

Black Ruthenium 474

Operating Instructions

Edition: 29 April 2002

- acidic black ruthenium electrolyte
- decorative black layers up to 0.5 µm coating thickness
- good colour constancy
- simple bath maintenance
- easy to use batch type

Bath Characteristics

Black Ruthenium 474 is a strongly acidic electrolyte from which decorative coatings of a anthracite colour up to 0.5 µm thickness can be deposited. The bath is easy to operate and the deposited coatings have a high colour constancy and retain their brightness.

Black Ruthenium 474 is used as a final coating for decorative applications, e.g. in the industries producing jewellery, spectacle frames and writing utensils. The wear resistance of coatings is good.

Pregilding as an undercoat under the black ruthenium layer is recommended (see "Special Process Hints", paragraph "Pretreatment"). If palladium-nickel or palladium is used as an undercoat, pregilding is not necessary.

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| Bath type: | acidic |
| Ruthenium content: | 5 g/l (batch type) |
| pH value: | 1.4 (1.0 - 1.5) at 70 °C |
| Temperature: | 70 °C (65 - 75 °C) |
| Current density: | 1,0 A/dm ² (0.5 - 2.0 A/dm ²) |
| Deposition speed: | approx. 0.08 µm/min at 5 g/l and 1 A/dm ² |

Coating Characteristics

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| Coating: | black ruthenium |
| Colour: | anthracite (black) |
| Density: | approx. 12 g/cm ³ |
| Max. coating thickness: | 0.5 µm |

Form of Supply

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| Bath makeup: | for 1 l of bath Black Ruthenium 474 Initial Concentrate (with Ru), 32 g/l Ru, 156 ml containing 5 g Ru Storage stability: min. 2 years |
| Bath replenishment: | Not available, batch type |
| Bath corrections: | For pH corrections diluted sulphuric acid p.a. and ammonia solution p.a. should be available. |

Bath Makeup

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| Makeup sequence: | To make up 1 l of bath, stir 156 ml of Black Ruthenium 474 Initial Concentrate slowly into 800 ml of deionized water and fill up to 1 l with deionized water. Heat up the bath to 70 °C and then measure the pH value with a glass electrode. If required, adjust the pH to 1.4. |
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Operating Conditions

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| Ruthenium content: | 5 g/l (batch type) |
| pH value: | 1.4 (1.0 - 1.5) at 70 °C 1.0 (0.6 - 1.1) at 25 °C Corrections of the pH are carried out with sulphuric acid p.a. or ammonia solution p.a. (both diluted 1:1). |
| Operating temperature: | 70 °C (65 - 75 °C) |
| Bath density: | 1.02 g/cm ³ at makeup 3 °Bé |
| Product agitation: | strong agitation required! At least 5 - 10 cm/s! Air agitation is recommended. |
| Bath agitation: | bath circulation required, min. 2 bath volumes per hour |

Important: See paragraph "Bath Monitoring and Corrections/pH-value"!

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| Filtration: | continuous filtration through polypropylene filters recommended. Active carbon cartridges must not be used. |
| Current density: | 1 A/dm ² (0.5 - 2.0 A/dm ²) |
| Deposition rate: | approx. 10 mg/Amin (8 - 12 mg/Amin) |
| Deposition speed: | approx. 0.08 µm/min at 1 A/dm ² at standard conditions, proportional to current density |
| Density of the coating: | approx. 12 g/cm ³ |

Calculation of Coating Thickness and Plating Time

The coating thickness should not be higher than maximally 0.5 µm.

Coating weight in mg = $\frac{\text{surface in cm}^2 \times 1.2 \times \text{coating thickness in } \mu\text{m}}{\text{coating thickness in } \mu\text{m}}$

Plating time in minutes = $\frac{\text{required coating weight in mg}}{10 \times \text{current in amperes}}$

Bath Monitoring and Correction

pH-value:

The pH value should be daily controlled with a glass electrode and adjusted with sulphuric acid or ammonia solution (both p.a. quality and diluted 1 : 1), if required. The pH-value should be measured at operating temperature (70 °C). At room temperature (25 °C) the pH is approx. 0.3 - 0.5 units lower.

Caution: If the pH-value is too high the bath becomes instable!

Metallic impurities:

The bath attacks the usual basic materials and is sensitive to metallic contaminants like copper, zinc, lead and other metals. See "Special Process Hints", paragraph "Pretreatment". The common metallic impurities can be removed by dummy plating at 0.1 A/dm² with wavy metal sheets or by a special precipitation method. The precipitation should be performed only after analytical control and consulting the supplier.

Special Process Hints

Pretreatment: The acidic bath attacks the usual basic materials and is sensitive to metallic contaminants. The parts, particularly materials containing copper, iron and zinc, must be protected by a gold strike bath. If palladium-nickel or palladium is used as an undercoat, pregilding is not necessary.

Post-treatment: Important!
After plating, the parts should be rinsed in a spare rinse and then under running water. Then post-treatment for approx. 2- 4 minutes in a 60 °C warm diluted sodium hydroxide solution (50 g/l NaOH). This post-dip solution neutralizes electrolyte rests on the surface of the parts. The solution itself is removed by longer rinsing under running water. The last rinse before drying should be carried out with deionized water.

Loading of the bath under voltage: As far as possible, the parts should be immersed with the current switched on.

Spare rinse: The spare rinse solution must be adjusted to a pH-value lower than 2. A higher pH results in precipitates (hydrolysis) which are not soluble in the bath and produce faulty (spotty) deposits.

Barrel: Due to the reasons stated above under "Pretreatment", the bath is only limitedly suitable for barrel applications.

If the parts have a complicated geometry, a 100 % protection of the parts by pregilding can possibly not be reached, which will lead to faulty deposition during the subsequent ruthenium plating and to contamination of the bath.

Therefore a use of the bath for barrel applications should be previously tested. In no case the usability in one special case indicates the usability for other articles.

Equipment

Bath tanks: acid-resistant plastic (polypropylene)
All parts coming into contact with the electrolyte must be resistant to strong acids. Plastic equipment, e.g. tank, pump, hoses, filter cartridges, etc. before use should be rinsed in diluted acid (e.g. 5 % sulphuric acid) for several hours and subsequently cleaned of the contaminated acid by intensive rinsing with water which should be changed several times.

Heating: adjustable immersion heater with coating of porcelain, quartz, or Teflon.

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| Anodes: | platinized titanium, e. g. PLATINODE coated with 2.5 µm of platinum |
| | anode surface : article surface at least 2 : 1 |
| Racks: | with acid-resistant coating, contact points of stainless steel or suffi- ciently gold-plated |
| Exhaust system: | an efficient exhaust system is 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!

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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