E-Line Refiner

SULFAMATE NICKEL GRAIN REFINER / HARDING AGENT

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E-Line Refiner is an electronic grade grain refiner and hardening agent specified for optical electroforming using sulfamate nickel baths. E-Line reduces the grain size of electrolytically deposited nickel by influencing crystalline structure formation, allowing the creation of smoother, low - stress electroforms with increased hardness. In addition, electroforms such as optical media stampers, that are made using E-Line Refiner have far greater resilience in punching and molding operations.

Used at concentrations as low as 0.02 – 0.10% by volume, E-Line Refiner helps to improve deposit smoothness, subsequently reducing back sanding requirements.

As a result of its grain refining characteristics, E-Line Refiner aids in maintaining low internal stress of deposit (0 – 4000 PSI Tensile) for better control of overall stamper flatness. E-Line Refiner will not increase the tensile stress of the bath and contains no butyne diol.

Because E-Line Refiner works by allowing nickel crystals to grow in geometrically planar structures, deposits obtained with the E-Line Refiner additive show an overall greater hardness, potentially increasing the life of electroformed replication media.

SPECIFICATIONS

Start Up Concentrations:

E-Line Refiner is typically added to a new bath at 0.05 - 0.20% by volume (0.5 ml E-Line Refiner / liter of bath solution). Maintain as needed. IMPORTANT NOTE: In situations where deposit stress is very low or compressive, E-Line Refiner should be added in increments of 0.02% to prevent inducing compressive stress. Verify stress using the Hull Cell method described below whenever possible.

Hardness Improvements:

Sulfamate Nickel (SN) Bath	*Rockwell-B (HRB)*	*Vickers (HV)*	*Knoop (HK)*
Typical SN deposit hardness	70-80	127-154	141-166
SN deposit hardness using 0.05% E-Line Refiner	90-95	193-220	203-230

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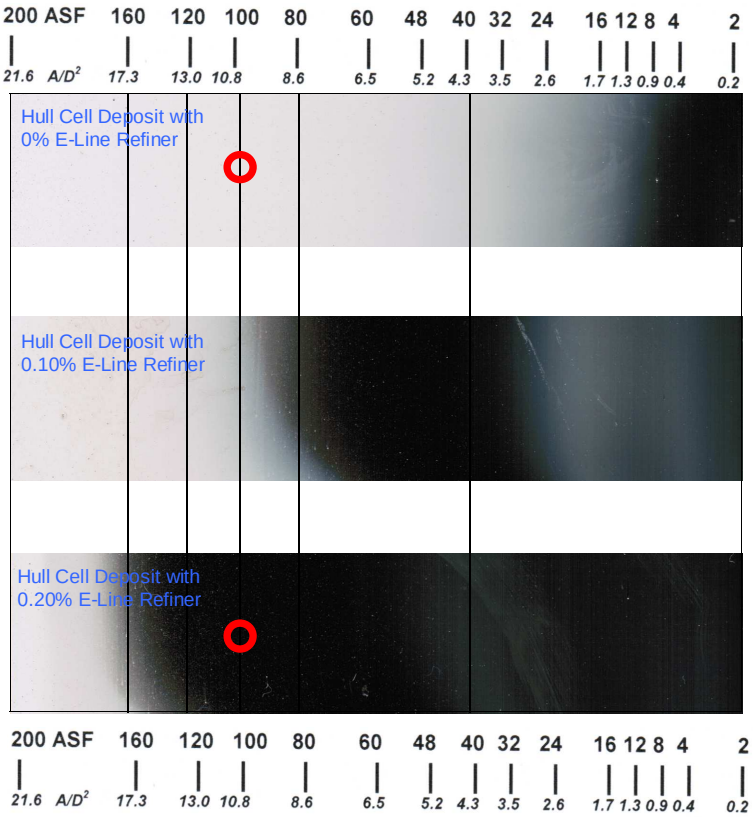
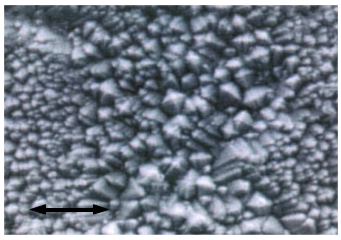
Deposit Crystalline Grain Structure

Electroplated / electroformed deposits are formed through the electrolytic deposition of nickel atoms carried from the anodes to the cathode[1]. The size and distribution of the resulting micro-crystalline nickel structures can vary significantly over current density. In addition to currently density, characteristics of the nickel micro-structures are strongly dependent of on bath composition, anode condition, temperature and pH.

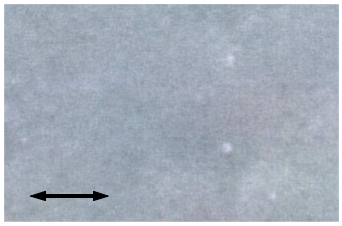
Grain size tends to increase with current density. This is believe to be due to decreasing electrolytic efficiency as current density increases. This results in a relative decrease in the availability of nickel ions at higher current densities. E-Line Refiner is thought to work by impeding active bonding sites at the cathode, providing for greater deposition efficiency at high current densities while encouraging planar (versus columnar) crystalline growth. Deposits formed in the presence of E-Line Refiner have been shown feature substantially refined crystalline grain structures with higher tensile strength, lower internal stress, greater ductility, and improved corrosion resistance. Furthermore, the planar growth of the nickel structures provides for a more amorphous surface finish.





5 µm Magn. 3000X

Nickel crystalline structure at 100 ASF.
No E-Line Refiner



5 µm Magn. 3000X

Nickel crystalline structure at 100 ASF.
0.20% v/v E-Line Refiner

Nickel Sulfamate Hull Cell deposit obtained with 0 - 0.20 %v/v E-Line pH
(Electroplating Bath Control Conditions: Nickel: 100 g/L, Boric Acid: 50 g/L,Temp 55℃, pH 4.0)

[1] *The source of nickel ions in sulfamate nickel electro-deposition is the anodes. This is not true of all electroplating formulations.*

MAINTENANCE AND CONTROL:

Since E-Line Refiner co-deposits with the nickel ions, it can be replenished on consumption per ampere hour basis. E-Line Refiner is typically consumed at a rate of 100 mL per 3000 – 4000 amp/hrs. In most cases, the E-Line Refiner concentration in the bath can be maintained by measuring and monitoring deposit hardness.

The E-Line Refiner concentration can also be determined through UV Spectrometer of Hull Cell testing[2].

E-Line Refiner Analysis By UV Spectrometer:

A. Equipment:

UV Spectrometer
1 cm Quartz Cell
5 mL Pipette
100 Volumetric Flask

B. Reagent:

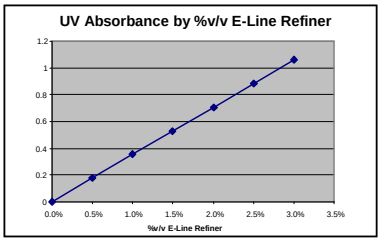
DI water

C. Procedure:

1. Create a calibration curve using fresh nickel sulfamate and E-Line Refiner to make the UV absorbance standards. Standards are made by filling a volumetric flask to 90 mL's of sulfamate nickel concentrate (180 g/L Ni) and adjusting the pH to 4.0 or above. It is critical that the pH be 4.0 or higher. Make six standard sets, adding the following amount of E-Line Refiner to each standard:

   a. 0.0% v/v (0 mL E-Line Refiner)
   b. 0.5% v/v (0.5 mL E-Line Refiner)
   c. 1.0% v/v (1.0 mL E-Line Refiner
   d. 1.5% v/v (1.5 mL E-Line Refiner)
   e. 2.0 % v/v (2.0 mL E-Line Refiner)
   f. 3.0% v/v (3.0 mL E-Line Refiner)

2. Compete each standard by filling the volumetric flask to 100 mL's.
3. Take a 5 mL sample of each standard and dilute to 100 mL using DI water.
4. Measure the absorbance of each diluted sample set using a 1 cm quartz cell at 270 nm, then plot the data to create the calibration curve.



5. Calculate the bath concentration of E-Line Refiner:

%v/v E-Line Refiner = Absorbance at 270 nm / 0.353

E-Line Refiner Determination By Hull Cell:

Maintained at 0.1% by volume, E-Line Refiner will provide a bright Hull Cell deposit below 60 ASF on a 2 ampere / 10 minute panel (see technical bulletin TECE9903. Where the Hull Cell panel brightness occurs below 60 ASF, additions are made, typically in increments of 0.02 % / volume, until the desire Hull Cell deposit characteristics are achieved. After monitoring the bath in this way, the required replenishment concentration can be correlated to ampere hours for automated feeder replenishment.

% / Volume E-Grain needed per amp hour =

$$\frac{\text{\% / Vol. E-Line Refiner required for Hull Cell replenishment}}{\text{Total amp hours used in plating bath}}$$

Note: when calculating amp hour consumption rates, be sure that replenishment occurs on the same scale as is used by the actual bath. In other words, amp hours per cell, amp hours per common sump, etc.

E-LINE REFINER REMOVAL

E-Line Refiner is readily removed from the bath by carbon treatment. Because wetting agents are preferentially removed by activated carbon, it is necessary to perform back to back carbon treatments where wetting agents are used.

[2] For information on Hull cell testing and equipment, contact your DisChem representative.

Back to Back Carbon Filtration:

The back to back carbon filtration process uses two stages of carbon filtration in combination with a peroxide breakdown process. Please note that the use of hydrogen peroxide will not cause an appreciable increase in the ammonium ion content as long as the peroxide is not used in quantities greater than list herein. This process may also be used for the removal of stubborn organic contaminants, but is not recommended as part of a normal bath maintenance procedure.

1. Carbon filter bath, 2 – 3 hrs, at normal operating temperature. All residual wetting agent will have been removed when the surface tension is greater than 65 dynes / cm.
2. Heat to 57℃ (135℉) and change filters.
3. Add 0.025 % by volume hydrogen peroxide (30%) and filter with agitation for 2 – 3 hours.
4. Heat to 65℃ (150℉) and maintain temperature for 1 hour to drive off residual peroxide. Caution: Exceeding 70℃ for several hours may cause a breakdown of the sulfamate nickel solution, releasing ammonium ion into the bath.
5. Cool to normal operating temperature.

PRODUCT AVAILABILITY

E-Line Refiner is available as a concentrate in 1 gallon (3.79L) and 5 gallon (19L) containers.

Part No.	Description
EFR05	E-Line Refiner, 5 Gallon (19L)

Ordering Information:

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MANUFACTURERS STATEMENT:

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