NB SEMIPLATE SN 150

Pure tin plating process

INTRODUCTION

NB SEMIPLATE SN 150 is a high-purity electroplating process which produces fine-grained, matte, pure tin deposits. It is especially formulated for use in the fabrication of circuit patterns and bumps on semiconductor wafers. The process contains no fluoborates or formaldehyde.
This product is specially designed for the operation with **inert anode** systems without negative impact on plating quality or sensitive compounds such as organic additives. However, the operation using soluble anodes is not excluded. The solution is available as ready-to-use solution and can be used with either soluble or insoluble anodes.

READ ENTIRE TECHNICAL DATA SHEET BEFORE USING THIS PRODUCT.

MATERIALS REQUIRED

The following materials are normally recommended for a typical start up and operation:

Product Name	Product Coed	Comment
NB SEMIPLATE Sn 150	202150-00	• Ready-to-use solution; contains tin, additives and methanesulfonic acid in nominal values
SN 150 REFINER	202150-50	• grain refiner to produce fine grained, matte deposits
SN 150 REPLENISHER	202150-10	• Replenisher used for replenishing tin metal and additives (refill or bleed and feed modus); contains 125% of the NB SEMIPLATE SN 150 contents
SN 150 ACID	202150-30	• high purity solution containing 942 g/l free methanesulfonic acid. It provides acid for the operating solution and is necessary for solution stability and conductivity.
SN 150 TIN CONC	202150-20	• is a high purity concentrate containing 300 g/l tin. It provides the tin ion concentration for heavy drag out situations, if regular replenishment using the SN 150 REPLENISEHR is not sufficient
SN 150 P CONC	202150-40	• consist of Sn 150 REPLENISHER plus high concentrated inert anode conditioner (IAC) for operation with inert anode system
SN 150 MAKE UP	202150-60	• make up solution identical to the ready to use solution NB SEMIPLATE Sn 150, but without any SN 150 REFINER

Materials are purified and packaged for semiconductor applications in clean room compatible packages.

EQUIPMENT REQUIRED

The following section is a guide for usual operation conditions. The specific conditions and requirements may depend on tool vendor specifications and application.
Acidic solutions are highly corrosive. Therefore, exposed metal materials in the fab area should be protected from the effects of these solutions. Several coats of a vinyl coating can provide adequate protection.

Tanks PVC, PVDC, polypropylene or Teflon tanks can be used.

Leaching	Tanks, filter cartridges, anode bags, and peripheral equipment must be leached prior to installation. Depending on tool status, this may include degreasing, base solution treatment, DI water rinse, acidic treatment and final DI-water rinse.
Agitation	Solution agitation is necessary to achieve the best results. Solution agitation, without air, is recommended. Increased solution flow rate can be important for uniform plate distribution and plating rate.
Heating and Cooling	Cooling coils may be considered for temperature adjustment. Cooling and heating coils made of titanium or Teflon-coated copper may be used. Teflon tube bundles, immersion type heat exchangers or external heat exchangers are preferred.
Filtration	Continuous filtration is necessary for maintaining low particle counts of the solution. Use woven Dynel or polypropylene filter cartridges (with a polypropylene core) with a 5 micron or less retention. Cotton filters must not be used. Filter cartridges must be leached before installation in the tool (refer to next section).
Ventilation	Ensure sufficient exhaust (acidic mists) and check with local regulations.
Rectifiers	Direct current or pulse rectifiers (direct or reverse mode) may be used. Make sure to use power supply without current ramping and ripple less than 2%. Consult NB Technologies GmbH for specific application recommendations.
Anodes	Boron Doped Diamond anodes (BDD anodes) are preferred as inert anode system. As an alternative, platinated titanium can be used, however, gas evolution and protective additive consumption may be higher. Inert anodes must be degreased and clean. If soluble anodes are used, titanium baskets with new anode bags made of PP are recommended. New anode bags have to be leached out. Do not introduce anode bags used before. Anode material: Sn-Domes or Sn-Wire. Use materials with high purity only. Metallic impurities have negative impact on the plating process.

MAKE UP PROCEDURE

1. Proper leaching and cleaning of the tank is mandatory. Depending on the tool status (used or first time of use), the tank must be leached with a solution containing 45 g/l trisodium phosphate and 7.5 g/l sodium hydroxide heated to 60 °C for 4 to 8 hours. Scrub tank lining with solution to remove any dirt, oils or surface soils. Be careful to flush thoroughly with several rinses to remove all residues of sodium (filled and drained).
2. Then leach with 10% by volume SN 150 ACID heated to 50°C for 8 hours. Again flush tank with water.
3. Empty the tank.
4. Soluble and inert anodes may be installed after degreasing with NaOH, short soak in 10% by volume SN 150 ACID and thorough cleaning with DI-water.
5. Leach string wound polypropylene filter cartridges at this time by immersing in boiling DI-water for 30 minutes, followed by thorough rinsing with deionized water. This process must be repeated until there is no evidence of foam or turbidity of the boiling water. The cartridges must then be immersed in a solution of 10% by volume SN 150 ACID to which 20 ml/l of SN 150 REFINER has been added. The cartridges must be allowed to soak for 1 hour. For sub-micron filters follow the manufacturer recommendation for preparation prior to installation.
6. Install leached filter cartridges.
7. Carefully pour or pump recommended amount of NB SEMIPLATE SN 150 solution (or 80% SN 150 REPLENISHER plus 40ml/l SN 150 ACID plus 20% DI-water; or SN 150 MAKE UP + 38ml/l SN 150 REFINER) into the tank and start filtering the solution. The solution is now ready for production operation.
8. Take a sample of the solution to check for final additive concentrations, optionally send to NB Technologies GmbH for analysis.

BATH PARAMETERS

The following table shows the bath parameters, which should be maintained and checked with regular sample analysis.

	NB analysis	Units	Max. upper limit	Upper action limit	Optimum	Lower action limit	Lowest limit
Sn^{2+} concentration	X	g/l	45	40	35	32	30
SN 150 ACID	X	ml/l	230	220	200	185	175
SN 150 REFINER	X	ml/l	70	50	38	25	20
Inert anode conditioner (IAC)	X	ml/l	100	75	50	37.5	25

PLATING CONDITIONS

Parameter		**Optimum**	**Range**
Cathode current density	[A/dm²] (ASD)	3	1 - 4
Flow (depending on tool)	[l/h]	-	500 - 1000
Anode to cathode area ratio BDD/platinated titanium soluble anode		 4:1 3:1	 3:1 or higher or higher
Anode to cathode spacing (depends on tool and wafer size)	[cm]		5 - 15
Temperature	[°C]	24	20 - 27

DEPOSITION RATES:

The bath yields 2.21 g Sn per Ah. The efficiency is 99.9% up to 3 ASD.

Current density [A/dm²]	Layer thickness per 1minute plating time [µm]	Time needed for 1µm deposit [min]
1 ASD	0.51 µm	1:57
2 ASD	1.02 µm	0:58
3 ASD	1.53 µm	0:39

OPERATION

SN 150 REPLENISHER

SN 150 REPLENISHER is a ready to use solution with higher concentration of tin metal and all additive contents, so that it can be used for replenishing the tin metal and additives in a bleed and feed modus. The SN 150 REPLENISHER contains 125% of the NB SEMIPLATE SN 150 solution (44 g/l tin, 47.5 ml/l SN 150 REFINER, 62.5 ml/l inert anode conditioner (IAC).

NOTE: There is no increased content of SN 150 ACID in the SN 150 REPLENISHER. SN 150 ACID content is at nominal value of 200 ml/l corresponding to 188.4 g/l methanesulfonic acid.

SN 150 REFINER

The consumption rate of SN 150 REFINER will be a function of the type of plating equipment used. However, the replenishment rate will normally be 0.2 to 0.5 ml per Ah. If deposits start to show coarse grain, SN 150 REFINER may be added in 5 ml/l to 10 ml/l increments. A slight overdose is not critical.

SN 150 P CONC / inert anode additive (IAC)
The inert anode conditioner (IAC) is needed for the operation with inert anode systems. The IAC normally is sufficiently replenished with the replenishments using SN 150 REPLENISHER. SN 150 P CONC contains 500 ml/l IAC and is added only upon analysis result, when heavy consumption of the IAC was not compensated with regular replenishments.

SN 150 ACID
The acid concentration should not fall below 70ml/l. For replenishment add the correspondent volume of SN 150 ACID.
Low levels of acid concentrations effect higher anode potential, poorer uniformity and higher rate of tin(IV) generation. Higher levels of acid concentration reduce the maximum plating current density, but do not impact the plating quality in general.

SN 150 MAKE UP
Sn 150 MAKE UP is used for mixture. It is identical to the ready to use solution NB SEMIPLATE Sn 150, but contains no SN 150 REFINER.

Tin concentration **when using soluble anodes**
The tin concentration of the electrolyte will change slightly with use and time. If there is an excessively high anode to cathode ratio, or if the solution is infrequently used, the concentration of tin in the electrolyte will rise steadily. When a solution is used infrequently and/or is taken out of service for longer than 2 weeks, remove all anodes and store in a tank of clean, DI-water. If left in the electrolyte, the high free acid will dissolve the tin.

Temperature
Control the temperature of the solution between 20 and 27 °C. The optimum operating temperature is 24 °C. Lower temperature effects better thickness uniformity, but limits the maximum current density to be used and effects bigger grain size. Higher temperature enables higher plating currents and finer grains, but effects poorer uniformity.

Filtration
Continuous filtration for the removal of particulate matter is strongly recommended. Clean and leach cartridges or filter bags prior to use according to the solution make-up section of this document. Do not operate continuously with carbon filter cartridges, or addition agent will be removed from the solution.
Capacity of the pump and filter must be sufficient to turn over the complete volume of solution at least once per hour, preferably two or more times per hour. Pumps, fittings, pipes, valves, connections and filter must be of inert acid resistant materials. Plastic and hard rubber are recommended for pumps. PVC, PVDC, polypropylene and approved grades of rubber are suitable materials of construction for filter chambers and baffles.

Anodes
Maintain the anode area in the indicated ratio to the cathode area (wafer). Lower anode to cathode area ratios may effect undesired gas generation and may cause defects especially in fountain plater arrangements. Exercise care in the original determination of the anode area and take into consideration the increase in area due to fine features including vias and trenches. Anodes facing tank walls are only 85% anodically effective of their full surface area.

Soluble anodes:
Establish a maintenance program to replace anodes as consumed to keep the anode to cathode ratio within the operating limits.

Anode-to-Cathode Spacing
Normal anode-to-cathode spacing for wafer plating is 5 to 15 cm depending on wafer size and anode shape.

Current Density
The normal current density range of 10 to 30 mA/cm² (1.0 to 3.0 ASD) is recommended for most applications.

CONTAMINATION PRECAUTIONS

Make sure to prevent sulfuric acid to enter the bath. The bath will be destabilized at small concentrations, such as 0.4g/l H_2SO_4 or 4ml per liter H_2SO_4 (10%).

REPLENISHMENT & MAINTENANCE

Sn^{2+} concentration

When inert anodes are used, the tin concentration needs to be replenished according to the amount of metal plated or Ah processed.

There are two options to replenish the solution:

Option 1: Replenish by bleed and feed using SN 150 REPLENISHER

After plating certain Ah and depositing the corresponding mass of tin, a pre-calculated volume is removed from the bath and replaced by the same amount of SN 150 REPLENISHER.

The volume V_R of the bath, which has to be replenished at a chosen level of reduced concentration c_R, at which the replenishment is performed, is given by:

$V_R = V_N * c_N * (1 - z) / (c_R - z * c_N)$, where V_N is the nominal bath volume, c_N is the nominal concentration, c_R the concentration of the replenishing solution SN 150 REPLENISHER and z the ratio of the concentration at depletion level c_{depl} to the nominal concentration c_N

This ratio can be chosen depending on the desired level of stability of the concentration.

$z = c_{depl} / c_N$, (e.g. z = 0.97)

The corresponding mass of metal plated is:

$m_p = V_N * c_N * (1 - z) = I * t Ä_e$, where $Ä_e$ is the electrochemical equivalent.

Example: Replenishment of the bath at a depletion concentration of tin at 97% of the nominal concentration of 35 g/l and a nominal bath volume of 40 litres.

The volume to be replenished is:

$V_R = (40 l * 35 g/l * (1 - 0.97) / (44 g/l – 0.97 * 35 g/l) = 4.18 l$

The mass of tin metal plated up to this point is: $m_p = 40 l * 35 g/l * (1 - 0.97) = 42 g$

The electrochemical equivalent $Ä_e$ is 2.21 g/Ah, so the replenishment has to be performed after:

$Q = m_p/Äe = 42 g / 2.21 g/Ah = 19.0 Ah$

As a result in this example: After plating 19.0 Ah, the tin concentration has dropped to 97% of the nominal value of 35 g/l. The bath is replenished by taking out 4.18 litres of the depleted bath and refilling 4.18 litres from SN 150 REPLENISHER.

Option 2: Replenish plated metal using SN 150 TIN CONC

The depleted amount of tin is replenished from the concentrated tin concentrate SN 150 TIN CONC:

The SN 150 TIN CONC contains 300 g/l Sn^{2+} .

In the example above 140 ml SN 150 TIN CONC are needed to replenish 42 g tin metal to the solution after 19.0Ah.

Please note that the concentration of SN 150 REFINER needs to be replenished separately.

SN 150 REFINER
The SN 150 REFINER is added along with the SN 150 REPLENISHER solution. The assumed consumption rate of SN 150 REFINER is 0.22 ml/Ah (or 0.1 ml per g tin deposited).
If the consumption of SN 150 REFINER is higher than this value or tin replenishment is performed using SN 150 TIN CONC, separate dosage of SN 150 REFINER can be performed. Especially in new tool setup situations, the consumption rate may be higher due to the absorption of organic compounds in filter units and other plastic materials.

Inert anode conditioner (IAC)
For the replenishment of the inert anode conditioner (IAC), substitute with the corresponding amount of SN 150 P CONC needed for proper IAC concentration from the volume of SN 150 REPLENISHER to be replenished as described in option 1 above. (1 litre SN 150 P CONC contains 500 ml IAC.)

Example: 40 litres nominal bath volume, IAC level has dropped to 37.5 ml/l

IAC amount needed: 40 l * (50 ml/l – 37.5 ml/l) = 500 ml
SN 150 P CONC needed: 1 litre (containing 500 ml IAC)

SN 150 REPLENISHER needed for replenishing Sn^{2+} and additives according to option 1 above:
4.18 litres.

Replenish the bath nominal volume of 40 litres with 4.46 litres bleed and feed:
1. Take out 4.18 litres depleted solution.
2. Replenish 3.18 litres SN 150 REPLENISHER
3. Replenish 1.00 litres SN 150 P CONC

Methanesulfonic acid
Methanesulfonic acid is replenished upon analysis according to the nominal values using the SN 150 ACID.

For increasing the concentration of SN 150 ACID the following formula applies:

$$V_R = V_N * (c_N *- c_{depl}) / (c_R – c_N) = V_N * \triangle c / (c_R – c_N)$$

V_R:	volume to add from SN 150 ACID
V_N:	nominal volume of the bath
c_N:	nominal concentration
c_{depl}:	depleted or actual concentration
c_R:	concentration of replenishing solution
$\triangle c$:	increase of concentration

Example: Bring SN 150 ACID concentration from 185 ml/l back to 200 ml/l in a bath of 40 litres

V_R = 40 l * (200 ml/l – 185 ml/l) / (1000ml/l – 200ml/l) = 0.75 l
(setting V_N to 1 liter, the result V_R = 12,5ml represents the value for increasing by $\triangle c$ = 10 ml/l per liter)

As a result, 750ml of SN 150 ACID are needed to restore the nominal concentration of 200 ml/l in the bath of 40 liters. Note, that while the acid concentration per liter is accurate, the total volume has increased by the volume replenished.

Acid replenishment may also be anticipated, if drag out loss data is consistent.

Example: Assuming 3 ml bath drag out per wafer, 0.3 ml SN 150 ACID is dragged out per wafer processed. After 100 wafers processed, 30 ml SN 150 ACID need to be replenished, corresponding to 28.26g methanesulfonic acid

SPECIFIC PROCEDURES

- Oxygen plasma before plating
- chemical pre-treatment not recommended/normally not needed
- Cleaning of all items with DI before insertion in electrolyte
- Wetting of wafer surface with DI water before insertion into bath (check for wetting)

CUSTOMER SUPPORT

Further customer support on the process with this product is available by contacting NB Technologies GmbH.

BATH ANALYSIS SERVICE

NB Technologies supports the bath analysis and provides special shipping kits including shipping box, sample bottles and labels.

DATA LOGGING

Keep a record of ampere-hours of use to determine replenishment volumes. Examples of process log sheets are available by contacting NB Technologies GmbH.

HANDLING AND SAFETY INSTRUCTIONS

For detailed information consult the material safety data sheets for this product.
Please read material safety data sheets carefully before using this product.

DISCLAIMER

All recommendations and suggestions in this bulletin concerning the use of our products are based upon tests and data believed to be reliable. Since the actual use by others is beyond our control, no guarantee expressed or implied, is made by NB Technologies GmbH, its subsidiaries of distributors, as to the effects of such use or results to be obtained, nor is any information to be construed as a recommendation to infringe any patent.

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