

ProcessLab for monitoring nickel-plating baths in surface engineering



Pure nickel is a silvery-white metal that is extremely hard, corrosion resistant, shiny and ductile. Due to these remarkable characteristics, the metal is largely used in coating and surface engineering. Excellent polishing characteristics also recommend nickel coatings for optical and aesthetic applications (e.g., decorative items and furniture fittings). Superb mechanical and optical characteristics make nickel very attractive for the machine and electronics industries – the coatings can even be soldered.

In the nickel-plating process, the elemental properties of nickel are transferred to the surface of the workpiece. Functional properties such as hardness, corrosion resistance, color or ductility can be specifically influenced via the process parameters and the addition of chemical additives. For example, very shiny nickel layers are achieved by the addition of so-called brighteners. Other specific characteristics, such as color or a further increase in corrosion resistance, can be achieved by applying other metals below or above the nickel layer; this is done in alloy wheel rims, for instance, which often feature a surface layer of chromium on top of the nickel adhesion layer (base layer). Applications are found in nearly all industries.

ProcessLab from Metrohm for bath analysis

The coating can be performed by the company itself or by a specialized service provider. In any case, the nickel-plating process must be monitored and documented for complete traceability to be able to guarantee a consistent quality and to meet the requirements of the contractual partner.



With the Metrohm ProcessLab analysis system, the entire process from preparatory baths to the nickel plating baths and rinsing baths can be analytically monitored.



ProcessLab system for the analysis of nickel-plating baths. A completely automated system with sample changer is recommended for the fast and convenient analysis of samples from several production lines.

The ProcessLab analysis system offers a multitude of analysis techniques for monitoring the entire plating process. Typical parameters and process steps that can be analytically monitored with ProcessLab include:

- Content of components in preparatory baths
- Alkalinity and acidity in degreasing baths
- Content of major components in nickel-plating baths
- Contamination of rinsing and cleaning baths
- Physical properties such as density, etc. (through the integration of additional devices)

The custom-tailored ProcessLab is installed on site in very close proximity to the process. All measurement and traceability data are saved in the integrated database, which provides information about the course of the process at any time. Measurements can be examined using a control chart and exported for further processing. The operation of ProcessLab is very easy and intuitive via touchscreen. ProcessLab is based on Metrohm's established and well proven components from the laboratory sector contained in a robust, splash-proof housing.

ProcessLab offers you many advantages:

- Consistent traceability and documentation
- Independence from the laboratory
- Minimal response times in case of problems with your baths
- More narrow process window thanks to precise control
- Longer bath life, resulting in lower disposal costs

Main process: Nickel electroplating and electroless nickel plating

In nickel electroplating, nickel is deposited on the surface of the workpiece through the application of electricity; in electroless nickel plating, a reducing agent is used for the same purpose. The two processes significantly differ in the composition of the baths and the deposition speed as well as in the uniformity of the deposit and the surface properties of the finished workpieces. The selection of the plating procedure is therefore largely based on the application area of the produced workpiece.



The aluminum cooling fins, copper heat pipes and the copper base of this CPU cooler are completely nickel-plated.



Transmission, hydraulic and pump parts, valve housings, pressure cylinders, etc., are typically finished with electroless nickel plating because of the more precise coatings obtained.

Nickel electroplating

In electroplating, nickel is electrolytically deposited from the nickel bath onto the workpiece using electrical current. Quality and thickness of the deposited layer can be easily controlled through current density. The electrolyte solution of the nickel bath can be prepared from nickel salts or nickel-containing concentrates. A typical bath includes nickel sulfamate and nickel sulfate or nickel chloride as well as boric acid and other chemical additives.

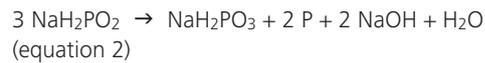
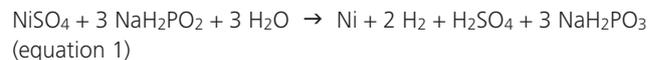
In nickel electroplating, very high workpiece throughputs can be achieved, and a regular analytic monitoring of the baths is essential. The following parameters can be determined on site using ProcessLab:

- Nickel content (EDTA titration)
- Chloride content (precipitation titration with AgNO_3)
- Boric acid content (acid/base titration)
- pH value (direct measurement)

Electroless nickel plating

Electroless nickel plating involves nickel deposition without current. The electrons needed to reduce nickel ions can be generated directly in the electrolyte solution via a chemical reaction, typically using the reducing agent sodium hypophosphite (see equations 1 and 2). Baths commonly contain nickel salt in the form of nickel sulfate. Nickel is deposited autocatalytically. Since phosphorus is also deposited, a nickel-phosphorus alloy results. Depending on the phos-

phorus content in the deposited layer (typically 1...14%), workpieces may be very hard and wear resistant (low phosphorus content) or very corrosion resistant (high phosphorus content).



The advantage of the electroless procedure lies in the slower deposition rate which results in more precise coatings. An excellent uniformity of the coating thickness with no edge build-up is obtained. Plastics such as acrylonitrile-butadiene-styrene (ABS) can also be nickel plated after pretreatment (e.g., multi-layer nickel chromium systems for logos and cooler grilles made by automobile manufacturers).

Equations 1 and 2 show that the chemical composition of the bath changes over time; the nickel ion and hypophosphite content decreases, and the concentrations of sulfate and orthophosphite (NaH_2PO_3) – products of increasing salinity – rise. As more nickel than phosphorus is deposited, more sulfuric acid than sodium hydroxide is formed, so that the pH continually decreases. The longer the bath is used, the more the concentration of impurities increases, which negatively affects surface quality and layer thickness. For process parameters to be met, the composition of the bath must remain



Nickel electroplating is used for connections, plugs, contacts, etc. A highly uniform coating thickness is not essential in these areas.

within certain concentration limits (process window), so precise control is absolutely necessary. If the exact concentrations in the nickel bath are known, suitable measures (replenishment of consumed bath components, addition of auxiliary agents, separation of contaminants formed) can be taken to increase the quality of the final product and extend the bath life.

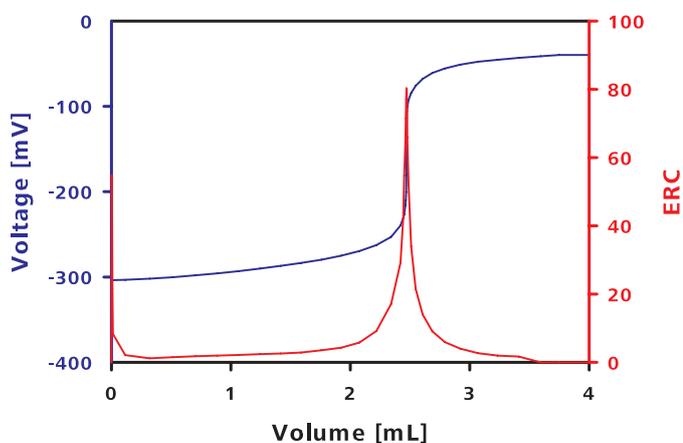
With ProcessLab, the parameters of electroless nickel baths can be easily and efficiently monitored:

- Nickel content (titration with EDTA)
- Hypophosphite concentration (indirect titration with sodium thiosulfate)
- Sulfate content (precipitation as barium sulfate with subsequent EDTA titration)
- Alkalinity (acid/base titration)

Summary

Using ProcessLab helps you to find out what's happening in your bath. You always know the exact concentrations of all substances involved in the process – whether you choose to coat your workpieces electrolessly or with electroplating. This allows you to take early measures to increase quality and extend bath life. So, ProcessLab helps you to save costs and protect the environment by generating less waste. Metrohm's process analysis system can be easily operated.

ProcessLab is able to analyze a multitude of different baths in the plating industry – your Metrohm representative will be happy to provide you with more information.



Hypophosphite determination – titration curve of the back titration of excessive iodine with thiosulfate solution.