SurTec® 691
Black Passivation for Alkaline Zinc Plating

Properties

- hexavalent chromium free black passivation based on chromium(III)
- specially developed for plating of bulk commodity
- produces uniform black layers on alkaline zinc coatings
- excellent corrosion protection and friction coefficient, comparable to hexavalent passivations
- easy waste water treatment
- usable with conventional equipment
- IMDS-number: 174923051

Application

SurTec 691 is used for barrel application.
The process includes the following products:

- SurTec 691 I Make-up Solution
- SurTec 691 II Make-up Additive
- SurTec 691 III Maintenance Additive
- SurTec 691 IV Maintenance Solution

make-up values:

SurTec 691 I Make-up Solution	8 %vol	(7 - 9 %vol)
SurTec 691 II Make-up Additive	2 %vol	(1.5-3 %vol)

analytical value: chromium 2.65 g/l
(for a make-up concentration of 8 %vol SurTec 691 I)

temperature: 25°C (15-35°C)

pH-value: 2.3 (2.0-2.5)
adjust with nitric acid (HNO_3)
or caustic soda

application time: 30 s (20-40 s)

agitation: barrel rotation

tank material: lined with PVC or PP

make-up: Steps for make-up:

1. Fill the calculated amount of SurTec 691 I Make-up Solution into the tank.
2. Dilute with deionised water to about 90 % of the final volume, mixing well.
3. Add the calculated amount of SurTec 691 II Make-up Additive slowly under continuous stirring.
4. Fill up to the final volume with deionised water.
5. Check the pH-value and adjust if necessary.

recommended process sequence:

1. alkaline zinc process SurTec 704
2. cascade rinsing
3. activation in 0.5 %vol nitric acid, pH 1.4-2.0
4. rinsing
5. **Black Passivation SurTec 691**
6. cascade rinsing
7. post-dip SurTec 544 or sealing e.g. SurTec 555 S
   (SurTec 544 I: 150 ml/l, SurTec 544 IV: 5-10 ml/l; at **40-45°C**)
   min. 10-15 s dropping time prior to drying
8. hot air drying 60°C (40-70°C)

The rinsing methods have to be adapted to the plating line.

Technical Specification

(at 20°C)	Appearance	Density (g/ml)	pH-value (conc.)
SurTec 691 I	liquid, dark violet	1.224 (1.17-1.28)	< 1
SurTec 691 II	liquid, colourless	1.205 (1.15-1.25)	2.8 (2.5-3)
SurTec 691 III	liquid, colourless	1.226 (1.18-1.27)	2.5 (2-3)
SurTec 691 IV	liquid, dark violet	1.255 (1.21-1.29)	< 1

Maintenance and Analysis

Check the pH-value regularly. Analyse the concentration of chromium regularly and adjust by adding SurTec 691 IV Maintenance Solution.

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.

Chromium – Analysis by AAS

equipment: atomic absorption spectrometer (AAS)
wave length: 357.9 nm

reagents: hydrochloric acid (1:1)
chromium standard solutions

procedure: Prepare a device-specific dilution, e.g. 1:5000:

1. Pipette 2 ml bath sample into a 100 ml volumetric flask.
2. Add 50 ml deionised water.
3. Acidify with 2 ml half conc. hydrochloric acid.
4. Fill up to 100 ml with deionised water.
5. Pipette 5 ml from this dilution into a 500 ml volumetric flask.
6. Add approx. 5 ml half conc. hydrochloric acid.
7. Fill up to 500 ml with deionised water.
8. Measure against chromium standard solutions using an AAS.

correction: rise by 0.1 g/l chromium = addition of 3.1 ml/l SurTec 691 IV

hints: Prepare the dilution of the sample according to the specified range of the measuring device.

The measuring can also be done via ICP.

Chromium – Analysis by Photometry

equipment:
spectrophotometer or
filter photometer with 371 nm filter unit (± 50 nm)
50 ml and 100 ml volumetric flask
100 ml beaker
blue ribbon filter
1 cm cuvette

reagents:
NaOH solution (10 %)
H_2O_2 (30 %)

procedure:
Prepare a chromium standard with SurTec 691 concentrate in a 100 ml volumetric flask:

Fill 8 ml SurTec 691 I and 5 ml SurTec 691 II up to 100 ml with deionised water.

For each analysis prepare and measure the standard in the same way as the bath sample (standard = absorbance **S**).

Sample preparation:

1. Pipette 10 ml bath sample into a 100 ml volumetric flask.
2. Fill up with deionised water.
3. Pipette 2.0 ml of this dilution into a 100 ml beaker.
4. Add:
   10 ml deionised water,
   0.2 ml NaOH solution and
   2 ml H_2O_2 (30 %)
   and mix by rotation.
5. Cover the beaker with a watch glass and let react for 5 minutes (gas evolution!).
6. Then heat up the solution and boil for 1 minute, and let it cool down.
7. Pour the solution quantitatively into a 50 ml volumetric flask, fill up with deionised water and mix.

Sample measurement:

1. Filtrate the so prepared solution through a blue ribbon filter.
2. Fill the filtrated solution into the same 1 cm cuvette that was used for measuring the standard.
3. Measure it in the photometer at 371 nm (bath sample = absorbance **P**) against deionised water.

calculation:
2.61 · (absorbance **P**) / (absorbance **S**) = g/l chromium

correction:
rise by 0.1 g/l chromium = addition of 3.1 ml/l SurTec 691 IV

Chromium – Analysis by Titration

Important:	While preparing the samples, nitrous gases will be formed – therefore an extractor hood is absolutely essential!
reagents:	sulfuric acid (conc.) silver nitrate solution (ca. 0.3 mol/l) ammonium peroxodisulfate potassium iodide 0.1 N sodium thiosulfate solution (= 0.1 mol/l) starch solution (1 %)
procedure:	1. Pipette 5 ml bath sample into a 250 ml beaker. 2. Acidify with 5 ml sulfuric acid. 3. Heat up the solution and boil for approx. 35 minutes, until no nitrous gases will be formed any more. 4. Let it cool down and fill up to 100 ml with deionised water. 5. Add 5 ml silver nitrate solution. 6. Add 2 g ammonium peroxodisulfate. (The solution becomes yellow/brown.) 7. Heat up again and boil for at least 20 min (at least 60 ml of the solution should remain in the beaker). 8. Add 2 g potassium iodide. 9. Titrate with 0.1 mol/l sodium thiosulfate solution to a weak yellow colour. 10. Add a few drops of starch solution (colour changes to blue). 11. Continue titrating until the colour disappears.
calculation:	consumption in ml · 0.347 = g/l chromium
correction:	rise by 0.1 g/l chromium = addition of 3.1 ml/l SurTec 691 IV

Consumption and Stock Keeping

The consumption depends heavily on the drag-out. To determine the exact amounts of drag-out, see SurTec Technical Letter 11.

The following values per m^2 can be taken as estimated average consumption:

SurTec 691 III	10 ml
SurTec 691 IV	40-50 ml

(ratio SurTec 691 III : SurTec 691 IV = 1:4-1:5)

The following values per 100 kg bulk commodity can be taken as estimated average consumption:

screws (100 kg)	SurTec 691 III	SurTec 691 IV
size M3	0.40 l	1.6-2.0 l
size M4	0.30 l	1.2-1.5 l
size M6	0.20 l	0.8-1.0 l
size M8	0.15 l	0.6-0.75 l

hint: Only if the pH-value of the passivation solution is already too low (1.8-2.0), add SurTec 691 I instead of SurTec 691 IV or a mixture of both (10 ml/m^2 SurTec 691 I + 30 ml/m^2 SurTec 691 IV).

contamination limits:

zinc (Zn):	15 g/l
iron (Fe):	200 ppm
copper (Cu):	5 ppm

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

SurTec 691 III	25 kg
SurTec 691 IV	125 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:

product	*hazard designation*	*water hazard class*
SurTec 691 I	T - Toxic N - Dangerous for the environment	WHC 2
SurTec 691 II	C - Corrosive	WHC 1
SurTec 691 III	C - Corrosive	WHC 1
SurTec 691 IV	T - Toxic N - Dangerous for the environment	WHC 2

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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