WANL-TME-628
DECEMBER 17, 1963

SUBMITTED BY:

**Westinghouse Electric Corporation
Astronuclear Laboratory
Pittsburgh 36, Pennsylvania**

INFORMATION CATEGORY Unclassified	
<i>D.C. Goldberg</i>	<i>2/20/64</i>
Authorized Classifier	Date

PREPARED BY:

H.O. Hanzlik

H.O. Hanzlik, Process Eng.

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H.E. Ricks
H.E. Ricks

MASTER

APPROVED BY:

F.D. Seaman

**F.D. Seaman, Adv. Eng.
Process Engineering**

D.C. Goldberg

**D.C. Goldberg, Manager
Structural Materials & Processes**

**DEVELOPMENT OF A GOLD
PLATING PROCESS TO
WITHSTAND A 1350°F
BRAZING CYCLE**

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INTRODUCTION

All instrumentation leads must be sealed against leakage where they pass through the wall of the pressure vessel. To accomplish this seal, trunks made up of from 16 to 66 leads are brazed into cylindrical seal blocks (which in turn are mechanically sealed to the pressure vessel). No detectable leakage is permitted through the braze under 750 psi at temperatures as low as -196°C .

In order to form this critical bond with the copper, silver, phosphorous brazing alloy used for the application* the leads are gold plated. The requirements for this plate are:

1. Close control of thickness (and uniform thickness along the lead) to maintain brazing clearances.
2. Sound bonding between the plate and the base metal of the various leads (all leads are sheathed with type 347 stainless steel and pressure probes are 347 stainless steel tubing). It is important that the bond remain sound after exposure to the 1350°F brazing cycle. Otherwise a leak path parallel to the brazed joint is formed.
3. Freedom from included gases from the plating solutions, particularly freedom from hydrogen which would evolve during brazing and blister the plated film.

*TME-624 describes the brazing process.

SCOPE

This report covers the evaluation of two processes and the development of one of these for the gold plating of approximately 500 instrumentation items for NRX-A-1.

MATERIALS

The items that were plated are described below:

<u>Type</u>	<u>Outer Sheath</u>		<u>Wire</u>	
	<u>Size (inches)</u>	<u>Material</u>	<u>Size (inches)</u>	<u>Material</u>
Thermocouple	.040-.045 x .006 wall	AISI 347	.006 dia.	Chromel-alumel
Thermocouple	.040-.045 x .006 wall	AISI 347	.006 dia.	Copper-constantan
Thermocouple	.062-.065 x .006 wall	AISI 347	.011 dia.	Chromel-alumel
Thermocouple	.062-.065 x .006 wall	AISI 347	.011 dia.	Copper-constantan
3-Conductor Cable	.062-.064 x .011 wall	AISI 347	.010 dia.	Copper (2)
			.010 dia.	Nickel clad copper(1)
Tri-axial Cable	.088-.090 x .010 wall	AISI 347	32 AWG	Copper center conductor
(Inner Sheath)	.040	AISI 347		
4-Conductor Cable	.088-.090 x .012 wall	AISI 347	.012 dia.	Copper (4)
Pressure Probe Tube	.125-.130 x .020 wall		None	---

Instrumentation, with the exception of the pressure probe tubing, was made with internal magnesium oxide insulating material.

Plating solutions used to produce the gold plated surface varied with two processes evaluated. In one process, a brush or localized type, proprietary solutions were utilized. The other process (electrolytic bath type) used gold foil anodes of 99.999 purity and a potassium gold cyanide compound (67% gold-plating grade).

EVALUATION OF PROCESSES

Only 1-2 inches of lead must be plated for brazing. However, the complexity of installation was such that it was not possible to pre-determine the location of the braze joint along the length of the lead. As a result, to assure the presence of plating at the braze location, it appeared necessary to plate a 96 inch length on each instrumentation item prior to installation.

(1) Localized Plating Process

The requirement for plating a 96 inch length prompted an inquiry into a means of plating after the instrumentation was installed and the braze location determined. This would reduce the plated length from 96 inches to 1 (or 2) inches.

The most promising localized plating process (sometimes referred to as "brush" plating) is one whereby coatings are applied by flowing the solutions through a felt tipped container. The container is filled with a proprietary electrolyte. The felt tip in conjunction with an electrode applies the coating. With the cooperation of Sifco Metachemical Inc. of Cleveland, Ohio, 32 6-inch lengths of the .125 diameter tubing were plated with .002 inches of gold. (Known as the "Dalic" process).

To compare this type of plate with the tank immersion type electroplating, a seal plug containing 30 of the tubes was brazed.

During the brazing cycle it was noted that, prior to reaching the brazing temperature, a number of the Dalic plated tubes evidenced blistering of the gold plate. The plug was leak tested at 100 psi and found to leak profusely. Bend tests performed on two as-received Dalic plated tubes revealed considerable plate cracking and flaking when bent 5-10 degrees. As noted in the following portion of this report, similar parts batch electroplated could be bent 180 degrees without any evidence of cracking or flaking.

Apparently the brush plate type failed because of either, (1) improper cleaning prior to applying the nickel strike or gold plate, or (2) insufficient activation of the stainless steel surface.

Proper activation of a surface governs the integrity of any electrofilm deposited thereon¹. Because stainless steel is not an active surface, an intermediate active metal coating must be applied to produce a surface upon which an adherent gold coating of high integrity can be deposited. Such a coating is called a strike ("nickel strike" in this case). Proper striking technique requires that the surface to be plated be exposed uniformly to the striking solution and also that the surface thus exposed be supplied with ample solution. Under the conditions imposed by brush plating it is difficult to achieve either condition and impossible, without a destructive check to verify the existence of a proper bond once the strike has been accomplished. Additionally, the rapid plating action which characterizes brush plating requires very high current densities. Excessive current density produces brittle coatings containing large amounts of hydrogen which may have contributed to the flaking that was observed. Finally unless the operator is very conscientious he may inadvertently change the rate of movement across the surface or interrupt the plating. Either action results in loss of adherence or variation in thickness.

Restrictions on using any water in the vicinity of the reactor also ruled out the brush plating process.

For the reasons mentioned above the brush plating technique was not considered applicable.

(2) Batch Type Electroplating Process

During the development phase six-inch pieces were plated in the Westinghouse - Research and Development Center plating laboratories.

As noted earlier, instrumentation leads range in diameter from 40 mils to 125 mils in diameter and wall thicknesses range from 6 to 20 mils. Hence, any cleaning and pickling treatment must produce a thoroughly cleaned surface with a minimum of material removal or reduction in wall thickness.

Preliminary screening tests on the effect of different anodic cleaning treatments on the adhesion of gold plate were made using 6-inch lengths of 63 mil diameter tubing. The results are shown in Table I.

TABLE I
Effect of Anodic Treatment on the Adhesion of
Gold Plate to Stainless Steel Leads

Anodic Treatment	Adhesion of the Gold Plate
1. Dalic proprietary solution anodic treatment followed by Dalic gold plating. Samples supplied by Dalic representative	Poor adhesion; the gold plate flaked off when sample was bent through 180°.
2. Reverse etch in 25% H ₂ SO ₄ followed by nickel strike, gold strike and gold plate	Excellent adhesion; but etching was excessive in local areas. There is danger of etching through the thin wall sections.
3. Reverse etch in a Wood's nickel strike solution followed by nickel strike, gold strike and gold plate	Excellent adhesion; but etching was excessive in local areas. There is danger of perforating the thin wall sections.
4. Reverse etch in 50% H ₃ PO ₄ followed by nickel strike, gold strike and gold plate	Excellent adhesion; etching was uniform on all areas.

As noted in Table I, Process 2, 3 and 4 produced good adhesion of the subsequent plated films but the 4th etching process was the best process from the standpoint of uniform etching. This was the process that was used for plating the 500 instrument leads and was written into a process specification which is attached (P.S. WANL 294582-1).

With a proper etch and close attention to the achievement of proper striking conditions¹ (discussed above) the nickel strike solution (nickel chloride-hydrochloric acid type) was selected, because, from experience, this solution produced maximum activation due to the high hydrochloric acid concentration.

As will be noted, a gold strike solution was used between the nickel strike and final gold plate. The gold strike was formulated with a low gold ion concentration and high cyanide concentration. Plating only occurs in a solution such as this under optimum electroplating conditions (not by inadvertent chemical deposition as would be the case in high-efficiency, concentrated, standard gold solution). Thus any plate deposited must also exhibit good adherence. However, because the required plating conditions are also inefficient, the strike plating thickness is kept to a minimum.

The gold plating solution selected is a high gold, low cyanide type for high plating efficiency². Additionally it is operated at 160-170°F to improve its efficiency. Because the gold strike film is so thin (and the concentrated gold plating solution is hot) it is necessary to immerse the work into the gold plating solution with the current on. This practice avoids the possibility of dissolving the highly adherent gold strike and replacing it with a chemically deposited plate out of the highly concentrated standard gold plating bath.

DESCRIPTION OF THE PROCESS

To plate the instrumentation parts, stainless steel tanks 12 feet long x 20 inches wide and 6 inches deep were used. In order to immerse the parts a wooden frame was made to be used as a rack (Figure 1). The rack design is such that parts to be plated can be secured at each end as well as guided into the solutions with a minimum of bending. The racks, which could accommodate a maximum of 17 parts were moved from tank to tank through the plating cycle. The total cycle requires approximately 3-1/2 or 4 hours. The

sequence used to plate the parts is as follows:

1. Rack parts
2. Wipe parts clean with acetone
3. Make contact to each wire by means of alligator clips at each end and in the middle
4. Clean electrolytically in 50% phosphoric acid (2-3 min)
5. Rinse in tank of circulating tap water (20-30 seconds)
6. Apply nickel strike (2-3 min)
7. Rinse in tank of circulating tap water (20-30 seconds)
8. Apply gold strike (1 min)
9. Apply gold plate (3-3-1/2 hrs)
10. Rinse in tank of circulating tap water (20-30 seconds)

Samples from the above process have been satisfactorily subjected to both the brazing process and to a 180° bend followed by an inspection for cracks. No cracks were observed after bending when the process was followed. An in-process check (Q.C. Request No. 159) by Quality Control was made with the following results, (a) plating thickness at four locations ranged from .0018 to .0020; (b) no cracking or flaking at 100X when specimens were bent 90°. One test, not required, was to heat a 2 inch plated specimen at 650°F for 2 hours, water quench, and examine at 100X for cracking or flaking. Results revealed no cracks or flaking. A number of brazed seal plugs containing instrumentation plated by the process subsequently used were helium leak tested at 100, 1000 psi (room temperature) and 1000 psi (cryogenically-liquid nitrogen) with no leaks reported. (see TME-624)

Since adherence is so important, consideration was given to a test that could be applied to each part to determine adhesion. Various adhesion tests were considered and it was decided that a burnish test on a non-significant area was the most logical approach. This test consists in burnishing through the gold plate with a burnishing tool such as the back of the blade of a pen knife; after a one inch length of the coating is removed by

burnishing down to the bare stainless steel tubing, the knife point is used to attempt to peel the plate from the leads. If the plate cannot be peeled, the adhesion is satisfactory. The test has the advantage that the adhesion on each plated part can be tested.

During the plating cycle parts were diametrically checked at the ends and center to control thickness. Using data obtained by WANL Quality Control, Table II shows the process variation that was encountered during production. These values reflect variation along the length of the lead.

TABLE II
Plating Thickness Variations

Size of Component	Plating Thickness Recorded			Number of Pieces Tested
	Lowest Value	Highest Value	Average	
.0410 inch	.0028	.0033	.0030	14
.0631 inch	.0024	.0028	.0027	125
.0886 inch	.0023	.0035	.0029	99
.1279 inch	--	--	.0028	15

Note: The drawing requires .002 to .004 inch plating (diametrically)

The critical aspects of the process have been documented in WANL 294582-1 which is attached as Appendix I.

CONCLUSIONS

1. The localized or brush plating process evaluated is not suitable for this application because neither the amount or composition of solution or the rate of deposition can be controlled well enough to obtain the high quality plate that is required for this application.
2. A standard, properly operated electroplating process which involves a nickel strike, gold strike and gold plate (as described by WANL 294582-1) produces a surface compatible with the 1350°F brazing process.
3. Maintenance of proper cleaning and striking conditions are essential to assure bonding.

REFERENCES

1. "Electroplating on Nickel and Nickel Alloy" by W. W. Sellers and C. B. Sanborn, Preprint from Technical Proceedings 1957, American Electroplaters Society, Inc.
2. "Note on Electrodeposition of Thick Gold Deposits" by Charles Bauer, Monthly Review of American Electroplaters Society, Volume 39, Number 12 (1952).

BIBLIOGRAPHY

"Principles of Electroplating and Electroforming" by William Blum and George R. Hogaboom (Third Edition) 1949.

FIGURE 1 - PLATING RACK AND TANK

APPENDIX I

PROCESS SPECIFICATION WANL 294582-1

Westinghouse Electric Corporation
Astronuclear Laboratory
P. O. Box 10864
Pittsburgh, Pa. 15236
(Fed. Ident. Code No. 14683)

PROCESS SPECIFICATION WANL 294582 Revision No. 1
(Not for Publication)

November 1, 1963

GOLD PLATING OF STEEL

1. SCOPE

This specification covers procedures for plating a layer of gold on austenitic stainless steel for corrosion resistance or to provide a coating compatible with low temperature brazing materials, designated as follows:

Designation

Description

294582-1

Procedure for plating a layer of gold from 0.0005 to 0.0050 in. thick by electrodeposition on austenitic stainless steel parts immersed in a bath containing a gold compound.

294582-2

NOTE: This process is to be used only for repair work and only with the approval of WANL Quality Control and of Materials.

Procedure for plating a layer of gold from 0.0005 to 0.0030 in. thick on austenitic stainless steel in air by electrodeposition from an absorbent pad containing a gold solution (the Dalic process).

NOTE 1: Unless otherwise specified, the following requirements apply to both designations.

NOTE 2: The thickness of plating desired is to be specified on WANL drawings. If a specific thickness is called out, the tolerance shall be $\pm 25\%$ of that specified.

2. GENERAL REQUIREMENTS

2.1 COMPLIANCE: No change shall be made from these procedures without first obtaining the approval of the purchaser.

2.2 MATERIALS (294582-1)

2.2.1 Acetone designated in Section 3.2.1

2.2.2 Electrolytic etching bath described in Section 3.2.3

2.2.3 Nickel electroplating bath described in Section 3.2.4

2.2.4 Gold plating bath described in Section 4.1

2.2.5 Pure gold anodes for plating bath

294582-2 MATERIALS

2.2.6 Apparatus containing source of direct current and controls of voltage and amperage with two leads, either of which can be made anodic or cathodic:

2.2.6.1 Lead which can be attached to work piece

2.2.6.2 Lead with stylus pad attached. Pad is composed of material which will absorb a considerable amount of liquid.

2.2.7 Electrocleaning solution, Code No. *1010, designated in Section 3.2.6.

2.2.8 Etching solution, Code No. *1022, designated in Sections 3.2.7 and 3.2.8.

2.2.9 Nickel plating solution Code No. *2080, designated in Section 3.2.9.

2.2.10 Gold plating solution, Code No. *3020 (alkaline solution), designated in Section 4.2.

2.3 SAFETY REQUIREMENTS

2.3.1 Acetone (Sections 3.2.1 and 3.2.5) has a flash point of 15°F. Sources of flames or sparks must not be allowed in the vicinity of open vessels containing acetone.

2.3.2 Potassium cyanide (Section 3.2.5 and 4.1) is extremely poisonous. Solutions containing it must not be allowed to contact the skin.

3. PREPARATIONS FOR GOLD PLATING

3.1 SURFACE CONDITION

3.1.1 The surface finish of parts to be plated shall be 63 micro-inches (arithmetic average) or finer.

3.2.2 The surfaces to be plated shall be free of contamination such as mill scale, heat treatment oxide, or dirt.

* Code numbers are designations given by Sifco Metachemical Division of Steel Improvement and Forge Company identifying the proprietary solutions supplied by them.

3.2 CLEANING AND UNDERCOAT PLATING

294582-1 PROCEDURE

- 3.2.1 The parts shall be wiped with a clean cloth soaked with acetone.
- 3.2.2 The parts shall be attached at both ends and the middle using alligator clips (or use spring contacts) to insure uniform current distribution.
- 3.2.3 Parts shall be electrolytically etched in a solution composed of $50\% \pm 5\%$ by volume of phosphoric acid, commercial grade ($84\% \text{H}_3\text{PO}_4$), and 50% by volume of water at room temperature with the parts as anodes. A current density of 50 to 60 amperes per square foot shall be continued for 2 to 3 minutes. Parts shall be rinsed in cold water after removal from the bath.

NOTE: If parts are not uniformly cleaned, wipe with a clean cloth and repeat the phosphoric acid anodic etch. It shall not be repeated more than two times (a total of three times).

- 3.2.4 A nickel strike shall be electroplated on the parts in a bath containing 32 ($\pm 10\%$) oz.* nickel chloride per gal. and 16 ($\pm 10\%$) liquid oz. hydrochloric acid (specific gravity-1.135 min.) per gal. at room temperature. The current density shall be 50 to 60 amp. per sq. ft. for 2 to 3 minutes. Parts shall be rinsed in cold water after removal from the bath.
- 3.2.5 A gold strike shall be electroplated on the parts using a current density of 50 to 60 amps per sq. ft. for 1 minute. The parts shall be immersed with the current on in a bath at room temperature of the following composition:

Gold (in potassium gold cyanide), $1/4$ Troy ($\pm 10\%$) oz. per gal.

Potassium cyanide, 3 ($\pm 10\%$) oz. per gal.

The part shall be transferred immediately to the gold plating bath without rinsing.

294582-2 PROCEDURE

- 3.2.6 The parts shall be degreased with acetone.

* Avoirdupois unless otherwise noted.

- 3.2.7 Electroclean the part with the current regulator in reverse position using Code 1010 solution for 15 seconds at 10 to 15 volts. Rinse the part with water after removing the clip.
- 3.2.8 Etch the part with the current regulator in reverse position with Code 1022 solution in the pad for 15 seconds at 10 to 15 volts. Rinse the part with water after removing the clip.
- 3.2.9 Remove passive film from the part with the current regulator in the forward position using Code 1022 solution in the pad for 15 seconds at 12 to 20 volts. Rinse the part with water after removing the clip.
- 3.2.10 Immediately after rinsing start plating the nickel strike on the part with the current regulator in the forward position using Code 2080 nickel solution in the pad at 8 to 30 volts. The pad should be moved over the part at 20 to 100 ft. per min. Continue plating until 0.0001 to 0.0002 in. nickel is deposited. Rinse the part with water after removing the clip.

4. GOLD PLATING PROCEDURE

294582-1 PROCEDURE

- 4.1 The parts shall be immersed in an electroplating bath immediately after removal from the gold strike bath, with the current on at a temperature of 160° to 170°F of the following composition:

Gold (as potassium gold cyanide), 2 to 3 Troy oz. per gal.
Potassium cyanide, 16 (+ 10%) oz. per gal.
Potassium hydroxide, 0.45 to 0.55 oz. per gal.
Potassium sulfite 0.45 to 0.55 oz. per gal.

The anodes shall be commercially pure gold (99.99% gold min.).

The current density shall be 2 to 4 amperes per square foot.

If a center clip is used, it should be removed after 20 minutes plating time.

After plating is completed, the plated surfaces shall be rinsed in cold water and air dried.

294582-2 PROCEDURE

- 4.2 Gold plate the part with the current regulator in the forward position using Code 3020 alkaline solution in the pad at 4 to 20 volts, usually about 10 volts. The anode pad should be moved over the part at 30 to 100 ft. per minute. Continue until the thickness specified by the applicable drawing is deposited. Rinse with cold water after removing the

clip and allow the part to dry.

5. ACCEPTANCE STANDARDS

294582-1 ACCEPTANCE STANDARDS

- 5.1 The gold plating shall be visually inspected. If the plating is not uniform and shows treeing or plate buildup, the part is subject to rejection.
- 5.2 The gold plating close to the end of the plated portion of each part shall be scraped off to the basic metal and an attempt made to peel the plating. If it peels, the part is rejected.
- 5.3 At least one of each lot of 100 parts shall be destructively tested.
 - 5.3.1 A cross section shall be mounted in plastic, polished and examined at 1000X on a metallographic microscope and the plating thickness shall be measured. If the thickness does not conform to drawing requirements, the lot is subject to rejection.
 - 5.3.2 A plated section shall be bent 90° over a radius equal to twice the diameter of the part and examined at 5X magnification. Flaking or cracking of the plating shall be cause for rejection of the lot.

294582-2 ACCEPTANCE STANDARDS

- 5.4 In addition to the tests specified in Sections 5.1 and 5.2, a section of each plated part shall be bent about 5° over a one-inch length (12 in. radius approximately).
- 5.5 The bent section shall be visually inspected at 5X magnification.
- 5.6 If cracks are observed in the plating, the part is subject to rejection.
- 5.7 REPAIRS (294582-1 and 294582-2): Proposals to repair rejected parts and the method of repair shall be submitted to WANL for approval.

6. RECORDS AND REPORTS

- 6.1 PROCESSING DATA (294582-1): The compositions of baths, bath temperatures, processing time periods, and current densities used in processing parts according to Sections 3 and 4 shall be recorded and reported to WANL.

(294582-2): The solutions used (as identified in Sections 2.2.8 to 2.2.10), the time periods for applying solutions, and current densities used in processing parts according to Sections 3 and 4 shall be recorded and reported to WANL.

- 6.2 TEST RESULTS AND SAMPLES: The conditions of testing and the results of tests specified in Section 5 shall be recorded and reported to WANL. Parts destructively tested in accordance with Section 5.3 and the specimens prepared therefrom shall be submitted to WANL.