Palladium-Nickel 462

Operating Instructions

Edition: 15 November 2002

Ammoniacal palladium-nickel bath

- **up to 10 µm bright and crack-free**
- **low-pore, ductile coatings**
- **corrosion- and tarnish-resistant**
- **high hardness of 620 HV_{10}**
- **suitable for decorative and technical applications, for rack and barrel**

Bath Characteristics

The Palladium-Nickel bath 462 usually deposits alloy coatings of the composition Pd/Ni 70/30 (% by weight).

By increasing the palladium content, coatings of the composition Pd/Ni 80/20 (% by weight) can also be deposited.

The coatings possess a pleasant white colour and are perfectly bright even at layer thicknesses of over 10 µm. At a hardness of approx. 620 HV_{10} their porosity is low, they are ductile, corrosion- and tarnish-resistant, and crack-free even as thick deposits.

If only low coating thicknesses are deposited, a lowering of the palladium content to 7 g/l is permitted. Then coatings of the composition Pd/Ni 65/35 are produced.

The bath requires a minimum of maintenance and is suitable for both decorative and technical applications in rack and barrel operation.

Bath type:	alkaline, ammoniacal	
Palladium content:	7 g/l	(6 - 8 g/l for Pd/Ni 65/35)
	10 g/l	(9 - 11 g/l for Pd/Ni 70/30)
	15 g/l	(13 - 17 g/l for Pd/Ni 80/20)
Nickel content:	10 g/l	(9 - 11 g/l)
Temperature:	35 °C	(33 - 37 °C) for Pd/Ni 70/30
	25 °C	(20 - 30 °C) for Pd/Ni 80/20 and Pd/Ni 65/35
pH-value:	8.2	(8.0 - 8.4) at 35 °C
	8.5	(8.4 - 8.7) at 25 °C
Current density:	1 A/dm²	(1 - 2 A/dm²) for rack and barrel operation
Deposition speed:	approx. 0.22 – 0.25 µm/min 1 µm in approx. 4 - 4.5 min	

Coating Characteristics

Coating:	palladium-nickel	
Alloy composition: (approximately)	70 wt.% Pd 30 wt.% Ni	standard Pd content (10 g/l Pd)
	80 wt.% Pd 20 wt.% Ni	high Pd content (15 g/l Pd)
	65 wt.% Pd 35 wt.% Ni	low Pd content (7 g/l Pd)
Density of the coating:	approx. 11 g/cm³	
Colour:	white	
Hardness:	approx. 620 HV_{10}	

Further Coating Properties

Max. coating thickness:	over 10 µm can be deposited crack-free, very good abrasion resistance

Form of Supply

Bath makeup:

a) Palladium-Nickel 462 Initial Concentrate
(free of precious metal)
200 ml for 1 l of bath
Storage stability: min. 3 years

b) Palladium Solution 460
100 g/l Pd
100 ml for 1 l of bath (10 g/l Pd)
Storage stability: min. 3 years

c) Palladium-Nickel 462 Brightener
(free of precious metal)
30 ml for 1 l of bath
Storage stability: min. 2 years

Bath replenishment:

d) Palladium Solution 460
100 g/l Pd
10 ml per 1 g of Pd
(equivalent to 1.4 g of Pd/Ni 70/30 or 1.25 g of Pd/Ni 80/20)
Storage stability: min. 3 years

e) Palladium-Nickel 462 Replenisher Solution 1
(free of precious metal)
100 g/l Ni
4.0 ml per 1 g Pd (for Pd/Ni 70/30)
2.5 ml per 1 g Pd (for Pd/Ni 80/20)
5.0 ml per 1 g Pd (for Pd/Ni 65/35)
Storage stability: min. 3 years

f) Palladium-Nickel 462 Replenisher Solution 2
(free of precious metal)
1 ml per 1 g of Pd
Storage stability: min. 2 years

Corrections:

g) Palladium Density Correction Salt 4
approx. 20 g/l per 0.01 g/cm³
Storage stability: unlimited

h) For the adjustment of the pH-value, ammonia solution 25 % and dilute sulphuric acid (both chem. pure) should be available.

Palladium-Nickel 462

Bath Makeup

Makeup sequence:	To make up 1 l of bath, add exactly 100 ml of Palladium Solution 460 and 200 ml of Palladium-Nickel 462 Initial Concentrate to approx. 500 ml of deionized water with stirring, and afterwards add 30 ml of Palladium-Nickel 462 Brightener. Then fill up to 1000 ml with deionized water. It is absolutely essential to filter the bath after the makeup. Then it should be allowed to stand for one night without being used and then again be carefully filtered.
pH adjustment:	If required, adjust the pH-value with ammonia solution 25 % or dilute sulphuric acid (both chem. pure). Please note the hints under "Operating Conditions".
Deposition of thick coatings:	If the deposition of particularly thick coatings (> 5 µm) is planned, an active carbon treatment has to be carried out prior to use. In this case first make up only the basic bath with Palladium Solution 460 and Palladium-Nickel 462 Initial Concentrate, without adding the Palladium-Nickel 462 Brightener. Then stir in 2 g/l of Active Carbon 1 and continue stirring for approx. 2 hours. After the carbon has settled overnight, draw off the bath and filter carefully. Only then add the full amount of Palladium-Nickel 462 Brightener and adjust the pH-value. Palladium and nickel are not removed by the active carbon treatment.

Operating Conditions

Palladium content:	10 g/l (9 - 11 g/l) standard (for Pd/Ni 70/30) or 15 g/l (13 - 17 g/l) (for Pd/Ni 80/20) or 7 g/l (6 - 8 g/l) (for Pd/Ni 65/35)
Nickel content:	10 g/l (9 - 11 g/l)
Operating temperature:	35 °C (33 - 37 °C) for Pd/Ni 70/30 25 °C (20 - 30 °C) for Pd/Ni 80/20 and for Pd/Ni 65/35
Bath density:	1.053 –1.06 g/cm³ (7.3 – 8.2 °Bé) when made up, rising during operation. Max. permissible bath density = 1.085 g/cm³. The bath density can be corrected with Palladium Density Correction Salt 4. Approx. 20 g/l to raise the density by 0.01 g/cm³.
pH-value:	8.2 (8.0 - 8.4) measured at 35 °C, at 20 - 25 °C (room temperature) the pH-value of the bath is approx. 0.3 units higher, i.e.: 8.5 (8.4 - 8.7) measured at 25 °C **Important:** See "Special Process Hints/pH-Value"
Product agitation:	Product agitation of approx. 5 cm/s is advantageous in most cases. Too strong agitation is disadvantageous. Also see "Special Process Hints".
Bath agitation:	Agitation of the electrolyte as uniform as possible is required.
Filtration:	Continuous filtration through 1 µm filter cartridge (not through active carbon). The cartridges must be cleaned in hot dilute sulphuric acid prior to use.
Current density:	1.0 A/dm² (1.0 - 2.,0 A/dm²) valid for rack and barrel operation
Deposition rate:	approx. 24 mg/Amin at 70/30 approx. 26 – 28 mg/Amin at 80/20
Deposition speed:	approx. 0.22 – 0.25 µm/min 1 µm in approx. 4 – 4.5 min
Loading per litre:	up to 1 A/l (1 dm²/l)

Calculation of Coating Thickness and Plating Time

Coating weight in mg =
surface in cm² x 1.1 x coating thickness in µm

Plating time in minutes =

$$\frac{\text{required coating weight in mg}}{\text{deposition rate in mg/Amin x current in amperes}}$$

Palladium-Nickel 462

Bath Replenishment

Replenish at the latest after depositing 1 g/l of Pd:

a) Alloy 70/30 (7 - 10 g/l of Pd):
1 g of Pd is approximately equivalent to 1.4 g of alloy and a current flow of approx. 58 Amin.

To replenish this, add in any order:

10 ml of Palladium Solution 460
4 ml of PdNi 462 Replenisher Solution 1
1 ml of PdNi 462 Replenisher Solution 2

b) Alloy 80/20 (15 g/l of Pd):
1 g of Pd is approximately equivalent to 1.25 g of alloy and a current flow of approx. 52 Amin.

To replenish this, add in any order:

10 ml of Palladium Solution 460
2.5 ml of PdNi 462 Replenisher Solution 1
1 ml of PdNi 462 Replenisher Solution 2

c) Alloy 65/35 (7 g/l of Pd):
1 g of Pd is approximately equivalent to 1.54 g of alloy and a current flow of approx. 64 Amin.

To replenish this, add in any order:

10 ml of Palladium Solution 460
5 ml of PdNi 462 Replenisher Solution 1
1 ml of PdNi 462 Replenisher Solution 2

Bath Monitoring and Correction

Analytical control:
The content of palladium and nickel should be regularly controlled. If there are shifts in the concentrations, corrections can be carried out with:

Palladium Solution 460, concentration 100 g/l of Pd and PdNi 462 Replenisher Solution 1, concentration 100 g/l of Ni

Contaminants:
The bath must be kept clean. Continuous filtration is required. Metallic and organic contaminants interfere and should be avoided. Also see "Special Process Hints".

Special Process Hints

pH-value:

Important: The pH-value must not drop under pH 8.0 during operation and idle times otherwise the lifetime of the bath will be clearly reduced (dark coatings). During operation and idle times the pH drops slightly. Therefore it must be measured every day and adjusted with ammonia solution 25 % (chem. pure).

The bath must be sealed tightly with a cover when the bath is not operated.

During longer idle periods the bath should be drawn off and sealed. A monthly control is then sufficient.

With continuously high bath load the volume of the bath can increase. In this case the pH-value can be adjusted by blowing in ammonia gas by means of suitable dosing equipment. We recommend this method!

Alloy 80/20:

Bath with 15 g/l of Pd at 25 °C

By increasing the palladium concentration and altering the temperature it is possible to deposit alloys with approx. 80 % by weight of palladium. For this purpose, increase the Pd concentration to 15 g/l (total: 150 ml of Palladium Solution 460 for 1 l of bath) without altering the Ni concentration, and lower the temperature of the bath to 25 °C. Current density 1 - 1.5 A/dm². For the modification of the replenishment, see "Bath Replenishment".

Alloy 65/35: (Low Pd content)

Bath with 7 g/l of Pd at 25 °C

If only low coating thicknesses are deposited, lowering the palladium content to 7 g/l is permitted. Then coatings of the composition Pd/Ni approx. 65/35 will be produced.

Active carbon treatment:

If there are organic contaminants, we recommend an active carbon treatment with at least 2 g/l of Active Carbon 1. The Brightener will be removed by this treatment and has to be readjusted to specified value by adding the full amount of Palladium-Nickel 462 Brightener. Palladium and nickel are not removed.

Wetting agent:

If there should be wetting problems, Wetting Agent 3 can be used in concentrations of up to 10 ml/l.

Palladium-Nickel 462

Hints for bright and haze-free coatings:	To obtain perfect brightness at a high coating thickness, it is essential to maintain optimum agitation conditions and optimum current density. Extremely low current density (< 0.5 A/dm²) will lead to irregularly dark coatings. In the case of articles with a high electrolyte exchange, i.e. profiled parts that have a strong stirring effect or parts with narrow surfaces (e.g. watch cases), it is advisable to work with very little agitation (3 cm/s) and possibly at a higher current density (up to 2.0 A/dm²). In the reverse case, parts with large closed surfaces require increased bath agitation. With older baths, a slight loss in brightness on areas of the articles with particularly low current density may occur. In most cases this effect can be remedied by increasing the current density and/or slowing down product agitation. In the case of a considerable loss in brightness and also to remove organic contaminants, an active carbon treatment of the bath is recommended (see above). Afterwards add the full amount of PdNi 462 Brightener.
Pre- and post-treatment:	On the usual basic materials including nickel and nickel alloys, the bath does not require any specific pre-treatment steps. PdNi coatings can be subsequently adhesively plated with PdNi. Intermediate degreasing of PdNi coatings in any case has to be anodic! Cathodic degreasing will lead to cracks.
Anode film:	brown-yellow anode films should be regularly removed by dipping into a cyanide solution. The anodes must be rinsed thoroughly before inserting them back into the bath.
Drag-out rinse:	Turbidity in the drag-out rinse is avoided by setting the pH to 10 with ammonia solution.

Equipment

Bath tanks:	Plastic , preferably polypropylene (PP) natural. The bath must be sealed tightly with a cover when the bath is not operated.
Heating:	Immersion heaters sheathed with porcelain, quartz, or Teflon. Equipment for temperature control.
Filtration and bath agitation:	Continuous filtration through 1 µm filter cartridge (not through active carbon). The cartridges must be cleaned in hot dilute sulphuric acid prior to use.
Anodes:	Graphite anodes. E.g. from Conradty / Nürnberg or SGL Carbon AG / Meitingen (both Germany)
Exhaust system:	required

Note

Our information relating to the storage stability refers to storage in closed original storage containers under the conditions stated on the label.

Precautionary Measures/Safety Hints

For information on safety. please see the corresponding Material Safety Data Sheets! The valid accident prevention regulations and safety information must be observed.

Reference to

Analytical control:	available on request

Umicore Galvanotechnik GmbH

P.O. Box 12 40 • D-73502 Schwaebisch Gmuend

Delivery address:
Klarenbergstraße 53-79 • D-73525 Schwaebisch Gmuend
GERMANY

Telephone +49 (0) 71 71 / 6 07 - 01
Fax +49 (0) 71 71 / 6 07 - 2 88

e-mail: galvano@eu.umicore.com
www.umicore-galvano.com