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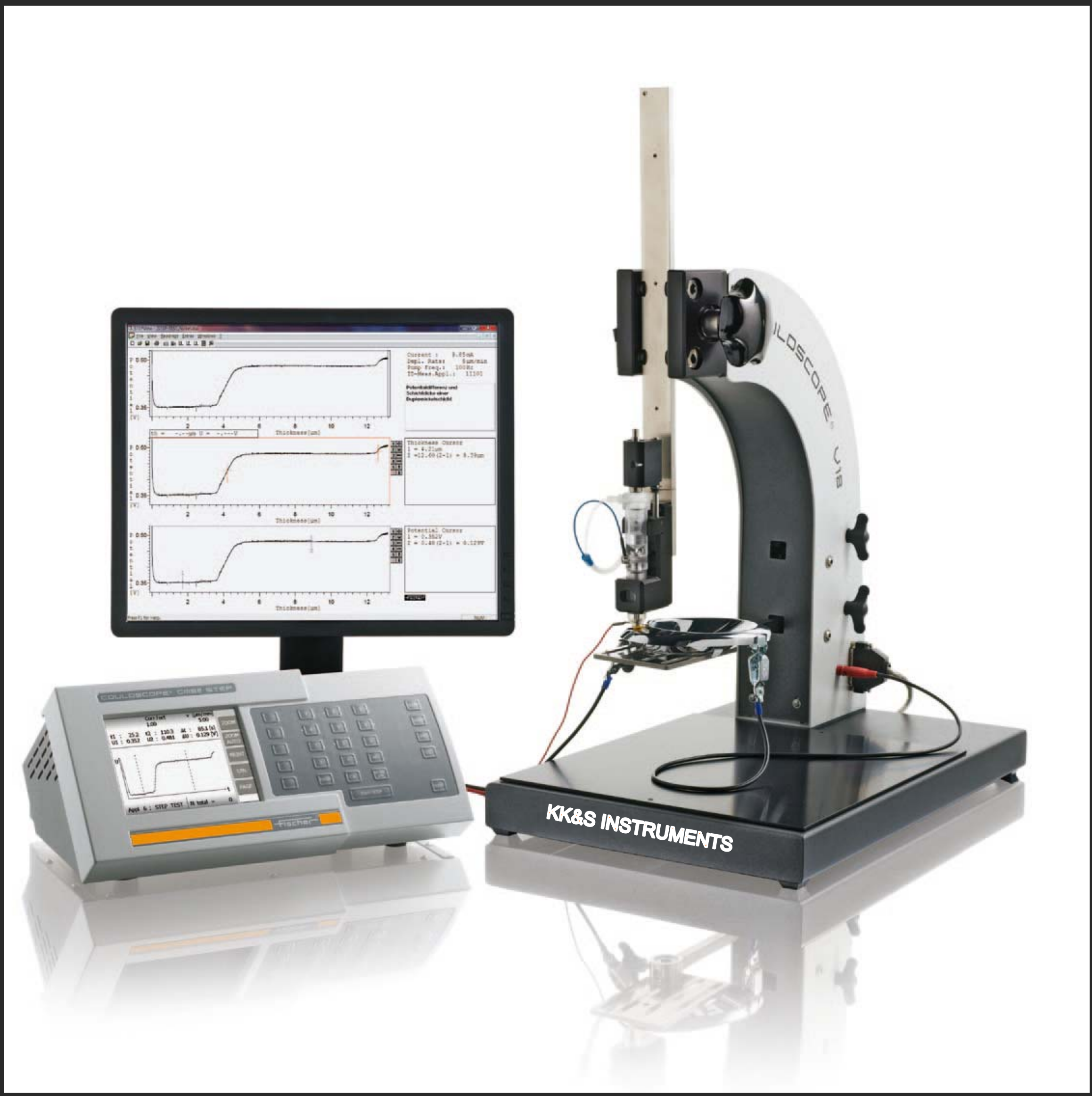
The Probe

KK&S INSTRUMENTS - July / September 2014 Issue



In This Issue:

COULOSCOPE® CMS2 & COULOSCOPE® CMS2 STEP

Measurement of coating thicknesses and electrochemical potentials according to the coulometric method.

Coulometry is an electrochemical analysis method that is simple and easy to execute and can be applied in order to determine the thickness of metal coatings.

While primarily used for checking the quality of electroplated coatings, this method is also suitable for monitoring the thickness of the remaining pure tin on printed circuit boards.

STEP Test measuring cell placed on a specimen

Features of the COULOSCOPE® CMS2

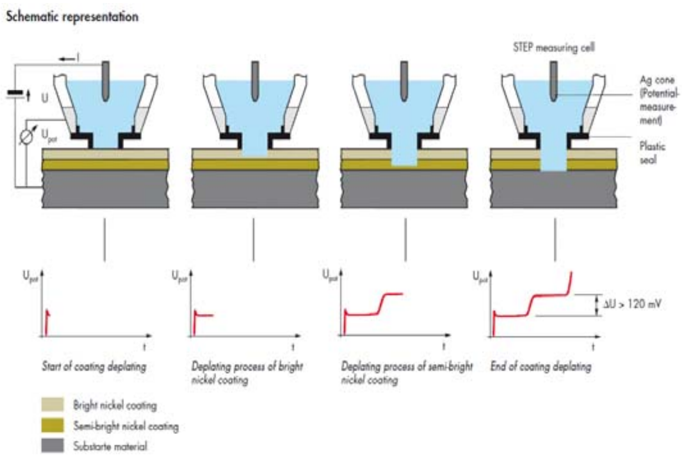
- Large, high-resolution colour display
- Simple instrument operation and graphically supported user guidance
- Partially-automated measurement with support stand V18
- Simple selection of;
  - de-plating speed (0.1 – 50 µm/min)
  - de-plating area (0.6 – 3.2 mm O)
- Graphic display of the voltage profile in the measuring cell
- Graphic and statistical analysis options
- Various languages and measurement units to choose from

Additional features - COULOSCOPE® STEP

- Simultaneous measurement of coating thickness and differences in potential
- Simple conditioning of the silver reference electrode
- Adjustable de-plating current

COULOSCOPE® CMS2

The CMS2 measures the thickness of virtually any metallic coating, including multi-layer, on any substrate material; it works according to the coulometric method by anodic dissolution (DIN EN ISO 2177). The simple handling and menu-supported operator guidance makes the CMS2 the ideal solution for both production monitoring in the electroplating industry and incoming inspection on finished parts. The device comes equipped with nearly 100 predefined measuring applications for different coating systems (e.g. zinc on iron, nickel on brass), as well as various de-plating speeds (e.g. 1, 2, 5, and 10 µm/min). These can also be combined for measuring multi-layer systems.



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News – Feritscope / Echometer 1076

The FMP30 Feritscope now comes with a Coloured Display and Free DataCentre

The reliable Helmut Fischer FERITSCOPE® FMP30 still features;

☑ **Small Diameter Probe** (5 Options)

☑ **Industry Recognised FMP30; Accuracy, Repeatability & Reliability**

☑ **The only accurate Feritscope % / FN on the market;**
- 0.1 to 80 Ferrite-percentage "%"
- 0.1 to 110 Ferrite numbers "FN"

☑ **Traceable Calibration to international IIW secondary standards.**
Feritscope therefore meets all requirements of;
- ANSI/AWS A4.2
- DIN EN ISO 8249
- BASLER Standard.

Karl Deutsch launches their new Basic version Ultrasonic Thickness Gauge, the ECHOMETER 1076

ECHOMETER 1076-Basic **Features**

☑ Ex Large Numbers 50 x 27mm display with illumination
☑ Made in Germany
☑ Sensitivity Adj. to the sound damping of different materials
☑ Three display modes: Standard, Difference & Minimum
☑ Limit values with acoustical and optical alarm
☑ User-friendly menu structure and measuring parameters
☑ Wall thickness & Sound velocity modes
☑ Velocity 100 to 19,999 m/s range stated in mm steel
☑ All parameters remain after switch-off and battery change.
☑ Readings in resolutions 0.1 mm, 0.01 mm or 0.001 inch
☑ Battery lifetime up to 130 hours
☑ Integrated pop-up stand
☑ Automatic recognition of probes
☑ Splash-proof housing according to IP 54
☑ Miniature probe (10 MHz): 0.5 to 25 mm
☑ Standard probe (4 MHz): 1.2 to 250 mm
☑ Low frequency probe (2 MHz): 5.0 to 400 mm

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Measurement of Multilayer Nickel Coatings
After the coating process

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For the quality control of multilayer nickel, a specific type of measurement equipment is required in order to measure the thickness as well as the electrochemical potential right away after the coating process. Therefore, a measurement system was developed, which is specifically suitable for the difficult measurement conditions in companies of the electroplating industry with an especially adapted easy handling of the reference electrode. Time-consuming salt-spraying-tests will then only be required for small spot checks.
The nickel plating is used for the decorative anti-corrosion protection and for the improvement on the mechanical surface properties, e.g. hardness. The parts to be protected mostly consist of steel, aluminium or synthetic material. Especially in the car industry, nickel plated parts must be extremely resistant against corrosion. For this matter, simple nickel layers are not sufficient. Therefore, a type of complex layer systems was developed, consisting of two, three or even four different types of nickel layers as well as further chromium and copper layers. The corrosion characteristics are checked with Cass- or Corrodkote-tests, with specific salt spraying methods. As these tests are very time-consuming, a new testing method was developed, where fast and reliable conclusions about the corrosion behaviour can be made right after the coating process.

Features of chromium plated multilayer nickel

Figure 1 shows the principle structure of such a layer system. However, the very sulphurous nickel inter-layers are not so frequently used at present. The technical requirements on such coatings can be taken from DIN EN 12540: corrosion protection of metals

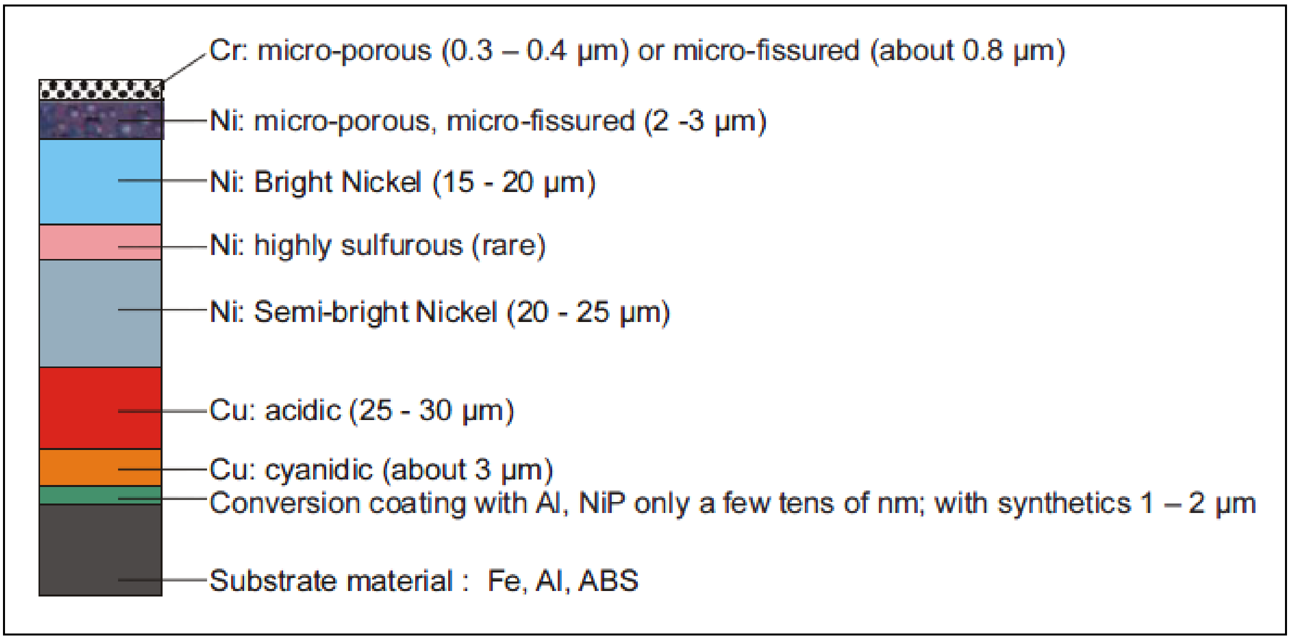

Figure 1: Principle structure of multiple nickel layers

The different type of coatings have the following features:

Micro-porous or micro crack chromium-layer:
This extremely hard layer is passivated due to the air oxygen. A thin oxygen layer is built up of an electrochemical potential of about 1.3 V. This enables a type of corrosion behaviour like for noble metals and the chromium layer keeps its bright appearance. Through the pores or cracks, a homogeneous anodic base of the less noble nickel layer underneath is created. Therefore, a local corrosion attack on this layer is less probable.

Micro porous or micro crack nickel layer
The crack nickel layer is more noble than the beneath underlying bright nickel layer (cathodic behavior). A corrosion attack therefore mainly concerns the bright nickel layer. The potential difference between both layers is according to acc. to TL 196 of Volkswagen AG in the range between 20 - 40 mV.

Bright nickel layer
According to TL 196 of Volkswagen AG and acc. to DBL 8465 of DaimlerChrysler AG, it must have an at least 120 mV lower electrochemical potential than the semi-bright nickel layer. Therefore a surface-like corrosion is achieved.

Only when this anodic operating layer has mostly corroded, a corrosion attack on the more noble semi-bright layer takes place. The surface-like corrosion of the bright nickel layer, especially at the bounders of the semi-bright nickel layer, can be improved, according to figure 1, by depositing highly sulphurous (%S > 1,5 acc. to DIN EN 12540) nickel layer with a higher negative potential in comparison to bright nickel.
The corrosion characteristics of such a layer system is not only determined by the layer thicknesses acc. to figure 1, but also by the differences of the electrochemical potentials. The potential differences should be determined immediately after the coating process, for the purpose of fast reaction and to prevent any rejects.

Simultaneous measurement of thickness and electrochemical potential

This in acc. with ASTM B 764-86 standardized measurement method STEP TEST (Simultaneous Thickness and Electrochemical Potential determination) enables by coulometric de-plating, with the use of a reference electrode the simultaneous measurement of thickness and electrochemical potential of the individual nickel coatings. (More information on this can be obtained in: Helmut Fischer product information: CMS STEP). It is important to use a suitable electrolyte, with the following composition: 300 g/l $NiCl_2 \cdot 6H_2O$, 50 g/l NaCl, 25 g/l H_3BO_3 with a pH-value of 3.0. The working group ISO TC 107/SC 3 is currently preparing a new edition of this standard ISO/DIS 16141. In this standard proposal, the reference electrode is recommended as a luggin capillary like in ASTM B 764-86. Due to the sensitive mechanical structure of this capillary, it is not very well suited for the handling in the electroplating industry. Therefore it was examined, whether similar results may be obtained with an electrode, that is easier to handle and more rugged.

Development of a practice-oriented measurement system

The luggin capillary consist of a thin silver wire with a silver chloride surface layer (Ag-AgCl), which is situated in a glass capillary, with a smaller diameter at the end, filled with a saturated KCl-solution. The glass capillary is sealed with a diaphragm at the end towards the electrolyte. This shall enable the movements of ions, which are necessary for the measurement of the potential, and will prevent a „contamination“ of the reference electrode with the detached nickel of the electrolyte. There are also designs, consisting of only an Ag-AgCl-wire inside a small plastic tube. A disadvantage of those designs is, that to keep a constant distance to the deplating surface is very difficult. But this constant distance is necessary in order to obtain comparable results with the potential measurements.

The disadvantages of such a measurement device shall be prevented with this kind of measurement system (see figure 2). It consists of the COULOSCOPE® CMS STEP, Helmut Fischer GmbH + Co.KG, Sindelfingen, which includes all the features of a coulometric coating thickness measurement. With that device, the layer thickness of chromium above nickel or copper beneath the nickel layers can be measured. Further the system is equipped with a measurement module to monitor the electrical voltage of a reference electrode. The de-plating cell for layer deplatement is fixed at a suitable measuring stand (figure 3).

Figure 2: With the measurement system COULOSCOPE® CMS STEP the potential development during the detachment of a layer can be depicted and analyzed.

The electrode under current is injected from above in the deplating cell. The reference electrode, made of massive silver, is a conical part, which also forms the lower part of the deplating cell. Due to this design, the active part of the reference electrode has the shape of a ring.

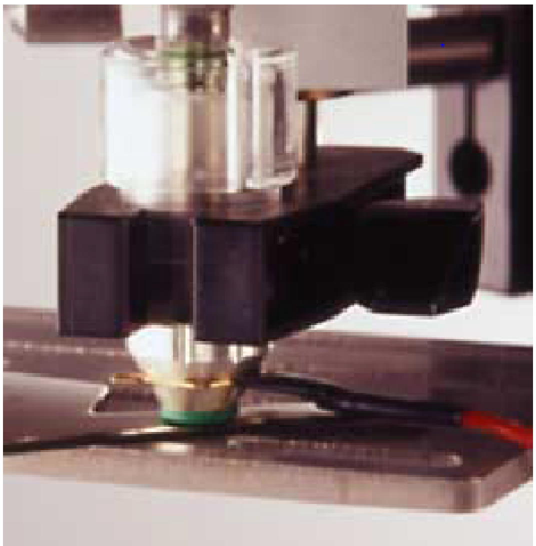

Figure 3: Deplating cell with new, conically formed reference electrode.

On the conical part the necessary measuring gasket is plugged in, which on the one hand grants the electrical insulation between the reference electrode and the test sample and on the other hand it defines the de-plating surface. With this design the same distance between the reference electrode and the test sample is ensured without any additional manual adjustment, so the measurement in practice are easier and much more reliable. The electrical connection between the reference electrode and the input connector of the measurement module consists of a simple insulated copper wire, which contacts the silver reference electrode with a fork-like plug.
The potential curve, which corresponds to the de-plating of the layers, is shown directly on the large LCD screen. With the help of freely to be positioned electronic cursors, the thickness of nickel layers and their potentials can be determined. This procedure is much more comfortable with the Fischer software STEP-View, running under Windows on a PC. As shown in figure 4 (4-layer nickel system), the potential curve can be shown in total. The cursor positions allow to determine the nickel layer thicknesses (figure 4, centre part) as well as the electrochemical potential differences (figure 4, see below part). The graphs can be saved with the results and comments in selectable graphs formats. Further the data transfer of the potential curve into an Excel–spreadsheet is possible.

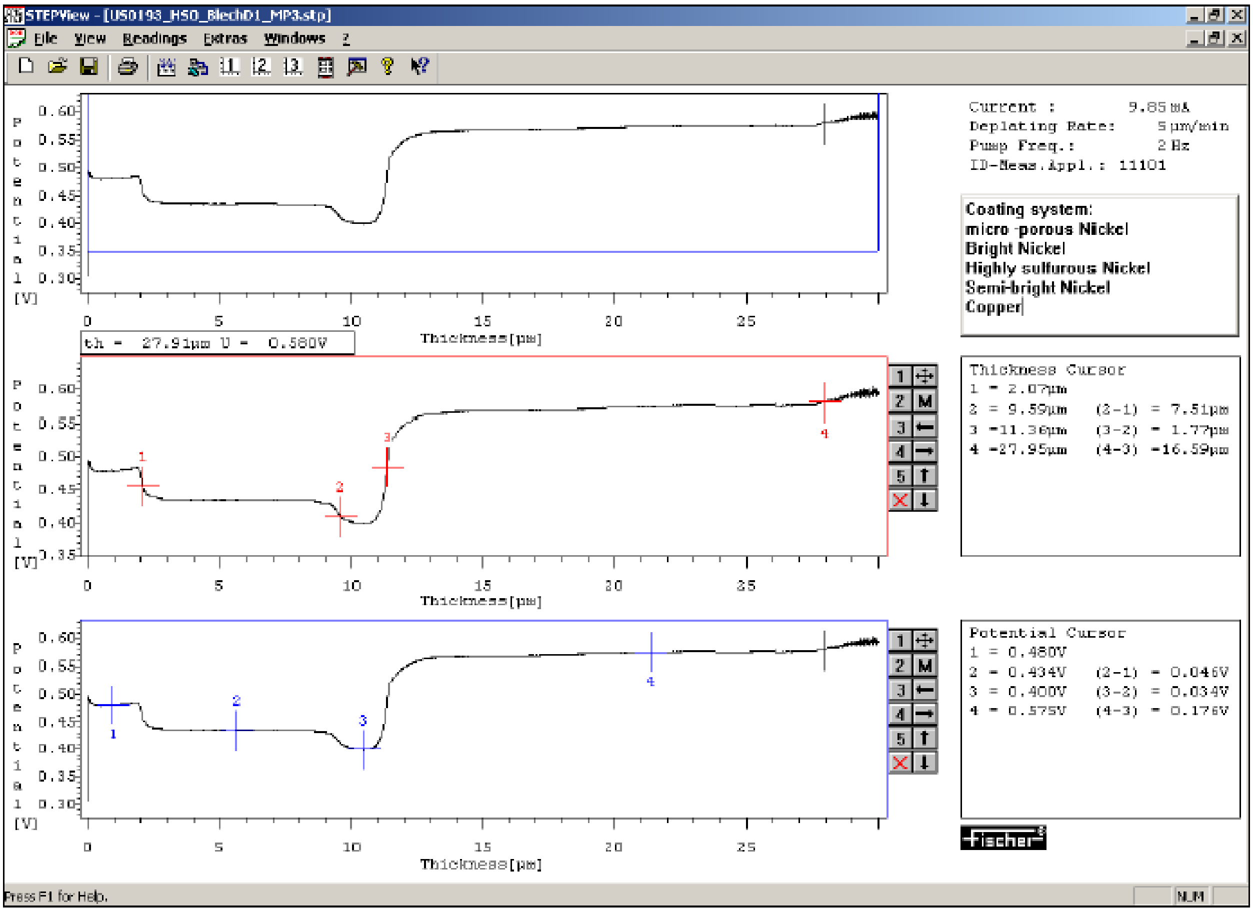

Figure 4: Display of the potential curves of a 4 layer nickel system with the PC software STEP View. With STEP view the individual nickel layers and the potential differences can be determined.

Extensive comparison measurements show that the conical silver reference electrode, used by Fischer, will lead to the same potential curves as with other existing reference electrode versions. The Fischer conical silver reference electrode is easier to use and much more rugged.

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