined to determine the viability of substitution. Information was obtained regarding necessary changes to the procedures, equipment modifications, spent material disposal, cost, etc. Standard Operating Procedures were developed and written for all the procedures and for the safe handling of hazardous materials. Through writing these procedures, personnel were made aware of previously unknown, unsafe practices, and allowed personnel to work more safely. Additionally, these procedures had the accompanying benefit of process improvement. Personnel took ownership for their own safety.

ENVIRONMENT

During the studies, it was discovered that the environment was being negatively impacted by the use of lined evaporation ponds, methods of waste disposal, use of ozone depleting chemicals, and waste of natural resources. It was apparent that the lined evaporation ponds that contained the chemical rinses were not environmentally sound. For this reason, the Naval Air Weapons Station (NAWS), China Lake, California, was ordered to "Cease and Desist" discharges into the ponds on or before 1 October 1991. Drastic changes had to be made to comply with the order.

Training courses were attended to investigate the different technologies available for wastewater treatment/reuse to minimize or eliminate chemical discharge into the ponds. This education covered the scope of treatment from metal surface finishing applications to circuit board manufacture. A quasi-benchmarking study was performed to look at what industry was doing with regard to wastewater treatment. Various facilities were contacted and systems evaluated for their feasibility in the PWB shop. In each case, inquiries were made into operational safety, system effectiveness, system efficiency, maintenance, and operational cost. Additionally, facility managers were asked to be candid and reveal any shortcomings of their systems and how they were handled. (This was crucial to minimize unexpected expenditures and problems after installation.) All information was recorded and categorized for evaluation.
During evaluation, it was discovered that not all of the material alternatives tried were acceptable. Additionally, some of the alternatives (i.e., metal precipitation) accomplished nothing more than changing the physical state of the hazardous materials and increasing the process steps. Ultimately, this technique was deemed unacceptable.

The next hurdle addressed was that of site modifications to accommodate the system components. Site modifications were defined as relocation of existing processes, existing plumbing, existing utilities, and any excavation to accommodate new plumbing and tanks. A site assessment was performed to identify the types of modifications needed to install the required equipment. To accomplish this task, a worse-case scenario was visualized in which the majority of the existing equipment and plumbing had to be relocated. A floor plan of the relocated equipment and ancillary plumbing was drawn and tentative locations for the system components with maximum allowable dimensions were identified. These dimensions included a reasonable amount of space around the perimeter of the components for maintenance and safety requirements. (Because of high cost, excavation for new storage tanks was not considered a viable alternative.) The gathered information was combined into a data package and the procurement phase was initiated.

Hazardous waste disposal was the next area addressed. As previously stated, bath dumps were drummed, labeled, manifested, and sent off-station for disposal. Costs of transportation and disposal of hazardous material were enormous. Also, there was the liability issue associated with the disposed hazardous materials to be considered since this facility was responsible for any damage done to the environment by the hazardous materials generated. This responsibility began at the time the material was generated and continued indefinitely.

A more efficient method to control the plating procedures, (and thus, the hazardous waste) was researched. Analytical methods were researched to provide a method to both identify and quantify all species in the plating bath. A capability of this type would drastically reduce the amount of hazardous waste requiring disposal. Obviously, a reduction in waste brought a concomitant reduction in operating cost and liability. A High Performance Ion Chromatograph (HPIC) was chosen because it would meet and exceed the analytical requirements.

Another area addressed was that of using ozone depleting chemicals (ODC). This facility used 1,1,1-Trichloroethane to remove rosin flux residues after fusion of tin/lead plated deposits and as a developer for photosensitive aluminum. This issue was easily resolved since there are several alternatives available to ODCs that were suited to this facility's needs. First, a rosin flux was replaced with a water-soluble flux. This eliminated the necessity of ODC solvent cleaning after fusing the tin/lead deposit. Next, the photosensitive aluminum, which required solvent-based developing, was replaced by another material that was developed with an aqueous-based chemistry. Only one liter of the new aqueous-based developer was used in a system that operated at room temperature. This is in contrast to 25 gallons of solvent that were necessary to develop the old material at 165°F.

Natural resource conservation was the next area addressed. With simple plumbing modifications to the rinse tanks, overflow rinses were eliminated and counter-current rinsing was incorporated. In counter-current rinsing, the water flows from one rinse tank to another against production flow. As the water cascades against the production flow, contaminants are
carried away from the critical process baths. Contaminant drag-in was also eliminated. Water reaching the end of the line was treated and discharged into the ISS.

Rather than directing the treated water to the sewer, it was possible to close the loop and recycle the water. Installation of the Wastewater Treatment System (WWTS) provided a very efficient conservation system (see Figure 1). Additionally, flow restrictors were installed in the rinse water supply lines. These devices limited the flow of water to only 2 gallons per minute as compared to 15 1/2 gallons per minute prior to installation.

Finally, the cooling water issue was addressed. Research was performed to identify a method that would reduce or even eliminate the excessive outflow and still maintain control over the process temperature. A water chiller with a recirculating pump was identified as the solution to the problem. Prior to the installation of the recirculating pump, this facility was discharging approximately 8,000 gallons of water per week into the lined evaporative ponds. The water chiller provided better temperature control and eliminated any discharge into the ponds.

FIGURE 1. Counter Current Closed-Loop Rinse System.

NOTE:
1. Closed loop for water conservation and process improvement.
2. Production travels left to right.
3. Rinse water flows right to left.
CURRENT OPERATION PRACTICES

WASTE WATER TREATMENT

Within the past few years, advances in ion exchange resins have opened the door to wastewater treatment. Ion exchange has been used extensively in water purification technology.

The WWTS that was installed at this facility consisted of ion exchange, carbon pack filtration, depth filtration, bag filtration, electrolytic recovery, and pH adjustment. Each component was installed at the source of discharge throughout the shop. This reduced the plumbing and auxiliary transfer pumps that would have been required had the installation been centralized. As the wastewater flows through the resin system, contaminant ions are captured and replaced with hydrogen (H+) and hydroxyl (OH-) ions. The hydrogen and hydroxyl ions then combine to form a molecule of water (see Figure 2).

During the ion exchange process, care must be taken that there are no oils or other types of organics in the water as these materials foul the exchange resins. Pretreatment is necessary.
to remove organic contaminants from the wastewater. This pretreatment is accomplished by carbon pack filtration. The carbon pack filters consists of a bag of activated carbon contained in a canister. As wastewater is pumped through the filter bag the organic materials are adsorbed.

The next treatment technology is depth filtration. This procedure removed suspended solids down to 5 microns. Depth filtration is necessary if treated water is to be reused. A build-up of suspended solids will interfere in the plating process, as well as clog the spray nozzles of other process equipment.

The last type of filtration incorporated into the WWTS is bag filtration. A bag filter is essentially a large sock. As the wastewater passes through the filter, particles are captured from the stream. The captured metal particles are then sold to offset some of the operational cost. The metals that are captured in this process are regulated and, therefore, justify the reclamation effort.

Upon ionic saturation of the ion exchange resin, the cannisters are physically transferred to a regeneration system for recovery of the metal ions. This is accomplished by washing the resins with a dilute acid solution that releases the metal ions from the resin. These metal ions are then pumped into an electrolytic recovery cell for reduction to solid metals. Recovered metals are reclaimed and sold (offsetting the operational cost of the PWB shop (see Figure 3)).

![FIGURE 3. Metals Reclamation System.](image)

Cannisters containing other ions (e.g., chlorides and sulfates) are similarly washed, but a dilute alkaline solution is used, which is later neutralized for environmentally safe discharge into DSS. The final step before discharge of the wastewater into the DSS is adjusting the pH. The shop's pH adjuster is a module consisting of three tanks with an attached control panel. Neutralization is accomplished by pH sensors, mixer, metering pump, alarm, and recorder (see Figure 4).
As the wastewater enters tank #1 of the module, the pH is measured. The controller then activates the metering pump which adds the pH adjusting chemical into the tank. The wastewater and pH adjusting solution are mixed together resulting in chemical neutralization. As more wastewater enters the tank for neutralization, the overflow runs into tank #2. This process continues and then overflows into tank #3. Located in the tank #3 is a pH probe that is connected to a recorder and alarms. The recorder maintains a hard-copy record of the pH at discharge into the DSS. Alarms will sound if the pH of the water does not meet the required value for discharge into the DSS.

The WWTS became fully operational on 1 August 1991, 2 months prior to the Cease and Desist order. All shop personnel have been trained in system operation and maintenance.

HAZARDOUS MATERIALS MINIMIZATION

A majority of the hazardous materials used have been substituted for materials that are less hazardous. This substitution directly affects the hazardous materials that are sent to disposal and also reduces the facility's operational costs. Some of the substitutions are:

1. Chrome trioxide/sulfuric acid copper etchant was substituted with an ammonia-based mixture. The replacement ammonia etchant removed the copper without effecting the tin/lead plated pattern. The etchant has no carcinogenic properties but does have an ammonia odor.
2. Methylene chloride photoresist stripper was replaced with a dilute solution of ethanolamine. A conveyorized photoresist stripping module was installed. Rather than standing over an open tray of methylene chloride, the process utilized an input conveyor in an enclosed system. Exposure occurred only during the replenishment of the chemistry and maintenance of the system. Hazards associated with this substitute were slight respiratory and skin irritation.

3. Xylene-based photoimagable resist was substituted with a high resolution dryfilm photoresist. The dryfilm provides the same capability, but the hazards are greatly reduced. Since the lamination equipment is well ventilated, the only hazard is skin irritation.

4. Ammonium persulfate/sulfuric acid microetchant was replaced with a hydrogen peroxide/sulfuric acid solution. This replacement eliminates the severe respiratory hazards and allows the microetch system residue to be recycled. The solution is regenerated by chilling and allowing the copper to precipitate as the sulfate. After decanting and analysis, proper chemical additions are made to return the solution to optimum condition.

RECYCLING

Rather than send the tin/lead solder pot dumpings and dross to salvage, this shop participates in an industry reclamation program. The reclamation program allows credit for scrap solder and dross, which offsets the shop's operational costs. The program sponsor provides the containers, manifests, labels, and pays for the cost of shipping to the refiner's facility. Refined materials are then sold at hardware stores for home-use soldering applications.

PROCESS CONTROL

Wet chemistry process control of all the bath chemistry was replaced with an HPIC. This instrument analyzes anions, cations, organics, and transition metals. The HPIC quantitatively identifies all chemical species contained in the baths. Data generated by this analysis are used in this facility's Statistical Process Control (SPC) program. In addition to the process bath analysis, HPIC is also used to analyze the contents of the wastewater before and after treatment. Quantities of hazardous waste sent to disposal are steadily declining while the quantity of material sent to reclamation is rising. The HPIC is a very powerful analytical tool that has made the dream of bath longevity into a reality.

Another control implemented was supplying each process line in the plating area with deionized water. Deionized water is used to generate new baths when necessary and to replace evaporation loss. This shop progressed from using four individual tap water lines to one line that feeds the deionizing system. Using deionized water dramatically improves the shop's plating processes.

Because bath temperatures are now controlled with recirculated chilled water, the shop's processes are more efficient and natural resources are being conserved. No longer are 8,000 gallons of water per week being discharged to the sewer, but rather the water is continually being recycled.
The photoresist developer and stripper have had pH monitors installed. Chemical additions are dependent on solution pH, rather than on product attributes exiting from the systems. Although pH probes require frequent maintenance (e.g., filling and calibration), the extra effort is worthwhile as defects from the processes have been significantly reduced.

**PROBLEMS ENCOUNTERED**

There were only two significant problems encountered: etcher rinse water and scrubbing/deburring treatment.

1. **Etcher rinse water.** Analysis during the WWTS identification phase detected only traces of copper in the etcher rinse. For this reason, the rinse water was only pH adjusted and then discharged. Upon start-up of the system, the pH adjuster water became turbid. An analysis revealed that the turbidity was caused by a build up of precipitated copper hydroxide. The metal hydroxide was formed during the neutralization reaction of the wastewater. To eliminate this problem, the first-stage rinse water was passed through an ion exchanger to capture the copper ions prior to pH adjustment.

2. **Scrubbing/deburring treatment.** After two days of operation, the bag filter system would become filled with fiber from the scrubber wheel. Since this is a closed-loop system, the product from this process was coated with fiber. Contaminated panels are unfit for further processing. The solution to this problem was to increase the filtration capacity. Until a new filter manifold could be procured, a secondary bag filter was installed. This extended the time that the product from the process was acceptable to one week. To reduce unscheduled downtime, cleaning the bag filters was placed on the "Weekly Maintenance Schedule."

**FUTURE PLANS**

Future plans for the Wastewater Treatment System is to continuously improve the system one small step at a time. Some examples of these plans are

1. Install solenoid actuated valves for inlet rinses. This improvement will allow the rinses to be run on certain tanks only when the process timers are operational.

2. Install pH controllers for the photoresist developer and stripper. This will fully automate the addition of replenishment chemistry.

3. Install a closed-loop system for the treated rinse on the developer and stripper. This will allow the reuse of water in the process.

The Environmental Project Office and this facility have been discussing controlling the shop's air emissions. The near-future goal is to accomplish similar achievements for air emissions that have been accomplished for wastewater.
CONCLUSION

Presently, less hazardous materials are being used in a more environmentally conscious manner. The discharge of untreated wastewater has been eliminated and replaced with recycling of treated water. Archaic attempts at process control have given way to state of the art analytical methods. Waste of water for process cooling has been replaced with recirculated chilled water. Waste of process metals has been replaced with metals reclamation. Large quantities of hazardous waste for disposal has been drastically reduced. Attitudes of "that's the way we've always done it" have been replaced with attitudes of "how can we do it better." The PWB shop has come a long way, but there is always room for improvement.