ENVIRALLOY NI 12-15

Alkaline Zinc Nickel Plating Process

Product Code: 174351
IMDS Number: 736126

DESCRIPTION

ENVIRALLOY NI 12-15 is an alkaline process for the electrodeposition of zinc nickel alloys with a nickel content of 12-15%.

It produces zinc nickel deposits with a consistent alloy composition and thickness across a wide range of current densities and operating conditions.

The zinc nickel deposit achieves exceptional corrosion protection with high temperature resistance that meets the demands of the automotive industry, even after heat treating (thermal shock). The deposit is compatible with light metals and is an ideal base for hexavalent chromium-free post treatments that meet the demands of the European Unions 'End of Life Vehicle' directive.

The process is easy to operate and control in volume production, being highly suited to barrel applications. Under optimum conditions, its high cathode efficiency ensures that fast plating rates can be achieved without high current density burning, dramatically improving production output.

READ ENTIRE TECHNICAL DATA SHEET BEFORE USING THIS PRODUCT

FEATURES & BENEFITS

- Consistent Nickel alloy
- Even thickness distribution
- High efficiency and plating rate
- Exceptional corrosion resistance even after heat treating
- Trivalent passivate post treatments

OPERATING PARAMETERS

	BARREL
Zinc	1.0 – 1.4 oz/gal
Nickel	0.85 – 1.8 g/l
Sodium Hydroxide	15.5 – 18.5 oz/gal
Sodium Carbonate [1]	< 9.5 oz/gal
Sodium Sulphate [1]	< 70 g/l
Cathode CD	3 – 10 A/ft^2
Temperature	25 – 30°C (77 – 86°F)
Filtration	Continuous filtration through 5 – 10µm filter is recommended (2 –3 turnovers per hour)

1) High carbonate and sulfate levels reduce efficiency and increase risk of burning at high cd. Combined sodium carbonate and sodium sulphate should not exceed 120g/l.

MAKE UP PROCEDURE

	BARREL
Isobrite 408 [2]	114 ml/l (11.5%)
Sodium Hydroxide	11.0 0z/gal
Envirozin Replenisher	10 ml/l (1%)
Enviralloy Ni 12-15 Pt A	25 ml/l (2.5%)
Enviralloy Ni 12-15 Pt B	40 ml/l (4%)

	BARREL
Enviralloy Ni 12-15 NiR	8 ml/l (0.8%)
Enviralloy Ni 12-15 Pt D[3]	3 – 5 ml/l (0.3- 0.5%)
Enviralloy Ni 12-15 LCD	0.2 ml/l (0.02%)
Envirowetter	1 ml/l (0.1%)

2) Isobrite 408 is a concentrated zincate base solution, containing 75 g/l (10 oz/g) zinc metal and 330 g/l (44 oz/g) sodium hydroxide (free caustic).

3) At zinc concentrations above 1.3 oz/gal Enviralloy Ni 12-15 Part D may be replaced with 0.2 ml/l of G2 Part D to achieve a smooth deposit.

Procedure

1. Select a suitable clean tank[4] and fill to 50% of working volume with water[5].
2. Add the required amount of Sodium Hydroxide and mix thoroughly.
3. Add the required amount of Isobrite 408 and stir to mix thoroughly.
4. Ensure the solution temperature is below 30°C (86°F)before proceeding with the solution make-up.
5. Start filter pump and circulation system
6. Make addition of the following additives:
   Envirozin Replenisher, Enviralloy Ni 12-15 Part A, Part B, NiR, Part D.
7. Add Envirowetter to reduce excessive mists and sprays.
8. Place steel anodes onto the anode bars and adjust solution level to the final working volume
9. Adjust solution temperature to within working window.
10. **Electrolysis of the solution up to 0.5 Ah/l is beneficial to achieve optimum brightness and performance.**

4) Tanks should be leached with a 5 – 10 g/l sodium hydroxide solution for a minimum of 4 hours prior to use.
5) In areas of poor water quality, deionized water can be used.

Note:
This make-up procedure will achieve a nominal base solution containing:

Zinc	Nickel	Sodium Hydroxide
8.5 g/L (1.13 oz/gal)	0.8 g/L (0.1 oz/gal)	120 g/L (16 oz/gal)

EQUIPMENT

Anodes

Steel
Mild steel anodes should be used in the plating tank. The recommended anodic current density is between above 40 ASF. Round or square shape steel bars are recommended, concaving shaped are beneficial.

Zinc
High purity zinc, minimum 99.99%, in steel cages. Zinc metal dissolution recommended through use of an externally mounted “zinc dissolving tank” as below.

Tank
Mild steel lined with alkaline resistant rubber, PVC or polypropylene or polyethylene.

Fiberglass linings are not recommended and should not be used.

Pipes
Should be constructed from alkaline resistant welded or threaded PVC, reinforced PVC/Polyester, polyethylene or polypropylene.

Cooling/Heating
Temperature control is required to ensure the solution remains within the operating range. Heat exchanges or heaters made from PTFE or titanium are recommended. Alternatively, steel sheathed electrical immersion heaters, steam or hot water coils are suitable.

Do not connect cooling coils directly to the main water supply.

Agitation
Uniform solution movement is recommended.
In barrel plating installations a rotation speed of 2-6 revolutions per minute is recommend.

Never use air agitation.

Filter
Continuous filtration is recommended at 2 to 3 solution volumes per hour.
Filter media must be alkaline resistant, rated at 10 microns.

Racks/Baskets
Polypropylene, polyethylene or stainless steel.

Fume Extraction

Required

Zinc Dissolving Tank

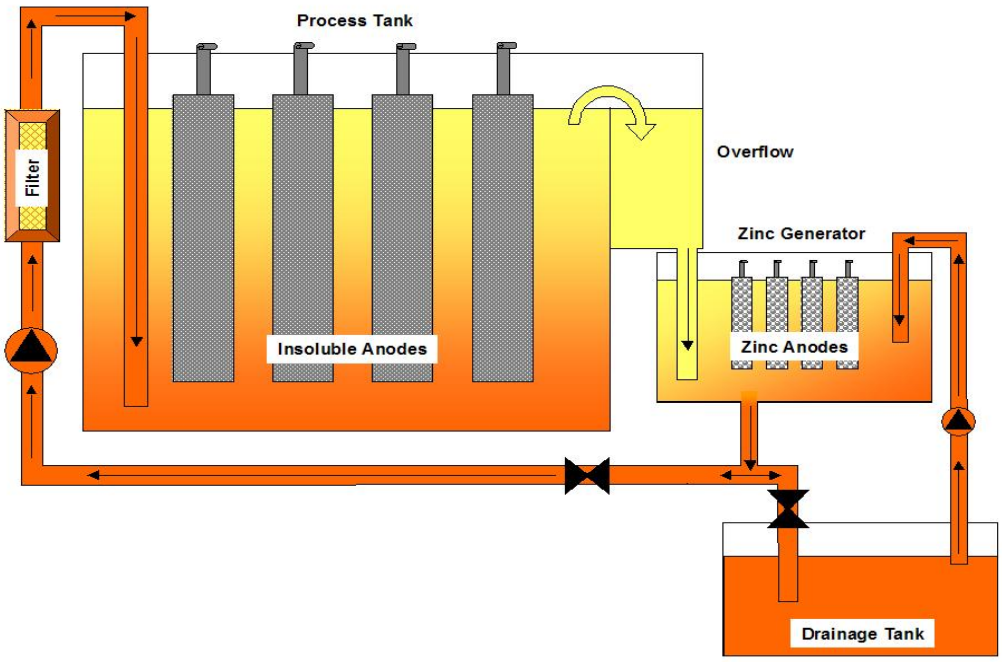
1. The use of a separate zinc dissolving tank is required when operating **Enviralloy Ni 12-15** in production applications, to enable optimum control of zinc metal in the plating solution. A typical volume of the dissolving tank system should be around 10 – 20% of the total volume of the plating tank.

2. The system should be designed to ensure the plating solution weirs from its tank into the dissolution unit, which is loaded with high purity zinc dome anodes contained in carbon steel mesh cages. The area of zinc under the solution should be adjusted until the concentration remains stable; this quantity is typically 10 times greater than daily usage.

3. Solution from the dissolution tank should then be transferred through a filter pump back into the plating tank.

4. **During periods without production, it is recommended that the zinc anodes are removed from the tankl.**

5. **It is essential that no chemical additions are made directly into the dissolving tank, therefore the use of a separate mixing tank solely for chemical additions is recommended or added directly to the plating tank**



SOLUTION MAINTENANCE

Enviralloy Ni 12-15 additive consumption rates are based upon practical experience. Adjustments may be necessary to address local operating conditions and substrate material types.

Process additives should be dosed through the use of ampere hour based automatic dosing equipment, and is highly recommended for all production installations. Additives should not be dosed directly into the zinc dissolving tank and should not be premixed prior to addition.

Increased consumption rates will occur if the plating solution is operated above the recommended temperature range.

	Barrel
Envirozin Replenisher	2 – 3 L per 25 kg Sodium Hydroxide
Enviralloy Ni 12-15 Pt B	0.75 – 2 L / 10 kAh
Enviralloy Ni 12-15 NiR	7 – 10 L / 10 kAh
Enviralloy Ni 12-15 Pt D #	1.5 – 2.5 L / 10 kAh
Enviralloy Ni 12-15 LCD	0.0 – 0.7 L / 10 kAh

The addition of 1 – 3 ml/l Enviralloy Ni 12-15 Part D is may necessary to achieve immediately good production results after longer production brakes.

Function of the Enviralloy additives:

Enviralloy Ni 12-15 Part A used on initial make-up, is added to improve the solubility of nickel in solution. An excess of this material will reduce cathode efficiency and lower plating rate.

Enviralloy Ni 12-15 Part B is added to improve the low current density performance of the solution. An overdose of part B will severely reduce the cathode efficiency.

Consumed by solution drag-out. Therefore, regular replenishment can be made based upon consumption of sodium hydroxide instead of auto dosing. In this case, the addition rate lies between 3 – 6 L **Enviralloy Ni 12-15 Part B** for every 25 kg Sodium Hydroxide added.

Enviralloy Ni 12-15 NiR is a concentrated version of the Part C nickel source. Consumed under electrolysis therefore regular ampere-hour dosing replenishment is recommended.

1 ml/l of NiR will increase nickel concentration in the solution by 0.1 g/l.

Enviralloy Ni 12-15 Part D is the main brightening component and refines the high current density areas of the deposit. Consumed under electrolysis therefore regular ampere-hour dosing replenishment is recommended.

G2 Part D is a leveling component which can replace Enviralloy Ni 12-15 Part D especially if the process is operated at zinc concentrations above 10 g/L. G2 Part D smoothens the deposit resulting in a more even and bright finish. Overdosing G2 Part D can cause stress in the deposit and reduced plating efficiency.

Addition rate: 0.5-1.2 L / 10 kAh

Enviralloy Ni 12-15 LCD is a secondary brightener additive to control extremely low current density areas.

Envirozin Replenisher is a water softening agent that does not contain strong chelators. Consumed by solution drag-out therefore regular replenishment should be made based upon consumption of sodium hydroxide or by amp hours.

Envirowetter

Used to overcome caustic fumes during instances of excessive mists and sprays from the electrolyte solution or zinc dissolving tanks.

OPERATING GUIDE

Carbonate and sulfate removal

Provision should be made for the periodic removal of carbonates and sulfates which build in alkaline based electrolyte solutions. Suitable off-line chilling is suggested to lower the solution temperature below 0^0C (32°F) to enable freeze-out of the carbonates.

Process Sequence

Effective cleaning of the substrate is essential to achieve good adhesion of the zinc nickel deposit. Approved alkaline cleaners must be used as the initial process step to remove heavy oil or grease. Substrates exhibiting rust or scale should be acid pickled prior to plating. If electrocleaning is used, the preferred method is to treat the components anodically. After suitable water rinsing, immersion into a dilute sodium carbonate or sodium hydroxide solution is recommended prior to zinc nickel electroplating in the **Enviralloy Ni 12-15** electrolyte solution.

After electroplating, components must be water rinsed. Application of subsequent post treatments provide colour and enhance functional properties of the deposit such as corrosion resistance.

Note: Please refer to MacDermid Technical Service for specific pre-plate sequences, as this will vary dependant upon the nature of the substrate and the degree of soiling.

PROCESS SEQUENCE

Typical pre-plate sequence for steel substrates:

- Soak clean
- Electrolytic clean (anodic)
- Water rinse 2x
- Acid pickle
- Water rinse 2 x
- 2nd Stage Electrolytic clean (anodic)
- Water rinse 2 x

Option A	Option B
• Alkaline dip – sodium carbonate or sodium hydroxide • **Enviralloy Ni 12-15** electroplate • Post treatment	• Mild Hydrochloric acid • Water rinse 2X

ANALYTICAL PROCEDURES

Concentration of Zinc and Nickel

The preferred method for Zinc and Nickel metal measurement is by **AAS**. If analysis by AAS is not possible, the following methods listed below can be used.

Concentration of Zinc Metal

Reagents:

- 0,1M EDTA volumetric solution
- Hydrochloric acid 12.5% solution
- Sodium Bifluoride
- Hydrochloric acid, concentrated
- Buffer solution pH 5.5 (90 g of sodium acetate and 15 ml pure acetic acid diluted to 1 L with DI water)
- Xylenol Orange Indicator

Titration Method:

1. Pipette a 5.0 ml** sample of the plating solution into a 250 ml flask and add 50 ml DI water.
2. Add 5 ml Hydrochloric acid 12.5% solution.
3. Add 1 – 2 g Sodium bifluoride.
4. Add 20 ml pH 5.5 buffer solution.
5. Add Xylenol Orange indicator.
6. Titrate immediately against 0.1M EDTA until a color change from a red to a yellow/orange end point.

Calculation:

ml of 0.1M EDTA *** x 0.173 = oz/gal Zinc

Concentration of Sodium Hydroxide

Reagents *

- Hydrochloric acid 1.0M solution
- 11-13 indicator = prepared as 4 – (4-nitrophenylazo) resorcinol (CAS No 74-39-5) 0.02% w/v in a 20% v/v solution of isopropanol in water.

Titration Method:

1. Pipette a 5.0 ml** sample of the plating solution into a 250 ml conical flask.
2. Add 25 ml DI water.
3. Add a few drops of 11-13 indicator
4. Titrate against 1.0M Hydrochloric acid until color changes from purple to a yellow end point.

Calculation:

ml of M Hydrochloric acid ***x 1.07 = oz/gal Free Sodium Hydroxide

* Unless otherwise stated, these will be analytical grade substances.
** Volumes given exact to a decimal point should be measured with a volumetric pipette.
*** For calculation purposes, volumetric solution factor assumed to be 1.0 and is therefore does not need to be taken into account.

Concentration of Nickel Metal

Reagents:

- Hydrochloric acid 12.5% solution
- Buffer solution pH 5.5 (90 g of sodium acetate and 15 ml pure acetic acid diluted to 1 L with DI water)
- Ammonia citrate 20% solution
- Gum Arabic 5%
- Nioxime 0.5% (freshly prepared)

Titration Method:

1. Pipette a 10.0 ml** sample of the plating solution into a 100 ml volumetric flask. Make to the mark with DI water.
2. Pipette 2.0 ml** of this diluted solution into a beaker.
3. Add 5 ml Hydrochloric acid 12.5% solution.
4. Add 20 ml pH 5.5 buffer solution, confirm pH is between 4 and 6.
5. Add 2 ml Gum Arabic solution followed by 2 ml of Nioxime 0.5% solution.
6. Dilute to 100 ml with DI water and stand for 15 min.
7. Measure 520 nm absorbance with a photometer.

Calculation:

Determine g/l of nickel using standard nickel curve.

* Unless otherwise stated, these will be analytical grade substances.
** Volumes given exact to a decimal point should be measured with a volumetric pipette.
*** For calculation purposes, volumetric solution factor assumed to be 1.0 and is therefore does not need to be taken into account.

Concentration of Sodium Sulfate

Equipment & Reagents

- Pipette 0.5 ml, 1 ml and 5 ml
- Volumetric flask 20 ml
- Beaker 50 ml
- Syringe and filter Sartorius Minisart NML 0.2 µm
- AAS, ICP or XRF
- Hydrochloric acid 5 mol/L (16%)
  Mix 500 mls concentrated hydrochloric acid with 500 mls DI water
- Barium acetate Solution 0.2 mol/L
  Dissolve 51.084 g Barium acetate in 1 L DI Water

Procedure

1. Pipette 5 ml DI water in a 20 ml flask
2. Add 1 ml Hydrochloric acid 5 mol/L
3. Add 0.5 ml Enviralloy Ni solution
4. Add 10 ml Barium acetate solution 0.2 mol/L. After the addition a thick white precipitate will form. Add DI water up to 20 mls.
5. Stir well and heat for 15 min in a water bath (70 – 90°C)
6. Filter (0.2 µm) some mls of the decant in a beaker
7. Determine the barium content of this solution by AAS, ICP or XRF

Calculation

- Sodium Sulfate concentration in g/L = 1.48*(383.6 - 28 x Barium concentration)
- This method works well in a concentration range of 30 -150 g/L Sodium Sulfate, deviation is below 10%.

Concentration of Sodium Carbonate

Equipment & Reagents

- Pipette 5 ml
- Conical flask 500 ml
- Barium chloride Solution 30%
  Dissolve 300 g Barium chloride-Dihydrate in 1 L DI Water
- Methyl orange Indicator solution 0.1%
  Dissolve 100 mg Methyl orange in 100 ml DI water
- Hydrochloric acid 1.0 N (mol/L) solution
- Sodium hydroxide 1.0 N (mol/L) solution

Procedure

1. Pipette 5 ml plating solution in a 500 ml flask
2. Add about 200 ml DI water
3. Heat to 70 - 90°C
4. Add 20 ml Barium chloride solution 30%. After the addition a thick white precipitate will form.
5. Stir well and boil for 15 minutes
6. Filter the solution well using a filter paper. Wash the barium carbonate precipitate in the filter well.
7. Place the filter paper and precipitate back into the conical flask and add about 200 ml DI water
8. Add 25 ml Hydrochloric acid 1.0 N solution and wait 10 minutes
9. Add some drops Methyl orange Indicator solution, the solution turns orange
10. Titrate with Sodium hydroxide 1.0 N solution to a yellow endpoint

Calculation

- (25 - ml Sodium hydroxide solution consumption) x 1.4 = oz/gal Sodium carbonate

Hull Cell Testing

Evaluation of the plating solution by the use of a Hull Cell is recommended during each production shift.

A 1A 20 minute Hull Cell steel test panel should be produced at 25°C (75°F) The resultant deposit can then be evaluated by visual inspection and by the measurement of thickness of the deposit at defined points.

The deposit thickness is measured by XRF at three locations:

A = 40 A/ft^2 B = 20 A/ft^2 C = 5 A/ft^2

It is essential to use a steel anode and a pre-cut template in order to remain consistent when evaluating deposit thicknesses.

Hull Cell 1A 20 min		
Zinc & Nickel Conc.:	Optimum	
Temperature:	77°F (25°C)	
A:C Ratio:	Optimum	
A	B	C
X	X	X
40ASF	20ASF	5ASF

Point	CD (A/ft^2)	Thickness (microns)	Nickel (%)
A	40	6.0 – 10.0	12 - 15
B	20	4.0 – 6.0	12 - 15
C	5	2.0 – 2.6	12 - 15

Ratio of thickness A:C should normally be in the range 3:1 to 4.5:1. In case of higher ratios, additions of Enviralloy Ni 12-15 Part B in 5ml/l increments can be tested in hull cell.

SAFETY & WARNING

MacDermid Enthone recommends that the company/operator read and review the Safety Data Sheets for the appropriate health and safety warnings before use.

Safety Data Sheets are available from MacDermid Enthone.

WASTE TREATMENT

Prior to using any recommendations or suggestions by MacDermid Enthone for waste treatment, the user is required to know the appropriate local/state/federal regulations for on-site or off-site treatment which may require permits. If there is any conflict regarding our recommendations, local/state/federal regulations take precedent.

ORDER INFORMATION

Product	Code
Isobrite 408	118326
ENVIRALLOY NI 12-15 Part A	174351
ENVIRALLOY NI 12-15 Part B	174352
ENVIRALLOY NI 12-15 NiR	187282
ENVIRALLOY NI 12-15 Part D	174354
ENVIRALLOY NI 12-15 LCD	187331
G2 Part D	187798
Envirozin Replenisher	174379
Envirowetter	174371

CONTACT INFORMATION

To confirm this is the most recent issue, please contact MacDermid Enthone

macdermidenthone.com/industrial

North America
245 Freight Street
Waterbury, CT 06702, USA
203.575.5700
americas@macdermidenthone.com

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