Integration of Chemistry with Equipment Improves Process and Environment

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Utilizing innovatively designed equipment with specific plating processes improves the quality and consistency of the plated part while reducing the processes impact upon the environment. Plating processes varying from cleaners, electroless and electrolytic nickel, and hexavalent and trivalent chromium are benefiting from this integration of equipment with chemistry. The plater and consumer both benefit through process consistency and reduced costs and wastes.

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Traditionally, platers considered equipment only as tools to make their operation more efficient. They could automate their line, filter and carbon treat their solutions, increase the plating speed and improve thickness distribution through auxiliary anodes, use pumpable liquid cleaners, etc. all to improve the utilization of their process. Any process and equipment would be used together. Recently, a trend has developed to combine innovatively designed equipment with a specific plating process, each uniquely depending upon the other for operation. These systems are designed to improve the plating operation while minimizing the impact upon the environment. Several examples of systems are presented.

High-speed Chromium Plating with Uniform Deposit Distribution

Functional hexavalent chromium deposits are essential in our modern industrial world because they increase service life through their unique good corrosion resistance, high hardness, anti-galling surface, and low co-efficient of friction. Compared to alternative deposits, hexavalent chromium deposits offer these benefits at a relatively low price. However, there are disadvantages such as a low plating rate, expensive pre- and post-plating preparations, and HES concerns. Hexavalent chromium plating is highly regulated because hexavalent chromium ions are a strong oxidizer and carcinogenic.

Figure 1 graphs the current density range and resulting plating rate of hexavalent chromium electroplating processes used to produce functional hexavalent chromium deposits. The plating rate for the conventional process is limited because of the narrow current density range that produces deposits with the required physical properties. Organic catalyzed processes plate faster than conventional processes because of their higher plating efficiency and slightly wider current density range. Both processes have a poor thickness distribution over a part's current density range compared to nickel and copper electroplating processes. The high current density areas of the part have much thicker deposits than the low current density areas. Because of this phenomena, shielding, special racking, and masking are usually required. Even with these procedures, the part typically must be machined to size after plating to obtain the required thickness and physical properties.

Integrating specialized equipment with a customized hexavalent chromium processes provides high volume parts such as shock absorber and strut rods, an environmental friendly, high production process, with minimal post- plating mechanical operations. A chemistry and equipment system approach makes it possible to build a specialized high speed plating line that can be integrated inline with other equipment required to produce rods. The environmentally enclosed 9-meter long unit, Figure 2, can be placed directly in the production area next to the production people because of its minimal HES impact.

Rods can be automatically loaded, plated and unloaded at the rate of 1000 rods per hour using approximately 50 % of the floor space of a conventional plating line. Rods that are 150 to 640 mm in length with 10 to 25 mm diameters are automatically processed without operator intervention. In approximately 6.5 minutes of plating, they receive 20 microns of hard chromium at 160 amps per square decimeter and 70 ^{0}C, Figure 1. Using integrated shielding, the rods receive a uniform deposit that requires no post grinding to size, only the normal super finishing. The system generates almost no wastewater because of a built in rinse water evaporator. It also has its own air scrubber designed to meet all government regulations. Because of the evaporator, scrubber, chromium recycling, and no post grinding, less chromium is consumed and with reduced environmental impact.

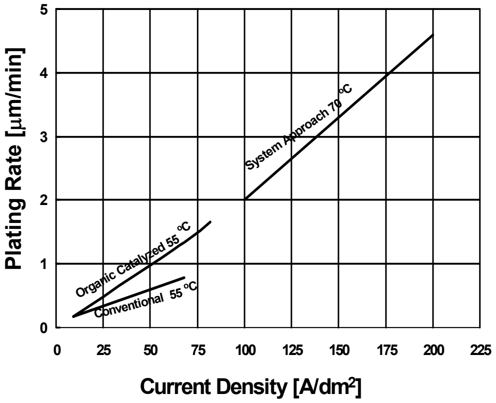

Figure 1, Plating rate of various hexavalent chromium processes

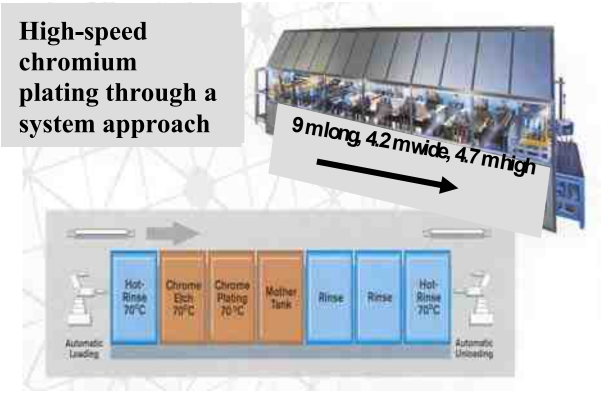

Figure 2, System approach for a high-speed hexavalent chromium plating process

Continuous Removal of Metallic Impurities From Trivalent Chromium Electroplating Process

Historically, the color of trivalent chromium electrodeposits has been darker than the standard for chromium deposits, the blue-white color of hexavalent chromium electrodeposits. Metallic contaminates such as iron, copper, nickel, and zinc can darken trivalent chromium deposits. However, in contrast to hexavalent chromium processes, these metallic impurities can easily be removed with a systems approach using ion exchange and a specific trivalent chromium chemistry. This ion exchange system can efficiently remove these metals even while electroplating is in operation. The specially designed resin is regenerated and reused for many years without replacement. A dilute solution of ammonium hydroxide is used to remove the copper from the resin. Dilute hydrochloric acid removes the other metallic impurities from the resin. Automatic equipment or lower cost manual equipment similar to ion exchange water purification units, Figure 3, are readily available and easily maintained. With ion exchange to remove metallic impurities and carbon to remove normal organic contaminates, trivalent chromium electroplating solutions can last “indefinitely” even while returning dragged out solution to the tank. Typically, hexavalent chromium plating solutions are periodically dumped due to build up of contaminants.

Prior to the availability of ion exchange, trivalent chromium electroplating solutions had to be purified either by chemical precipitators or dummying. These methods do remove the common metallic impurities but they have operational problems. In most cases, the chemical purifiers remove all of the common metallic impurities but the resulting precipitate must then be filtered out or it could cling to the part and leave an objectionable film on the part. This hard to filter precipitate must then be safely disposed of. If improperly or over used, chemical purifiers might permanently damage the trivalent chromium plating electrolyte. This purification procedure must be conducted while the bath is not in operation resulting in lost production. Also, since this is a messy and time consuming operation, some platers tend to let the deposits get darker due to the build up of metallic impurities until they can no longer accept the color.

Another old method to remove metallic impurities is to low current dummy them out on surfaces such as nickel plated corrugated steel sheets. This operation is slow and unless a separate electro-purification tank is used, it cannot be conducted while chromium electroplating. It also wastefully consumes expensive additives, electricity, and some chromium.

Figure 3, Typical design for an ion exchange and filter system used with trivalent chromium

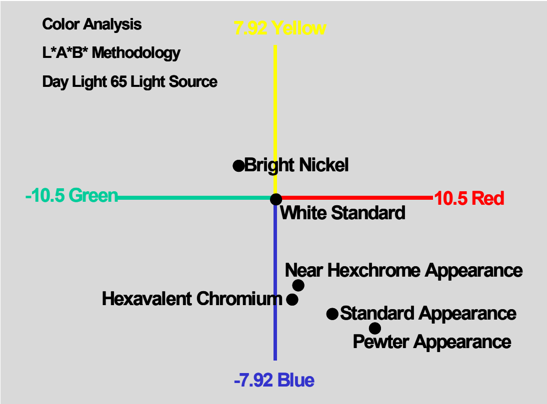

Figure 4, Color comparison between hexavalent and trivalent chromium deposits

With the advent of the ion exchange system, it is possible to consistently produce different colored deposits from trivalent chromium electroplating processes. Figure 4 graphs the color analysis of some commercially available trivalent chromium deposits. A standard white is used as the reference. The deposit listed as “near hexchrome appearance” is being intermixed with hexavalent chromium deposits on the same commercial part with very little difference in appearance. The “standard appearance” deposit is very pleasing to the eye making it very acceptable for most parts. The “pewter appearance” deposit has a darker appearance that appears to have a pleasing depth to the metallic deposit.

Continuous Purification of Acid Copper without Carbon

The physical properties of electrodeposited copper are very important to the overall performance of the part. Copper deposited from organic contaminated acid copper electroplating solutions might not have sufficient ductility to offer optimum thermal expansion. The deposit might also have pitting, reduced brightness, loss of leveling, poor metal distribution, and even jeopardize adhesion to the subsequent nickel deposit. Under normal operating conditions, the deposit’s quality can start to deteriorate after about 700 to 1000 amp-hours of plating per liter of operating solution. Typically, a batch treatment with 6 to 10 g/l activated carbon is then required. Peroxide or permanganate might also be used if highly contaminated. These purification procedures usually work but they require that the solution be taken out of production for about a day. They also generate a significant amount of copper and carbon waste. There might also be startup problems due to balancing the additives again, anode conditioning, and roughness.

A system purification procedure combining chemistry and equipment is now available for some acid copper processes that eliminate the need for batch carbon treatments. This system approach uses ultraviolet (UV) light and hydrogen peroxide (H_2O_2). The use of UV light for organic purification is an established procedure for water, organic waste and nickel plating solutions

Continuous purification with UV/H_2O_2 can be accomplished by a bleed-and-feed operation. As an example, Figure 5, a 6000-liter acid copper tank can be purified by bleeding out 3000-liters of operating solution while an equivalent amount of purified, ready to use, solution is added to the tank. This can be done as needed during production. When the treatment tank is full, the treatment cycle begins. This sequence is repeated based upon the buildup rate of organic contaminates in order to maintain an almost pure acid copper plating solution.

Experience with other UV purification operations demonstrates that the lamp will last between 125 and 150 acid copper treatments while consuming approximately 0.08 KWhr per liter of treated solution. Approximately 4 ml of hydrogen peroxide per liter of copper solution treated is added to the UV chamber each treatment cycle. A comparison between UV/H_2O_2 and carbon purification, Table 1, shows that the UV/H_2O_2 system has the advantage of having a higher efficiency with a lower loss of plating solution and no loss of production time.

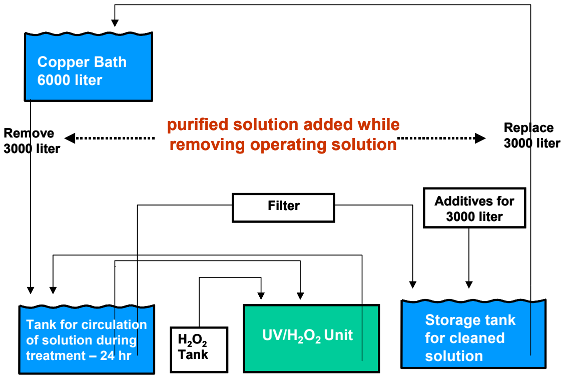

Figure 5, Ultraviolet light/hydrogen peroxide purification of acid copper electroplating solution

Table 1, Comparison of carbon vs. UV/H_2O_2 *treatments of acid copper plating solution*

	Carbon	**UV/H_2O_2**
Removal of organic impurities	40-60 %	90^+ %
Loss of plating solution	5 – 15 %	nearly 0 %
Sludge generated	yes	no
Roughness at start-up	possible	no
Lost production	yes 1^+ days	no
Anode conditioning	possible	no

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Continuous Purification of Nickel Electroplating Solutions

With the increased demand for higher performance from nickel electroplating systems at a lower cost, plating solution purity becomes even more important. The purity of nickel electroplating solution determines the physical properties of the deposit such as ductility, leveling, appearance, internal stress, chromability, and corrosion rate (sulfur concentration in deposit and STEP). These have a direct relationship to the performance of a part in service.

For years batch carbon treatments have been performed at scheduled intervals, which might have been before or after it was actually needed. Others have waited until one or more of the physical properties were bad enough to either be out of specification or cause performance problems. If the solution is very contaminated, hydrogen peroxide or even permanganate is used during the batch carbon treatment to increase the removal of the impurities. Batch carbon treatments are expensive due to lost production, labor, and the replacement of lost additives and nickel. Also, the cost of disposing of the nickel and carbon containing sludge that is generated during the treatment is increasingly more expensive because it is classified as a hazardous waste. For these reasons, many platers do not treat their nickel as often as required. This jeopardizes the performance of their parts in service. In some cases, such as platers for the automotive industry, poor performing or out of specification parts could result in the termination of their contract or loss of future ones. Even if they do not lose the contract, many times they have to pay to replace the failed part in service. This could include the labor, which is usually much more expensive than the part itself. In many cases, treating plating solutions is less expensive than not.

A very successful system has been developed to completely eliminate batch carbon treatments for the routine purification of nickel electroplating solutions. It is used on rack and barrel insulations. It can be used on semi-bright, bright, and particle nickel solutions. The system utilizes a special adsorber polymer to remove organic impurities and break-down-products without interrupting the plating operation. One 8 hour treatment a week of less than 10 % of the bath is usually sufficient to maintain nickel electroplating solutions in an “almost new” operating condition. With this system, there is no longer a problem with fluctuating critical physical properties of the nickel deposits caused by varying levels of organic contaminates. For example, a deposit ductility of 100 % for semi-bright nickels and greater than 11 % for bright nickels can be consistently maintained. After each adsorption purification cycle, the polymer is regenerated. The polymer has a service life of at least five years based upon actual plant experience

The polymer is designed to favor the removal of break down products and other organic impurities found in nickel electroplating processes. In particular, it removes the break down products from PPS type baths that some platers find hard to remove by batch carbon treatments. Table 2 lists the removal preference for carbon and adsorber polymer treatments. Since the adsorber polymer has a stronger affinity for break down products, it removes less active ingredients than carbon. With the adsorber polymer system, very little of the additives are lost if the level of Carrier in the bath is monitored when establishing the treatment sequence for the adsorber polymer.

HPLC can be used to monitor the break down products and Carrier levels. Figure 6 shows HPLC curves for a bright nickel process being purified by the adsorber polymer. Curve A is for the solution prior to treatment. Curve B is for the nickel solution as it exits the adsorber polymer column. The break down products, labeled X in Figure 6, are completely removed. Since a completely pure solution is not necessary, the bath was purified until the physical properties were well within automotive specifications. Only 5 percent of the bath needed to be purified, curve C. The same procedure is followed for semi-bright and particle nickels. Since the break down products can be continuously removed, platers can return as much dragged out nickel solution as they can capture without the danger of increasing the level of organic contamination. This is becoming more important as the cost of nickel increases. Inert anodes are available to eliminate nickel build up if the amount of nickel returned becomes very high.

Table 2, Removal preference for organics from nickel electroplating solutions

Carbon Treatment	**Adsorber Polymer**
Wetting Agents	Break Down Products
Carrier	Carrier
Bread Down Products	Wetting Agents
Brightener	Brightener

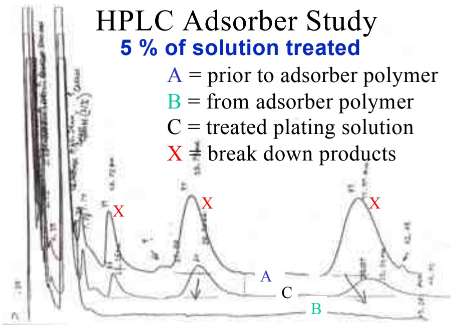

Figure 6, Using HPLC to monitor organic removal by adsorber polymer treatment

Figure 7 shows the design of a typical adsorber polymer unit. Nickel solution is automatically pumped directly from the plating bath through the column and back into the plating tank. After a set time, which varies depending upon plant conditions, the column is automatically regenerated and made ready to start again with a push of a button. To demonstrate the adsorber polymer's ability to purify a semi-bright nickel solution, a "very dirty" one was treated by five adsorption cycles, Table 3. Within two cycles, the percentage of sulfur in the nickel deposit was within specification. Within three cycles, the ductility of the nickel deposit met automotive specifications.

Figure 7, Design of an Adsorber Polymer Unit with its three regeneration tanks

Table 3, Adsorber Polymer Purification of a "Very Dirty" semi-bright nickel and automotive specifications

		Cycles of Adsorber Polymer				
	At Start	1	2	3	4	5
Ductility	<0.01	0.1	0.3	0.4	0.5	0.5
% Sulfur	0.008	0.007	0.003	0.003	0.003	0.003

Automotive specifications for semi-bright nickel deposits

Physical Properties	**Automotive Specification**
Ductility	≥ 0.4 (67%)
% Sulfur	≤ 0.005 %

"Never Dump" Soak Cleaner Using Micro-organisms

Conventional high performance aqueous soak cleaners simply displace and emulsify oils and other complex organics from the surface of the part. The cleaner becomes increasingly contaminated, cleaning effectiveness suffers, and eventually saturation can occur. This results in frequent dumping, costly replacement, down time, and the disposal of the cleaner sludge. Even worse is that the cleaning action potentially varies from over cleaning when new to inadequate cleaning prior to dumping. This is especially true if the type and amount of soils are not consistent. Since a properly cleaned part is essential for adhesion and appearance, this variation could lead to rejects.

Combining bioremediation technology and high emulsifying surfactants with equipment has produced an economical, steady state, never-dump soak cleaner. This system, Figure 8, removes and consumes a wide range of oils and soils by converting them through enzyme action into carbon dioxide and water. Depending upon the oils present, environmentally safe short chain fatty acids and alcohols may also be produced. The bacteria that consume the soils are naturally occurring and can be found in sewage treatment sledges and dairy and brewing plants. They multiply in the cleaner indirect proportion to the amount of their food supply, the emulsified oil. The surfactant/cleaner system emulsifies the oils and then the bacterium consumes the oils from the oil/water emulsion.

The equipment provides and maintains the ideal environmental conditions for the naturally introduced microorganisms to thrive. Using liquid additives, this soak cleaner is continuously rejuvenated and replenished to optimum condition automatically by the system's controls, Figure 8. This permits greater efficiency and lower cost than traditional soak cleaners. The low temperature (40 – 55 ^{o}C), mildly alkaline (pH 8.8 – 9.4) cleaner is compatible with most base metals. Built in filters, Figure 8, remove the dead microorganisms and other solids from the cleaner as a low volume, safely disposable sludge.

Figure 8, Side and top view of a four filter unit that is used to regenerate the never dump soak cleaner system using microorganisms

Pretreatment System Prior to Powder Coating

A new pretreatment system prior to powder coating has been devolved. The problems associated with conventional iron phosphate, the formation of scale and sludge, high operating cost temperature and high volumes of sludge can be eliminated. Traditional pretreatment for powder coating consists of iron phosphate. This method generates sludge and scale as a by-product of the phosphate reaction. This sludge creates many maintenance issues such as: clogging spray nozzles, scaling of the insides of the washer and blocking pipes and pumps. This new system does not cause sludge, which in turn can save maintenance issues associated with the sludge.

The system operates at low temperatures (43-52 ^{o}C), which is 10 – 15 degrees lower than conventional iron phosphate systems. With the lower operating cost, users have reported energy saving greater than the total chemical costs for operating the system. It also provides a more comfortable work environment by reducing the hazards associated with working around hot process solutions.

This system, Figure 9, utilizes a highly emulsifying cleaner combined with bioremediation, which eliminates the need for regular dumping of the process solutions due to build up of the oils and soils. Bioremediation is a natural process that we live with every day. Naturally occurring microorganisms have the ability to convert organic molecules, such as oil and other complex organics, into less complex innocuous substances such as carbon dioxide and water. The system contains neither hard chelators nor alkyl phenol surfactants.

The system provides all of the above maintenance and coast advantages while providing performance equal to or superior to conventional iron phosphate, Figure 10.

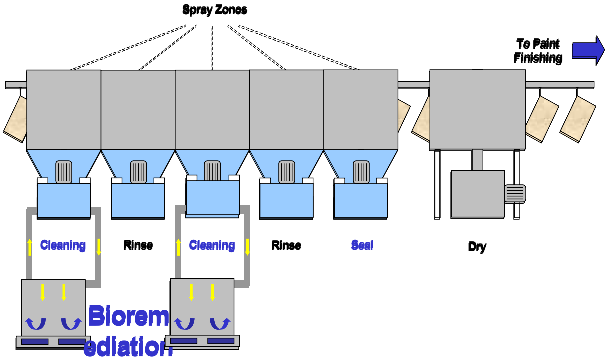

Figure 9, Five-stage washer layout

Figure 10, Powered coated panels scribed and NSS tested for 168 hours
- Left panel new system pretreatment
- Right panel conventional phosphate

Typical Process Cycle- Three Stage Washer

1. Cleaner – 2 to 3% by volume, 40 – 55 ^{o}C, 1-2 minutes, 14-20 psi
2. Rinse
3. Seal – 2-4% by volume, ambient to 49 ^{o}C, 30-60 seconds

Mechanical Plating Systems

Mechanical plating, also referred to as peen plating, is an effective means of applying zinc, tin, or other ductile metals or mixture of ductile metals to metal substrates- usually steel. Thicknesses are typically 0.2 to 0.6 mils. In the mechanical plating process, impact energy is transferred from a rotating open-ended oblique barrel through glass beads, resulting in the cold welding of fine metal dust particles to the substrates.

Figure 11, Typical mechanical plating system

Figure 12, Typical parts mechanically plated

The resulting deposit is slightly pours, semi-bright in finish, and provides corrosion protection to the articles plated without introducing hydrogen embitterment into the part. It is therefore used widely to provide corrosion protection to high strength fasteners, Figure 11.

Mechanical galvanizing is an extension of the well-established mechanical plating process. This process is nearly identical except that the amount of metal plated (zinc) is substantially greater. Galvanized coatings begin at a coating thickness of 1 mil, which is equal to 0.59 ounces per square foot. The coating can go up to approximately 2.6 mils, which is equal to 1.5 ounces per square foot.

Most commercial work is done to a thickness target of about 2mils or about 1.2 ounces per square foot. Generally parts to be mechanically galvanized can be up to 1 pound in weight and up to 6 inches long.

Parts, Figure 12, to be mechanically plated or galvanized are bought to a barrel-loading hoist in some type of bulk container. Impact media (mixture of glass beads ranging in size from 0.1mm to 5mm) is transferred from the overhead reservoir into the plating barrel. Proprietary chemicals, water and fine metal powder are added to the barrel. The plating barrel is then rotated to thoroughly mix the parts, chemistry and media. Mechanical energy produced in the rotating mass causes the impact media to "cold weld" the extremely fine metal powder to the parts. The process continues until the desired thickness is achieved. The load is then discharged into the surge hopper. Water sprays wash the impact media away from the parts as they travel through the separating section. A magnetic conveyor is used for small parts. The impact media is diverted into the media sump for reuse. The processed material continues with the parts passing through an optional vibrating chromater and on to the optional vibrating dryer to the collection bin, Figure 13.

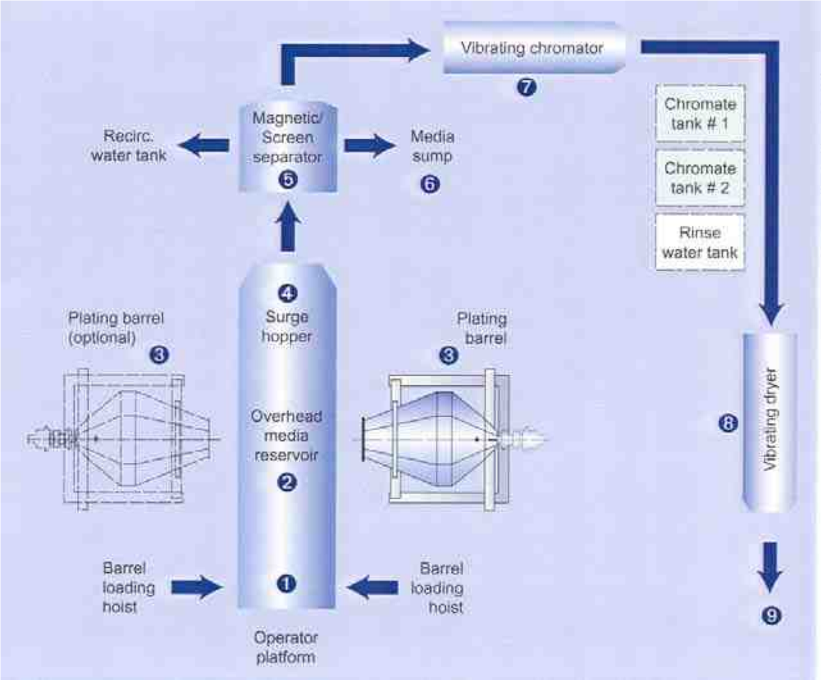

Figure 13, Typical configuration of a mechanical plating system

The heavier the coating, the greater the cost advantage of mechanical plating and mechanical galvanizing over electroplating. Thicker coating require more time in electroplating while mechanical plating does most of thickness in about the same time. Mechanical plating is more cost effective than electroplating at comparable thickness above 0.5 mils. Mechanical Plating Systems are ecology minded processes, there are no cyanides, chelated chemistries or strong odors with mechanical plating.

Continuous Removal of Metallic Impurities From a Trivalent Chromium Passivate

Zinc Platers have used chromium passivate to improve the corrosion protection of the deposit. Chromium passivate also offers resistance to finger prints while improving the brightness of the deposit, also in some cases as a base for paint adhesion. Chromate colors that have been in use for different industries include yellow, clear, blue, olive drab, and black.

Historically the chemistry has been hexavalent chromium based with different industries using trivalent where possible. The chromate would be dumped on a regular basis based on the accumulation of iron, in some cases as much as each shift. Due to the low concentrations and cost of the chromate, this was an economical method to achieve the salt spray requirements that were required.

With the European End-of-life (ELV) Scrap Vehicle Directive, banning the use of hexavalent chromium ion passivates in automotive applications, zinc platers are required to meet or improve the testing requirements with new chemistries that did not have the same characteristics or economy of the hexavalent chromates.

A new ion exchange system has been developed for the removal of iron contamination from a trivalent passivate that meets the requirements of automotive, with the economics of a potential never dump. In operation, the trivalent chromate solution is pumped through the filter chamber, Figure 14, to the resin columns where solution flows through the bottom to the top of the column, through the resin bed, removing iron contamination from the chromate solution. The solution is then returned to the process bath from the top of the column through a header system.

The system has a full and active resin charge (7.5 liters per column) that will remove 400 grams of iron before the resin reaches its absorption capacity. At this point the resin is need of regeneration to return to the active condition. This is accomplished by flushing the resin with dilute hydrochloric acid, followed by a rinsing cycle with pH 2.0 water. It is suggested that the storage tanks be provided for regenerating that are capable of holding a minimum of 250 liters of 15-20% by volume hydrochloric acid and 250 liters of water with a ph of 2.0 for the regenerations cycles. The regenerating acid can be used 3 to 5 times.

Figure 14, Typical four-stage ion exchange

The frequency for the regeneration will be determined by the rate at which the metallic impurities are introduced to the process bath. The preferred method is to analyze the bath for iron on start-up of the unit and to continue analyzing the solution on a daily basis until metallic impurity build reaches 400grams of iron. This time frame should then become the maximum period between schedule resin regeneration with similar production throughput. It should be noted that the discharge from the system of regenerate chemical and the lower pH water rinse water will contain iron and will be at low pH, requiring treatment prior to discharge to sewer.

A Continuous Electrodialysis Process for Electroless Nickel

Electroless nickel-phosphorus alloy coating offer outstanding distribution of the plating with constant layer thickness - even on, complex parts. Depending on the alloy composition, they can also offer high chemical resistance, outstanding wear and corrosion properties, and an x-ray amorphous structure. These characteristics have given this process a constantly increasing market share. Today it is used in all industrial sectors that depend on maximum quality. Electroless metallization processes have system-related disadvantages. Instead of being supplied from the outside, the electrons needed to deposit the nickel come from a reducing agent (hypophosphate). The chemical reaction produces a by-product, orthophosphite, which is a waste product that interferes with the process. Also nickel is added as a salt solution, usually nickel sulfate, and the anions of the nickel salt accumulate in the bath. This causes a permanent impairment of the deposit properties, and a steadily decreasing rate of the deposition. An ideal process would remove the interfering salts continuously and allow a higher bath loading. The result would be consistent high quality of the deposited nickel layers.

Various methods on the market have been discussed and tested for a long period of time:

- Precipitation process
- Ion exchange
- Electrodialysis
- Direct cathodic reduction
- Bleed and feed methods

None of these methods has proved satisfactory so far. The cost of treatment is often too high, other interfering materials are added, a waste disposal problem results, or the loss of valuable substances cannot be adequately separated from the interfering materials with the standard design until now.

A new development for Electrodialysis extends the bath life, while minimizing loss of material by a special procedure, choice and design of the membrane, Figure 15. In the Electrodialysis process, the bath is pumped through a stack with multiply types of chambers. The two different types of chambers are arranged alternately, and are separated by anion-exchange a cation-exchange membranes. A direct current is applied to the ends of the stack. It causes the anions and the cations to migrate in opposite directions. In contrast to ordinary Electrodialysis systems, this system separates the interfering anions and cations from the useful ones and removes them from the system into the concentrate tank.

This new system, Figure 15 and 16, consists of:

- Membrane module
- Fully automated work module
- Bath module with integrated nickel controller and heat exchanger to heat and cool the feed flow.

The pump station and the stack can be installed away from the bath, or on another floor for instance. The feed and heat system is placed adjacent to the bath. The system operates continuously production is not interrupted. The bath module removes a substream from the working solution, cools it in countercurrent flow by a flow returning to bath, and pumps it to the ED unit. The heat introduced from the feed stream and the system components is conducted away by a chiller. At the same time, bath replenishment automatically takes place. This eliminates time-consuming cooling, intermediate storage, and adjustment of the regenerated bath.

The system is designed to attain economic and ecological objectives:

- Potentially unlimited bath life, with consistent bath parameters
- Higher throughput possible compared to conventional systems
- Reduced labor cost
- Simple automation.

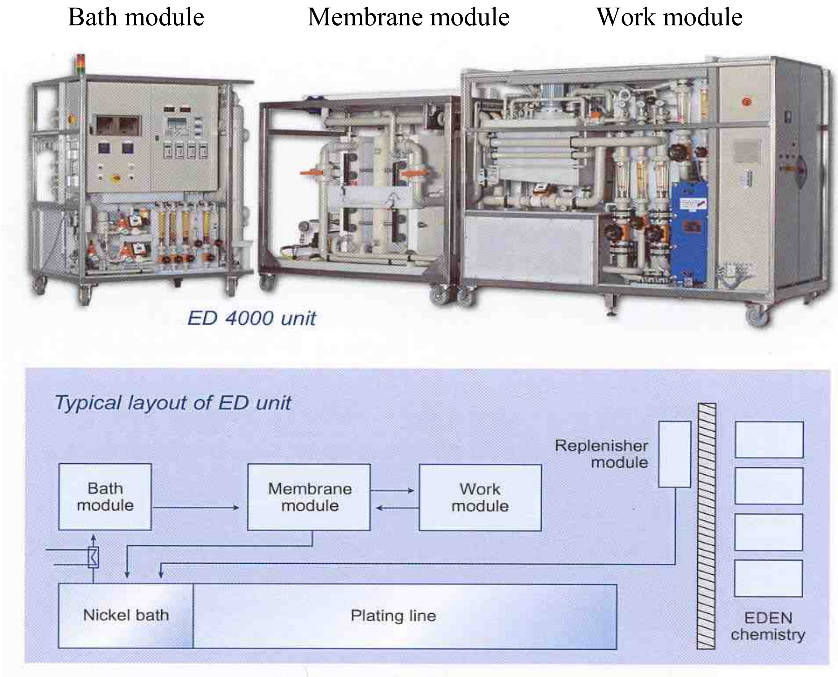

Figure 15, ED Unit with typical plant layout

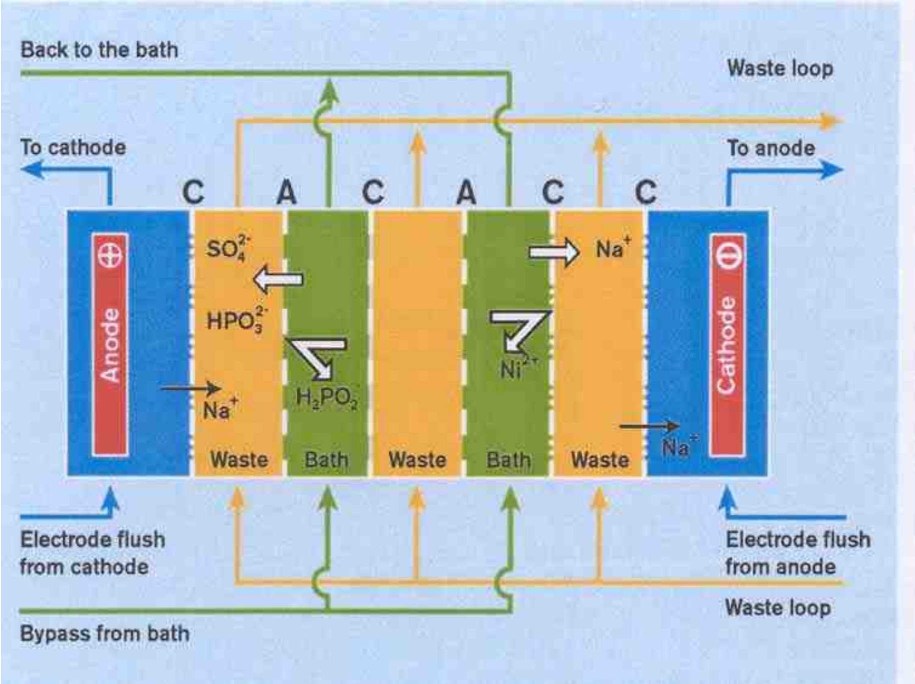

Figure 16, Membrane schematic used with ED system