



Application Note AN-PAN-1012

无电解镀镍液中镍离子和次亚磷酸含量的在线分析

纯镍是一种银白色金属,具有非常高的硬度、耐腐蚀性和延展性。由于这些显著特点,这种金属被广泛应用于涂层和表面工程领域。化学镀镍是一种在固体工件表面沉积一层镍磷合金的自催化化学技术。该工艺依赖于还原剂(次磷酸钠)的化学存在,还原剂与金属离子发生反应,从而进行沉积。然而,电镀槽化学品的寿命是

有限的,因此需要对化学品消耗进行自动监控,这也是关键的工艺控制要求。随着电镀槽使用时间的延长,电解液中的反应产物会过量,从而对工件的表面和镀层特性产生负面影响。

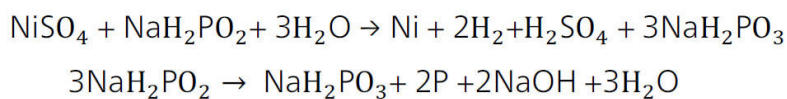
本工艺应用报告介绍了一种定期监控化学镀镍液中活性成分的方法,以确保沉积出均匀的镍磷合金层。

INTRODUCTION

Electroless nickel plating baths facilitate the chemical reduction of nickel ions to the metal in acidic electrolyte baths. Here, sodium hypophosphite (NaH_2PO_2) is used as the reducing agent; with its help, a very corrosion-resistant nickelphosphorus alloy is deposited on the material surface.

The decisive reaction is the chemical reduction of the nickel and hydrogen ions by the hypophosphite leading to the deposited nickel and hydrogen gas (**Reaction 1**). Little hydrogen

gas formation points to a missing or a slow nickel deposition. The quicker this reaction occurs, the lower the amount of phosphorus in the coating. On the other hand, more phosphorus is contained in the coating when the reaction is slowed down. Coatings with high amounts of phosphorus (10–14%) are very resistant to corrosion, whereas higher abrasion resistance is more readily achieved with a low phosphorus content (3–7%).



Reaction 1. Reaction of electroless nickel deposition.

As nickel ions and hypophosphite are continuously consumed during the deposition process, the concentrations of these components must be kept within defined tolerances and continuously replenished to maintain consistent quality in the final product. When the plating bath is in use, the concentrations of sulfate and sodium phosphite (NaH_2PO_3) steadily increase; this becomes the limiting factor when the bath is in use for a long time. As more nickel is deposited than

phosphorus, more sulfuric acid than sodium hydroxide is formed as the process continues. This leads to a decrease in pH during nickel deposition which must be increased again by the addition of sodium hydroxide or ammonia. Only exact and reproducible determination of the process-relevant parameters can ensure that the consumed bath components can be replenished correctly to guarantee optimal process control.

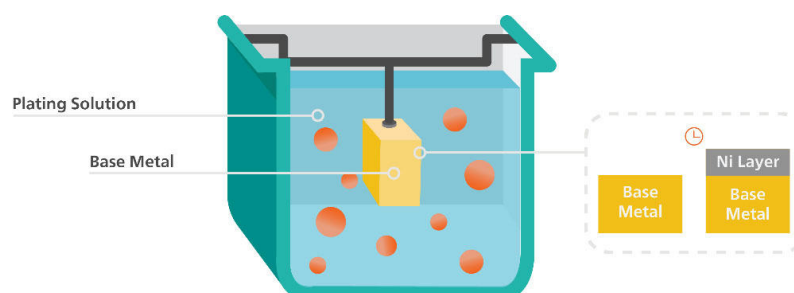


Figure 1. Schematic diagram of the electroless nickel plating process.

APPLICATION

Online monitoring of the pH, nickel, and hypophosphite content is possible with the **2060 Process Analyzer** from Metrohm Process Analytics (**Figure 2**). All liquid handling steps such as taking sample aliquots, dosing of reagents, titration, and cleaning are performed by pumps and burets controlled by the process analyzer.

The analysis consists of transferring a sample aliquot either to the vessel for alkalinity and nickel analysis or to the vessel for sodium hypophosphite determination.

The 2060 Process Analyzer enables simultaneous, monitoring of diverse bath parameters with a single measurement, increasing measurement frequency. Nickel and pH are determined by online titration (**Figure 3**), and sodium hypophosphite is determined by potentiometric titration using a platinum electrode.



Figure 2. 2060 Process Analyzer

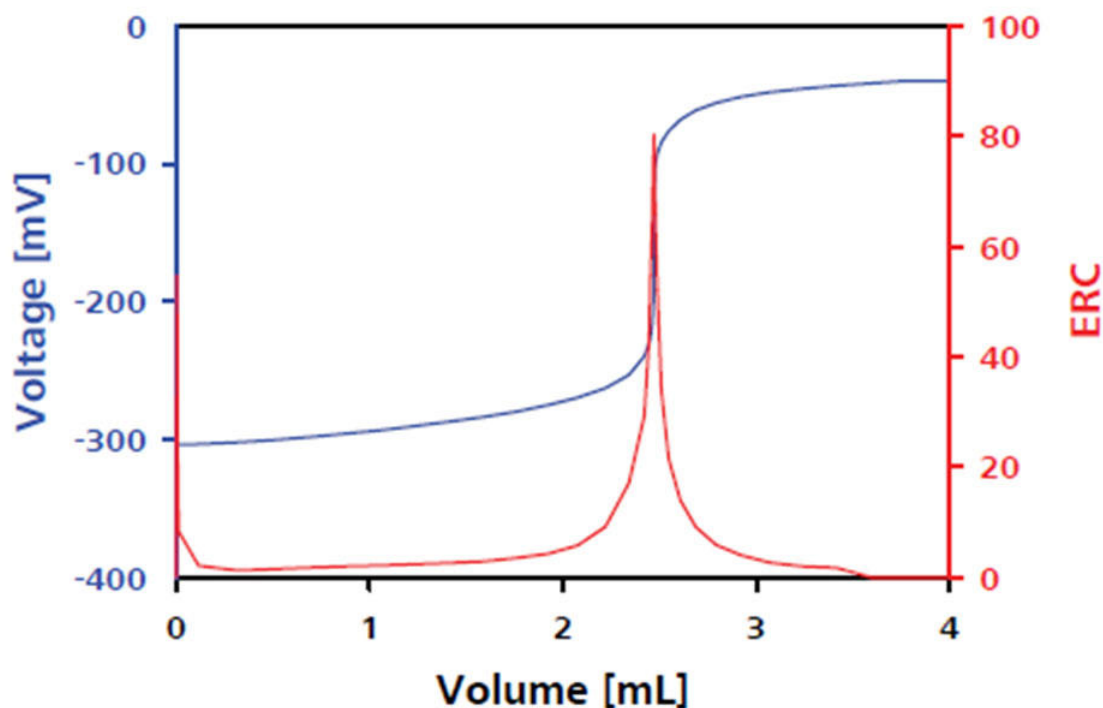


Figure 3. Back-titration curve of iodine using thiosulfate. ERC: Equivalence point Recognition Criterion.

Manual data collection can affect product quality, reduce yield, and expose personnel to hazardous conditions. This robust process analyzer has the flexibility to automatically recognize the titration endpoint to guarantee reproducibility of results, and high reliability and

dispense accuracy of the bath constituents. The 2060 Process Analyzer can be programmed to acquire data at regular intervals without needing to wait for laboratory results, and out-of-specification readings can immediately inform operators to take direct action.

Table 1. Parameters to monitor in electroless nickel plating baths

Analyte	Range
Ni as nickel sulfate (NiSO ₄)	<10 g/L
NaH ₂ PO ₂	1–12 %
pH	4.5–5.0

CONCLUSION

Knowing the exact concentration of the active bath constituents in an electroless nickel plating bath is crucial since early measures can be taken if necessary. This includes the timely replenishment of the consumed components to ensure an even coating deposition and the

separation of formed contaminants. Online monitoring of plating baths ensures the quality of the final product, meaning higher yields and less downtime as well as a reduction in operation costs by extending the bath life.

FURTHER READING

[Determination of acids, bases, and aluminum: galvanic industry – metal surface treatment](#)

[Online and atline analysis of acids and iron in pickling baths](#)

BENEFITS FOR TITRATION IN PROCESS

- Increased final product quality and metal turnover (MTO) due to online determination of bath parameters
- Fully automated diagnostics – automatic alarms for when samples are out of specification parameters
- Safer working environment and automated sampling



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CONFIGURATION



2060 Process Analyzer

2060 Process Analyzer 是一种在线湿化学分析仪,适用于无数应用。此过程分析仪提供了一个新的模块化概念,由一个称为«主机柜»的中心平台组成。

主机柜由两部分组成。上部包含触摸屏和工业计算机。下部含有柔性取样部,其中放有用于实际分析的硬件。如果主取样部容量不足以应对分析挑战,那么主机柜可以扩展为多达四个额外的取样部机柜,以确保有足够的空间来应对具挑战性的应用。附加机柜的配置方式使每个取样部机柜可以与具有集成(非接触式)液位检测的试剂柜组合使用,以增加分析仪的正常运行时间。

2060 Process Analyzer 提供不同的湿化学技术:滴定法、卡尔费舍尔滴定法、光度测定、直接测量和标准添加法。

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