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Staples

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[54] **CALIBRATION SOURCE EMITTING HIGH ENERGY BETA PARTICLES**

[75] Inventor: **Bruce A. Staples,** Idaho Falls, Idaho

[73] Assignee: **The United States of America as represented by the United States Atomic Energy Commission,** Washington, D.C.

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[52] **U.S. Cl.** ..... **204/15,** 29/196.6, 204/29, 204/32 R, 204/35 R, 204/40, 204/43 N, 250/493

[51] **Int. Cl.** **C23b 5/48,** C23b 5/50

[58] **Field of Search** ..... 204/43 N, 40, 29, 32 R, 204/35 R, 15; 250/493, 496, 497; 29/196.6

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Primary Examiner—G. L. Kaplan

Attorney, Agent, or Firm—John A. Horan; Arthur A. Churm; Frank H. Jackson

[57] **ABSTRACT**

A radiation source emitting high energy beta particles which is useful for film badge calibration is prepared by simultaneously electrodepositing nickel and ruthenium-106 onto a stainless steel disc, said ruthenium-106 being carried by natural ruthenium, washing the disc and electrodepositing nickel on the disc.

3 Claims, No Drawings

CALIBRATION SOURCE EMITTING HIGH ENERGY BETA PARTICLES

CONTRACTUAL ORIGIN OF THE INVENTION

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

BACKGROUND OF THE INVENTION

This invention relates to a method of preparing a calibration source. In more detail, the invention relates to a method of preparing a radiation source emitting high energy beta particles for film badge calibration.

Film badges are a type of personnel dosimeter containing a pack of sensitive photographic film worn by atomic energy workers to provide a record of radiation exposure. Since radiation acts to darken the film, an approximate determination can be made of the radiation dose that has been received by the wearer by comparing the film pack with control specimens of like material exposed to known amounts of radiation. Well calibrated sources of radiation similar to that which might be met with in an atomic energy plant are thus needed. Presently, no well calibrated source emitting high energy beta particles is available for film badge calibration.

SUMMARY OF THE INVENTION

According to the present invention, a calibration source emitting high energy beta particles is prepared by electroplating a stainless steel plate with nickel and a ruthenium carrier containing a small amount of radioactive ruthenium-106 from a solution of nickel chloride and nickel sulfate, washing the plate with water and electroplating the plate with nickel to seal the radioactive ruthenium within the calibration source. Ruthenium-106 with a half-life of one year decays to rhodium-106 with a half-life of 30 seconds giving transient equilibrium conditions and emitting a beta particle of 3.55MeV energy with a 79 percent branching ratio.

SPECIFIC EMBODIMENT OF THE INVENTION

In response to the request from Health Physics Section personnel at the National Reactor Testing Station, Arco, Idaho, for a high-level $^{106}Ru—^{106}Rh$ source for film badge calibration, tests were made on the best method of plating ruthenium on stainless steel. Metal surfaces on which electroplating will be performed must be descaled of oxidation products. Therefore, the surfaces were immersed in 10% H_2SO_4 at a temperature of 180°F. for a few minutes after gas was evolved in the solution. Next, the surfaces were rinsed in water, then immersed in 10% HNO_3 — 2% HF at 150°C. until they etched to a white color. The surfaces were then rinsed in water before electrodeposition. It was found that ruthenium would electrodeposit best from an 0.1 N HNO_3 — 0.005 N HCl solution onto descaled 347 stainless steel. However, the ruthenium could be mechanically removed from the surface of the steel regardless of the plating solution and current density. Nickel was found to electroplate easily from a nickel chloride — nickel sulfate solution onto a descaled 347 stainless steel surface using a carbon steel anode and a current density of 2 mA/cm². Ruthenium then could be plated using a platinum anode and a current density of 3 mA/cm² from a chloride solution onto the nickel surface. However, a permanent nickel plate to prevent decontamination could not be electrodeposited over the ruthenium surface.

Success was attained by simultaneously electroplating nickel and ruthenium on a descaled stainless steel surface and overplating with nickel. After preliminary tests which showed that nickel and ruthenium could be permanently electroplated together onto a stainless steel surface using a platinum anode and a current density of 3 mA/cm², a full-scale electrodeposition cell 11.4 cm in diameter and 10.2 cm high was constructed. A stainless steel disc 11.4 cm in diameter and 0.0127 cm thick at the bottom of the cell was employed as cathode; a rotating platinum disc, 7.5 cm diameter, positioned 1 cm from the cathode, was the anode. The plating solution, 1.25 M $NiSO_4 \cdot 6\ H_2O$ — 0.21 M $NiCl_2 \cdot 6\ H_2O$ — 0.66 M H_3BO_3, contained about 0.8 mg of ruthenium carrier and enough ruthenium 106 to exceed the desired disintegration rate on the stainless steel disc. Current density was 3 mA/cm². During the electrodeposition of nickel and ruthenium the ruthenium-106 activity in the solution was monitored by taking small aliquots which were gamma scanned for ruthenium-106. When 75 percent of the ruthenium-106 activity was removed from the solution by electrodeposition, the plating was stopped and the solution immediately removed from the electrodeposition cell. The cell was rinsed with water and nickel chloride — nickel sulfate solution added to the cell and nickel was immediately plated for 20 minutes on the nickel-ruthenium surface. In this time, approximately 160 mg of nickel was plated on the surface, giving a nickel density of about 2 mg Ni/cm². Nickel appeared not to electroplate on an area about 2 cm in diameter at the center of the source. The source was removed from the cell and the unplated area was covered for one minute with 1 ml of 10% HNO_3 – 2% HF at 100°F. A gamma-scan of a small portion of this solution revealed that very little ruthenium-106 had been removed from this surface. The source was next rinsed with water to remove acid and returned to the cell which was filled with plating solution. A smaller platinum anode, 1 cm in diameter, was placed 1 cm above the area on the source onto which nickel did not plate. The electrodeposition cell was then operated at 3 mA/cm² for 20 minutes during which time nickel plated onto the area washed with 10% HNO_3 — 2% HF. The ruthenium-106 source produced by this procedure read 7 R/hr β—γ at its surface. No ruthenium-106 activity could be smeared from the surface of the plate and the protective nickel layer was thin enough so a beta particle degradation problem did not exist. Because a source of greater than 4 R/hr was requested by Health Physics Section personnel, no attempt was made to plate a specific amount of ruthenium-106 onto its surface. Initial platings using a full-scale system illustrated that about 3 R/hr of ruthenium-106 beta activity could be plated onto a source from a plating solution containing 0.25 mCi of ruthenium-106. The source emitting 7 R/hr of ruthenium-106 beta activity was obtained when a plating solution containing 0.5 mCi of ruthenium-106 was used.

Several 347 stainless steel sources emitting ruthenium 106 activity were prepared by this method. In preparing some of these sources, areas where nickel would not plate onto the nickel-ruthenium surface were found. However, after treating these areas with 10% HNO_3 – 2% HF, as in preparation of the 7 R/hr ru-

thenium-106 activity source, these areas were overplated with nickel surfaces.

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

1. A method of preparing a calibration source which emits high energy beta particles, comprising simultaneously electrodepositing nickel and ruthenium-106 onto a stainless steel disc, said ruthenium-106 being carried by natural ruthenium, washing the disc with water, and immediately electroplating the disc with nickel.

2. A method of preparing a ruthenium-106 film badge calibration source comprising:

a. preparing an electrodeposition cell consisting of a stainless steel disc as cathode, a rotating platinum disc disposed one centimeter from the cathode as anode, and containing an electrolyte solution consisting of 1.25 M nickel sulfate — 0.21 M nickel chloride — 0.66 M boric acid and including 0.8 mg ruthenium carrier containing ruthenium-106

b. operating the electrodeposition cell at a current density of 3 mA/cm^2 for a time sufficient to deposit 75 percent of the ruthenium-106 activity on the stainless steel disc;

c. immediately removing the solution from the cell;

d. rinsing the cell including the cathode with water;

e. adding a nickel chloride — nickel sulfate electrolyte solution to the cell; and

f. operating the electrodeposition cell at a current density of 3 mA/cm^2 for 20 minutes to seal the ruthenium-106 activity within the calibration source by electroplating a nickel coating thereon.

3. A method according to claim **2** wherein the source is removed from the cell, covered for one minute with 1 ml of 10 % HNO_3 — 2% HF at 100°F., washed with water, returned to the cell which was filled with plating solution, and plated with nickel using a platinum anode, 1 cm in diameter, above an area on the source onto which nickel did not originally plate.

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