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Improving the Quality of Black Oxide on Flexible Circuitry by Optimization of Filtration and Agitation Systems By C.R. Reichert , C.E.F., Regional Manager, SERFILCO, Ltd.

Black oxide on printed circuit boards is used throughout the industry as a means of maximizing the bond between copper clad laminates in multilayer boards.

SERFILCU

Although it is hard to conceive, the black oxide process itself may be a source of contamination which can cause short circuits in printed circuit boards. Today we report the case of a large manufacturer, Zycon of Santa Clara, California. Zycon produces approximately 20,000 flexible boards per month and applies black oxide to same. The black oxide used is a conventional sodium chlorite/sodium hydroxide solution which operates at 160°F (71°C).

This solution is known to be highly corrosive and a great generator of particulate in the solution. As the black oxide forms, some of it sloughs off of the board, especially in flexible circuits and creates particulate which is larger in diameter than the separation between lines in the board.

The original set-up of the black oxide line consisted of a programmed hoist machine manufactured by Chemcut. This particular unit has approximately a 400 gal. (1514 l) black oxide tank. A 1.0 HP pump powered the filtration system. This pump would turn the tank over approximately 4 times per hour. Five micron filtration was required to remove all particulate large enough to cause rejects on the board. The filter chamber used with this pump was fabricated from titanium and held five, 30" long polypropylene string wound, five micron cartridges. This chamber provided approximately 52.5 square feet (4.88 m²) of depth filtration surface area.

A sales representative working in conjunction with the personnel from Zycon analyzed the situation and found two distinct problems with the filtration system. The first was that particulate was not being removed from the solution fast enough to prevent adhesion in areas of the boards where shorts could form. The other was that the amount of particulate being generated would quickly load the filter elements, significantly reducing the flow through the filtration system and causing solids build-up in the solution beyond acceptable levels in a very short period of time.

Observation of the process confirmed some suspicions. First of all, if the filtration system was running prior to startup of the line, the solution was perfectly clear and water white so that the bottom of the tank could be seen. Once the first fixture of boards was immersed in the tank, the solution would instantly become turbid and dark. Upon removal of the rack, the solution would gradually begin to clear. The original setup relied on a trial and error method which determined that rejects decreased with successively denser filtration media until at five micron the minimum reject rate was attained. However, when this old set-up was operating, Zycon still experienced an average of about 100 rejects per month on the flexible boards. These boards are $15" \times 20"(381 \text{mm x} 508 \text{mm})$. The scrap rate obviously was at an unacceptable level with a reject rate of 0.5%.

It seemed that a significant portion of the rejects occurred during filter change. Still, a certain portion were random and not attributable to any specific cause. Our conclusion is that we must both maximize the interval between filter changes to eliminate spikes of rejects associated with filter changes and also increase the turnover rate to eliminate random rejects by removing a greater proportion of the particulate generated as quickly as possible.

THE FILTRATION SYSTEM

A new filtration system was proposed. Zycon agreed that new equipment was necessary to resolve the problem, so

they placed the order. When the new equipment was installed, it consisted of a double mechanical seal end suction centrifugal pump of polypropylene with EPDM elastomers and sili-

> con carbide seal faces. The filter chambers are all polypropylene with stainless steel external hardware. The sys-

tem is plumbed with polypropylene pipe and mounted on an

FRP base. The two filter chambers are plumbed in series with 50 and 5 micron filter cartridges in successive chambers.

The pump turns the tank 12 times per hour. The larger pre-filter/post filter approach increases dirt holding capacity 15 times.

Now let's spend a few minutes examining those factors which impact solution clarity and quality of the finished part.

DESIGN CONSIDERATIONS FOR THE FILTER SYSTEM

We will now investigate the design considerations which lead to well engineered filtration systems. We can choose surface media, such as a paper on membrane filter or a depth media such as a wound cartridge. Each has its place. Surface media does well at removal of very fine particulate down to 0.1 micron where dirt loads are very low. Typical applications might include beverage packaging, fuel dispensing and final filtration of chemicals prior to use or packaging. Let's took at a depth wound cartridge to see how it does what it does. The passages of a depth cartridge are wide at the exterior and narrow through the windings to the pore size equal to the micron retention rating.



Typical pores of a depth wound cartridge

Figure 1

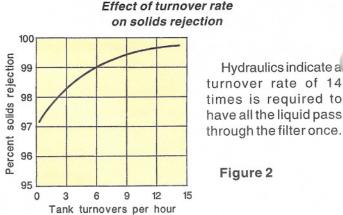
Not all of the contamination in the tank is the same size. The frogs, birds and large fish are trapped in the outer pores. Since these large particles are unlikely to blind off the entire pore, smaller particles stick further in. The minnows and polliwogs are then trapped in the inner windings.

Upon analyzing the particle sizes in the Zycon black oxide tank, it was apparent that the particulate ranged from 1 to 100 micron. They also knew from their earlier experimentation that five micron was the most dense filtration needed to provide a clean solution. We decided to use a pre-filter to remove the large particulate and extend the lifetime of the five micron cartridges. Arbitrarily, we chose 50 micron as the pre-filter density, and were very pleased with the results. A five micron, 10" long cartridge when used efficiently has a capacity of approximately 1¼ ounces (50g) of dirt. A 50 micron cartridge at the same flow rate will have a 12 oz. (340 g) dirt holding capacity per 10" length of cartridge.

On a 10 micron cartridge, the outer pore may measure 1/8" x 1/16" (3mm x 1.5mm). A particle of 100 micron will lodge in the inner pores and perhaps trap several 20 to 30 micron particles. As the pores fill, smaller and smaller particles are trapped. Statistically with one 10 micron cartridge, 80 to 90% of the 10 micron particles are trapped in the first pass. Also, since the pores fill gradually, a significant number, say 30 to 50 percent, of the particles of less than 10 micron will be trapped.

In the original set-up, one would normally assume that 1 to 4 turnovers per hour in a process like black oxide would be more than adequate to maintain good quality. However, our study shows that 12 turnovers per hour are needed to maintain the quality of the boards. This confirms the results of a nearly 20 year old study on hydraulics illustrated in Figure 2. The percent of solids removed is related to tank turnovers per hour and, as we see, at about 14 turnovers per hour we have statistically filtered 100% of the solution.

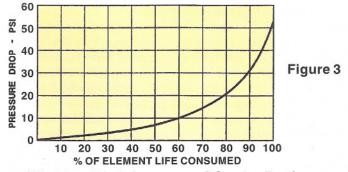
Thus, successive passes serve to further reduce the particulate. If a 10 micron cartridge removes 40% of 5 micron particles on each pass, the second turn leaves 36%, the third turn 22%, fourth turn 13% and by the fifth turn more than 92% of 5 micron particles will be removed! What has this taught us? High turnover rates remove sufficient amounts of particulate and will yield good quality plating. This is all accomplished with media that is economical and long lasting. Figure 2 shows the significance of turnover rate in particle removal in hydraulic fluid filtration. Statistically, in this study, 14 turnovers assure 100% of the fluid has passed through the filter.



The next issue in design is pressure drop. Any time you pass a fluid through a filter, a pressure drop occurs that is analogous to the potential difference across a resistor as you pass current. We want the lowest initial or 'clean' pressure drop possible as every bit of initial pressure drop is depleting the life of the media. As Figure 3 shows, a 5 PSI 'Clean' drop will translate to using 40% of the life of the element. The cutoff pressure drop is 25 PSI, so we are able to use 85% of the design life of the cartridge if we have done our design work correctly.

We must also consider the sizing of the filter into the equation. The less time spent changing media, the better. Also, the lower the velocity of liquid through the filter, the more we increase dirt holding capacity. If the flow through a 20 micron filter is cut in half from say, 1 gal (3.8 l)/minute to 0.5 gal (1.9 l)/minute, the dirt holding capacity increases by 40%.

Element life as a function of applied pressure drop



What does this do for economy? Quadrupling the amount of media doubles dirt holding capacity of each 10" (25.4 cm) cartridge. However, now the time between cartridge changes will be 8 times longer. This will cut annual consumption of cartridges by 50%. Labor cost also takes a significant drop! Figure 4 illustrates these points:

Economics of filter chamber oversizing

OVER- SIZING FACTOR	NUMBER OF CARTRIDGES IN CHAMBER *	DIRT HOLDING FACTORS PER CARTRIDGE	TIME BETWEEN CARTRIDGE CHANGE	CARTRIDGE CONSUMPTION/ CARTRIDGE COST REDUCED BY:	LABOR COST, DOWNTIME & SOLUTION LOSS REDUCED BY:
1	С	D	т	0	0
2	2xC	1.4D	3T	29%	67%
3	3xC	1.7D	5T	42%	80%
4	4 x C	2D	8T	50%	87.1%

* Based on average sizing [i.e. 1x10" (25 cm) cartridge per 50 gallons (200I)]

The original filtration unit used 15 - 10" cartridges. The lew unit uses 21 - 50 micron cartridges in the pre-filter and 21 - 5 micron cartridges in the fine filter. Dirt holding capacity of the original filtration system is approximately 1.17 pounds (.53 kg). The new system has a dirt holding capacity of 17.4 pounds (7.9 kg). Space consideration kept us from strictly following the guidelines of our sizing theory. Practical constraints such as available chamber sizes and, in this particular case, a very special need to maximize the turnover rate did not allow us to apply all of the optimization we could have in this particular matter.

To summarize:

- Filters should have as much surface area as you can practically afford. The long term savings are immense.
- Use sufficient turnover on critical baths to keep particulate at a steady state low level.
- Utilize pre-filters when dirt loads are excessively high and particle sizes are well distributed over a wide range.
- Choose the proper media, both material of construction and micron retention. Depth cartridges are forgiving in that at even relatively high micron retention levels, smaller particles are removed in large quantities.

AGITATION AS A QUALITY TOOL

Air agitation provides the vigorous agitation needed, but has some major drawbacks. The use of plant air is out of he question, as compressors will dispense aerosols of oil not the tank. The invention of the regenerative blower eliminates this concern but others remain.

One serious drawback is that whatever is in the shop air will be sparged through the tank. Granted, most blowers have a filter, but it is less effective than your typical furnace filter, considering its cross section area versus the CFM pulled through it.

Another concern is the noise from the blower itself. Although accessory mufflers are available, the noise level with the muffler may or may not be in an acceptable DBA range. Without the muffler, these units are almost always out of compliance.

Another limitation to regenerative blowers is that they will only deliver a specific volume of air at a certain pressure. If a special job requires less agitation, we cannot throttle back the discharge of the blower without damaging the blower. The excess air volume must be vented, creating more noise.

In the case of black oxide solution, the problem is further complicated by the fact that a solution with a potentially noxious fume will give off even larger volumes of the fume, requiring a much larger ventilation capacity to remove it.

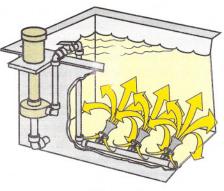


Figure 5

Tank Mixing Eductors have provided a new solution to these problems. The TME is a specially designed thermoplastic device which, when placed in a tank at the discharge of a pump or filter system, will deliver 5 GPM (19.4 I/min) of flow for every 1 GPM (3.8 I/min) fed to it.

Furthermore, a 400 gallon (1514 l) black oxide tank operating at 160°F (71°C), when properly air agitated, could conceivably lose 30,000 - 70,000 BTU's per hour. Obviously, that amount of heat would need to be made up by the heating system. Also, in this particular case, since it is not a tremendous amount of agitation that is needed, the existing pump / filter set-up will more than adequately provide the gentle agitation that the flexible boards need.

This brings up another issue with air. The only way to assure adequate mixing of a solution by use of air agitation is to vigorously agitate the solution with excessive air. This would be unacceptable with the black oxide process on flexible boards in that the boards would move too much in their fixtures and could conceivably touch each other, destroying the finish. Therefore, what we need in this particular case is the very slow, steady agitation of the solution provided by the eductors. In the case of the 400 gallon (1514 I) black oxide tank, we use six eductors with three flowing horizontally at the mid-depth of the tank and three at the bottom of the tank angled up at a 45° angle to provide a swirling agitation. These two sets of eductors provided the smooth controlled flow of solution which was necessary for proper quality.

The eductor agitation system assists a filter system by sweeping the horizontal surfaces of the tank and anything in the tank so that no particulate matter accumulates to cause further problems later. If all particles in the tank are in continuous motion they cannot reside on the bottom of the tank, on heating coils or on any other permanent fixtures in the tank.

The instantaneous replenishment of bath chemistry at the part provides much faster formation of the black oxide and also prevents loose, non-adherent deposits which may flake off during the lifting of the parts from the tank. Along with this benefit, the fact that temperature stratification of the solution is virtually eliminated, again promotes the even formation of the deposit. Eventually this elimination of thermal stratification can allow experimentation with operating temperature and a resultant lowering of the operating temperature would obviously save energy and further reduce operating costs. In processes other than black oxide, it has been our experience that eductor agitation prevents oxidation of organic additives and metallics in the solution along with the prevention of gas bubbles which can cause foaming of heavily wetted baths. Since eductor agitation is airless, air bubbles are not ingested in the pump suction and homogenized by the pump. This can cause skips and pits on the surface of the part.

Let us summarize the results of this study:

- Reduction of scrap rate on 20,000 flexible boards from an average of 100 per month down to an average of 2.25 per month. This is a reduction from a scrap rate of 0.5% to a scrap rate of 0.011%. This translates to a reduction in the scrap rate of 97.75%.
- Reduced filter changes from 1 to 2 times daily to approximately once every four days on the five micron cartridges and once every ten days on the 50 micron cartridges.
- Reduction of total annual cartridge usage from 5,475 per year on the old system to 2,685 cartridges per year on the new system. That is a savings of \$6,000 per year in cartridge cost.

Methods of accomplishing cost savings and quality improvement:

- Since particle formation in the bath is rapid, high turnover rates are necessary to prevent deposition of particulate on the boards.
- Large surface area filters provide sufficient capacity to minimize downtime of filter change-out.
- Utilization of pre-filter to remove coarse particles extends the overall life of the final filter media and makes the filtration system more efficient.
- Use of string wound depth filtration cartridges provides optimum dirt holding capacity.

Lastly, eductive tank mixers prevent settling of particulate, stratification of solution, and inconsistent deposition of oxide.

ECONOMIC JUSTIFICATION

The rejects experienced during the operation of the old filtration system amounted to \$100,000 to \$150,000 in product loss per year. The new system saved about \$6,000 in media cost over and above the product loss savings. Therefore, at the minimum, since the filtration system came in at a cost of \$7,700.00, the unit paid for itself in less than one month.

FUTURE IMPROVEMENTS TO THE SYSTEM

In the implementation stages right now, the customer has opted to purchase an additional filter chamber and process piping so that the fine filtration at five micron will be nearly continuous. It takes a certain finite amount of time to change filter cartridges and, considering that the five micron cartridges are changed on a 3-4 day cycle, the new system will seldom be off-line. In order to change cartridges, the user switches one chamber to by-pass and does not have to shut down the unit. The only time the filter system will need to shut down is on a ten day interval to change the 50 micron cartridges. Also, it will be possible to run the system with both five micron chambers in operation together in parallel, which should bring the change-out interval for the five micron cartridges to a ten day interval as with the 50 micron cartridges.

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Process Engineering Supervisor of the black oxide line who provided size and application data allowing us to readily solve these problems.

Biography

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