REPORT AE63-0013

INVESTIGATION AND ANALYSIS OF THE ELECTROPOLISHING PROCESS FOR SATURN DUCTING

Task Order No. M-ME-TLB-C-2
Contract No. NAS 8-818

Prepared by

GENERAL DYNAMICS/ASTRONAUTICS
San Diego, California

31 January 1963

GPO PRICE $ ______

OTS PRICE(S) $ ______

Hard copy (HC) #3.00

Microfiche (MF) #0.75

FACILITY FORM 602

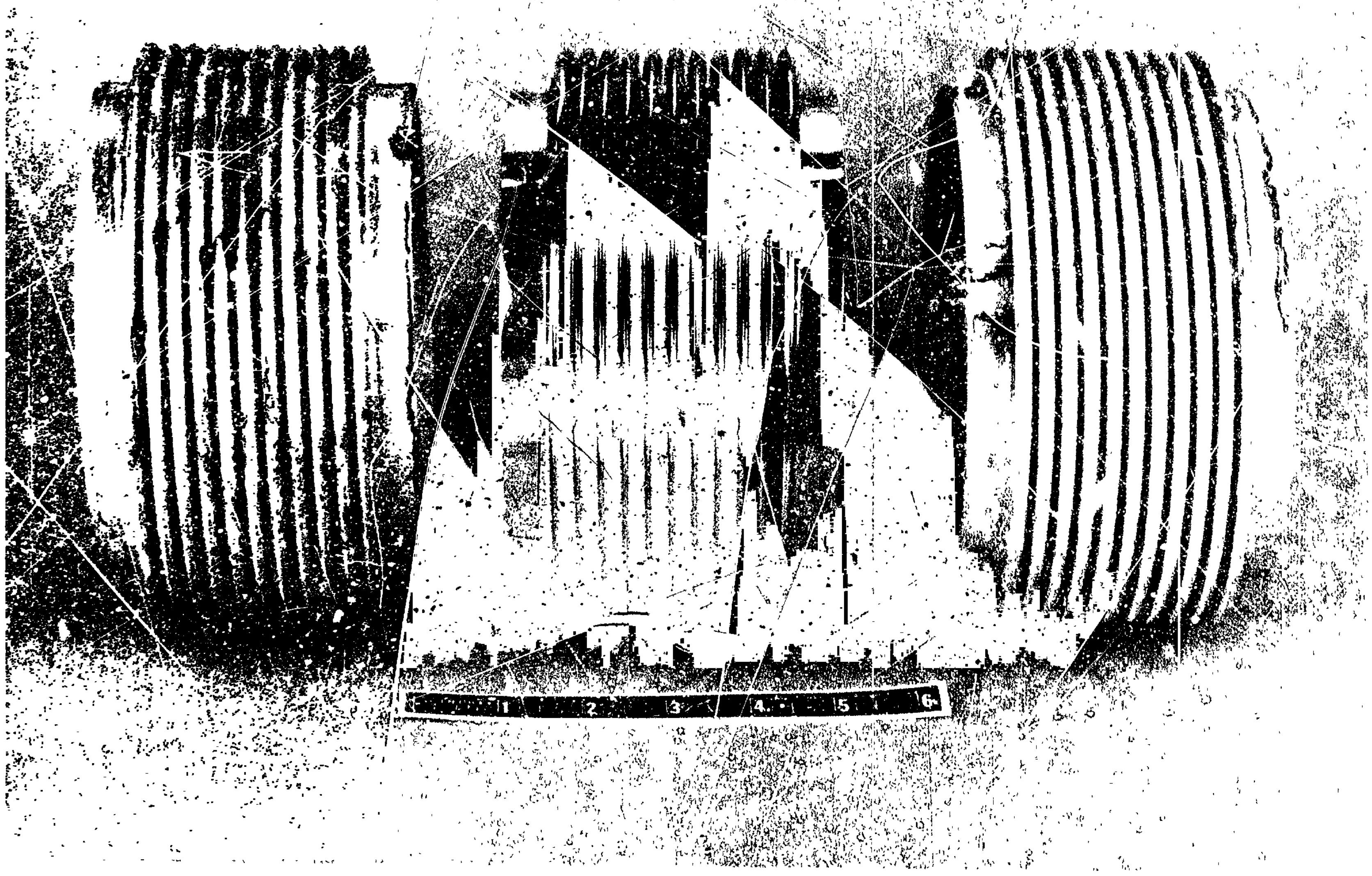
N65-22641 (ACCESSION NUMBER)	(THRU)
82 (PAGES)	1 (CODE)
CR 57864 (NASA CR OR TMX OR AD NUMBER)	17 (CATEGORY)

Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
George C. Marshall Space Flight Center
Fabrication and Assembly Engineering Division
Huntsville, Alabama

FACILITY FORM 602

(ACCESSION NUMBER)	(THRU)
(PAGES)	(CODE)
(NASA CR OR TMX OR AD NUMBER)	(CATEGORY)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AE63-0013
31 January 1963

FOREWORD

This report describes the work performed on Task Order M-ME-TLB-C-2 and Amendment 2 to Contract NAS 8-818, "The Study of the Corrosion of Metals for Saturn." This is the final report on the subject contract. The experimental program was performed by the materials research group, General Dynamics/Astronautics under the sponsorship of the Fabrication and Assembly Engineering Division, Technical Liaison Office, Marshall Space Flight Center, Huntsville, Alabama.

The Task Order extends the effort on the investigation of the electropolishing process and protective coating systems for stainless steel propellant ducting and components initiated under the prior Task Order of this study program.

The work discussed in this report was performed during the period from 15 March 1962 to 31 December 1962.

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AE63-0013
31 January 1963

ABSTRACT

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Type 321 and Type 302 stainless steels and certain high-nickel alloys which include Inconel 718, Hastelloy C, and René 41 were electropolished in a series of formulated and vendor baths. After electropolishing, samples were subjected to investigation and analyses including salt spray, salt atmosphere, and seacoast environment corrosion tests, profilometer, and weight-loss measurement tests. The immersion and impingement methods of electropolishing were evaluated for use on propellant ducting and flexible metal hoses and sections of bellows. Electropolishing greatly increases the resistance of stainless steels to corrosion, and its use is recommended for this purpose. Nine types of organic coatings were evaluated for protection of propellant ducting at cryogenic temperatures. Air-drying epoxy and acrylic formulations were found satisfactory for large assemblies that cannot be baked, and a heat curing modified silicone resin was found to be best for small assemblies or parts that can be baked.

The impingement method of electropolishing, whereby large parts (any dimensions) can be polished, was proven successful. This constitutes a breakthrough in electropolishing since it eliminates the use of tanks, which are necessarily limited in size and capacity.

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SECTION 1

INTRODUCTION

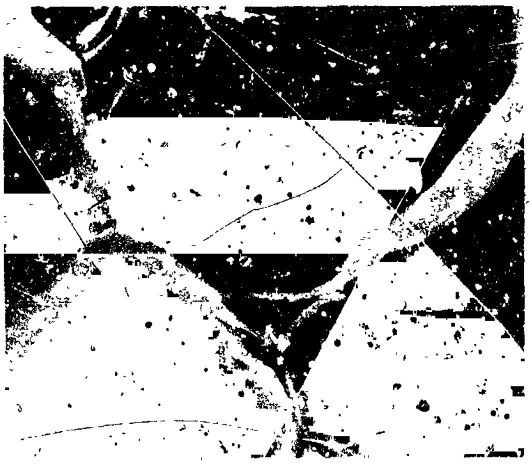
It was found during a previous study under this contract (1) that electropolishing eliminates the selective corrosive attack so noticeable in the heat-affected zone of stainless steel weldments. It was also noted that certain organic formulations used as protective surface coatings, stood up well in cyclic tests from room temperature to -320°F when applied to stainless steels, and that they provided remarkable resistance to corrosion. Task Order M-ME-TLB-C-2 was outlined to obtain additional information on the electropolishing process and protective coating systems for possible use on Saturn propellant ducting.

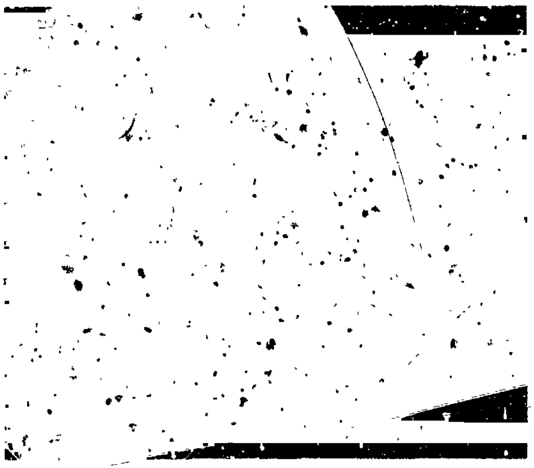
The Task Order was divided into two phases: Phase I, Investigation and Analysis of Electropolishing Processes; and Phase II, Evaluation of Protective Coating Systems for Saturn Ducting. An amendment was added to the Task Order to investigate the feasibility of electropolishing large components by the impingement method, and to include in the program the electropolishing of certain high-nickel alloys. This work was completed as a continuation of Phase I.

Adequate rinsing is an important phase of the electropolishing process. Corrosion staining in many instances can be attributed to improper rinsing after electropolishing. Therefore, rinsing techniques were investigated during this program.

Pretreatment cleaning cycles prior to electropolishing were evaluated. They included solvent cleaning, alkaline cleaning, chemical pickling, and vapor honing. These are reported fully in Appendix B.

The following photomicrographs show clearly the advantages to be gained with electropolishing. The photographs are of two normal 321 stainless steel samples (pickled and passivated; magnification, 6000 times). Note that in the electropolished sample (shown on right) potential corrosion sites have been virtually eliminated.





1.1 DETAILED ANALYSIS AND EVALUATION OF ELECTROPOLISHING SOLUTIONS (PHASE IA). Electropolishing baths commonly in use in this country are composed of sulfuric, phosphoric, and chromic acids(2). Since sulfuric or phosphoric acids are the major constituents, the baths are referred to as the sulfuric-phosphoric type. Perchloric-acetic anhydride baths are also used commercially, especially in Europe. This type of bath is mentioned because it polishes a wider range of different metals. Jacquet (3) obtained considerable success with a mixture of perchloric acid and acetic anhydride. Young and Brytczuk (4) reported reflectivity as high as 40 percent using the perchloric batch, while 32 percent was the highest reported value using the sulfuric-phosphoric mixture. (See Figures 1, 2, and 3.)

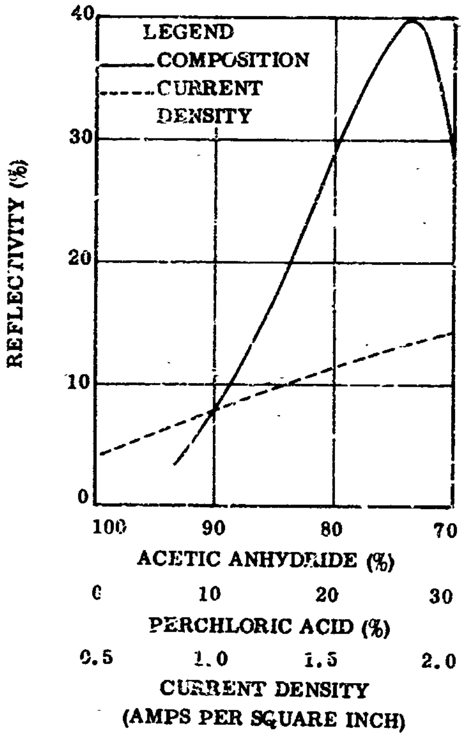


Figure 1. Reflectivity as It Is Affected: 1) by the Composition of the Solution (Perchloric-Acetic) and 2) by Current Density.

Perchloric acid and acetic anhydride baths have not been accepted for commercial work in this country for these reasons:

a. Perchloric acid is a very strong oxidizer and can become a safety hazard because of its violent reaction to mixing.

b. These baths require a low operating temperature (not over 30°C), which necessitates the use of refrigeration.

c. These electrolytes are more expensive.

Therefore, this investigation was restricted to the standard sulfuric-phosphoric type bath presently used on stainless steels.

1.2 LIMITING CONDITIONS IMPOSED BY THE DESIGN OF THE TEST SPECIMEN (PHASE IB). Propellant components (consisting of bellows with open and closed convolutes, flexible metal hoses of various diameters, ducting, and disconnects) were typical test parts that were electropolished and subjected to accelerated corrosion tests. These were selected because they incorporated the sizes, geometries, recessed

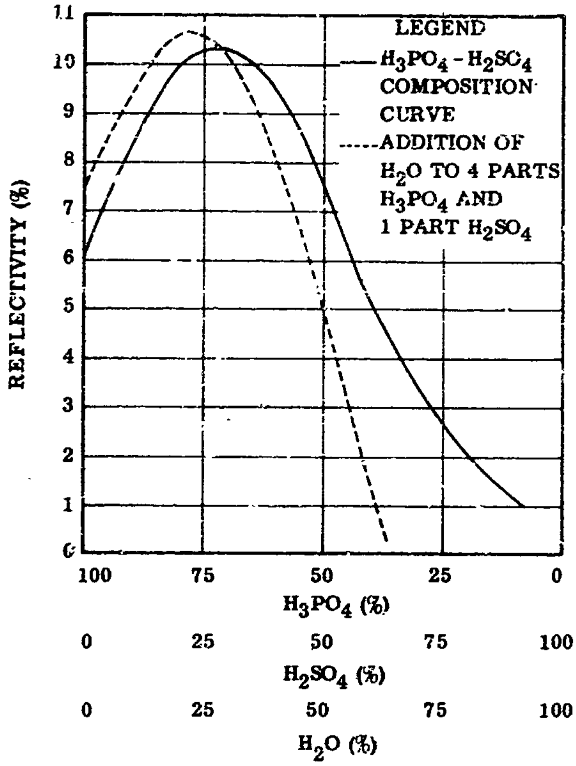


Figure 2. The Effect on Reflectivity of Varying the Amounts of Phosphoric and Sulfuric Acids in the Bath.

areas, etc. which are found in standard missile propulsion systems. Auxiliary cathodes were designed to determine the limitations of electropolishing recessed areas.

Previously, the size of parts which could be electropolished was limited by the size of tanks available in commercial electropolishing facilities. Therefore, an investigation of the impingement method of electropolishing was conducted. By this method. an electrolyte is forced through a perforated cathode nozzle and impinged directly on the article to be polished. This method eliminates the need for an electropolishing installation that provides immersion in a tank (of a specified size) filled with solution. Thus. electropolishing by the impingement method can be accomplished quickly on any part, regardless of size.

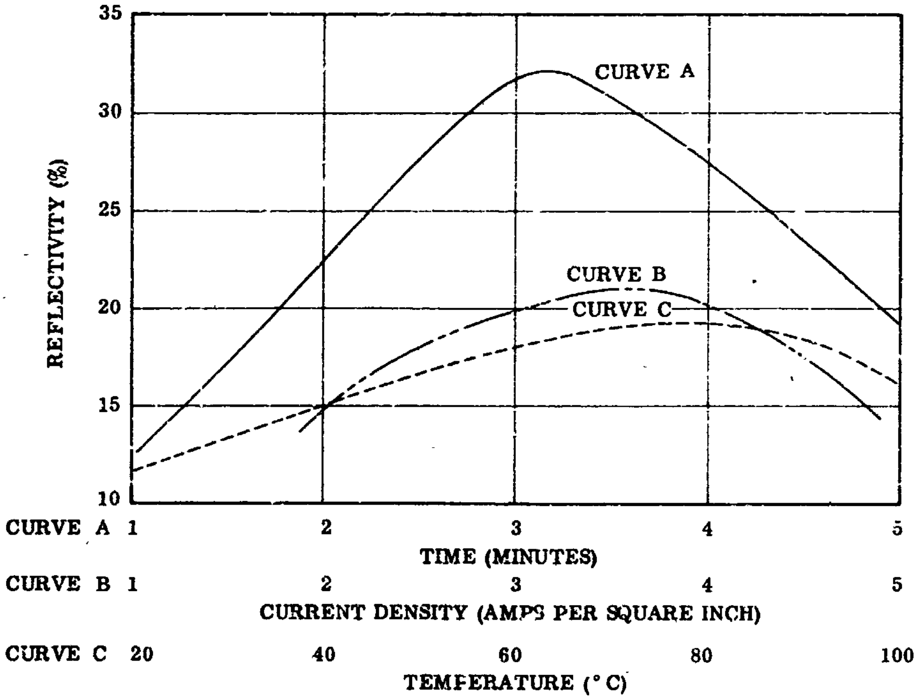


Figure 3. Effect of Time, Current Density and Temperature on Reflectivity.

1.3 THE EFFECT OF ELECTROPOLISHING ON MECHANICAL PROPERTIES (PHASE IC). Engineering requirements (General Dynamics/Astronautics Specification 0-75089) states that electropolishing shall not adversely affect physical strength and ductility properties of the parts involved. The effect of electropolishing on the mechanical properties of austenitic stainless steels and Type 321 and Type 304 stainless-steel alloys was studied at 78°F and -320°F.

1.4 PROTECTIVE COATING SYSTEMS (PHASE II). The formulations under consideration are relatively durable protective coatings including FEP Teflon, Vinylidene fluoride, silicones, and modified alkyds. Air-dry formulations such as epoxy, acrylic, polyurethanes, and modified nitro-cellulcse, all presently used on missile components, are also included in the evaluation.

During the test program, evaluations were made of hardness, flexibility, adhesion, resistance to cleaning solvents, and resistance to water immersion. The ability to withstand cryogenic cycling from -320°F to room temperature without loss of adhesion to stainless steel was a prime requisite of all the coatings tested.

SECTION 2

MATERIALS

The materials under study were limited to Type 321 and Type 304 stainless steels and certain high-nickel alloy materials presently being considered for use in the Saturn propellant ducting system.

2.1 BASE METALS. The stainless steels used in this investigation conformed to AISI compositional limits and to MIL-S-6721B. The stainless steel specimens were composed of Type 321 and Type 304 ELC (Extra Low Carbon). The tempers studied in each alloy were:

a. AISI Type 321, annealed, in form of 3/8-inch diameter rod, 4-inch diameter tubing, in sheet form.

b. AISI Type 304 ELC, annealed, in form of 3/8-inch diameter rod, in sheet form.

c. AISI Type 304 ELC, in 70-percent cold-worked sheet.

Since certain of the high-nickel alloys are being considered as base materials for liquid propellant ducting and components, in combination with the above alloys, investigations were also conducted on Inconel 718, Inconel X, Hastelloy C, and René 41 alloys in the form of annealed sheet.

All welds were butt welds made with the Tungsten Inert Gas (TIG) method. The high-nickel alloys include Inconel 718, Inconel X, Hastelloy C, and René 41. The nominal chemical compositions and thicknesses of all alloys are listed in Table I.

2.2 ELECTROLYTES. Five vendor and five baths formulated by General Dynamics/Astronautics were used in this investigation. The five vendor baths were:

Number 1. Electro Glo "300" by Electro-Glo Co., 621 Kolmar Ave., Chicago 24, Illinois.

Solution: Phosphoric-Sulfuric acid type (concentrations not revealed)
Cathode: Lead
Voltage: 4 to 9 volts
Current Density: 50 to 216 amps/square foot
Temperature: 110 to 135°F

Number 2. Electro-Gleam 55 by MacDermid Corp., Waterbury 20, Connecticut.

Solution: Phosphoric-Sulfuric acid type (concentrations not revealed)
Cathode: Lead
Voltage: 7 to 15 volts
Current Density: 100 to 300 amps/square foot
Temperature: 160 to 200°F

Number 3. Battelle 41/45 Electropolishing Solution for Stainless Steel by Battelle Memorial Institute, Columbus 1, Ohio.

Solution: Phosphoric-Sulfuric acid type (concentrations not revealed)
Cathode: Lead
Voltage: 7 to 15 volts
Current Density: 300 to 450 amps/square foot
Temperature: 190 to 220°F

Number 4. An undesignated solution (employed for larger components) by Electro Process and Engineering Corp., 3375 Hancock Street, San Diego 10, Calif.

Solution: Phosphoric-Sulfuric acid type (concentrations not revealed)
Cathode: Copper
Voltage: 7 to 15 volts
Current Density: 100 to 300 amps/square foot
Temperature: 125 to 140°F

Number 5. Molectrics, Inc., 459 N. Eucalyptus Ave., Inglewood 3, California.

Solution: Phosphoric-Sulfuric acid type (concentrations not revealed)
Cathode: Copper
Voltage: 7 to 15 volts
Current Density: 100 to 300 amps/square foot
Temperature: 125 to 150°F

The five baths formulated by General Dynamics/Astronautics were:

Number 6. Solution: 40 percent (by weight) orthophosphoric acid
52 percent glycerine (specific gravity 1.26)
8 percent water
Cathode: Stainless steel
Voltage: 4 to 5 volts for new solution, but on aging, rises to 7 to 8 volts
Current Density: 72 amps/square foot
Temperature: 230°F (±9 degrees)

Number 7. Solution: 15 percent (by weight) sulfuric acid
70 percent orthophosphoric acid
15 percent water
Cathode: Copper
Voltage: 8 to 15 volts
Current Density: 72 to 216 amps/square foot
Temperature: 180 to 190°F

Number 8. Solution: 57 percent (by weight) orthophosphoric acid
20 percent sulfuric acid
1-1/2 percent chromic acid
21-1/2 percent water
Cathode: Copper
Voltage: 3-1/2 volts
Current Density: 228 amps/square foot
Temperature: 125 to 150°F

Number 9. Solution: 55 percent (by weight) glycolic acid
30 percent sulfuric acid
15 percent water
Cathode: Copper
Voltage: 3-1/2 volts
Current Density: 228 amps/square foot
Temperature: 180°F

Number 10. Solution: 65 percent (by weight) orthophosphoric acid
15 percent sulfuric acid
6 percent chromic acid
14 percent water
Cathode: Lead
Voltage: 4 to 10 volts
Current Density: 300 amps/square foot
Temperature: 120 to 150°F

2.3 PROTECTIVE COATING SYSTEMS. The protective coating systems included in this study are:

Type of Resin	Manufacturer	Manufacturer's Designation
Fluoroethylene propylene	DuPont Co.	FEP-120
Phenolic fluorocarbon copolymer	Acheson Colloids Co.	Emralon-310
Vinylidene fluoride	Pennsalt Chemical Co.	RC-2525
Silicone (modified)	Glidden Paint Co.	Nubelon-S
Epoxy	Andrew Brown Co.	A423
Acrylic	Sherwin-Williams Co.	M49WC8
Polyurethane	Andrew Brown Co.	MIL-C-27227
Nitrocellulose	W. P. Fuller Co.	MIL-C-7178
Alkyd (modified)	Andrew Brown Co.	MIL-E-7729

All materials were commercial preparations with the exception of the vinylidene fluoride material, which was prepared by straining Pennsalt RC2525 resin through a 2-1/2 mm sieve, adding 70 grams of the strained resin to 30 grams of titanium dioxide, and then emulsifying to application consistency with approximately 800 ml of dimethylacetamide.

SECTION 3

EXPERIMENTAL PROCEDURES

The experimental procedures used in this program were, in the main, developed expressly for this program by General Dynamics/Astronautics.

3.1 THE ELECTROPOLISHING TEST CELL. An electropolishing test cell was developed for use in this program. The concepts involved in this cell were formulated by Bertein (11); however, these concepts and Bertein's miniature and inaccurate device have been altered and developed (during this program) until the test cell is now an effective measuring device. This test cell eliminates the voluminous research formerly required to determine the optimum operating conditions for polishing solutions chosen for chemical considerations.

On applying a fixed current to this newly developed cell, the anode (which is enclosed by a glass shield) shows zones of varying surface finish. Inspection indicates whether the solution is suitable for polishing. The optimum ranges of current density can be estimated roughly from the position and length of the satisfactorily polished zones, since the glass shield prevents the current from acting on the anode except by going around the glass shield. See Figures 4 and 5.

The cell has a four-liter capacity; it uses a two-inch diameter glass tube as a mask and a 3/8-inch diameter stainless-steel rod as an anode. The glass mask is submerged one inch below the solution and the polish on the anodic rod (set up as an extension of the polishing zone) is evaluated. The beaker (with a circular cathode of copper, lead, or stainless steel depending on the particular electrolyte involved) can serve as a small electropolishing tank if the glass-mark assembly is removed. Sections of 4-inch diameter tubing (3 inches by 4 inches) were polished in the various electrolytes with this arrangement and subsequently tested.

Type 304 and 320 stainless steel samples 10 inches long and 3/8 inch in diameter were used in the test cell to obtain current density versus amperage curves for the test solutions. Results are plotted in Figures 6 and 7.

3.2 ELECTROPOLISHING BY IMPINGEMENT. A method of electropolishing flat sheets and cylindrically shaped objects by pumping an electrolyte through the cathode and impinging it on the object to be electropolished, which is made anodic, was set up at the Electro Process & Engineering Corp., under the technical direction of the Materials Research Group, General Dynamics/Astronautics. The impingement electropolishing equipment and procedures developed by General Dynamics/Astronautics represent considerable improvement and simplification of a process developed previously by Yuganson (8).

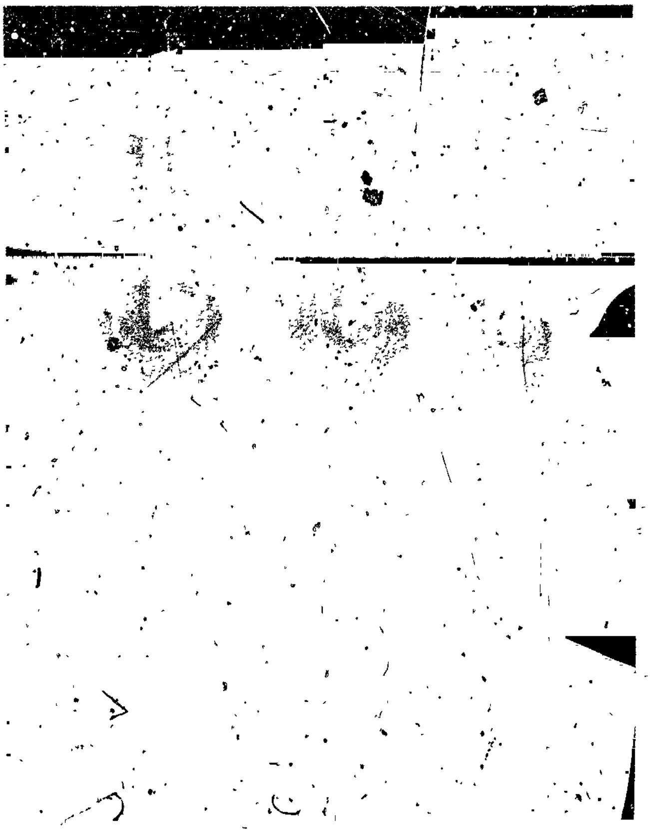


Figure 4. Glass Mask and Anode Used in Electro-polishing Cell. This apparatus indicates the bright range of the electrolyte.

The method consists of pumping solution through a hose to which a nozzle containing a perforated disc is attached. The disc is perforated to allow solution to flow on to the surface to be electropolished. Current is supplied to the disc by a bolt-type connection which is connected to the negative pole of a generator. The positive lead is connected to the object to be electropolished.

A clearance is maintained between the cathode and the surface to be polished by a layer of porous fiberglass cloth. The solution saturates the fiberglass and fills the gap between the work and the cathode, completing resistance and, consequently, the anode current density.

By this method, electropolishing of the surface directly under the cathode is possible. Rotating the work or the cathode selectively polishes the surface without restriction to the length or width of the part to be processed.

A set of operating conditions have been determined by tests that successfully electropolish stainless steel; however, the process is not restricted to any particular alloy. The operating conditions (see Figure 8A and 8B) include:

a. Control of the temperature of the electrolyte.

b. Solution flow-rate, time of polishing or metal removal rate.

c. Cathode head speed.

d. Distance between cathode and anode.

e. Current density.

3.3 ELECTROPOLISHING THROUGH THE USE OF ULTRASONICS. A 4 KW NARDA ultrasonic unit (laboratory model, 5-gallon capacity, transducers in bottom of tank) was used in high-frequency ultrasonic electropolishing experiments. Test specimens

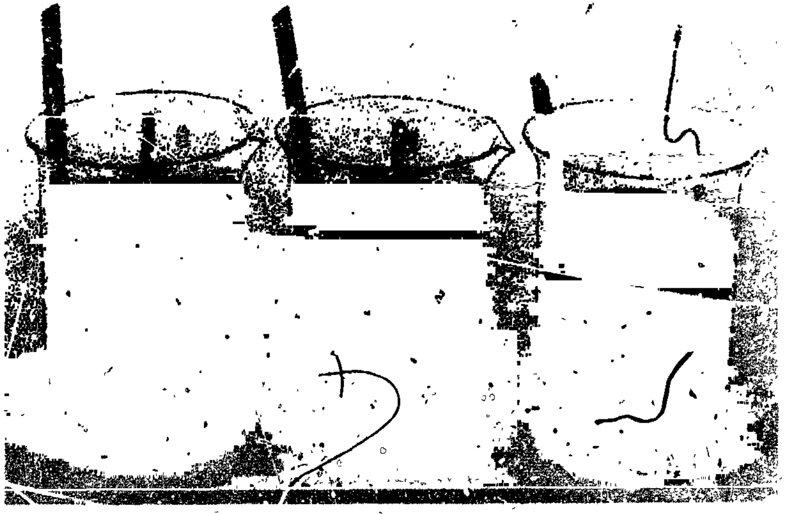


Figure 5. Electropolishing Test Cell. This cell was developed especially for this program. The cathodes are made of either copper or lead.

were electropolished in a phosphoric-sulfuric electrolyte with the unit "on" and then electropolished with the unit "off". Ultrasonics did not improve polishing to any significant degree at high frequencies; however, the scope of this contract did not permit experimentation extensive enough to enable any conclusions about ultrasonic electropolishing to be made.

Transducers attached to the sides of the tank convert high-frequency electrical energy into high-frequency sound waves. The sound waves travel through the liquid in a direction perpendicular to the face of the transducer. As the sound waves travel through the liquid they create an invisible stream of sub-microscopic bubbles which constantly form and collapse, bombarding the surface of the part. The most effective sound-wave frequency is dependent, to a great extent, on the contour of the part. At higher frequencies the stream of bubbles become more directional and travel in a narrow path. In this form, they are particularly effective in reaching deep, narrow recesses. More experimentation is required to determine the correct frequency and time to allow the bubbles to grow to the most effective size.

3.4 RINSING PROCEDURES. An evaluation of various rinsing methods was conducted including effect of tap water, deionized water, and chromates in faying surfaces after electropolishing.

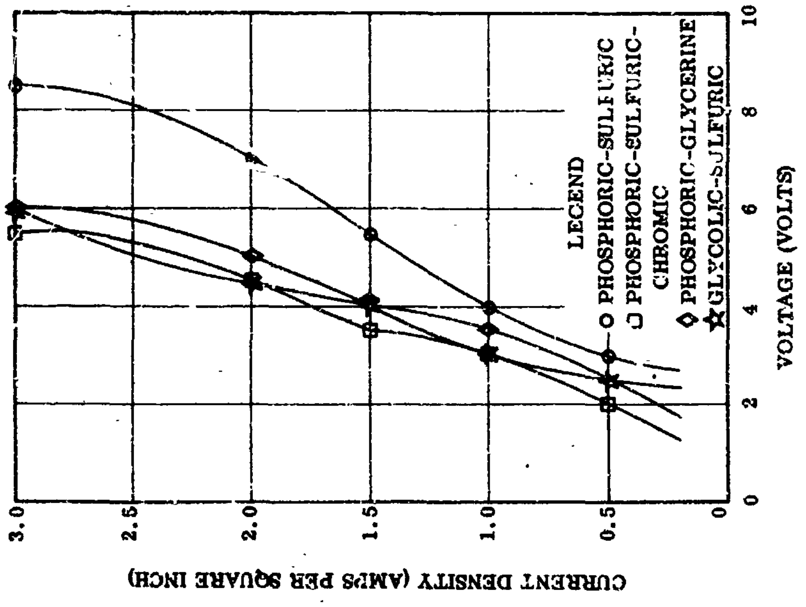


Figure 6. Current Density as a Function of Voltage, Type 321 Stainless Steel.

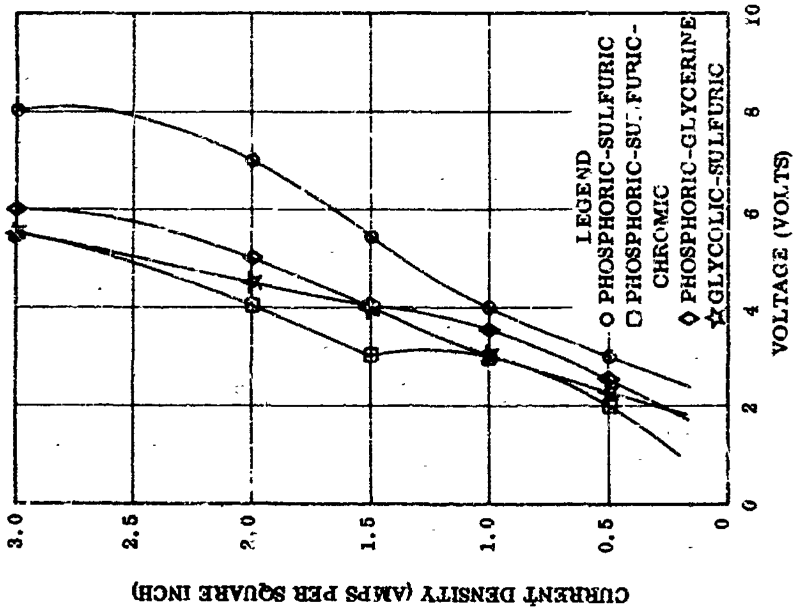


Figure 7. Current Density as a Function of Voltage, Type 304 Stainless Steel.

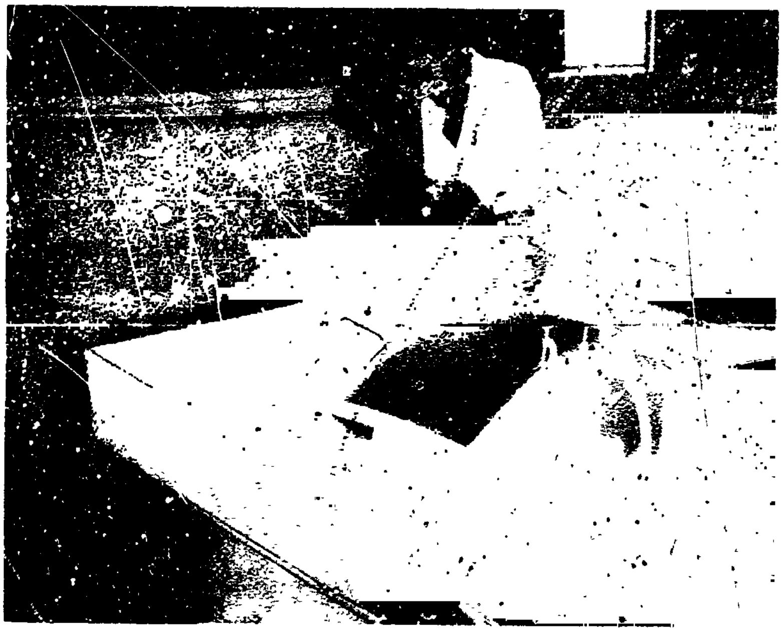


Figure 8A. The Impingement Process. Here, a large flat sheet, that would be polished only with difficulty by the immersion method, is polished easily with the impingement method.

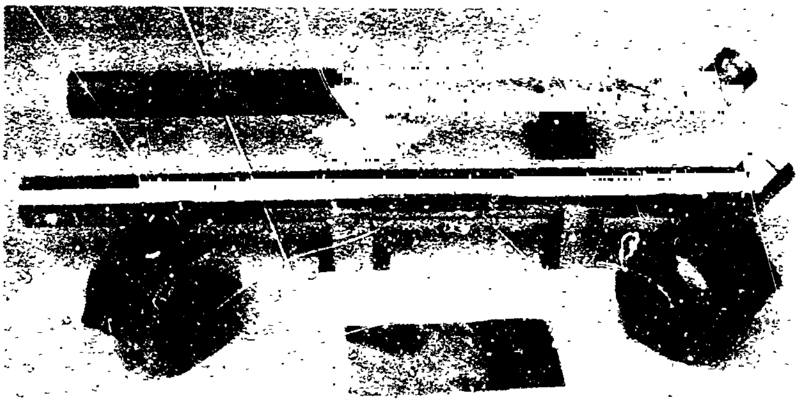


Figure 8B. Production Parts Electropolished by Impingement. Some parts were polished and some unpolished for purposes of comparison. Note the superior brightness of the electropolished parts. The longest part shown is 5 feet in length.

The rinsing methods evaluated were:

a. Rinse Method No. 1:

  1. Tap-water immersion followed by a forced tap-water rinse at room temperature.
  2. Immersion in a solution containing 10 percent sulfuric acid plus 2 percent sodium dichromate at 70 to 90°F. This removes a slight milky haze which sometimes forms on the surface of the work after the part has been rinsed.
  3. Warm tap-water rinse to facilitate drainage.
  4. Heated demineralized-water rinse to eliminate water spotting.

b. Rinse Method No. 2:

  1. Tap-water immersion followed by a forced tap-water rinse at room temperature.
  2. Demineralized-water rinse at room temperature.

c. Rinse Method No. 3:

  1. Tap-water immersion followed by a forced tap-water rinse at room temperature.
  2. Sodium bicarbonate and demineralized water rinse.
  3. Demineralized-water rinse at room temperature.

d. Rinse Method No. 4:

  1. Tap-water immersion followed by a forced tap-water rinse at room temperature.
  2. Heated demineralized-water rinse.

The test samples were dried with clean, filtered forced air and/or nitrogen gas. One set was dried in a vacuum oven (20 inches of Mercury) at 230°F, the other set dried at 230°F (no vacuum).

Baths were selected to represent the widest variation of chemical formulations used in electropolishing during this program. The overlapped test specimens were then subjected to salt spray tests and examined for corrosion staining, corrosion pitting, and residual salts left in the faying surface. All test specimens were electropolished for three minutes at 2 amps/square inch at the temperature recommended for each individual bath.

3.5 METAL REMOVAL BY ELECTROPOLISHING. Determination of the amount of metal removed by electropolishing was accomplished with a number of methods. To obtain information on the comparative rates of metal removal achieved in various electropolishing baths, duplicate samples of 3 inch long rods, 3/8 inch in diameter, of Type 304 and 321 stainless steel were immersed in baths 1 through 6 for 8 minutes at a current density of 2 amps/square inch and their losses in weight determined. The data are presented in Table II.

In other tests, the electropolishing bath, the material, the length of time, voltage, and current of electropolishing were varied and the amount of metal removed was determined by measuring the diameters of the rods before and after electropolishing. See Table III. Measurements were made with a vernier micrometer which permitted measurements to be made to an accuracy of ±0.0001 inch. The purpose of these tests was to obtain an overall picture of the metal removal by electropolishing over a wide range of operating conditions.

The final tests on determination of metal removal by electropolishing were conducted on Types 321 stainless steel and a number of the high-nickel alloys. Triplicate 3-inch square samples of the metals were electropolished in bath 10. This bath was found best in overall electropolishing performance on the high-nickel alloys; the electropolishing conditions were 4 minutes at 8 volts and 130°F bath temperature. The samples were carefully weighed before and after electropolishing, and the thickness of metal removed from one side of the specimens was determined from the equation:

$$t = \frac{\Delta W}{(2A + 4\ell t_o)\rho}$$

where

t = thickness of metal removed from one side of the sample

ΔW = weight loss

A = surface area of one side of sample

ℓ = length of one side of square sample

t_o = original thickness

ρ = density of alloy

The results of these tests are presented in Table IV.

3.6 SMOOTHNESS OF ELECTROPOLISHED SURFACES. Ten-inch long, 3/8-inch diameter rods of Types 304 and 321 stainless steel were electropolished in vendor baths 1 through 4 using the electrolytic test cell. The operating conditions corresponded to those described in Section 2.2 for each electrolyte. The surface smoothness before and after electropolishing was determined with the Mototrace Profilometer, Model 5, manufactured by the Physicists Research Co., Ann Arbor, Michigan. The results of the tests conducted on the stainless steel rods are shown in Table V.

Samples of the high-nickel alloys, 3 inches x 3 inches in size were electropolished in bath 10 for 3 minutes. Surface smoothness measurements before and after electropolishing were made along directions both parallel and perpendicular to the original rolling or grinding directions of the alloys, and the results are presented in Table VI.

3.7 EFFECT OF ELECTROPOLISHING ON MECHANICAL PROPERTIES (PHASE IC).

Types 321 and 304 stainless steel alloys were subjected to tension tests both in smooth and notched (stress concentration factor, $K_t = 6.3$) configurations to provide values of yield and tensile strength, elongation, and notched-unnotched tensile ratios of the base metal. The materials used in these tests included Type 321 stainless steel sheet, 0.032-inch thick, in the annealed condition, Type 304 ELC stainless steel sheet, 0.20-inch thick, in the annealed condition, and Type 304 ELC stainless steel sheet, 0.20-inch thick, cold-rolled 70 percent. Three to five replicate tensile tests using longitudinal coupons were conducted at room temperature and at -320°F, using both smooth and notched tensile test specimens having the dimensions shown in Figure 9. The notched coupon yields a stress concentration factor of 6.3 by virtue of its 0.0025-inch root radius and 0.20-inch width at the root of the notch.

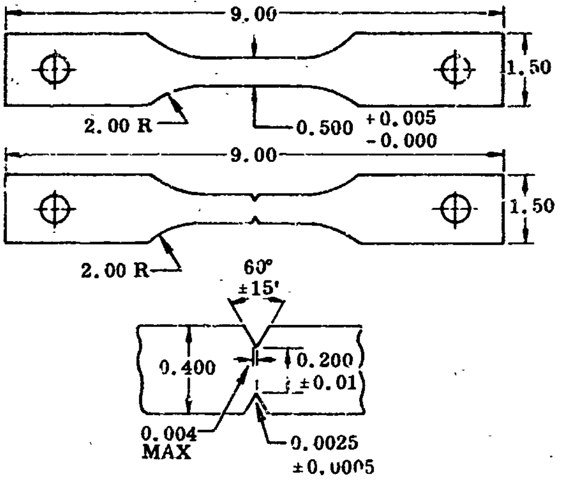


Figure 9. Notched and Flat Tensile Specimens.

The notched tension tests were included in this study to evaluate toughness, a measure of resistance to brittle failure (12). Toughness is a property of vital importance in missile design because missile propellant systems are subjected to shock-type loads as a result of hydraulic hammer caused by rapid disconnect, and to vibration from rocket-engine firing and quick-closing valves. In addition, missiles contain built-in stress concentrations of varying degrees of intensity due to welding defects, tool marks, assembly eccentricities, and random defects in the metal. These conditions all favor brittle failure, and become even more severe at low temperature since brittle fracture is more prone to occur at reduced temperatures (13).

3.7.1 Mechanical Properties Determination. Tension tests were performed on a 50,000 pound Baldwin tensile testing machine at a strain rate of 0.001 inch per minute to yield, followed by 0.15 inch per minute to fracture. Cryogenic temperatures were maintained by means of a low-temperature chamber which allowed the specimen to be completely immersed in liquid nitrogen (-320°F) during testing. All data points reported are the average of at least three tests (see Tables IX, X, and XI).

To complete the mechanical property evaluation, notched specimens of Type 321 annealed and Type 304 stainless steels in the 70 percent cold-rolled condition were electropolished and subjected to sustained-load notched rupture tests (14). See Table VI. The tests were conducted for a period of 2000 hours using an Arcweld Model EE creep rupture testing machine. The test specimens were subjected to constant loading of 90 percent of the ultimate tensile strength of the individual alloy. The high-strength alloy was included to obtain additional information about the effect of electropolishing on work-hardened areas.

3.8 COMPONENTS. Typical components found in propellant systems were electropolished by the immersion and the impingement method, and subjected to salt-atmosphere testing. These components ranged from flexible hoses as small as 1/2 inch in diameter to bellows as large as 12 inches in diameter. Flexible metal tubing, rigid welded tubing, omega joints, and gimbal joints were also tested. The components were selected as typical of assemblies found in liquid propellant missile systems.

Since some of the propellant component parts were so large, an outside vendor, Electro Process and Engineering Corp., San Diego, Calif., performed the electropolishing. The vendor designed racks and electropolished them in his proprietary solution, which is a variation of the phosphoric-sulfuric type commonly used on stainless steels. Figure 10 shows a portion of the parts electropolished; Figure 11 is a picture of the production tank used in the processing of the components. Figure 12 shows a commercial electropolishing unit used for polishing small parts.

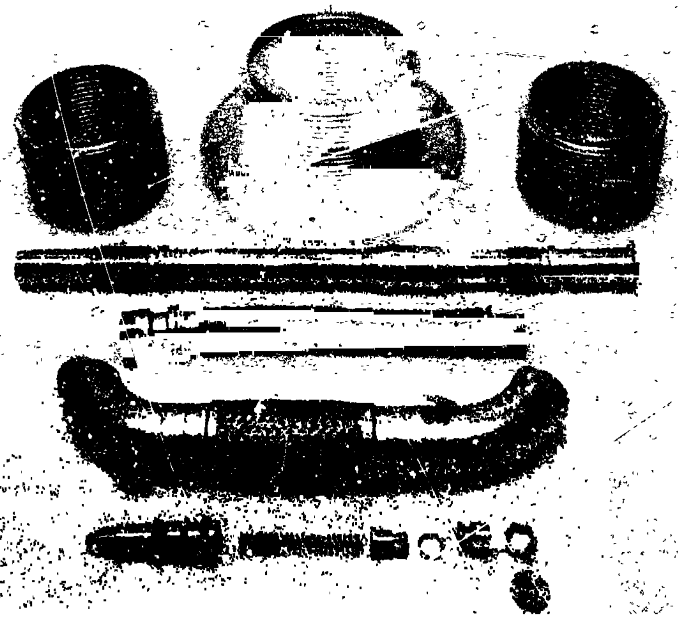


Figure 10. Typical Propellant Components after Electropolishing by the Immersion Method. The longest part shown, the helium-pressurization line, is about 5 feet in length.

Figure 11. Typical Commercial Immersion Electropolishing Facility. The parts are immersed for from 3 to 5 minutes.

Figure 12. Typical Commercial Electropolishing Unit for Small Parts. It features built-in rectification, and is designed for laboratory or limited production use.

The group of components were precision-cleaned and tested for liquid-oxygen cleanliness. The total weight and size of particles were measured after electropolishing as well as the total weight of hydrocarbons present (Table XII). The percentage of contaminants was within standard cleaning specifications.

3.9 ELECTROPOLISHING OF DISSIMILAR METALS. Panels of Type 304 annealed stainless steel, Monel, K-Monel, and Inconel were electropolished both singly and coupled together (see Table XII) using stainless steel fasteners of the 18Cr-8Ni type of steel. The couples were made by overlapping a one-inch section of two 3 inch x 3 inch panels and bolting them together with two sets of size 8-32 stainless steel bolts and nuts, 1/2-inch long, placed two inches apart. This type of mechanically fastened joint simulates actual production assemblies found in missile propellant ducting systems. A total of 15 single and 6 dissimilar metal coupled specimens were electropolished in a number of different electrolytes to determine the capability of the baths to polish different metals when they are coupled together in the same assembly.

In addition to the mechanically joined dissimilar metal coupons, a variety of dissimilar metal coupons were butt-welded together, using the Tungsten Inert Gas (TIG) welding method with no filler metal added. This type of weld joint is often employed in missile ducting systems to join dissimilar metals which are weldable to each other. The combinations which were tested include:

a. Inconel 718 welded to Type 321 stainless steel.

b. René 41 welded to Type 321 stainless steel.

c. Hastelloy C welded to Type 321 stainless steel.

d. Inconel 718 welded to Hastelloy C.

e. Inconel 718 welded to René 41.

The test specimens were made by butt welding together 3 inch x 3 inch samples of each of the above metals. Various electropolishing procedures were experimented with in an attempt to develop techniques which would successfully electropolish both metals of the above dissimilar metal combinations as well as each one of the metals taken singly.

3.10 CORROSION TESTING. Corrosion tests were conducted upon both flat test samples and on actual propellant-system components using salt atmosphere or salt-spray cabinets for laboratory type accelerated corrosion testing and by placing samples on exposure test racks at the University of California Scripps Institute of Oceanography, La Jolla California. At the Scripps seacoast exposure site, test racks are located on a pier 25 feet above sea level and directly above the shoreline (see Figure 13).

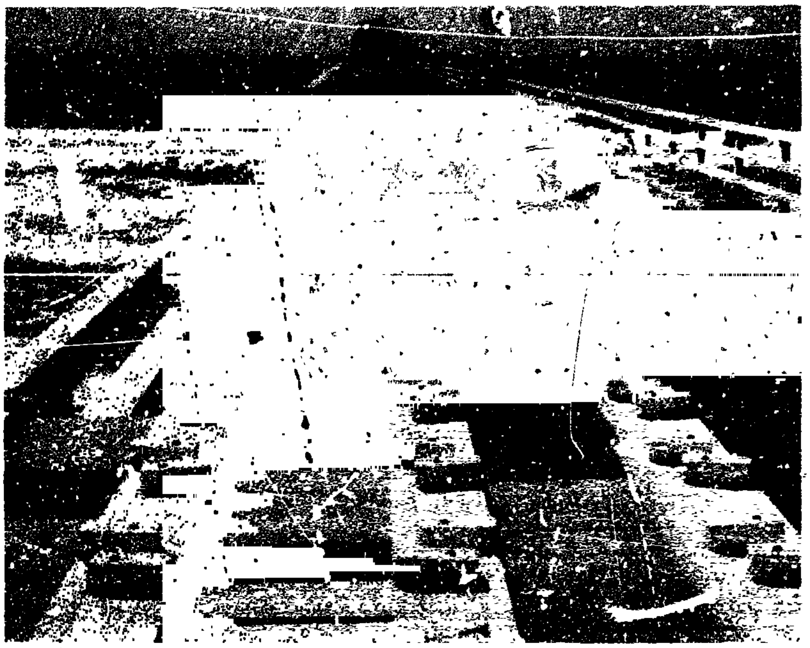


Figure 13. Test Racks Located on the Pier at the Scripps Institute of Oceanography. These racks provide actual seacoast exposure for testing electropolished samples.

The salt-atmosphere tests were conducted in a test chamber that was maintained at a constant temperature of 90 ±2°F and a relative humidity of 95 percent. The atmosphere in the chamber contained 2.3 percent sodium chloride.

The salt spray tests were conducted in accordance with the requirements of Federal Test Method Standard Number 141, Method 6061, and consisted of a 20 percent sodium chloride solution atomized under pressure to maintain a constant fall-out rate in a test chamber maintained at 95°F.

Significant seacoast exposure and corrosion test cabinet results are discussed below and references to pertinent tables and results given.

SECTION 4

RESULTS AND DISCUSSION

Excellent results were achieved with electropolishing both by immersion and by impingement. A bath versatile enough to be used with all alloys was developed.

4.1 DETAILED ANALYSIS AND EVALUATION OF ELECTROPOLISHING SOLUTIONS (PHASE IA). The results prove conclusively that austenitic stainless steels can be electropolished successfully and economically in acid-type solutions. The investigations were conducted with Type 321 and Type 304 stainless-steel alloys, using ten different electropolishing solutions (five of which are commercially available). These electrolytes were basically of four types: 1) phosphoric-sulfuric, 2) glycolic-sulfuric, 3) phosphoric-sulfuric-chromic, and 4) phosphoric-glycolic acid solution. The electrolytes were prepared and used in the manner prescribed by previous investigators, since this investigation was primarily concerned with selecting and perfecting electropolishing techniques rather than exploring electrolyte behavior. All ten electropolishing solutions gave satisfactory results when used with the two stainless steels listed above.

Acid and water concentrations of the phosphoric-sulfuric type baths which satisfactorily electropolish stainless steels are presented in Figure 14. The preferred acid concentration for the glycolic-sulfuric acid bath is given in Figure 15. The make-up of the

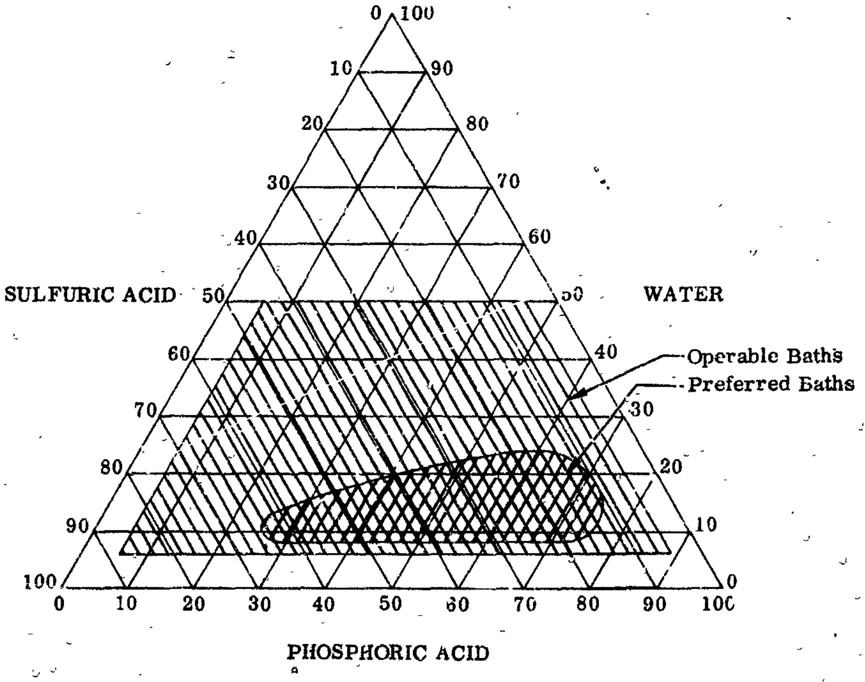


Figure 14. Composition of Sulfuric-Phosphoric Acid Electropolishing Baths for Stainless Steel Surfaces.

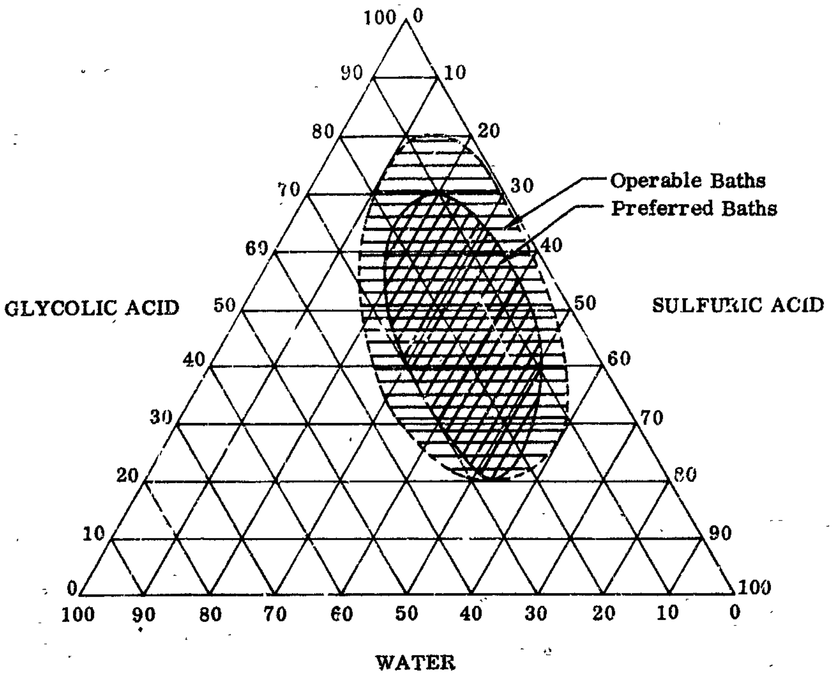


Figure 15. Electropolishing Electrolyte Composition Range, Plotted for Stainless Steel at 1 Amp/Square Inch; Temperature Range: 90°C-100°C; Tests Conducted for 3 and 5 Minutes.

phosphoric-sulfuric-chromic and phosphoric-glycerine baths that have been successfully used on stainless steel were taken from the literature(9). The others were commercially available. A complete listing of all baths is contained in Section 2.2.

Current density versus voltage curves were plotted for Types 304 and 321 stainless steels in the four different types of electropolishing solutions. All the electrolytes operated in a similar narrow current density and voltage range for the stainless steel alloys (see Figures 6 and 7). Satisfactory electropolishing was obtained with all solutions with current densities below 3 amps/square inch and at less than eight volts, which is a practical range for operation in any production cleansing line. Optimum length of time for electropolishing by immersion in electrolytes is from 3 to 5 minutes.

Acceptable electropolishing was obtained on all of the electrolytes tested. The phosphoric-sulfuric solutions exhibited superior brightness, throwing power (ability to polish recessed areas), and resistance to chemical etching. The current density and temperature ranges for phosphoric-sulfuric baths operating on the stainless steels were less critical.

Electropolishing baths commonly used for stainless steels did not electropolish the high-nickel alloys efficiently. Therefore a new bath was needed. After many trials, bright smooth surfaces were finally obtained by varying concentrations of phosphoric, sulfuric and chromic acids until an optimum bath was developed. This bath consisted of 65 percent (by weight) of orthophosphoric acid, 15 percent sulfuric, 6 percent chromic acid and 14 percent water. Fedot'ev (10) in the U.S.S.R. makes use of a similar bath in electropolishing steel alloys.

A set of operating conditions for the above electropolishing solution was established for both the immersion method and the impingement method of electropolishing. Using the solution described above for immersion electropolishing, the operating conditions are:

Temperature:	Room temperature to 130°F
Voltage:	6 to 10 volts
Time:	3 to 5 minutes
Cathode:	Lead

For the impingement method, operating conditions are:

Temperature:	Room temperature to 130°F
Voltage:	6 to 12 volts
Time:	2 to 5 minutes/square foot of area
Cathode:	Stainless steel
Cathode Nozzle Speed:	12 ft/minute
Solution Flow Rate:	1.0 to 1.5 gal/minute

Data on the comparative amounts of metal removed from Types 304 ELC and 321 stainless steel when electropolished in various solutions are shown in Table II. Each bath was operated at a constant current density of 2 amps/square inch for 8 minutes and at its recommended optimum temperature. While the amount of metal removed varied from bath to bath, it is noted that, except for bath 6, both steels underwent approximately the same weight loss in each bath. These results indicate that Types 304 ELC and 321 stainless steels exhibit similar electropolishing characteristics insofar as metal removal is concerned.

Table III presents an overall picture of the amount of metal removal to be expected from wide variations in electropolishing conditions, where the electrolyte, material, current density, voltage, and time are varied over rather wide ranges. Tests 1 through 6 were performed by a vendor who customarily employs a high voltage (in the range of 8 to 10 volts) and consequently a high current. The metal removal was high (in the range of 0.0025 inch to 0.0028 inch from the radius of a 3/8-inch diameter rod). Tests 7 through 26 were performed at General Dynamics/Astronautics and yielded metal removal in the range of 0.0004 inch to 0.0017 inch; the higher amounts of metal removal occurring at the higher currents and longer times of electropolishing. The General Dynamics/Astronautics tests showed that, at lower currents and voltages and shorter times of electropolishing, the metal removal was in the range of 0.0004 inch to 0.0007 inch.

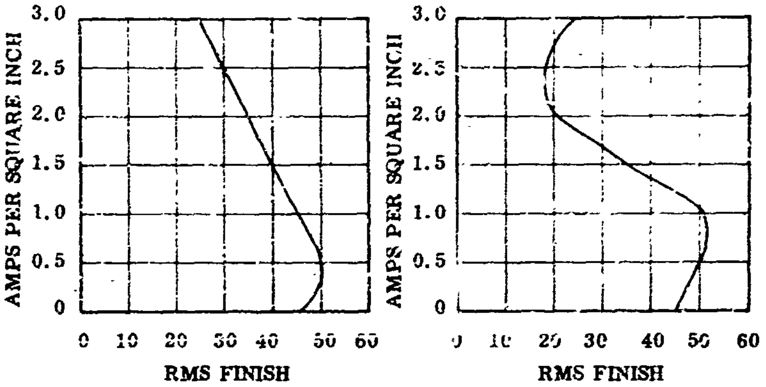
The high-nickel alloys are found to undergo a greater amount of metal removal during electropolishing than the Type 321 stainless steel (see Table IV).

For example, when Type 321 stainless steel is combined with the René 41 alloy, and electropolished in the phosphoric-sulfuric-chromic bath, 0.0002 inch more metal is removed from the René 41 alloy surfaces than from the Type 321 stainless steel surfaces. When Type 321 stainless steel is combined with Inconel X, Inconel 718, and Hastelloy C alloys, 0.0001 inch more metal will be removed from the high-nickel alloys than will be removed from the stainless steel surfaces. The Inconel X, Inconel 718, and Hastelloy C alloys may be coupled to each other and a fairly constant metal removal rate will occur on each of the alloys.

Although the metal removal is not excessive for any of the alloys tested, the variations in metal removal rates of the dissimilar metals must be considered when electropolishing components that include thin-walled sections in their designs.

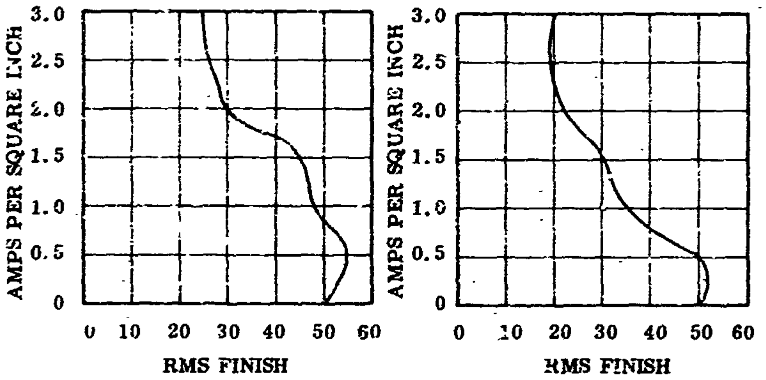
Profilometer measurements on polished and unpolished Type 321 and Type 304 stainless steel alloys are given in Table V. The alloys were electropolished with four different vendor baths. The operating conditions of the baths are given in Section 2.2. The data reveals that a marked improvement in surface smoothness is obtained by electropolishing in phosphoric-sulfuric baths. In general the Type 304 alloy exhibits greater smoothing characteristics than the Type 321 stainless steel. Average profilometer measurements, using the four types of bath, polished under identical operating ranges, are presented graphically in Figure 16 for Type 321 stainless steel. The graphs reveal that with increased current density a smoothing action can be expected from all four baths and that this smoothing action is similar with all four baths.

Profilometer measurements were taken before and after electropolishing on flat specimens of the high-nickel alloys. Table VI reveals that no improvement in surface finish was obtained on these alloys by electropolishing, however a very considerable improvement in brightness and reflectivity was obtained even though the overall smoothness was not improved. On examination a metal surface will be found consistently irregular in profile. For simplicity, these irregularities (microasperities and microrecesses) may be referred to as projections and impressions. Electropolishing eliminates the irregularities by selectively dissolving the projections (or leveling), thus producing brightness. Jamur (15) refers to leveling of the coarse projections as macropolishing, and the dissolution of much smaller microscopic irregularities as micropolishing. When macropolishing and micropolishing take place simultaneously, smoothing and brightness occur respectively. The degree to which this occurs depends on the nature of the metal, the original smoothness of the surface, and on the operating conditions of the electrolytic treatment. In certain instances, either can occur independently of the other; therefore, brightness does not always imply smoothness and, alternately, smoothness can exist without brightness.



a. Phosphoric-Sulfuric-Chromic Acid Solution

b. Glycolic-Sulfuric Acid Solution



c. Glycerine-Phosphoric Acid Solution

d. Phosphoric-Sulfuric Acid Solution

Figure 16. RMS Finish as a Function of Current Density, 3 Stainless Steel Specimens (Polished for 3 Minutes).

Adequate rinsing is an important phase of the electropolishing process. Corrosion staining, in many instances, can be attributed to improper rinsing after electropolishing. Parts that were corrosion-stained after polishing were reprocessed with special emphasis on complete rinsing, and corrosion staining was then eliminated. Table XV tabulates the results of the four methods of rinsing usually associated with the electropolishing process. Rinse method 4 which consisted of simple water immersion, followed by a forced tap water rinse, and a third rinse in 180° demineralized water resulted in no evidence of entrapped electrolyte in faying surfaces.

Vacuum drying of the test specimens did not show any beneficial effects of removing contaminating residuals in faying surfaces.

4.2 PROTECTIVE COATING TESTS. All candidate materials were first subjected to cryogenic flexibility testing. The test consisted of immersing two panels of each material in liquid nitrogen (-320°F), flexing them at that temperature, and allowing the panels to return to room temperature. This cycle was repeated at least five times.

The following tests completed the evaluation of the coating systems:

a. Hardness. The test panels were subjected to "marking" by a series of AW Faber-Castell drawing pencils of graduated hardnesses. The hardest grade of pencil that did not mark the coating was designated the material's pencil hardness.

b. Flexibility (Room Temperature). The test specimens were subjected to a 180-degree bend over a conical mandrel varying in diameter from 3/4 inch to 1/4 inch. The condition of the test material was examined visually; crazing, cracking, or loss of adhesion classified the coating material as brittle.

c. Adhesion. A Rondeau-Gardener tester was used. It consisted of a cutting edge attached to a spring bolted to a carriage. As the carriage is moved over the panel, the cutting edge is forced through the coating. The carriage is slid upward along an inclined plane, forcing the cutting edge through the coating and gradually decreasing the cutting action. The average of the "resistance length", i.e., the length of the specimen that is not scratched to the bare metal, is reported. Ten trials were made on each panel.

d. Resistance to Solvents. A measured amount of solvent was placed on the test material and allowed to evaporate. One minute after evaporation, the pencil hardness of the material exposed to the solvent was determined. Trichloroethylene, Oxylene, and Tec 901 solvents were used because these solvents are usually used at missile sites for cleaning.

e. Water Immersion. Test panels were placed in distilled water at a constant temperature of 100 ±5°F for a period of 600 hours.

f. Weatherometer. Test panels were placed in an Atlas weatherometer, Model XW, for 200 hours. The cycle of the weatherometer is 102 minutes of ultra-violet light and water spray. Initial and final gloss measurements were made with a Photovolt Glossmeter, Model 660. The panels were cleaned after exposure and gloss measurements were made again.

g. Seacoast Exposure. Panels were exposed on the pier at Scripps Institute of Oceanography, La Jolla, California, for a period of 60 days. The panels were subjected to a combination of salt air, fog, prevailing ocean winds, and intense sunlight. Periodic examinations were made to note any degradation effects due to any of the above environmental factors. Gloss measurements were taken before and after exposure. The results of the above tests are presented in Table XIV.

4.3 COMPONENTS (PHASE IB). Various propellant assembly components were electropolished to determine the versatility of the process. These included flexible lines, bellows, curved sections, straight sections, and complex fittings. All visible surfaces were polished to a bright, smooth finish.

The components are positioned in the electropolishing bath in such a manner that the maximum surface area is exposed to the cathode. Whenever possible, the cathode area should be at least two times the area to be electropolished. The solution operates most efficiently at this ratio.

The distance between the anode and cathode is variable and will range from two to nine inches in most instances. An even greater distance may be necessary on large components with sharp irregular configurations and when a minimum metal removal rate is desired. The electropolishing action is greatest at the points of highest current density. It should also be noted that higher voltages are required as the ratio of anode to cathode area is increased to overcome resistance of the solution.

Figure 10 illustrates the complex configurations that make up propellant systems designed to carry liquid fuels. The flexible sections as well as the rigid piping of the Atlas propellant ducting system are made of stainless steel. It is these sections that require the greatest care and ingenuity during the polishing process. In the cases of bellows section, for example, it is virtually impossible to build a conforming cathode for polishing deep recesses so that hydrogen and oxygen gases are not trapped. Polishing will not occur in a gas pocket.

Smooth, bright surfaces may be obtained on bellows sections with open convolutes in all areas except the extreme bottom sections of the convolute. For obtaining electropolished surfaces on all areas of bellows section, it is recommended that the tube section be electropolished prior to forming into the bellows. This is the only reliable method of obtaining bright, smooth surfaces in the recessed areas of the convolutes.

There are many advantages in electropolishing as an in-process step during the fabrication or manufacturing of the propellant components. The material is cleaner and the possibility of impregnating impurities in the base material during forming is reduced. The smoothing action does not leave any ridges of metal or extremely small burrs on the surface and a mechanically clean surface is obtained that is free from stressed, torn, or distorted metal thus reducing the tendency to crack during forming operations. This is a very important factor when forming multiple bellows; electropolishing is a method to reduce manufacturing defects that do not appear until the assembly is completed, resulting in major repairs. Bellows formed from electropolished tubing have maximum corrosion resistance; i.e., corrosion resistance in the recessed areas is equal to that of the external areas.

Figure 17 shows an example of a rather complex design found in propellant systems that require a chemically and mechanically smooth surface. The interior surfaces were electropolished by threading a series of copper wires throughout the tubing in such a manner that they were completely insulated from the outside surfaces. This may be done by spacing ceramic insulators at areas that will contact the side walls

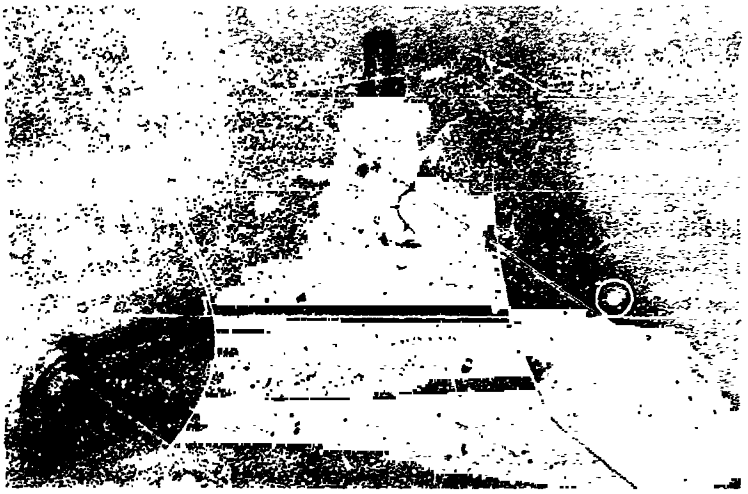


Figure 17. Electropolished Propellant Ducting. This ducting transfers missile fuels at cryogenic temperatures. The complexity of this ducting and its size (approximately 10 feet) illustrate the capability and versatility of the electropolishing process.

or by wrapping these areas with fiberglass. Whenever possible, it is best to design a rigid electropolishing rack with built-in conforming cathodes similar to those used in chromium plating.

The anode area should be as large as possible, but should allow adequate solution flow through the part and be designed so that the possibilities of gas pockets is eliminated.

The electropolished components listed in Table X were subjected to salt atmosphere environment tests to evaluate resistance to corrosion staining and pitting. Corrosion staining appeared in isolated areas after 96 hours of testing. The test was completed after 359 hours and the components were minutely examined for corrosion pitting. Stereo-microscopic examination of bellows sections revealed that surfaces electropolished in the "bright range" did not exhibit pitting. The surfaces that were not bright, such as the interior area of convolutes, were corrosion stained and occasionally pitted, though not as much as unpolished specimens. More protection can be achieved for these surfaces through the use of special techniques such as racking and auxiliary cathodes.

Control samples consisting of bellow sections that were not electropolished were corrosion stained and pitted to a much greater degree. There was no evidence of corrosion being selective in the unpolished bellows, which again pointed out the improved resistance to corrosion of the electropolished surfaces.

Bellows that were electropolished exhibited a remarkable improvement in resistance to pitting in corrosive atmospheres. One reason for this is the high degree of surface smoothness developed by electropolishing. Figure 18 shows the polishing action (left picture) that occurs on the surface of a bellows convolute compared to an unpolished surface of an equivalent bellows section (right picture). An electropolished surface has less tendency for liquids and particles to adhere to it than an unpolished surface. Thus the retention of foreign substances in minute cavities minimizes the likelihood of contact corrosion.

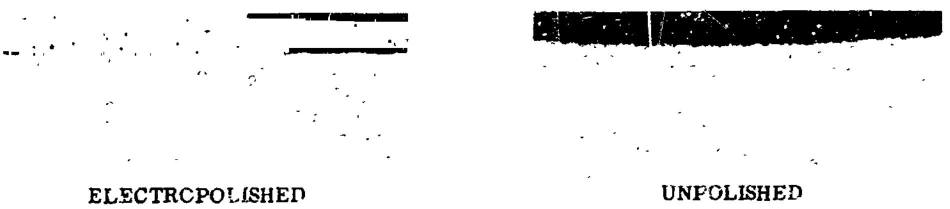


Figure 18. Comparison of an Electropolished Surface with an Unpolished Surface. These are photomicrographs of a cross section of a surface of an electropolished bellows section, magnified 250 times (unetched). Note that the electropolished surface shows a high degree of surface smoothness.

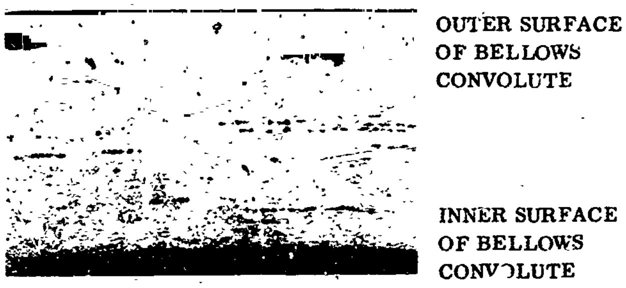


Figure 19. Typical Corrosion Pitting of Unpolished Surfaces. This bellows section was exposed for 368 hours in 2.5 percent salt atmosphere at 95° F. The section is magnified 250 times and has received a 10 percent oxalic etch to reveal the nature of the corrosion.

Figure 19 shows a typical pitting attack (at a magnification of 250 times) that occurs in unpolished stainless steel after 368 hours of exposure in the 2.5 percent salt atmosphere testing chamber. This was obtained on an unpolished bellows section and was typical of the attack found on both interior and exterior areas of the convolutes. The photomicrograph of the etched specimen reveals that the attack is of a pitting rather than an intergranular nature.

4.4 <u>AUXILIARY CATHODES FOR IMMERSION ELECTROPOLISHING.</u> Auxiliary cathodes were used in electropolishing the propellant components. They consisted mainly of sheet copper conformed to the configuration of the component. Small wires, rigid wafers attached to a spline, and wires coated with porous fiberglass were all used with little success to increase the bright range in rather close convolutes. Flexible hoses larger than one-half inch in diameter have been successfully electropolished with the use of internal cathodes. On other configurations, 1/2 inch x 12 inch is the minimum width and depth that can be electropolished without the use of complex racking.

4.5 <u>CATHODE NOZZLES FOR IMPINGEMENT ELECTROPOLISHING.</u> Special cathode nozzles are required for impingement electropolishing. Figure 20 illustrates the typical configuration designed for polishing flat sheet stock, bellows sections, curved piping, and ducting. The cathode nozzles were fabricated of stainless steel and copper. They were made of 3/16- to 1/4-inch-thick plate formed to the contour of the part to be polished and held within an area of approximately 20 square inches. The distance from the cathode to the anode should be from one to two millimeters. All surfaces of the cathode were coated with teflon except the area directly opposite the surface to be polished. This area is insulated from the work by wrapping the

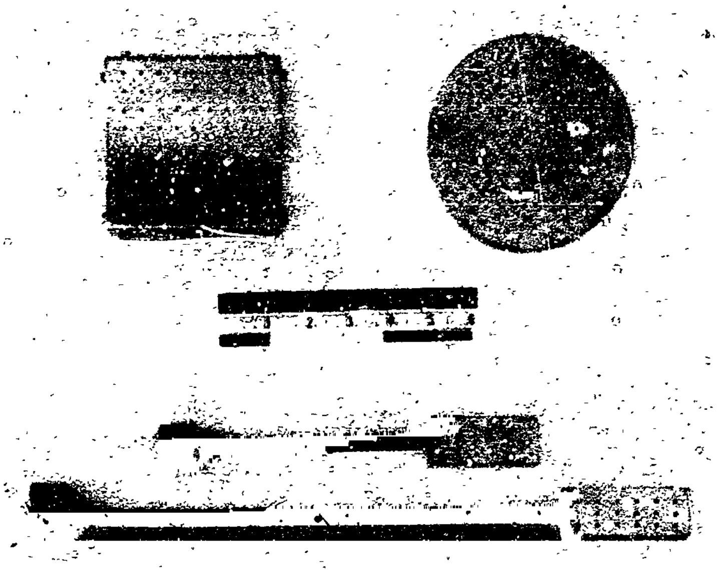


Figure 20. Impingement Cathode Nozzles. These perforated nozzles were designed to electropolish the interior and exterior surfaces of very large ducting lines and flat surfaces.

nozzle with fiberglass cloth and/or by teflon insulators placed at the outer periphery of the nozzle. This is very important since the operating voltages possible with this method can burn a hole through thin wall tubing if "shorting" occurs. The solution is pumped through the perforated cathode and forced on to the surface of the work area to be polished.

For example, for polishing flat sheets or exterior surfaces the perforations will be on the flat side of the cathode disc and when polishing interior surfaces the perforations will be at the outer circumference directly opposite the area to be polished. The cathode is moved at a speed slow enough to obtain polishing, but fast enough to prevent burning. Speed of movement is determined by solution flow, solution temperature, and current density.

4.6 ADVANTAGES OF IMPINGEMENT ELECTROPOLISHING.

a. Electropolishing may be selective in nature. Polishing of weldments, for example, can now be accomplished without polishing the rest of the part.

b. Because of the vigorous washing action of the electrolyte forced on the surface, current densities as high as 3000 amps/square foot are possible, resulting in a smooth, bright, corrosion-resistant finish.

c. The need for an electropolishing installation requiring large immersion tanks filled with expensive solution, bus bars, and expensive electrical power sources is eliminated.

d. Size of parts to be electropolished is no longer limited. Previously, size of parts which could be electropolished was limited by size of tanks available in commercial electropolishing facilities. Since parts do not require immersion, the process can be applied to almost any electropolishing problem.

4.7 PRECISION CLEANING OF ELECTROPOLISHED COMPONENTS FOR LIQUID OXYGEN SERVICE. After electropolishing, the parts were precision cleaned to meet the requirements of duct assemblies for fuel and liquid-oxygen service. The results of the "clean-room" contamination count is presented in Table XI. All components tested well within the maximum allowable contamination values as required by General Dynamics/Astronautics missile-cleaning specification 0-75002.

a. The maximum permissible weight of solid particles and fibers, not including hydrocarbons, shall be 2000 micrograms per square foot.

b. Fibers shall be less than 2000 microns in length and 350 microns in diameter.

c. The maximum particle size allowable must be less than 500 microns and there must be no more than four solid particles per square foot.

The electropolished parts did not have to be recycled, nor did they need additional cleaning, ultrasonic rinsing, or any other cleaning except rinsing with particle-free trichloroethylene prior to testing.

4.8 ELECTROPOLISHING DISSIMILAR METALS. Type 321 and 304 stainless steel may be coupled with other metals such as Inconel 718, Inconel X, Hastelloy C, and René 41 without affecting the electropolishing characteristics of the stainless steels. However, the metal removal rates may, as noted previously, be different for the different metals. Experimentation proved conclusively that bath 10 is an efficient and versatile bath. It electropolished the widest selection of alloys (coupled and uncoupled) and, perhaps of even greater significance, provides excellent impingement as well as immersion electropolishing.

Voltage versus temperature curves obtained with bath 10 on the high-nickel alloys revealed that a minimum voltage (6 volts) was necessary for effective electropolishing. The minimum temperature at which brightness was observed was approximately 115°F. René 41 alloy, however, required a temperature of 150°F at this voltage. By increasing the voltage, temperatures could be decreased to the lower range used with the other alloy. The optimum temperature and voltage for electropolishing each of the high-nickel alloys in combination with Type 321 stainless steel is in the range of 8 to 10 volts at 120 to 130°F (see Figures 21, 22, 23, 24, 25). Figure 26 shows dissimilar metal alloy results.

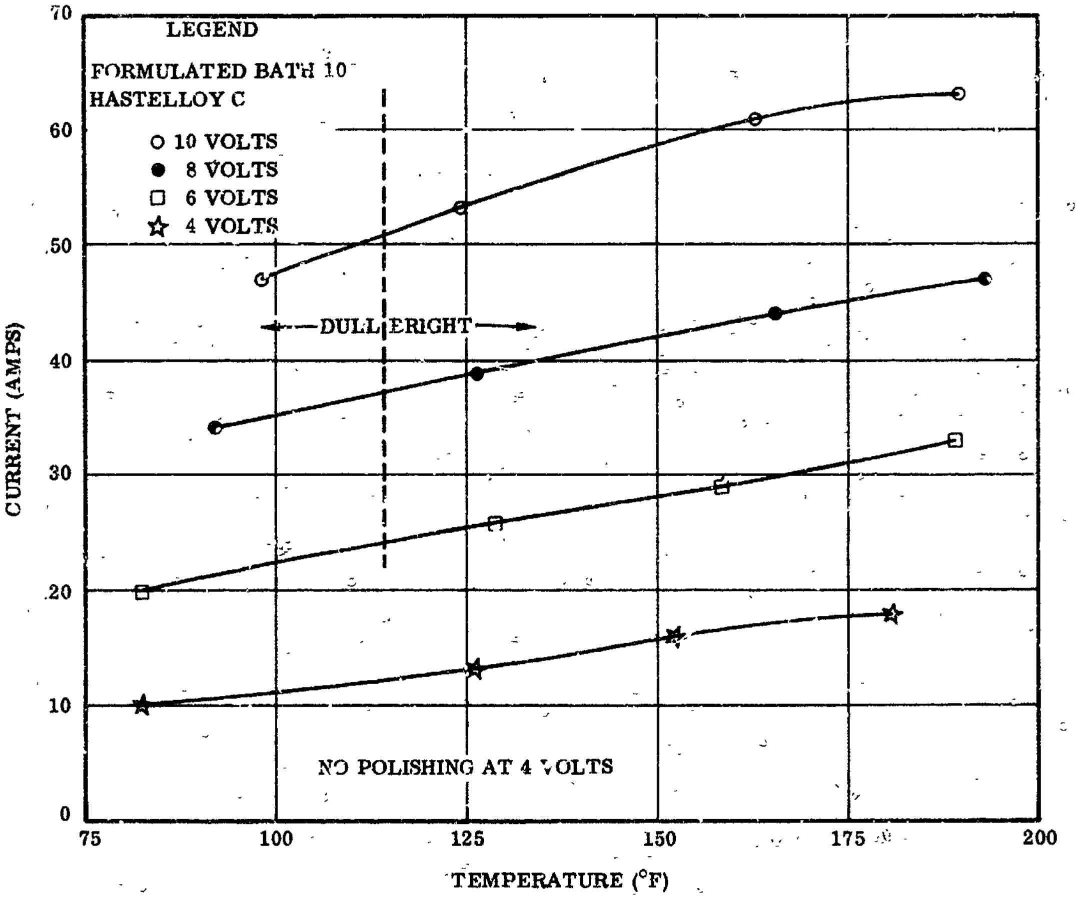


Figure 21. Electropolishing as a Function of Electrolyte Temperature and Current Density (Polishing Time, 3 Minutes).

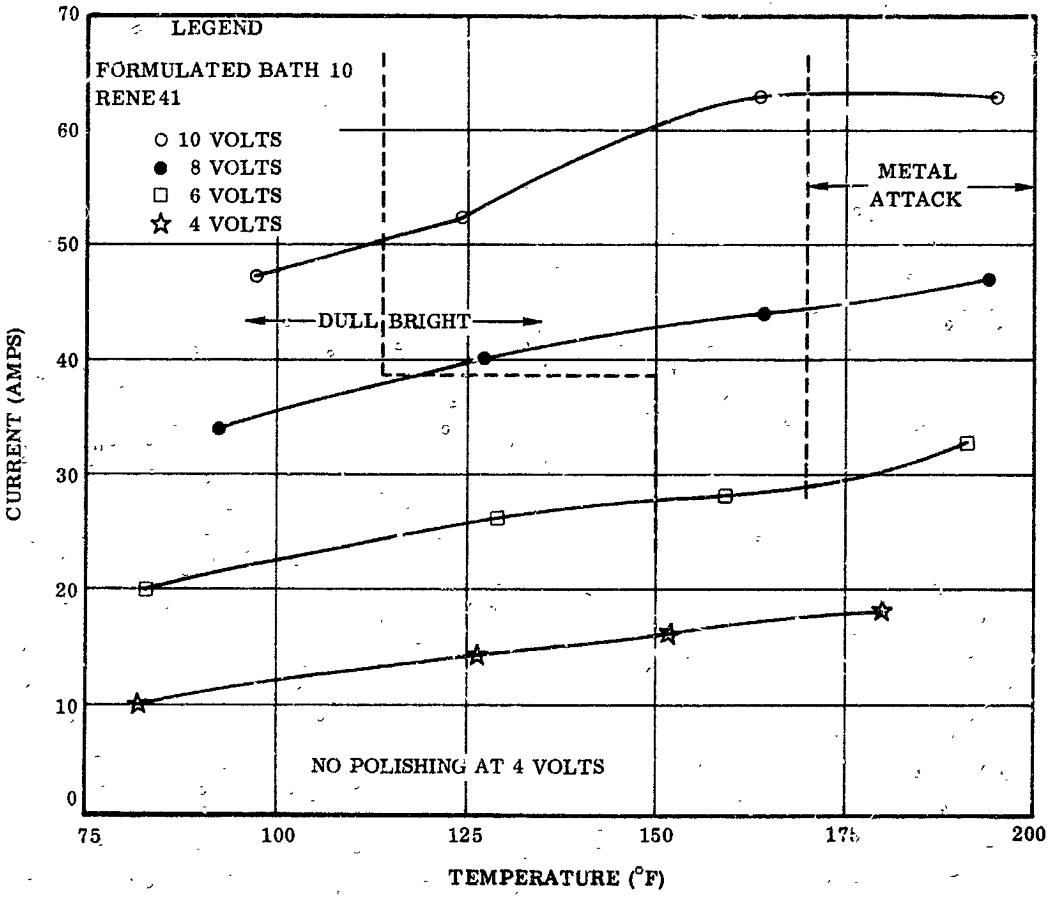


Figure 22. Electropolishing as a Function of Electrolyte Temperature and Current Density (Polishing Time, 3 Minutes).

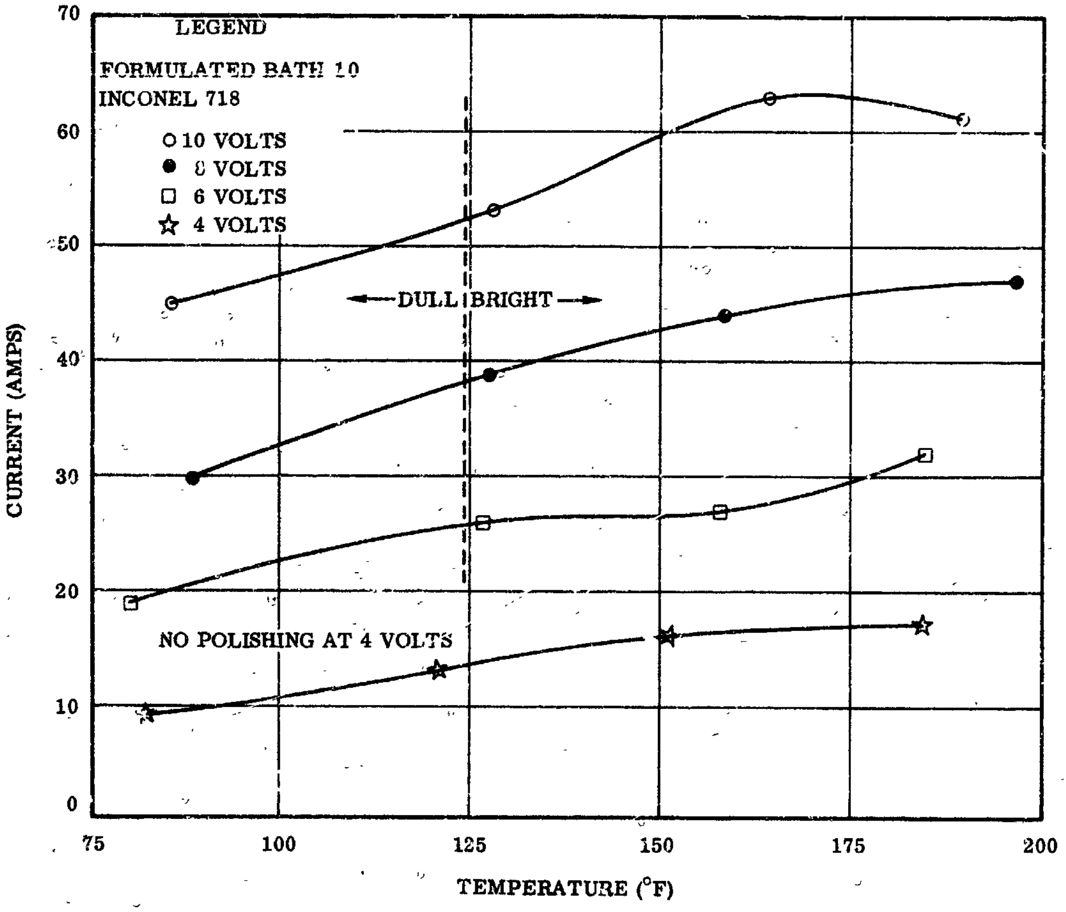


Figure 23. Electropolishing as a Function of Electrolyte Temperature and Current Density (Polishing Time, 3 Minutes).

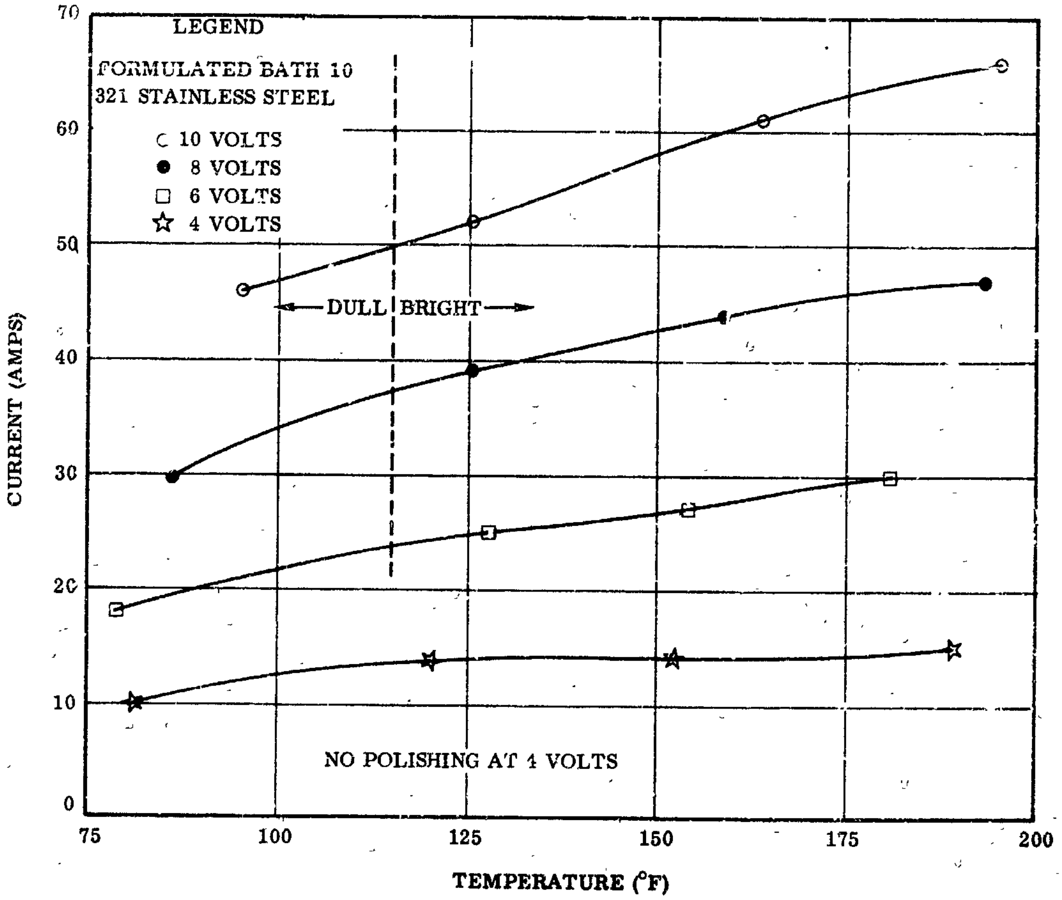


Figure 24. Electropolishing as a Function of Electrolyte Temperature and Current Density (Polishing Time, 3 Minutes).

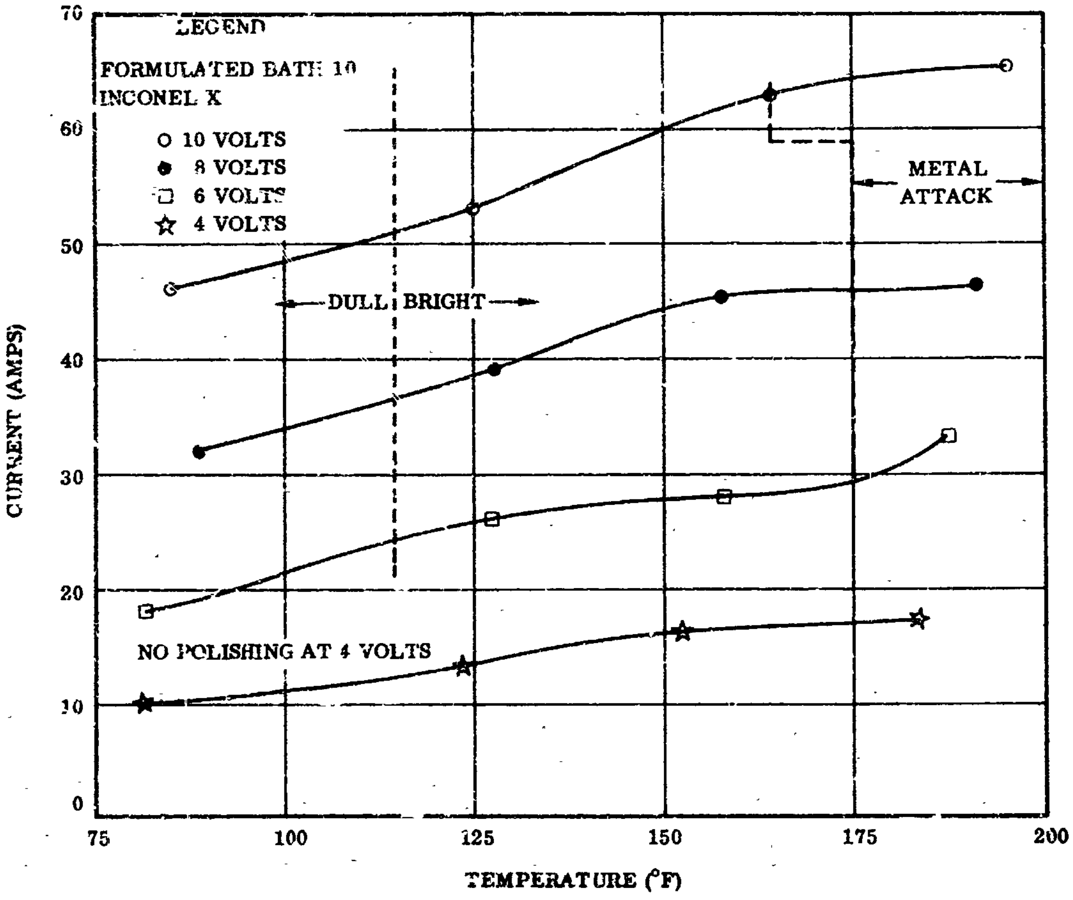


Figure 25. Electropolishing as a Function of Electrolyte Temperature and Current Density (Polishing Time, 3 Minutes).

Figure 26. Electropolishing Dissimilar Metal Alloys. This photograph illustrates the degree of brightness and smoothness attainable with electropolishing. (The bottom five panels are unpolished.) Stainless Steel (321), Inconel X, Inconel 718, Hastelloy C, and Rene 41 are pictured; these alloys can be polished singly or when coupled.

4.9 EFFECT OF ELECTROPOLISHING ON MECHANICAL PROPERTIES (PHASE IC).

Tensile and notched tensile ($K_t = 6.3$) yield and elongation properties at 78°F and at -320°F were obtained on samples as received and electropolished samples. They are presented for Type 321 annealed (Table IX) and Type 304 both annealed and in the 70 percent cold-worked condition (Tables X and XI, respectively).

The electropolishing process does not materially change the mechanical properties of the alloys tested. Data obtained on Type 321 indicated a slight decrease in tensile properties at -320°F; however, there was a marked improvement in toughness at both ambient and cryogenic temperatures. Elongation increased from 2.8 to 4.0 percent at room temperature after electropolishing of the Type 304 cold-worked material and increased from 28.6 to 32.5 percent at -320°F.

The notched/unnotched tensile ratio, which is an index of a material's susceptibility to brittle fracture, remained relatively constant after electropolishing. Therefore, we may conclude that electropolishing is not detrimental to the toughness of Type 321 and Type 304 stainless steels.

The sustained-load notched rupture test is considered a sensitive and reproducible method of determining the presence of hydrogen embrittlement. After 2000 hours of sustained load-testing there was no indication of failure either by abrupt rupture of the specimens or by the appearance of fine-line cracking originating at the notch on the specimens.

Since notched specimens of Type 321 (annealed) and Type 304 (70 percent cold-rolled) were electropolished and subjected to constant loading to 90 percent of the ultimate tensile strength of the individual alloy without deleterious effect, we may also conclude that the electropolishing baths do not cause hydrogen embrittlement in either of these alloys.

4.10 SEACOAST AND SALT SPRAY EXPOSURE. Accelerated testing and actual seacoast exposure test results consistently show the advantage of the electropolishing process for increasing the corrosion resistance of propellant system materials.

After six months seacoast exposure, the electropolished samples of Type 321 welded tubing showed remarkable resistance to corrosion when compared to unpolished samples. Figure 27 shows typical results obtained at the Scripps exposure site. The center panels were unpolished and the surrounding panels were electropolished with variations of the phosphoric-sulfuric type electrolyte. There were indications that some formulations give slightly superior corrosion resistance; however improvement in corrosion resistance was obtained with each of the electropolishing baths.

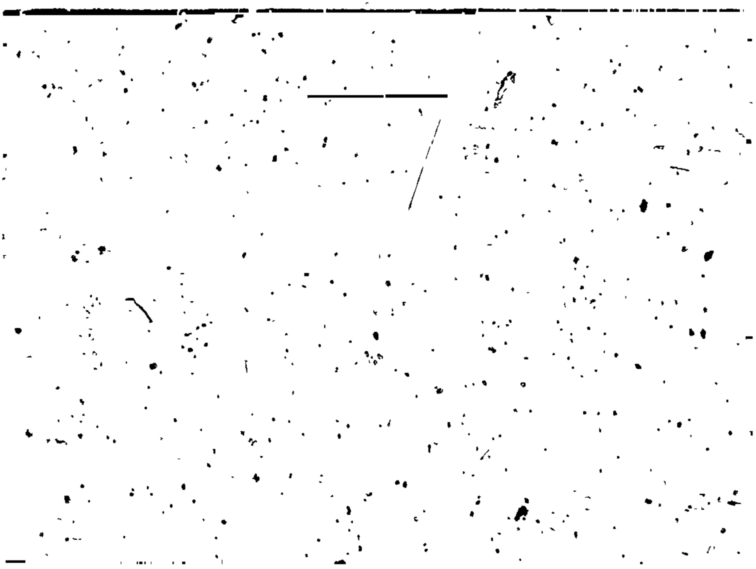


Figure 27. Seacoast Exposure of 321 Stainless Steel Test Panels. The panels were welded tubing. Note that the unpolished control tubing (center) was severely corroded after 6 months of exposure.

Salt-spray tests on Type 321 stainless steels with the vendor baths (see Table XV) did not materially affect the electropolished surface. Corrosion staining did occur but was the result of the fasteners used in bolting the two panels together for determining the effect of chlorides in the faying surfaces.

Butt-welded samples of 321 alloy did show a staining attack at the weld areas, however there was a marked improvement in the polished sample compared to the unpolished samples (see Table XVI).

The electropolished high-nickel alloys did not show any attack during 800 hours of salt-spray testing except for two corrosion pits on the Inconel X panel. The unpolished Inconel X panel also revealed a staining condition, but no pitting after testing for this length of time (see Table XVII).

The welded electropolished samples were not affected by the salt spray tests, however, Type 321 stainless steel welded to Inconel 718 resulted in corrosion staining at the weld in the unpolished condition. Type 321 stainless steel welded to Rene 41 and Hastelloy C were severely corrosion stained in the unpolished condition (see Table XVIII).

On test panels made of flat-sheet stock, seacoast exposure tests duplicated the results obtained in the tubing samples. Where corrosion staining did occur, it could be easily cleaned with 2F pumice and no indications of corrosion pitting was in evidence. This was true on both Type 321 and Type 304 stainless steel alloys.

The high-nickel alloys Inconel X, Inconel 718, Hastelloy C and Rene 41, in the welded and unwelded conditions, did not show any indications of corrosion attack after 30 days exposure to seacoast environment. The alloys were electropolished with baths 5 and 10.

Type 321 stainless steel panels, electropolished by the impingement method, were exposed to seacoast environment and proved that impingment of the solution on the work is an effective means of applying a passive surface on stainless steel. Figures 28 and 29, two panels electropolished by the impingement method, illustrate the corrosion resistance provided by this method. Exposure time was 30 days.

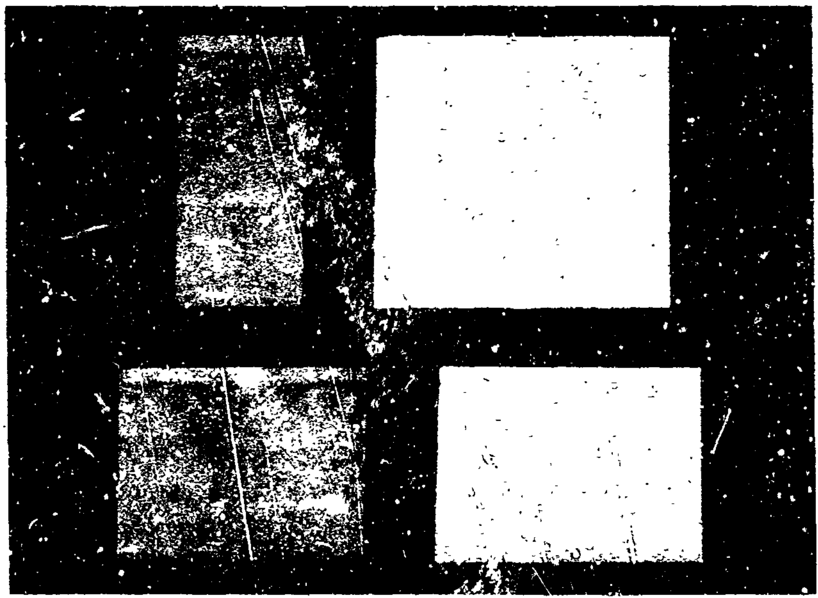


Figure 28. Two Sheets of 321 Stainless Steel, Exposed to 30 Days of Seacoast Environment. The sheets were divided into 2 panels with masking tape. The electropolished half of each sheet (see right side of figure) showed no corrosion whatever, whereas the unpolished areas were severely corroded.

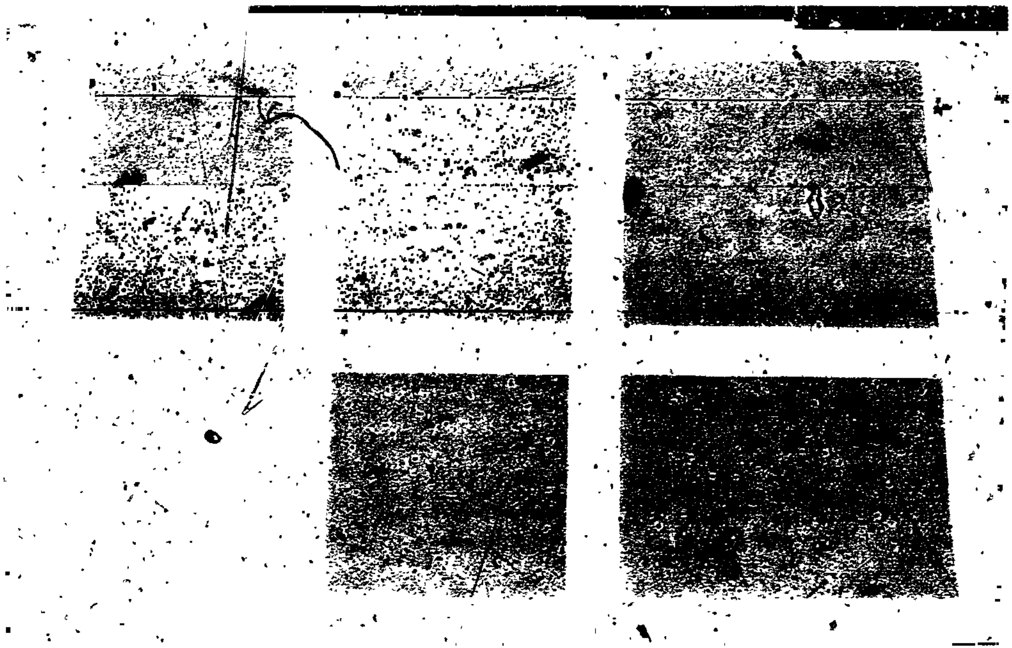


Figure 29. A Single Sheet of 321 Stainless Steel Electropolished by the Impingement Method. Six different areas were delineated with masking tape. One area (bottom left) was used as a control. Each of the other areas received a variety of voltages, exposure times, and temperatures. Impingement electropolishing was shown not to be limited to a narrow range of operating conditions as is immersion electropolishing.

Type 321 stainless steel panels, electropolished by the impingement method, were exposed to seacoast environment and proved that impingement of the solution on the work is an effective means of applying a passive surface on stainless steel. Figures 28 and 29, two panels electropolished by the impingement method, illustrate the corrosion resistance provided by this method. Exposure time was 30 days.

4.11 <u>PROTECTIVE COATING SYSTEMS (PHASE II)</u>. The coating systems that did not pass the liquid nitrogen (-320°F) flexibility tests were the MIL-E-7729 enamel system and an alternate material used for the protection of steel surfaces, the MIL-L-7178 nitro-cellulose lacquer system.

The heat-curing-type resins, flourinated-ethylenepropylene, vinylidene flouride, and modified silicone, all displayed excellent properties throughout the testing program. However, the epoxy and modified silicone exhibited the best properties of adhesion and corrosion resistance. Acrylic air-dry formulations, although providing less resistance to seacoast exposures, can be used as protective coatings for missile propellant systems.

The results of the liquid nitrogen flexibility test on the organic protective systems were positive (see Table XIV).

SECTION 5

CONCLUSIONS

1. Of the electrolytes investigated for use in electropolishing Type 321 and Type 304 stainless steels, the phosphoric-sulfuric acid solutions produced the best combinations of corrosion resistance, smoothness, brightness and resistance to chemical etching on welded and unwelded surfaces.

2. Satisfactory electropolishing on all of the immersion electrolytes investigated was obtained with current densities less than three amps/square inch, with polishing times in the range of three to five minutes. The temperature of the baths were dependent on the formulation of the electrolyte.

3. For electropolishing high-nickel alloys such as Inconel X, Inconel 718, Hastelloy C and René 41, a bath consisting of 65 percent phosphoric acid, 15 percent sulfuric acid, 6 percent chromic acid, and 14 percent water was satisfactory. The operating conditions of this bath are quite flexible and polishing occurs over a wide range of temperatures and current densities.

4. Type 321 stainless steel, in combination with the high-nickel alloys, can be electropolished successfully, however the operating conditions of the bath must be carefully controlled. Metal removal variations of 0.0002 inch per surface may be expected when electropolishing these dissimilar metals.

5. Stainless steel ducting, bellows sections, flexible metal hoses, and tubing assemblies that make up complex propellant systems may be electropolished by the immersion method. The interior surfaces of small diameter tubing and ducting necessitates the use of internal cathodes consisting of wires or rods insulated from the sides of the part by perforated acid-resistant plastic tubing or fiberglass cloth. Maximum polishing of recessed areas of ducting and bellows sections can best be accomplished by polishing prior to forming.

6. Cleaning tests after electropolishing exhibited excellent low particle count results when components were subjected to liquid-oxygen cleaning cycles. The improved smoothness of the surface does not allow liquids or large particles to adhere to the surface, thus reducing the possibility of allowing the liquid-oxygen to be contaminated.

7. The impingement method of electropolishing eliminates size limitations previously imposed on parts to be electropolished since it obsoletes large immersion tanks, bus-bars and expensive power sources. The surfaces obtained by the impingement process are smooth, bright, and equally as corrosion resistant as those obtained by the immersion method.

8. Electropolishing did not show any deleterious effects on the mechanical properties, including toughness, of Type 321 and Type 304 stainless steels. There was no indication of hydrogen embrittlement caused by electropolishing.

9. Seacoast exposure tests, as well as accelerated salt atmosphere or salt-spray cabinet exposure tests on samples and actual propellant components showed that electropolishing increases the corrosion resistance of stainless steels and high-nickel alloys in both the unwelded and welded conditions.

10. Of the nine types of organic coatings evaluated for protection of propellant ducting at cryogenic temperatures the air-drying epoxy and acrylic formulations were found satisfactory for large assemblies that cannot be baked and a heat-curing modified silicone resin was found to be best for small assemblies that can be baked.

SECTION 6

RECOMMENDATIONS

Based on experiments and information obtained to date, the following recommendations can be made:

1. The phosphoric-sulfuric acid electropolishing baths are recommended for use on Type 321 and Type 304 stainless steels to increase their resistance to corrosion staining and pitting when exposed to salt atmosphere.

2. For electropolishing high-nickel alloys in combination with 321 stainless steels, a bath consisting of 65 percent orthophosphoric acid, 15 percent sulfuric acid, 6 percent choromic acid and 14 percent water is recommended.

3. For obtaining maximum corrosion resistance of bellows, it is recommended that electropolishing be accomplished while the material is in the straight tube condition; i.e., after welding but before forming. This is the only reliable method of obtaining bright, smooth surfaces in the recessed area of the convolutes.

4. For maximum corrosion protection of exterior surfaces of stainless-steel propellant ducting and/or components, organic coatings are recommended. A heat-curing resin such as the modified silicone is recommended for comparatively small assemblies. For large ducting and installations that cannot be baked, the air-dry epoxy and acrylic formulations may be used as protective coatings.

5. It is strongly recommended that the impingement method of electropolishing be explored and developed further, since it has already been proved a superior electropolishing tool in the preliminary experiments performed under this contract.

6. Despite the fact that no positive results were achieved with ultrasonic electropolishing, there are enough indications and theoretical guideposts to warrant further investigation and development, particularly with regard to the lower frequencies, which were not explored within the confines of this contract.

SECTION 7

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Table I. Nickel Base Alloys

NOMINAL CHEMICAL COMPOSITION (%)	TYPE 304 ELC STAINLESS STEEL	TYPE 321 STAINLESS STEEL	INCONEL 718	INCONEL X	HASTELLOY C	RENÉ 41
Carbon	0.03	0.08	0.10	0.04	0.10	0.12
Manganese	2.00	2.00	0.5	0.7	0.8	-
Silicon	1.00	1.00	0.7	0.3	0.7	-
Chromium	18.00-20.00	17.00-19.00	19.0	15.0	16.0	19.0
Nickel	8.00-11.00	8.00-11.00	53.0	73.0	57.0	Balance
Cobalt	-	-	-	-	-	11.3
Molybdenum	-	-	3.0	-	17.0	10.0
Tungsten	-	-	-	-	4.0	-
Columbium	-	-	5.0	0.9	-	-
Titanium	-	5 × Carbon	1.0	2.5	-	3.0
Aluminum	-	-	0.6	0.9	-	1.52
Iron	-	-	Balance	7.0	5.0	-
Copper	-	-	0.75	-	-	-

Table II. Comparative Weight Loss by Electropolishing in Various Baths for 8 Minutes at Current Density of 2 Amps/Square Inch (Specimens: 3 Inch Long Rods, 3/8 Inch in Diameter)

BATH NO.	TYPE STAINLESS STEEL	WEIGHT LOSS (Grams)	TEMPERATURE (°F)
1	321	0.47544	125
2	321	0.58769	170
3	321	0.59452	180
4	321	1.33430	150
5	321	0.55580	230
6	321	0.52073	185
1	304 ELC	0.46419	125
2	304 ELC	0.66853	170
3	304 ELC	0.53102	180
4	304 ELC	1.32450	150
5	304 ELC	0.60362	230
6	304 ELC	0.92695	185

Table III. Metal Removed by Electropolishing

			ELECTROPOLISHING CONDITIONS			DIAMETER OF ROD		METAL REMOVED FROM RADIUS
BATH NO.	TEST NO.	MATERIAL	AMPS	VOLTS	TIME (MIN)	BEFORE (IN.)	AFTER (IN.)	(IN.)
4	1	321	Not furnished by vendor			0.3757	0.3707	0.0025
4	2	321				0.3756	0.3704	0.0026
4	3	321				0.3756	0.3706	0.0025
4	4	304 ELC				0.3755	0.3699	0.0028
4	5	304 ELC				0.3751	0.3698	0.0027
4	6	304 ELC				0.3750	0.3700	0.0025
2	7	304 ELC	10.0	6.0	8	0.3747	0.3725	0.0011
2	8	304 ELC	10.0	6.0	8	0.3747	0.3728	0.0010
2	9	304 ELC	10.0	6.0	8	0.3732	0.3723	0.0005
2	10	321	10.0	6.0	8	0.3743	0.3709	0.0017
2	11	321	10.0	6.0	8	0.3742	0.3728	0.0007
2	12	321	10.0	6.0	8	0.3743	0.3728	0.0008
7	13	321	8.0	3.5	5	0.3750	0.3737	0.0007
7	14	321	8.0	3.5	3	0.3750	0.3740	0.0005
7	15	304 ELC	8.0	3.5	5	0.3750	0.3737	0.0007
7	16	304 ELC	8.0	3.5	3	0.3748	0.3739	0.0005
7	17	321	15.0	4.0	5	0.3750	0.3723	0.0014
7	18	321	15.0	4.0	3	0.3750	0.3730	0.0010
7	19	304 ELC	15.0	4.0	5	0.3750	0.3718	0.0016
7	20	304 ELC	15.0	4.0	3	0.3748	0.3731	0.0009
5	21	304 ELC	5.5	4.0	8	0.3741	0.3730	0.0006
5	22	304 ELC	5.5	4.0	8	0.3740	0.3712	0.0014
5	23	304 ELC	5.5	4.0	8	0.3738	0.3725	0.0007
5	24	321	5.5	4.0	8	0.3750	0.3734	0.0008
5	25	321	5.5	4.0	8	0.3750	0.3742	0.0004
5	26	321	5.5	4.0	8	0.3750	0.3734	0.0008

Table IV. Metal Removed by Electropolishing in Bath No. 10 for 4 Minutes at 8 Volts at 130°F (Samples: 3 Inches by 3 Inches)

ALLOY	WEIGHT BEFORE ELECTROPOLISHING (Grams)	WEIGHT AFTER ELECTROPOLISHING (Grams)	WEIGHT LOSS (Grams, Average)	THICKNESS OF METAL REMOVED FROM ONE SURFACE (Inches)
Type 321 Stainless Steel	46.0016	45.1824		0.00034
	46.2943	45.4685		
	46.0176	45.1989		
Average	46.1045	45.2831	0.8214	
Inconel X	39.5184	38.4500		0.00041
	38.9894	37.9878		
	39.2680	38.2850		
Average	39.2586	38.2409	1.0177	
Inconel 718	53.5184	52.3976		0.00042
	54.0718	53.0347		
	53.2697	52.2925		
Average	53.6199	52.5748	1.0451	
Hastelloy C	53.0056	51.6681		0.00044
	52.7324	51.5983		
	53.0653	51.9230		
Average	52.9344	51.7298	1.2046	
René 41	51.9943	50.6560		0.00050
	52.3252	51.0017		
	52.6564	51.5464		
Average	52.3253	51.0680	1.2573	

Table V. Effect of Electropolishing for 8 Minutes on Surface Smoothness (Specimens: 10 Inch Rods, 3/8 Inch Diameter)

BATH NO.	ALLOY	BEFORE ELECTROPOLISHING (Microinches)	AFTER ELECTROPOLISHING (Microinches)
1	321	20-30	18-25
1	304	20-30	5-13
2	321	20-30	14-19
2	304	20-30	14-20
3	321	20-30	12-18
3	304	20-30	6-9
4	321	20-35	7-11
4	304	20-35	5-8

Table VI. Effect of Electropolishing on Surface Smoothness of High-Nickel Alloys*

ALLOY	SMOOTHNESS** BEFORE ELECTROPOLISHING LONGITUDINAL	SMOOTHNESS** BEFORE ELECTROPOLISHING TRANSVERSE	SMOOTHNESS** AFTER ELECTROPOLISHING LONGITUDINAL	SMOOTHNESS** AFTER ELECTROPOLISHING TRANSVERSE
321	4-6	8-10	6-8	6-12
Inconel X	8-10	9-11	8-10	9-12
Inconel 718	9-11	10-12	7-9	7-9
Hastelloy C	15-17	15-20	15-22	17-22
René 41	19-21	17-21	20-25	20-25

* Flat test specimens 3 inches by 3 inches electropolished in bath 10 for three minutes at 8 volts. Temperature of the bath was 130°F.

** Microinches.

Table VII. Notched/Unnotched Tensile Ratio

TYPE STEEL	TEMPERATURE (°F)	NOTCHED/UNNOTCHED TENSILE RATIO
Annealed 321 S. S.	78	0.873
As received	-320	0.785
Annealed 321 S. S.	78	0.939
Electropolished	-320	0.858
Annealed 304 S. S.	78	0.905
As received	-320	0.875
Annealed 304 S. S.	78	0.903
Electropolished	-320	0.764
70% Cold Rolled 304 S. S.	78	1.10
As received	-320	1.00
70% Cold Rolled 304 S. S.	78	1.11
Electropolished	-320	0.993

Table VIII. Sustained-Load Notched Rupture Tests

CONDITION	THICKNESS (Inches)	LOAD 90% YIELD	RESULTS
Electropolished*	0.032	32,800	No Failure
Electropolished*	0.032	32,800	No Failure
Electropolished*	0.032	32,800	No Failure
Unpolished*	0.033	32,800	No Failure
Unpolished*	0.033	32,800	No Failure
Electropolished**	0.019	155,700	No Failure
Electropolished**	0.019	155,700	No Failure
Unpolished**	0.020	155,700	No Failure
Unpolished**	0.020	155,700	No Failure

* Type 321 S. S. annealed - yield strength 36,500 PSI - duration of test 2184 hours

** Type 304 S. S. 70% cold-rolled - yield strength 173,000 PSI - duration of test 2184 hours

Table IX. Mechanical Properties of 321 Annealed Stainless Steel, at Ambient and Cryogenic Temperatures

	TEMPERATURE (°F)	DIRECTION	YIELD STRENGTH 0.2% OFFSET (1000 psi)	TENSILE STRENGTH (1000 psi)	ELONGATION (%)	NOTCHED TENSILE STRENGTH, 1000 psi ($K_t = 6.3$)	NOTCHED/ UNNOTCHED TENSILE RATIO
AS RECEIVED	78	Longitudinal	36.7	91.0	51.5	79.6	
			36.6	90.1	51.5	79.5	
			37.4	90.3	51.5	77.9	
			35.0	89.8	50.0	78.5	
			-	-	-	78.5	
		Average	36.6	90.3	51.1	78.8	0.873
	-320	Longitudinal	50.1	203.5	45.0	160.0	
			52.2	204.1	44.0	158.3	
			45.8	204.4	44.0	162.3	
			49.3	205.4	45.0	158.3	
			-	-	-	164.7	
		Average	49.4	204.4	44.5	160.7	0.785
ELECTROPOLISHED	78	Longitudinal	34.9	89.2	54.0	82.6	
			33.9	88.0	53.0	82.9	
			33.7	88.6	52.0	83.7	
		Average	34.2	88.6	53.0	83.1	0.939
	-320	Longitudinal	53.7	200.5	46.0	179.1	
			52.2	199.9	45.0	171.8	
			50.7	202.3	47.0	167.5	
			-	-	-	173.7	
			-	-	-	173.6	
		Average	52.2	200.9	46.0	173.1	0.858

Table X. Mechanical Properties of 304 ELC Annealed Stainless Steel, at Ambient and Cryogenic Temperatures

	TEMPERATURE (°F)	DIRECTION	YIELD STRENGTH 0.2% OFFSET (1000 psi)	TENSILE STRENGTH (1000 psi)	ELONGATION (%)	NOTCHED TENSILE STRENGTH, 1000 psi (K_t = 6.3)	NOTCHED/ UNNOTCHED TENSILE RATIO
AS RECEIVED	78	Longitudinal	-	-	-	80.4	
			31.9	87.3	36.0	79.3	
			32.4	90.1	48.5	81.4	
			32.0	87.7	38.5	80.7	
			31.9	89.7	51.0	79.7	
		Average	32.1	88.7	43.5	80.3	0.905
	-320	Longitudinal	-	-	-	154.3	
			-	-	-	161.0	
			49.3	173.7	42.0	162.1	
			56.8	178.4	42.0	158.2	
			60.0	187.9	41.0	151.7	
		Average	55.4	180.1	41.7	157.5	0.875
ELECTROPOLISHED	78	Longitudinal	-	-	-	76.8	
			-	-	-	78.7	
			31.8	86.4	61.0	78.2	
			32.8	87.8	60.0	80.6	
			32.5	86.8	62.0	78.8	
		Average	32.4	87.0	61.0	78.6	0.903
	-320	Longitudinal	62.8	215.8	36.5	159.1	
			66.2	217.3	38.5	167.6	
			67.5	214.5	32.5	155.3	
		Average	65.5	21[illegible].8	35.8	165.0	0.764

Table XI. Mechanical Properties of 304 ELC 70% Cold Rolled Stainless Steel, at Ambient and Cryogenic Temperatures

	TEMPERATURE (°F)	DIRECTION	YIELD STRENGTH 0.2% OFFSET (1000 psi)	TENSILE STRENGTH (1000 psi)	ELONGATION (%)	NOTCHED TENSILE STRENGTH, 1000 psi ($K_t = 6.3$)	NOTCHED/ UNNOTCHED TENSILE RATIO
AS RECEIVED	78	Longitudinal	170	190	3.5	209	
			172	192	2.5	217	
			171	190	2.5	218	
			176	192	2.5	206	
			176	193	2.5	208	
			174	195	3.0	210	
		Average	173	192	2.8	211	1.10
	-320	Longitudinal	197	273	29.0	277	
			209	273	29.0	274	
			207	273	28.0	274	
			208	280	29.0	278	
			213	282	28.5	-	
		Average	207	276	28.6	276	1.00
ELECTROPOLISHED	78	Longitudinal	159.5	185.8	4.5	206	
			164.7	185.3	4.0	206	
			164.5	186.3	3.5	207	
		Average	162.9	185.8	4.0	206	1.11
	-320	Longitudinal	189.7	276.7	31.0	273.8	
			195.8	279.0	35.0	272.2	
			185.8	269.4	31.5	272.8	
		Average	190.4	275.0	32.5	272.9	0.993

Table XII. Liquid Oxygen Cleaning Results

PART NAME	DIMENSIONS (IN.)	SERIAL NO.	EXTRACTION	PARTICLE COUNT-SIZE (MICRONS)					FIBER COUNT-SIZE (MICRONS)				WEIGHTS-MICROGRAMS FIBERS AND PARTICLES	HYDROCARBONS
				50-100	100-300	300-500	500-1000	1000	500-750	750-2000	2000-6000	6000		
ELECTROPOLISHED SAMPLES														
BELLOWS	10-3/4 ID × 9-3/4 L	1490	1			3	0	0		6	1	0	600	800
			2			0	0	0		0	0	0	100	<100
BELLOWS	10-3/4 ID × 9-3/4 L	415	1			5	1	0		23	2	0	200	
			2			0	0	0		0	0	0	<100	1100
BELLOWS	10-3/4 ID × 9-3/4 L	1142	1			1	0	0		28	2	0	400	1650
			2			0	0	0		0	0	0	<100	1200
BELLOWS	1-3/4 ID × 3-1/2 L	1336	1			0	0	0		0	0	0	300	3300
			2			0	0	0		0	0	0	300	<100
BELLOWS	6 ID × 4-1/2 L	1494	1			5	0	0		4	1	0	400	1300
			2			0	0	0		0	0	0	100	1000
BELLOWS	4 ID × 4-1/8 L	045	1			1	0	0		0	0	0	<100	<100
			2			1	0	0		2	0	0	<100	300
BELLOWS	2-5/8 ID × 7/8 L	355	1			2	0	0		4	3	0	-	-
			2			0	0	0		0	0	0	<100	400
BELLOWS	2-5/8 ID × 7/8 L	454	1			2	0	0		4	5	0	-	-
			2			0	0	0		0	0	0	300	200
BELLOWS	2-5/8 ID × 7/8 L	570	1			2	1	0		8	1	0	1800	200
			2			0	0	0		0	0	0	700	300
BELLOWS	2-5/8 ID × 7/8 L	3006	1			0	0	0		2	3	0	-	-
			2			0	0	0		0	0	0	500	<100
BELLOWS	11 ID × 10-1/4 L	2532	1			1	0	0		0	0	0	<100	1600
			2			1	0	0		0	0	0	100	<100
FLEX HOSE	1-1/4 ID × 14 L	1282	1			0	0	0		0	0	0	300	200
			2			0	0	0		0	0	0	<100	<100
FLEX HOSE	2 ID × 33 L	1686	1			0	0	0		2	0	0	200	500
			2			0	0	0		0	0	0	100	100
FLEX HOSE	4-1/2 ID × 38 L	2572	1			1	0	0		0	0	0	<100	<100
			2			0	0	0		0	0	0	300	300
FLEX HOSE	1 ID × 50 L	1080	1			0	0	0		0	0	0	<100	<100
			2			0	0	0		0	0	0	600	<100
DISCONNECT	APP. 9 × 2	2289	1			5	3	0		6	2	0	-	-
			2			2	0	0		1	0	0	300	100
PRESSURIZATION LINE	31 ID × 51 L	2303	1			1	0	0		1	1	0	300	1350
			2			0	0	0		0	0	0	<100	<100
FLEX METAL HOSE	3-1/2 ID × 30 L	270	1			0	0	0		0	0	0	<100	<100
			2			0	0	0		2	0	0	500	100
SAMPLES NOT ELECTROPOLISHED														
BELLOWS	10-3/4 ID × 9-3/4 L	2405	1	NOT COUNTED (HIGH)										
			2			1	0	0		0	0	0	100	1000
BELLOWS	10-3/4 ID × 9-3/4 L	2605	1	NOT COUNTED (HIGH)										
			2			0	0	0		0	0	0	<100	1650
BELLOWS	2-5/8 ID × 7/8L	1477	1			0	0	0		3	3	0	-	-
			2			1	0	0		0	0	0	600	200
BELLOWS	2-5/8 ID × 7/8 L	065	1			1	0	0		1	0	0	400	3400
			2			0	0	0		0	0	0	500	200

Table XIII. Dissimilar Metals Electropolishing Test

BATH NO.	MATERIAL	AREA (IN.^{2})	VOLTS	AMP/ IN.^{2}	TIME (MIN.)	RESULTS
			SINGLE COUPONS			
7	304 SS	16	3	2	3	Bright finish
7	K-Monel	16	3	2	3	Oxide film formed (dull finish)
7	Inconel	16	3	2	3	Pitted and uneven
7	Monel	16		1	3	Polished in areas of high C. D.
7	Monel	16		2	3	Pitted severely
7	Monel	16		3	3	Polished but mildly pitted
7	Monel	16		3	5	Polished but still some pitting
2	304 SS	16	5	2	3	Bright polish
2	K-Monel	16	20	2	3	Dull finish
2	Inconel	16	2	2	3	Dull finish
2	Monel	16	5	2	3	Dull finish
8	304 SS	16	4	2	3	Bright polish
8	K-Monel	16		2	3	Pitted and uneven
8	Inconel	16	4	2	3	Polished with slight pitting
			MECHANICALLY FASTENED COUPONS			
2	304 SS with K-Monel	28	8	2	3	Stainless steel, bright polish; K-Monel, dull finish
2	304 SS with Monel	28	8	2	3	Stainless steel, bright polish; Monel, dull finish
2	304 SS with Inconel	28	7	2	3	Stainless steel, bright polish; Inconel, dull finish
4	304 SS with K-Monel	28	-	-	-	Stainless steel, bright finish; K-Monel, dull finish
4	304 SS with Monel	28	-	-	-	Stainless steel, bright finish; Monel, dull finish
4	304 SS with Inconel	28	-	-	-	Stainless steel, bright finish; Inconel, smooth and bright

Table XIV. Protective Coatings Test Results

PAINT SYSTEM DESIGNATION	TOPCOAT	PRIME	(1) PENCIL HARDNESS	(2) FLEXIBILITY	(3) ADHESION RONDEAU-GARDNER CASE A	CASE B	FILM THICKNESS (MILS)	(4) RESISTANCE TO SOLVENTS* 1	2	3	(5) WEATHER-OMETER** 1	2	3	(6) GLOSS AFTER SALT ATMOSPHERE TEST PANEL 1	PANEL 2	PANEL 3	PANEL 5	PANEL 7	PANEL 9
A	DU PONT FEP TEFLON	NONE	B	NO AFFECT	0.5	0.1	1.1	B	B	B	52	18	28	50	50	46	--	49	--
C	PENNSALT VINYLIDEN FLUORIDE (SEE PROCEDURE)	NONE	H	NO AFFECT	1.9	1.9	1.6	H	H	H	8	4	4	--	3	--	--	--	--
E	EMRALON 310	NONE	HB	NO AFFECT	1.6	1.7	1.2	B	B	HB	30	3	14	15	24	--	-	28	--
F	NUBELON S	NONE	4H	NO AFFECT	1.6	1.8	1.8	4H	4H	4H	93	6	10	56	60	55	85	-	--
G	ANDREW BROWN A423 EPOXY	ANDREW BROWN TA862 (FMS 0003B)	4H	NO AFFECT	1.4	1.5	2.2	H	H	H	100	4	10	58	61	80	--	--	95
H	SHERWIN WILLIAMS M49WC8 FLAT WHITE ACRYLIC	WASH PRIME, MIL-C-8514	2H	NO AFFECT	1.2	0.9	2.5	<B	<B	2H	10	4		5	6	8	--	9	--
I	ANDREW BROWN POLY-URETHANE, MIL-C-27227	SHERWIN WILLIAMS WASH PRIME MIL-C-8514 COVERED WITH ANDREW BROWN TA862 (FMS0003B)	3H	CRACKS ON TENSION SIDE	2.6	2.9	2.2	3H	3H	3H	92	7	8	71	--	--	--	--	--
J	W.P. FULLER MIL-C-7178 LACQUER	W.P. FULLER ZINC CHROMATE PRIME, MIL-P-6889A	FAILED CRYOGENIC CYCLING TEST																
K	ANDREW BROWN MIL-E-7729 ENAMEL	SHERWIN WILLIAMS WASH PRIME, MIL-C-8514																	
L	ANDREW BROWN Z SPAR 100	ANDREW BROWN P-93 RED PRIME																	

* (PENCIL HARDNESS AFTER EXPOSURE AND 1 MINUTE RECOVERY)
1. TRICHLOROETHYLENE
2. OXYLENE
3. TEC 901

**
1. GLOSS BEFORE TEST
2. GLOSS AFTER TEST
3. GLOSS AFTER TEST AND WASHING

Table XV. Faying Surface Test Results After 300 Hours of Salt-spray Exposure

RINSING METHOD	FORMULATED BATHS	RESULTS
1	1	Trapped electrolyte present
1	2	Trapped electrolyte present
1	3	Trapped electrolyte present
1	4	Trapped electrolyte present
2	1	Trapped electrolyte present
2	2	Trapped electrolyte present
2	3	Trapped electrolyte present
2	4	Trapped electrolyte present
3	1	Slight trace of electrolyte present
3	2	Trapped electrolyte present
3	3	Trapped electrolyte present
3	4	Trapped electrolyte present
4	1	No trace of electrolyte present
4	2	No trace of electrolyte present
4	3	No trace of electrolyte present
4	4	No trace of electrolyte present

Note: Corrosion staining was evident in samples due to the failure of the fasteners in the salt-spray cabinet.

Table XVI. Evaluation of 321 Stainless Steel Butt-welded Specimens After 288 Hours of Salt-spray Exposure

BATH NO.	SPECIMEN NO.	EVALUATION
1	23	Corrosion staining on surface, clean weld
1	24	Corrosion staining on surface, clean weld
1	25	Corrosion staining on surface, clean weld
2	29	Slight corrosion staining in weld area
2	30	Slight corrosion staining in weld area
2	31	Slight corrosion staining in weld area
3	16	Corrosion staining in weld area
3	17	Corrosion staining in weld area
3	18	Corrosion staining in weld area
4	4	No attack
4	5	No attack, corrosion staining on edge
4	6	No attack, corrosion staining on edge
Not Processed	32	Generally corrosion stained and pitted
Not Processed	33	Generally corrosion stained and pitted
Not Processed	0	Generally corrosion stained and pitted

Table XVII. Salt-spray Results on Unwelded Samples After 800 Hours of Exposure

MATERIAL	RESULTS
Type 321 S.S. (Polished)	No Attack
Control	Corrosion stained
Inconel 718 (Polished)	No Attack
Control	No Attack
Inconel X (Polished)	Corrosion pitted on two areas
Control	Slightly corrosion stained
Hastelloy C (Polished)	No Attack
Control	No Attack
René 41 (Polished)	No Attack
Control	No Attack

Table XVIII. Salt-spray Results on Welded* Samples After 800 Hours of Exposure

MATERIAL	RESULTS
Inconel 718 and Type 321 S.S. (Polished)	No Attack
Control	Corrosion stained at weld area
René 41 and Type 321 S.S. (Polished)	No Attack
Control	Severe corrosion at weld area
Hastelloy C and Type 321 S.S. (Polished)	No Attack
Control	Severe corrosion at weld area
Inconel 718 and Hastelloy C (Polished)	No Attack
Control	No Attack
Inconel 718 and René 41 (Polished)	No Attack
Control	No Attack

* All specimens were butt-welded by the TIG method in a welding machine with no filler metal added.

APPENDIX A

CLEANING

Cleaning is essential for best electropolishing results. Electropolishing is conducted in much the same manner as plating, and surface conditions, regardless of base metal, determine to a large extent the degree of good surfaces obtained after processing. Equipment and materials for cleaning are the same as those generally used in the metal-finishing industry.

Vapor degreasing, solvent cleaning, and alkaline cleaning are used to remove surface soils such as greases, oils, fingerprints, and fine solid particles. Grease and oil films not entirely removed in cleaning are removed in the electropolishing bath thereby forming a film on the surface of the solution. If this film is not removed, it will cause uneven polishing due to the film's adhesion to the parts being immersed in the bath. This is especially true if the parts are immersed when dry.

Adequate rinsing after cleaning is necessary to prevent carry-over of alkaline solution wetting agents and other contaminating agents in the polishing electrolyte.

Water concentration of the electrolyte is rather sensitive and care must be taken that excessive "drag in" of water is not encountered. A secondary hot-water rinse or a clean dry-air blast may be necessary if parts are such that the designs include deep cavities or traps that can not be adequately drained before immersing in the electropolishing bath.

At times it becomes necessary to remove heat scale, weld scale, oxide coatings, and similar tenacious coatings prior to electropolishing. These are generally removed by pickling, salt-bath descaling, or by dry blasting or vapor-honing.

Pickling baths are formulated for specific alloys and care must be exercised when selecting this type of cleaning.

The cleaning process sequence and solution make-up to obtain bright parts that are free of visible oxide, scale, and other contaminants on Inconel 718 and Inconel X alloys are listed in Tables A-1 and A-2. Vapor degreasing and alkaline cleaning are typical processes that are used for all alloys; however, the pickling and bright dip solutions are specifically formulated for these alloys. They consist of a 15 percent solution hydrochloric acid bath to which cupric chloride is added, followed by a bright dip of sulfuric acid and sodium dichromate.

Table A-1. Inconel 718 Cleaning

OPERATION	TEMP (°F)	TIME (MIN)
Vapor degrease	180-190	as required
Alkaline clean	170-190	as required
Rinse, tap water	170-190	as required
Pickle	170-190	5-10
Rinse, tap water	room	as required
Bright dip	room	5-10
Rinse, tap water	room	as required
Dry	150-200	as required

Table A-2. Preparation of Solutions for Inconel 718 and Inconel X

SOLUTION	MATERIALS	CONCENTRATION
Alkaline Cleaner	Tap Water	as required
	Oakite 61A	4-8 oz/gal.
Pickle	Tap Water	as required
	Hydrochloric Acid	14.5-17.5 oz/gal.
	Cupric Chloride	2.5-3.0 oz/gal.
Bright Dip	Tap Water	as required
	Sulfuric Acid	21-24 oz/gal.
	Sodium Dichromate	14.5-17.5 oz/gal.

Type 321 stainless steel requires slightly different cleaning solutions. This consists of a nitric acid, hydrofluoric acid pickling followed by nitric acid passivating. The nitric acid passivating is included on stainless steel, since in production there is usually an extended time lag before further processing is completed. The processing sequence and solution made up for 300-series stainless steels are listed in Tables A-3 and A-4.

For very stubborn oxide scales, and staining (particularly in welded areas) it may be necessary to remove the contaminants by abrasive blasting. Wet abrasive blasting or vapor honing with a 100-mesh silica grit is recommended for use on stainless steel and high-nickel alloys.

Table A-3. Processing Operation for Type 321 Stainless Steel, Hastelloy C, and René 41

OPERATION	TEMP (°F)	TIME (MIN)
Vapor Degrease	170-198	as required
Alkaline Clean	170-190	2-10
Hot Rinse	170-190	3-5
Stainless Steel Pickle #1	60-130	10-20
Rinse Tap Water	room	3-5
Passivate	room	15-60
Rinse	room	5-10
Dry, Circulated Hot Air	150-200	as required

Table A-4. Solution Preparations

SOLUTION	MATERIALS	CONCENTRATION
Alkaline Cleaner	Tap Water	as required
	Oakite 61A	4-8 oz/gal.
Stainless Steel Pickle No. 1 (Chrome-Nickle Alloys)	Tap Water	as required
	Nitric Acid	20-34 oz/gal.
	Hydrofluoric Acid	2-4 oz/gal.
Passivate	Tap Water	as required
	Nitric Acid	26-36 oz/gal.
	Sodium Dichromate	1/2-3 oz/gal.

The process used in preparing samples for test specimens is as follows:

a. Degrease part as necessary.

b. Blast part.

c. Rinse loose abrasive from part with tap water.

The following results should be achieved without causing abrasions that interfere with operational movements:

a. Blasted parts shall have a clean, dull, uniform surface.

b. Parts shall be free of all loose abrasive.

To determine the effect of cleaning prior to electropolishing on the corrosion resistance of the polished surface, test panels were prepared with four different cleaning methods, electropolished, and submitted to salt-spray testing. The test consisted of electropolishing Inconel 718, Inconel X, Hastelloy C, René 41 and Type 321 stainless steel alloys after each of the following cleaning procedures: solvent cleaning, alkaline cleaning, chemical pickling, and vapor honing.

The test specimens were electropolished in the phosphoric-sulfuric-chromic bath at eight volts for four minutes at 130°F.

The test specimens were exposed to accelerated corrosion tests in a salt-spray chamber at 95°F with an atomized solution of 20-percent sodium chloride. The test was conducted in accordance with Method 811.1 of Federal Test Standard 151a.

After 800 hours of salt spray testing all the electropolished samples were unaffected. This impressively demonstrates that each of the above methods of cleaning prior to electropolishing are acceptable. The choice of cleaning method is dependent on the surface condition of the metal. If the base metal is free of scale and oxides, only degreasing is necessary. If chemical cleaning is necessary, electropolishing will polish the etched surface. Stubborn weld scale can be vapor-honed prior to polishing. It is necessary to use the above methods of cleaning since removal of scale is uneven or nonuniform in electropolishing baths and pitting results at metal sites exposed first, while scale remains on other sites. Correctly formulated pickling baths will not dissolve the base metal, but will attack the surface scale regardless of its thickness or chemical composition. (15)

Representative specimens of Type 321 stainless steel were examined by electron microscopy after pickling, passivating, and electropolishing. These specimens were compared with specimens that received the standard pickling and passivating treatment. The electropolished specimens were examined by means of dry-stripped collodion replicas, shadowed with palladium at an angle of approximately 45 degrees. The unpolished specimen was too rough to permit this technique to be used. Instead, a first-stage replica was made using nitrocellulose film moistened with amyl acetate. This was strong enough to be stripped easily from the specimen surface. This replica was then shadowed with palladium at 45 degrees, after which a carbon film was deposited. The nitrocellulose film was dissolved in amyl acetate. The pre-shadowed carbon film was then examined in the electron microscope.

Both techniques provide negative replicas of the surface to be examined; that is protrusions from the replica correspond to depressions in the specimen surface, and vice versa. The photo micrographs have been processed so that the shadows produced by surface irregularities are black, just as are shadows of objects in sunlight. Thus in Figure A-1, the electropolished surface shown is extremely even, with only the faintest indications of changes in relief between grains. Intergranular fissures have

Figure A-1. Electropolishing. Electron photomicrograph of normal 321 Stainless Steel (pickled and passivated; magnification, 6000 times) that has been electropolished. Note that potential corrosion sites have been virtually eliminated. The two spheres shown are for purposes of measurement.

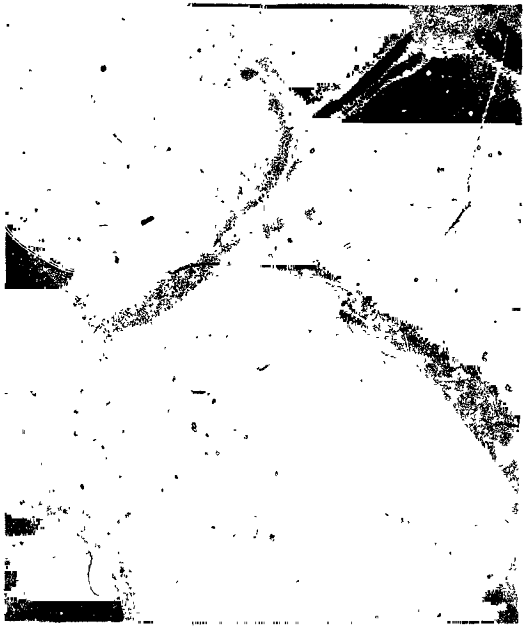

Figure A-2. No Electropolishing. Electron photomicrograph of normal unpolished 321 Stainless Steel (pickled and passivated; magnification, 6000 times). Note recessed areas which entrap corrosive elements, and result in corrosion.

been completely removed, and only shallow depressions of the order of 0.2 microns (8 microinches) are seen around a few particles in the matrix. Knowing the angle of shadowing to be approximately 45°, the height of an object above the replica surface is approximately equal to the length of the shadow it casts. If, however, the grain-boundary areas in the replica have bent over after stripping, due to lack of flexural strength in the thin plastic film, any height so calculated will be less than the true height, and the intergranular cracks in the metal will be deeper than calculated.

In contrast, an electron photo micrograph of the unpolished pickled and passivated specimen, Figure A-2, shows the grain boundaries raised above the surface of the replica. Since they produce shadows across the adjacent grains. Hence, it is determined that the grain boundaries in the pickled and passivated metal have been eroded to a depth of more than one micron (40 microinches) in some places. Small surface irregularities are also visible on the generally smooth grains. These may be extra phases in the metallic matrix, or may be surface contamination. No attempt was made to identify these irregularities. Surface relief of this magnitude is sufficient to provide opportunity for retention and concentration of corrosive materials at the grain boundaries.

This sensitive area is then susceptible to the initiation of corrosion pitting and may explain one of the reasons why electropolished surfaces are resistant to corrosion.

APPENDIX B

EQUIPMENT SETUP FOR IMMERSION ELECTROPOLISHING

Equipment setup and handling of work as to racking are similar to electroplating except that the material to be electropolished is connected to the positive lead from the direct-current source. Thus, the material is the anode in the electrolytic circuit. The cathode is connected to the negative terminal of the direct-current source and may be any metal or carbon that is chemically resistant to the electrolyte. The individual cathodes are placed so that current distribution is as uniform as possible.

The Battelle Electropolishing Process (16) for stainless steel requires the following equipment:

a. Alkaline cleaner tank (mild steel) for temperatures of 180 to 200°F, equipped with bottom drain (flanged), and electric heaters for steam platecoils and thermostat.

b. Cold water rinse tank (mild steel) equipped with dam-type overflow and bottom drain.

c. Battelle acid tank (mild steel, No. 8 chemical lead-lined), to operate at 75 to 130°F with lead-coated immersion heaters (General Electric) and thermostat. The tank should be insulated with 1 inch of fiberglass, covered with black iron (10 gauge), and equipped with propeller mixer (electric), plastisol-coated for agitation.

d. Cold water rinse tank (same as Item b except that it is lined with 3/32-inch PVC).

e. Dilute acid tank (mild steel, lined with 3/32-inch PVC) to operate at room temperature.

f. Cold water rinse tank (same as Item b).

g. Hot water rinse tank (mild steel, lined with CPD-10 neoprene 3/32-inch PVC) to operate at 180 to 200°F by electric immersion heaters (stainless steel) or stainless steel steam platecoils and thermostat.

h. Udylite selenium rectifiers, rated at appropriate ampere capacities to accommodate 300 amps/square foot at 18 volts, integral regulation with variable stepless control from 0 to 18 volts (one each for Tanks 3 and 5).

The size of the parts determines suitable tank dimensions and rectifier capacities as well as heater and steam-coil units.

Operating instructions are given with the bath; however, included here are comments on operation from the Battelle process that are typical of the controls necessary to obtain good electropolishing results.

TYPE 302 (AND OTHER 300 SERIES) STAINLESS STEEL

For these steels, the temperature of the fresh bath is kept between 170 and 190°F, and the specific gravity is 1.71 to 1.72 as measured at the operating temperature (if the specific gravity is lower, the bath is heated as previously described).

The current for the tank load is adjusted to 250 to 350 amps/square foot (1.7 to 2.4 amps/square inch); the tank is kept in continued use, the only control being the addition of fresh bath to replace dragout. The specific gravity increases slowly and is allowed to reach 1.74 to 1.76. At this point the tank is operating at 190 to 200°F and the dissolved iron from the work is about three percent (by weight) of the bath. The bath is maintained thereafter at this operating level.

SLUDGING

Sludging results from the precipitation of nickel and iron salts and accounts for about 10 to 40 percent of the metal dissolved from the material, depending on operating conditions. The sludge settles to the bottom of the tank and requires occasional removal. In a 750-gallon tank, operating at the heavy load of 10 amps/gallon, three shifts per day, using one program for Type 302 stainless steel, 14 inches of sludge were formed in 10 months of operation.

Iron dissolves in the III valence state from the work during electropolishing. This ferric iron is soluble in the electropolishing bath, and is reduced at the cathodes to ferrous iron. Ferrous sulfate is insoluble in the electropolishing bath and sludges out. To promote the most effective removal of iron by sludging, the cathode-current density should be less than 80 amps/square foot. Ferrous sulfate may form a crust on the cathodes. This will cause the tank voltage to increase; consequently, the crust must be removed by taking the cathodes out and dissolving the crust in hot water.

No reference is made to the dissolved-chromium concentration. Chromium does not precipitate, and with the dragout and/or "decant and replacement process" of control, the chromium concentration is taken care of automatically and should remain at about 0.8 to 1.5 percent.

ECONOMICS

One advantage in electropolishing is the savings in time which can be transferred directly into costs. Normal electropolishing is a short operation, the average time being from four to eight minutes. Precleaning cycles are reduced since degreasing or mild alkaline cleaning is usually all that is necessary prior to polishing. Chemical costs should be less, since pickling and passivating are not necessary.

Parts cannot be immersion electropolished as each part must be positioned individually on a rack. Special racking with conforming cathodes in irregular-shaped designs is required to obtain polishing in recessed areas. Racking of the parts are similar to the racking encountered in chromium plating.

Consistant results will be obtained, since close control of time, temperature, and current density is required.

Table B-1 lists some typical ducting bellows, flexible hoses, and couplings used in the test program shows the costs of the electropolishing process at a commercial installation. These prices did include some special racking, but did not include any packaging.

Table B-1. Electropolishing Costs

PART NAME	DIMENSION (IN.)	PRICE ($)
Bellows	10-3/4 ID x 9-3/4 L	5.25
	6 ID x 4-1/2 L	2.00
	4 ID x 4-1/8 L	1.50
	11 ID x 10-1/4 L + Flange	35.00
	2-5/8 ID x 7/8 L	1.00
	1-3/4 ID x 3-1/2 L	1.00
Flexible Hose	4-1/2 ID x 38 L	25.00
	2 ID x 33 L	20.00
	1-1/2 ID x 14 L	8.00
	1 ID x 50 L	10.00
	3/4 ID x 26 L	15.00
	3/4 ID x 28 L	10.00
Tubing	2-3/4 ID x 51.5 L	15.00 (per Linear Foot 1.50)
Coupling	Assorted parts	10.00

The Electro Process and Engineering Corp., San Diego, California, has been electropolishing production and test components for missile and space vehicles since 1959. Stainless steel valves, manifolds, complex housings, and miscellaneous small parts are being electropolished for both Atlas and Titan missile systems. Parts are being electropolished for various reasons which include: deburring of sharp edges, to prevent galling of threaded parts or close fits, to remove discoloration due to heat treatment or welding, and to improve corrosion resistance.

Listed in Table B-2 are typical components that are presently being electropolished during this program.

Table B-2. Typical Electropolishing Production Parts

MISSILE SYSTEM	PART NAME	PART NUMBER
Atlas ICBM	Propellant Utilization Manometer Housings & Related Hardware	7-43450-803 7-43450-801 27-43014-501 27-43015-501 55-43006-501
	Manifold and Attached Ducting	27-24033-9
	LO$_2$ Boil-off Valve Hub Assembly	27-80758-1
Titan ICBM	Valves	
	4" Valve Body	10381
	3" Valve Body	10002
	3" Valve Body	10385
	2" Valve Body	10003-1
	1-1/2" Valve Body	10389
	1/2" Valve Body	10403
	1/2" Valve Body	10122
	Mandrels and Large Sub Assemblies	S-760-3 S-7472 S-7163 S-700-2 10124 10383