

SurTec® 865

Acid Copper

Properties

- produces mirror bright deposits with excellent levelling and superior throwing power, especially at lower current densities
- ductile and low-stressed copper layers
- well suited for parts with deep recesses or complicate shapes
- for rack and barrel plating
- economical process: very efficient additives and low maintenance cost
- not sensitive to pitting or pin holes
- stable brightener system with much reduced break down products
- additives have different colours, to prevent any interchanges during addition
- IMDS-number: 736943

Application

The process SurTec 865 includes the following products:

- SurTec 865 M Make-Up
- SurTec 865 I Leveller
- SurTec 865 II Brightener

make-up values:

copper sulfate · 5 H ₂ O	200	g/l	
sulfuric acid, pure (96 %)	60	g/l	
sodium chloride, pure	100	mg/l	
SurTec 865 M Make-Up	6	ml/l	(4-8 ml/l)
SurTec 865 I Leveller	0.4	ml/l	(0.2-0.6 ml/l)
SurTec 865 II Brightener	0.4	ml/l	(0.2-0.6 ml/l)

analytical values:	copper	50	g/l	(45-55 g/l)
	sulfuric acid	60	g/l	(50-90 g/l)
	chloride	60	mg/l	(30-120 mg/l)

make-up:

Steps for make-up:

1. Dissolve the calculated amount of copper sulfate in a separate tank in 50 % of the deionised water.
2. Heat it up to 50-60°C, stirring thoroughly.
3. Filter the solution into a working tank through an activated carbon cartridge.
4. At 38-43°C add 1-1.5 ml/l hydrogen peroxide (30 %) and stir for 2 hours at this temperature.
5. Raise the temperature to 70°C and stir 2 hours, in order to remove excessive peroxide (rest of peroxide will disturb the copper plating!).

6. Add 4-6 g/l granular activated carbon and stir another 2 hours (still at 70°C).
7. Allow to settle for 8-12 h, then filter the solution carefully into the plating tank (rest of activated carbon will lead to rough deposits!).
8. Add sulfuric acid carefully (attention: solution becomes hot, safety goggles must be worn!)
9. At 25°C add the pre-dissolved sodium chloride.
10. Finally add the additives SurTec 865 M, SurTec 865 I and SurTec 865 II.

temperature:	23°C	(20-30°C)
pH-value:	< 1	does not need to be monitored
deposition rate:	0.65 µm/min at 3 A/dm ²	
cathodic current density:	3 A/dm ²	(1-6 A/dm ²)
bath voltage:	1.0-3.5 V (larger tanks may need up to 6 V)	
anodes:	copper anodes (99.9 %), containing 0.03-0.06 % phosphorous anode bags out of acid resistant material are needed	
agitation:	oil free air agitation of approx. 10-20 m ³ /h per meter cathode length, additional rack agitation can be used	
tank material:	steel with hard rubber coating, reinforced with PVC or PE/PVC	
filtration:	PP cartridge, 2-3 times the bath volume per hour; pore size: 5-10 µm; filter aid: cellulose	
heating:	if necessary, out of Teflon, titanium, PE or PVC	
exhaust:	required	
hints:	Until a stable anode film is formed, the chloride content should be analysed regularly.	

Technical Specification

(at 20°C)	Appearance	Density (g/ml)	pH-value (conc.)
SurTec 865 M	liquid, green, clear	1.030 (1.01-1.05)	< 2
SurTec 865 I	liquid, dark violet, clear	1.015 (1.00-1.03)	< 2
SurTec 865 II	liquid, blue, clear	1.035 (1.01-1.06)	< 2

Maintenance and Analysis

Replenish evaporation losses with deionised water.

Analyse and adjust the concentration of copper, sulfuric acid and chloride regularly. Add the additives according to Hull cell tests. To reduce the break down products and the dragged in impurities, activated carbon treatments are recommended in regular intervals.

Sample Preparation

Take a sample at a homogeneously mixed position. Let it cool down to room temperature. If the sample is turbid, let the turbidity settle down and decant or filter the solution.

Copper – Analysis by Titration

- reagents: 5 mol/l ammonia solution
0.05 mol/l EDTA solution (Titrplex III)
indicator: murexide (1:100 in NaCl)
- procedure: 1. Pipette 10 ml bath sample into a 250 ml volumetric flask.
2. Fill up to the mark and mix well.
3. From this dilution pipette 25 ml into a 500 ml Erlenmeyer flask.
4. Dilute with approx. 250 ml deionised water.
5. Add so much of the diluted ammonia solution, until the colour of the solution turns to deep blue.
6. Add a spatula tip of indicator.
7. Titrate with 0.05 M EDTA from reddish-yellow to deep violet.
- calculation: consumption in ml · 3.177 = g/l copper
- correction: rise by 1 g/l copper = addition of 3.9 g/l $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$
- note: If the iron content is too high in the electrolyte, there will be a precipitation of iron hydroxide while adding the ammonia solution. The analysis has to be done again, adding 3 g Seignette's salt before adding the ammonia solution.

Sulfuric Acid – Analysis by Titration

- reagents: 1 N sodium hydroxide solution (NaOH solution)
indicator: methyl orange solution (0.04 %)
- procedure: 1. Pipette 10 ml bath sample into a 250 ml Erlenmeyer flask.
2. Dilute with approx. 100 ml deionised water.
3. Add 5 drops of indicator solution.
4. Titrate with 1 N NaOH solution until the colour changes.
- calculation: consumption in ml · 4.9 = g/l sulfuric acid (H_2SO_4)

Chloride – Analysis by Titration

- reagents: 0.01 N mercuric(II) nitrate solution
0.1 N silver nitrate solution
diluted nitric acid (1:1)
- procedure: 1. Pipette 25 ml bath sample into a 250 ml Erlenmeyer flask.
2. Dilute with approx. 30 ml deionised water.
3. Add 30 ml diluted nitric acid.
4. Add 3-5 drops of 0.1 N silver nitrate solution to form a steady turbidity.
5. Titrate immediately under strong stirring with 0.01 N mercuric(II) nitrate solution until the turbidity clarifies.
- calculation: consumption in ml · 14.2 = mg/l chloride
- correction: rise by 10 mg/l chloride:
addition of 23.4 ml/l hydrochloric acid (37 %)
or:
addition of 16.48 mg/l NaCl
- The chloride content can be adjusted with NaCl or with HCl (both pure p.a. quality).
- note: The analysis of chloride can also be done by measuring the potential difference during the titration with silver nitrate (Hg-free method). Please ask for the description at SurTec.

Hull Cell Test

- material: rectifier with 10-30 V and 0-2.5 A
cables
air-agitated 250 ml Hull cell, phosphorized copper bar anode,
polished brass Hull cell panels (scratched in the middle with type
"000" emery paper)
- procedure:
1. Put the clean anode (evtl. activated in HCl and rinsed well) into the Hull cell and connect with the cable to the (+) pole of the rectifier; fill the cell with the original copper bath up to the Hull cell mark.
 2. Remove the plastic film of the brass panel mechanically and scratch it in the middle with the emery paper.
 3. Electro-clean the panel, immerse it into an acid dip, rinse well and put it into the cell. Move slightly to and fro in order to wet the panel properly. Then connect with the cable to the (-) pole of the rectifier.
 4. Use an aquarium pump for air agitation and plate the panel at 2 A for 10 minutes (full voltage, current adjusted to the desired value).
 5. Take out the panel, rinse it well and dry it with hot or compressed air.
- If the analysis of the bath values indicated that some inorganic ingredient should be adjusted, plate a second panel with these corrections.
- evaluation: A correct SurTec 865 panel should be completely bright and levelled.
- Make the corrections by the help of the trouble shooting list.

Consumption and Stock Keeping

The additives are consumed by drag-out as well as electrochemically, anodic oxidation and cathodic build-in. To determine the exact amounts of drag-out, see [SurTec Technical Letter 11](#).

The following values per 10,000 Ah can be taken as estimated average consumption:

SurTec 865 M	1.0-1.5 l
SurTec 865 I	1.5-2.5 l
SurTec 865 II	1.5-2.5 l

In order to prevent delays in the production process, per 1,000 l bath the following amounts should be kept in stock:

SurTec 865 M Make-Up	30 kg
SurTec 865 I Leveller	60 kg
SurTec 865 II Brightener	60 kg

Product Safety and Ecology

The safety instructions and the instructions for environmental protection have to be followed in order to avoid hazards for people and environment. The Material Safety Data Sheets (according to European legislation) contain explicit details for this.

The following hazard designations and classifications into water hazard classes (WHC) have to be taken into account:

<u>product</u>	<u>hazard designation</u>	<u>water hazard class</u>
SurTec 865 M	Xi - Irritant N - Dangerous for the environment	WHC 1
SurTec 865 I	-	WHC 1
SurTec 865 II	Xi - Irritant N - Dangerous for the environment	WHC 1

Warranty

We are responsible for our products in the context of the valid legal regulations. The warranty exclusively accesses for the delivered state of a product. Warranties and claims for damages after the subsequent treatment of our products do not exist. For details please consider our [general terms and conditions](#).

Further Information and Contact

In our forum, you can discuss topics of the surface technology:

<http://forum.SurTec.com/>

If you have any questions concerning the process, please contact your local technical department: <http://SurTec.com/International.html>

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Trouble Shooting

problem	possible cause	remedy
deposit tends to burn	a) bath temperature is too low (below 20°C)	increase the temperature to 24-28°C
	b) copper content is too low	increase the anode surface or add copper sulfate
deposit is relief-like in the hcd	deficit of chloride ions	analyse and adjust the chloride content
matt deposit in the lcd	a) bath temperature is too high (above 30°C)	cool the electrolyte
	b) organic impurities	add 50-100 mg/l sodium persulfate
levelling effect is too low	excess of chloride ions (more than 150 mg/l)	add 1 % silver sulfate solution (4.4 ml/l precipitate 10 mg/l chloride)
electrolyte responds badly to brightener additions	organic contamination	add 50-100 mg/l sodium persulfate and treat the electrolyte with 5-10 g/l active carbon; then adjust by Hull cell test
consumption of leveller is too high	a) bath solution is too warm	cool the electrolyte
	b) concentration of leveller and brightener are not in correct ratio	adjust the additives to correct ratio, using Hull cell tests
	c) filtering aid is unsuitable or excessive amount used	use filter aids (amount: 300-500 g/qm filter area)
	d) excessive sludge on the anodes	anodes unsuitable or anodic density is too high (not above 2.5 A/dm ²)
nickel does not adhere to the copper deposit	excess of leveller (more than 0.8 ml/l)	after copper plating electro-clean and reduce the addition of leveller
copper does not adhere to the nickel strike deposit	nickel solution contains sulfurous compounds	use sulfur-free semi-bright nickel
deposits are covered with fine pores	a) pump is sucking air	remove pump suction pipe from zone of agitated air
	b) air agitation system is incorrect	jets of air agitation pipes are too small; diameter must be at least 3 mm
deposit covered with particles	a) electrolyte is contaminated with suspended particles (e.g. activated carbon)	filter the electrolyte continuously and use the recommended filter aids
	b) the injected air is contaminated (oil, dirt)	check oil and dirt filter, use blower
	c) burnings on the parts (amorphous copper)	see under "deposits tend to burn"
	d) the copper sulfate replenishing solution is not filtered adequate	filter the copper sulfate solution as described under make-up
	e) unsuitable anodes (e.g. P-free anodes, seen at the dark red anode sludge)	use only phosphorous containing anodes
anodes are passive	a) sulfuric acid or copper content is too high	dilute electrolyte and adjust by Hull cell tests
	b) chloride content is too high	add 1 % silver sulfate solution (4.4 ml/l precipitate 10 mg/l chloride)
	c) anode bags are clogged or too fine	clean or replace the anode bags
	d) electrolyte is contaminated with lots of iron; therefore bath density is too high	dilute electrolyte and adjust by Hull cell tests