# United States Patent

**Knight et al.**

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## [54] REMOVAL OF NITRATE CONTAMINATION FROM NICKEL-PLATING SOLUTIONS


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[51] **Int. Cl.** ..... **C01d 7/34,** C23b 5/08

[58] **Field of Search** ..... 204/130, 102, 49



[56] **References Cited**

UNITED STATES PATENTS

1,212,062 1/1917 Hunt ..... 204/102

OTHER PUBLICATIONS

Electroplating Engineering Handbook, by A. K. Graham, 1962, pp. 245-246 Modern Electroplating, by F. A. Lowenheim 1963, pp. 292-293



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[57] **ABSTRACT**

A nitrate-contaminated nickel-plating bath is purified by heating the bath to between 65° and 70° C., agitating the bath and electrolyzing the bath while providing a 10 to 20 fold greater cathode area than anode area.


**1 Claims, No Drawings**

# REMOVAL OF NITRATE CONTAMINATION FROM NICKEL-PLATING SOLUTIONS

## CONTRACTUAL ORIGIN OF THE INVENTION

The invention described herein was made in the course of, or under, a contract with the United States Atomic Energy Commission.

## BACKGROUND OF THE INVENTION

This invention relates to purifying nickel-plating solutions and, more particularly, this invention concerns purification of a nitrate-contaminated nickel-plating solution or bath.

Nickel-plated uranium slugs are used as fuel in some nuclear reactors. The nickel plating may be performed by a standard Watts process with an all chloride bath. The standard Watts process for nickel plating is well known in the art as are the various types of bath compositions which may be used with the Watts process, see Vol. 2, Metals Handbook, 8th Edition, American Society for Metals (1964), pp. 432–443. In the nickel plating of uranium, it is very important to maintain the bath purity and, in general, contamination at only a very low level is acceptable. Because of prior operations on the uranium before the slugs enter the nickel bath, various contaminants are dragged in with them. The above-mentioned reference sets out purification procedures effective for various organic and inorganic contaminants, such as iron, copper, magnesium, lead, various oils and paints. All the enumerated purification procedures in the Metals Handbook reference use elevated temperatures and elevated pHs, some use filtering agents such as charcoal or clay and some use electrolysis.

The uranium slugs to be nickel plated have previously been in contact with nitric acid and it is probably this contact which is responsible for the nitrate contamination present in the nickel-plating bath. While purification procedures as enumerated above, are available for various contaminants, none of them reduce nitrate concentration, and they are useless where nitrates are present in sufficient quantities to hinder operation of the bath. A procedure for removing nitrates was reported in the March 1966 issue of "Plating" magazine which electrolyzed with low cathode areas at a pH of 0.5 and with high-current densities but such a process is inconvenient if the volume of additional acid required to obtain the pH is so great as to render the plating solution unmanageable.

## SUMMARY OF THE INVENTION

The process of this invention comprises maintaining a nitrate-contaminated nickel bath between 65° and 70° C., agitating the bath and electrolyzing the bath at its operational pH with a cathode area substantially larger than the anode area; a filter material may be provided as well as a dummy plating surface for various metallic impurities to plate onto.

## DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT



After several ineffective experiments with the prior art purification processes reported in the Metals Handbook, all of which required adding nickel carbonate to the bath to raise the pH to between 5.2 and 5.5 prior to electrolyzing at a cathode current density of about 5 amps/sq. ft., it was decided to initiate electrolysis at the operating pH of the bath. The all chloride bath used in the nickel plating of uranium slugs is operated at a pH of about 2.4 and it is at this general pH value which the purification procedure must start.

It was clearly unexpected that the simple step of starting electrolysis at the operational pH rather than first raising the pH would make the removal of nitrates possible, particularly when the fact is known that the pH of the bath rises during electrolysis so that at the end of the purification the pH is about 6. Various baths contaminated with from 360 to 730 parts per million (p.p.m.) of nitrates have been successfully purified with the process of this invention.

The baths are heated to between 65 and 70° C. prior to purification and agitated during the purification. While purification occurs at lower temperatures it is too slow to be practical, but agitation of the bath is required to effectively remove nitrate contamination. After the above conditions are satisfied, then electrolysis is started at current densities from about 1 to 6 amps/sq. ft. of cathode area, and the cathode area is 10 to 20 times that of the anode. Up to 0.03 lbs. of activated charcoal (about −325 mesh) per gallon of bath may be added for heavily contaminated baths, but if charcoal is used then the bath must be filtered. The above conditions have resulted in reduction of nitrate contamination from 730 to 70 p.p.m. and from 360 to 26 p.p.m. The process also removes the usual type of impurities present in a nickel-plating bath and a dummy plating surface may be provided if metallic ions, such as iron, copper or magnesium, are present. In all cases, it must be remembered that artificially raising the pH of the bath prior to electrolysis prevents effective removal of nitrate contamination.

If the dummy plating surface is present than the metallic ions will plate onto it and be removed from the bath. Exactly how the nitrates are removed is not known but it is believed that they boil off as ammonia. Since the concentration of nitrates is low and the baths are open to the atmosphere it is very difficult to collect off-gases from the bath for analysis, and the above explanation is advanced as only a possibility.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A process for reducing the nitrate concentration by approximately a factor of 10 in a nickel-plating bath utilized for the nickel plating of uranium slugs, said bath having a nitrate contamination of at least 360 p.p.m. nitrates, comprising heating said bath to between 65° and 70° C.; adding 0.03 lb. of −325 mesh activated charcoal per gallon of bath; and electrolyzing the bath at a current density of 1 to 6 amps/sq. ft. with a cathode area 10 to 20 times as great as the anode area, the pH of said bath initially being 2.4 and rising to 6 during said electrolysis while agitating said bath during electrolysis.

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