MultiScan MS 20

Stability Analysis of Electroplating Formulations

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Understanding Interfaces

*Electroplating is a commonly used technique to create a metal coating on a solid substrate to enhance its optical or material properties. To this end electroplating formulations are used which contain the coating as ions. The solid substrate is immersed in this electroplating formulation and an electric current is applied to the substrate. This induces the metal ions to be reduced to a metallic layer on the surface of the substrate. To guarantee a homogeneous coating the electroplating formulations needs to be stable throughout the coating process. To create a stable formulation that can be used for as many pieces as possible and a very long time, organic additives are used and the pH-value is being well adjusted. With the **MultiScan 20 (MS 20)** dispersion stability analysis system (Fig. 2) from DataPhysics Instruments stability changes can be detected and evaluated in a **quantitative** way **much faster** than any traditional shelflife test would permit. In the following application note we describe how changes of the pH-value influence the stability of the liquid used for a copper electroplating bath.*

Fig. 1. Example of an electroplating bath.

Keywords: MultiScan 20 (MS 20) ▪ Stability Analysis ▪ Electroplating Formulations ▪ pH-dependent Stability Issues

Technique and Method

The MultiScan MS 20 (Fig. 2) from DataPhysics Instruments is the measuring device for an automatic optical stability and aging analysis of liquid dispersions and the comprehensive characterisation of time- and temperature-dependent destabilisation mechanisms. It consists of a base unit and up to six connected ScanTowers with temperature-controlled sample chambers. The ScanTowers of the MS 20 can be individually controlled and operated **at different temperatures (4 °C to 80 °C).**

With its matching software MSC, the MS 20 is an ideal partner for the stability analysis since **even the slightest changes** within dispersions can be detected and evaluated. The MS 20 enables a fast and objective analysis of the dispersion stability as well as conclusions on possible **destabilisation mechanisms.**

Fig. 2: DataPhysics Instruments stability analysis system MultiScan MS 20 with six independent ScanTower.

Experiment

A small vial filled with the desired dispersion is placed in one of the ScanTowers of the MS 20. The scanning system is composed of a transmission and backscattering LED along with a detector. This system moves along the vertical axis of the vial (z-axis).

The obtained transmission and backscattering intensities are represented in an intensity-position diagram. The sample is scanned at regular time intervals. Changes in the detected measuring signal can provide explanations on the stability properties of the sample.

20 ml of each copper electroplating formulation (original, base additive, acid additive) were poured in a transparent glass vial and measured at every 5 min for 2 h 30 min. The measured zone is between 0 mm (bottom of the glass) and 57 mm (fill level of the vial). Fig. 3 shows the sample vials at the beginning and end of the measurement.

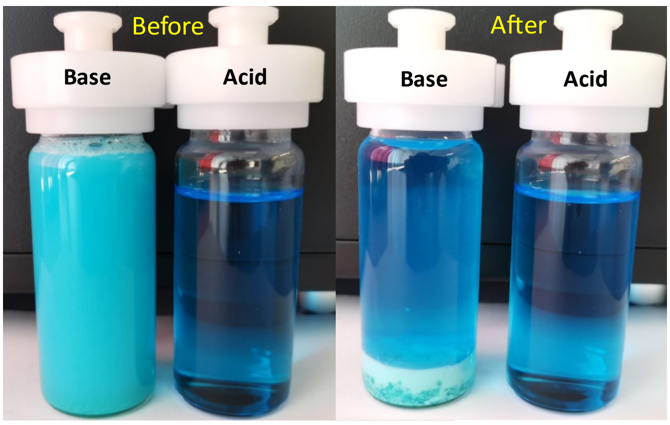

Fig. 3: Samples of electroplating solutions with base and acid additives before and after measurement.

Results

As the samples have a significant volume concentration, the transmission signal was very weak and provided no usable information throughout the measurment. Therefore the backscattering signal was analysed to study the stability of three copper electroplating formulations.

Fig. 4 shows the plot of the backscattering intensities against the position in the electroplating formulation with base additive. It shows a clearly time-dependent as well as position-dependent change of the signal, which increases at the bottom of the sample vial between 2 mm and 10 mm, indicating a typical precipitation process.

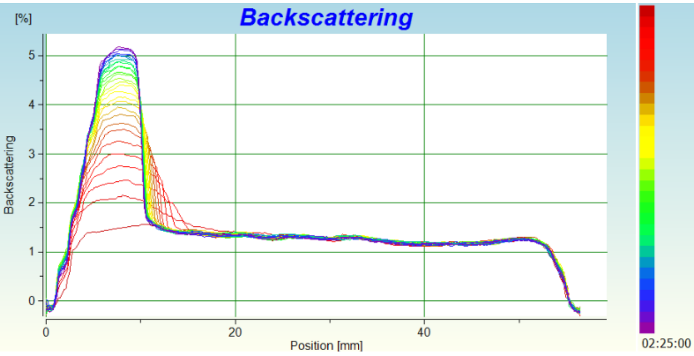

Fig. 4. Backscattering intensity diagram of the electroplating formulation with base additive.

Using the vast analytical capabilities of the MSC software, the change in migration front was analysed, resulting in an average sedimentation rate of 2.3 mm/h (in the first 50 min) and 0.35 mm/h (in the last 1 h 40 min) for the electroplating formulation with base additive (Fig. 5). The reason is that most of the particles have already precipitated in the first 50 min.

The other 2 samples were analysed accordingly, leading to the change rates as displayed in Fig. 6. Both the orignial electroplating formulation and the formulation with acid additive were very stable throughout the whole test with

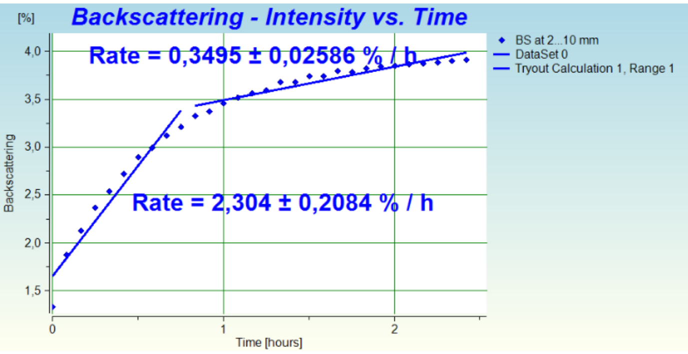

Fig. 5. Changes in migration front of the electroplating formulation with base additive over time.

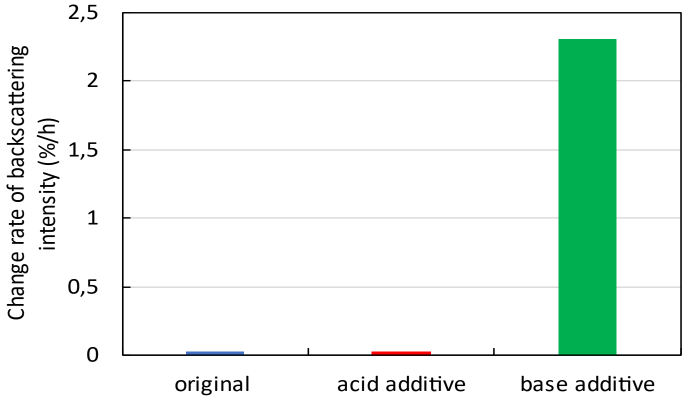

Fig. 6. Change rates of backscattering intensity over time.

similar stability. The base containing formulation was found to be the most unstable formulation.

Most notably, the MSC software can also provide an overall analysis by the **stabilty index** function. To **directly and simply** get the stability difference. These results can be displayed in a comparison plot (Fig. 7).

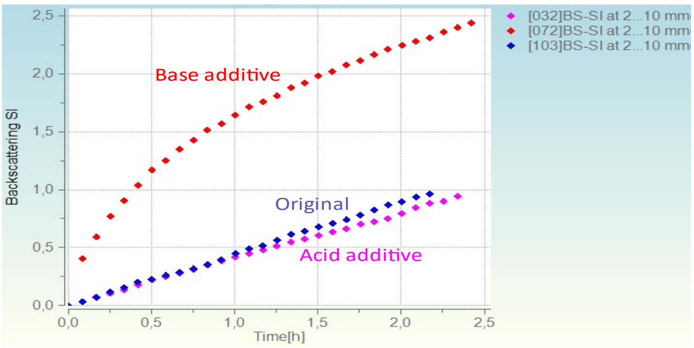

Fig. 7. Overlay of the backscattering stability index change over time.

In agreement with the previous results the stability index analysis shows that the electroplating formulation with base additive was the most unstable formulation, while the stability of the original electroplating formulation and the formulation with acid additive was much higher. These results underline **the excellent applicability** of the MS 20 to analyse and quantify stability issues of different formulations **with high reliability**.

Summary

Using the MS 20 stability analysis system and its corresponding MSC software, an **easy and fast way** to study the stability of electroplating formulations could be demonstrated. **Changes can be detected sensitively, easily, fast and reliably** which enables the producer to anticipate and quantify **stability issues** and thus guarantee time and cost optimal product development.

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