Converting to Lead Free, Cad Free Electroless Nickel

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This paper is a case study of a supplier to the automotive industry converting to new electroless nickel technology. It will address the driving force behind the change, and compare operating conditions, performance and deposit properties between electroless nickel systems with and without cadmium and lead additives.

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Background
Electroless nickel is commonly chosen by the automotive industry for a variety of properties and its ability to plate over a wide selection of substrates including complex geometries. These properties include excellent adhesion, hardness, wear resistance, lubricity, chemical stability, corrosion protection, magnetic characteristics, electrical resistance, solder ability, and deposit uniformity. It can be applied to a variety of steel alloys, cast iron, stainless steels, aluminum, copper, bronze, brass, powdered metals, magnesium, beryllium, titanium, ceramics, and plastics. Common applications in the automotive industry include differential pinions, gears, yokes, thrust washers, hose couplings, heat sinks, etc. to name a few.

Electroless nickel solutions are a complex mixture of components including nickel, reducing agents, accelerators, complexors, buffers, stabilizers, and brighteners. Traditionally, electroless nickel formulations have included trace amounts of cadmium and lead compounds to enhance the deposit appearance and bath stability. Lead is used as a stabilizer to prevent spontaneous deposition of nickel, and contributes to the brightness of the deposit. Cadmium is used primarily as a brightener, but detracts from the corrosion resistance. The levels in a working solution are very low – typically below 2 ppm of each metal. In a typical mid-phosphorus (6-9% phosphorus) electroless nickel deposit, the amount codeposited is 0.03-0.1% of lead and 0.1-0.2% of cadmium.

Reason for change
Lead and its compounds are classified as a toxic with known cumulative health hazards. Cadmium and its compounds are classified as toxic and a known carcinogen. It is estimated that over 7000 pounds of lead and 9000 pounds of cadmium are used annually in electroless nickel formulations.

Automotive requirements restrict the use of four hazardous metals: mercury, hexavalent chromium, lead and cadmium compounds in order to comply with the European Union end-of-life vehicle (ELV) directive, established in 2000. This directive was designed to minimize the impact on the environment, promote energy conservation, and reduce waste while remaining competitive. Eliminating the hazardous metals from vehicles will make them more easily recycled or recovered. The concentration of lead compounds and cadmium compounds in electroless nickel chemistry is below the limit requiring disclosure on the material safety data sheets. Because of the low concentrations, the automotive industry and their suppliers were unaware that they were exceeding the limits established in the ELV directive.

Subject
The subject of this paper is a Tier One and Tier Two supplier of mid-phosphorus alloy electroless nickel plated parts to the automotive industry. The facility offers rack and barrel plating, primarily on steel stampings and castings. Cathodic protection is utilized for the stainless steel

process tanks. The lines are operated by means of a manually controlled hoist system. Additions are determined and made on a manual basis. A large percentage of the work requires post-plate heat treatment.

Identification

In 2003, the facility identified the need to investigate their current electroless nickel system to determine compliance with the ELV directive. A simple phone call to the supplier of the electroless nickel technology confirmed the presence of cadmium and lead in their current process. A compilation of suppliers that offer ELV-compliant electroless nickel systems was made. The decision was made to evaluate and qualify a primary and secondary chemical supplier.

Selection and Testing

The objection of the testing was to select a process based on ability to meet deposit specifications, bath life, ease of use, compatibility with current equipment and process times, and cost. Multiple suppliers were chosen for comparison. Typical production parts were processed in accordance with the supplier's recommended operating parameters. There were subtle performance differences noted between the various systems, but they all met the deposit requirements. The objectives are described in Table 1.

Pretreatment

A typical pretreatment cycle has traditionally been used, incorporating alkaline immersion cleaning, electrolytic cleaning, and acid pickling prior to electroless nickel plating. No change in chemistry or operating conditions was required.

Plating bath solution control

The plating solution is manually controlled. Analytical procedures, optimum concentrations, and addition rates were similar before and after the change. The pH is still manually controlled with ammonium hydroxide additions, although the operating window was slightly reduced from 4.6-5.4 to 4.8-5.2 pH units.

Bath loading capabilities held true, capable of plating anywhere from 0.5-42.5 dm^2/L (0.2-1.7 $feet^2/gallon$). No indications of over- or under- stabilization were experienced.

Plating bath equipment control

The cathodic passivation was adequate in preventing plate-out of the process tank. The regular 5-micron filter bags were successfully used, with no difference noted in the replacement frequency.

The amount of clean, filtered air agitation was sufficient.

Increasing the temperature from 82 °C (180 °F) to 87 °C (195 °F) enhanced the deposit appearance and plating rate.

Post-treatment
The reactivation cycle for the passivated electroless nickel surface for subsequent plating operations did not change. Similar heat treat temperature and time in an inert atmosphere were employed, with the predicted resulting hardness.

Rework
The chemistry used to strip rejected parts did not require any adjustments in concentration, temperature, or time. Reprocessing parts followed the regular rework cycle.

Results
An acceptable replacement to the lead and cadmium containing electroless nickel system was identified. The deposit properties met the current requirements of the current electroless nickel specifications.

Comparison of Deposit

	Conventional system	**Lead and cadmium free system**
Nickel	82.8-90.2%	82.5-89%
Phosphorus	7.2% - 9.8%	7.5% - 11.0%
Cadmium	0.08 – 0.17%	0
Lead	0.02 – 0.04%	0
Balance	0.10-0.15%	0.15 – 0.20%
As-plated hardness	450 Hv	480 Hv
Hardness after heat treat	980 Hv100	980 Hv100
Corrosion resistance on 25 µm (1 mil) thick deposit	96 hours	96 hours
Melting point	890 °C (1634 °F)	890 °C (1634 °F)
Appearance	Bright to semi bright as bath aged	Remained bright throughout bath life

Conclusion
A successful transition can be made from the traditional electroless nickel systems utilizing lead and cadmium additives to an electroless nickel system that is free of lead and cadmium, and ELV-compliant. Modifications to existing equipment and process cycles are not required, as the bath is operated similar to the conventional system. The properties of the lead and cadmium free electroless nickel deposits meet the same requirements of the lead and cadmium bearing electroless nickel deposits.

