

Palladium 457

Operating Instructions

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Weakly alkaline palladium electrolyte for pure palladium coatings

- high-gloss, low-pore coatings
- coatings bendable crack-free up to 3 µm
- excellent corrosion-resistance
- stable electrolyte with wide operating range
- for rack and barrel

Bath Characteristics

Palladium 457 is a weakly alkaline palladium electrolyte for decorative and technical applications. High-gloss and light pure palladium coatings can be deposited from the electrolyte within a wide operating range of current density.

Bath type:	weakly ammoniacal
Palladium content:	10 g/l
pH-value:	7.7
Temperature:	42°C
Current density:	1.0 - 1.5 A/dm ²
Deposition speed:	approx. 0.25 µm/min at 1.0 A/dm ² approx. 0.38 µm/min at 1.5 A/dm ²

Coating Characteristics

The white low-pore coatings are brilliant up to 5 µm of coating thickness. The ductile, low-stress layers have high hardness and a very good wear resistance, as well as good corrosion and tarnish resistance.

Coating:	pure palladium
Percentage purity:	approx. 99.9 % Pd
Colour:	white, light/bright
Hardness:	approx. 300-350 HV 0.015
Coating density:	approx. 12 g/cm ³

Further Coating Properties

Wear properties:

Very good abrasive resistance in Bosch-Weinmann-test with abrasive paper, grain size 6/0 (wear approx. 1.5 mg/ cm²·1000 double strokes). Also good wear property on the pin-disc-tribometer (palladium with gold-flash).

Diffusion barrier:

The coatings are suitable as diffusion barriers, similar to nickel layers.

Ductility/bendability:

Up to 3 µm of coating thickness can be bent without cracking around a mandrel of 20 mm.

Contact resistance:

Low contact resistance of 1.1 mOhm (50 cN contact force).

Form of Supply

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|-----------------------|---|
| Bath makeup: | a) Palladium 457 Initial Concentrate
500 ml to make up 1 litre of bath
Storage stability: min. 1 year |
| | b) Palladium solution 460
100 ml g/l palladium
100 ml to make up 1 litre of bath with 10 g/l palladium
Storage stability: min. 3 years |
| Bath replenishment: | c ₁) Palladium solution 460 (as Pos. b)
1000 ml for the replenishment of 100 g palladium drag out
Storage stability: 3 years |
| | alternatively: |
| | c ₂) Palladium salt 41-43 % Pd
238 g salt for the replenishment of 100 g palladium drag out
Storage stability: unlimited |
| | d) Palladium 457 Replenisher Solution
200 ml for the replenishment of 100 g palladium drag out
Storage stability: min. 1 year |
| Correction chemicals: | e) Palladium Density Correction Salt 1
approx. 18 g/l per 0.01 g/cm ³ .
Storage stability: unlimited |
| | f) For the adjustment of the pH-value, ammonia solution and phosphoric acid (each p.a.) should be available. |

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Bath Makeup

Preparations: All materials coming into contact with the bath (bath tanks, filter cartridges, pumps, pipes, hoses, barrels, rack insulations, etc.) must be leached in sulphuric acid prior to first use and subsequently in caustic soda solution. Before filling the bath thoroughly rinse with deionized water to make it alkali-free.

Makeup sequence: To make up 1 litre of bath, add 100 ml Palladium Solution 460 to 500 ml Palladium 457 Initial Concentrate and fill up to 1000 ml with deionized water.

The solution is (continuously) filtrated and the pH-value is adjusted to the nominal value (ammonia solution resp. phosphoric acid, both p.a.).

Attention:

When making up the bath the personnel must take the necessary preventive measures, such as wearing goggles, protective gloves, protective clothing.

Example: To make up 100 l of bath:
50 l Pd 457 Initial Concentrate
10 l Palladium Solution 460
40 l deionized water

Operating Conditions

Palladium content:	10 g/l (8 - 12 g/l)
Operating temperature:	42 °C (40 - 45 °C)
pH-value:	7.7 (7.2 - 8.2)
Bath density: (20°C)	1.075 g/cm ³ (1.06 - 1.09 g/cm ³) 10 °Bé (8.2 - 12 °Bé)
Product agitation:	Required. 2-4 m/min, freely variable
Bath agitation:	Required. Via filter pump. Recirculation approx. 2-3 times the bath volume/hour. Air agitation is not allowed!
Filtration:	Continuous filtration through polypropylene filter cartridges, pore width ≤5 µm is recommended.
Current density:	Rack: 1 A/dm ² (0.5 - 3 A/dm ²) Barrel: 0.5 A/dm ² (0.2 - 0.8 A/dm ²)
Voltage:	approx. 1.5 - 2.5 V
Deposition rate:	approx. 30 mg/Amin
Current efficiency:	approx. 90 % (1 A/dm ²)

Deposition speed:	0.05 µm/min at 0.2 A/dm ² 0.12 µm/min at 0.5 A/dm ² 0.20 µm/min at 0.8 A/dm ² 0.24 µm/min at 1.0 A/dm ² 0.51 µm/min at 2.0 A/dm ² 0.79 µm/min at 3.0 A/dm ² These figures are only reference values.
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Exposition time: approx. 4 min for 1 µm

Loading per litre: up to approx. 1 A/l

Calculation of Coating Thickness and Plating Time

Coating weight in mg =
surface in cm² x 1.2 x coating thickness in µm

Plating time in minutes =
 $\frac{\text{required coating weight in mg}}{\text{deposition rate in mg/Amin} \times \text{current in ampere}}$

Bath Replenishment

Replenish at the latest if 10 % of the palladium content has been removed. 100 g Pd corresponds to a current quantity of approx. 3300 Amin (55 Ah). To install a Ah-counter is recommended in order to exactly keep the replenishment intervals. For the consumption per 100 g palladium the following reference values can be valid:

1000 ml Palladium Solution 460
alternatively: 238 g Palladium Salt 41-43 %
as well as
200 ml Palladium 457 Replenishment Solution

For continuous replenishment the operating conditions which are different from user to user, such as current density applied, temperature, agitation, filter performance, drag out, etc., must be observed and the approximate values mentioned above must be changed accordingly, if necessary.

Bath Monitoring and Correction

The **analytical control** is made according to the analytical control methods for Palladium 457 which can be obtained on request.

The **palladium content** must be checked regularly and if necessary corrected with Palladium Solution 460 to the nominal value.

The **pH-value** drops during operation of the bath and idle times. Therefore it must be measured every day and adjusted with ammonia solution p.a. resp. phosphoric acid p.a. The pH-value can be increased by blowing in of gaseous ammonia by means of an appropriate dosage unit. We expressly recommend this method!

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The **bath density** depending on the throughput should be regularly checked (e.g. weekly). To increase the bath density by 0.01 g/cm³ add 18 g/l Palladium Density Correction Salt 1.

Influence of **contaminants**:

Metallic, inorganic and organic contaminants interfere and should be avoided by appropriate measures (rinsing steps, etc.).

Very important: The bath is very sensitive to **cyanide** contamination! Any drag-in of cyanide must be avoided. The parts must be rinsed very well before placing them in the palladium bath, particularly in the case of a preceding gold strike bath or a cyanide containing process step. Any rinse water must be cyanide-free! Placing the palladium bath next to or in the vicinity of cyanide baths must be avoided. Cyanide contamination shows in the formation of cracks and unsightly coatings (hazy, matt).

Metallic impurities like copper, nickel and zinc interfere with the plating >10 mg/l (sum of metallic impurities) and result in dark/hazy matt coatings, especially in low current density areas. See "Special Process Hints/Metallic impurities".

Organic impurities often lead to hazy coatings and reduced cathodic current efficiency of the electrolyte and must be avoided by corresponding rinsing steps. Drag-in, e.g. from bright nickel baths are sometimes shown by a small amount of a whitish-yellow precipitation in the bath. This precipitation can be filtered off without loss of function.

Active carbon purification:

In case of brittleness or lack in brilliance caused by organic contaminations an active carbon purification is necessary. For this, stir 6 g/l Active Carbon 1 for one hour into the bath, allow to settle and filter off subsequently. By this active carbon treatment also important components are removed. Therefore after such an active carbon purification the electrolyte must be analysed and adjusted. Palladium is hardly removed.

Hull cell test:

A quick method to check the bath is the Hull cell test. The operating conditions are:

- 250 ml Hull cell with anode
- 0.5 A cell current
- 5 min plating time
- Magnetic stirrer (500 U/min, 25 mm stirring rod)
- Layer sequence: sheet brass / bright nickel / strike gold plating / palladium
- Normal working conditions of the bath

The test result should be shiny and without burnt area.

Special Process Hints

Pre-treatment: Place the parts in the electrolyte free from oxides and grease. Sufficient degreasing and acid dip should always be guaranteed in addition to sufficient rinsing steps. For adhesive depositions on non-precious metals (Ni, etc.) we recommend a palladium strike plating with Palladium 459.

Post-treatment: The coatings can be lightened by a treatment in hot water. Water temperature >90 °C. Immersion time 1 minute.

An electrolytic degreasing of the palladium coatings has to be in any case anodic. Cathodic degreasing causes cracks.

Removal of metallic contaminations: Copper and nickel can be almost completely removed by a treatment with Ion Exchange Resin 1. Zinc is removed only partially. Application: apply approximately 10 ml resin for 100 mg of impurities under continuous stirring max. 4 hours. Palladium will not be bound.

Economy rinse: Turbidity in the economy rinse is avoided by adjusting the pH to 10 with ammonia solution.

Equipment

Bath tanks: PPH, PVC, glass, teflon
Preferably polypropylene natural. The bath must be sealed tightly with a cover when the bath is not operated.

Attention:

Stainless steel, rubber or coloured plastic must not be used as tank material!

Heating: Immersion heaters out of porcelain, quartz glass or Teflon, with temperature control.

Filtration and bath agitation: Continuous filtration through polypropylene filter cartridges, pore width ≤5 µm, is recommended. Recirculation via the filter pump approx. 2-3 times the bath volume / hour. The pump used should be a magnet-coupled centrifugal pump out of alkali-resistant plastic. As material for the pumps and hoses, PP, PE or PTFE is recommended.

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Anodes:	We recommend to use our PLATINODE® 167 as anode material. Important: Stainless steel or platinized titanium must not be used under any circumstances. The use of such materials causes permanently considerable problems and result in new make up of the bath. Ratio of anode area : product area at least 2:1 The anode current density should be max. 1 A/dm ² .
Material:	Materials out of copper, nickel, brass or stainless steel must not be exposed to the bath steams and must not come into contact with the bath itself. These metals are attacked by the bath and the ammonia steams. We recommend titanium.
Rectifier:	Infinitely variable, with current display and Ah meter. Residual ripple <5 %.
Exhaust system:	Effective 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 instructions must be observed.

Reference to

Analytical control: Methods available on request

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