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Electroforming of a Throat Nozzle for a Combustion Facility

J. W. Dini, H. R. Johnson

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ELECTROFORMING OF A THROAT NOZZLE FOR A COMBUSTION FACILITY

(NASA Langley Reimbursable Program)

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AESTRACT

Special procedures were developed and then utilized for plating nickel over channels of a throat nozzle section of a NASA Langley combustor facility. When tested hydrostatically, the part failed in the stainless-steel substrate and not at the interface between the plating and substrate. The procedures used for plating the part are detailed as are high-temperature property data which show that the part can withstand long-term, high-temperature exposure without suffering degradation of the plated bond.

ACKNOWLEDGMENTS

A number of people contributed to the work described in this report. We would particularly like to acknowledge the support of the following at Sandia: D. E. Brown and J. R. Helms, plating; L. Reis and W. Young, machining; D. R. Adolphson, guidance and faith; and Pete Dean, technical writing assistance. From NASA-Langley, Dr. J. D. Buckley provided program direction and much encouragement, B. H. Wilfong, many helpful discussions and high-temperature property data, W. A. Wright and W. K. Gerdes, NDE of the nozzle section, Roger Buchanan, property data, and J. C. Miller and his people, helpful dialogue.

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Summary

Procedures were developed for plating thick nickel on throat nozzles for combustor facilities. To demonstrate that the process works with parts of varying curvature, a 15.2 by 66 cm section of an actual nozzle was plated at SLL, hydrostatically tested, and then sent to LRC for further evaluation. Before plating the nozzle section, quantitative adhesion tests were made on test samples to help pinpoint the best procedures to use. The chosen technique works equally well with nickel as substrate material so that full-scale LRC nozzles which contain nickel inserts can also be treated by this method. Essentially, the process consists of wedging aluminum strips over the channel cavities and then cleaning, activating, and plating the part. Upon completion of plating, the aluminum strips are chemically dissolved. The key to the process is the use of a nickel sulfamate-sulfamic acid solution which prepares the stainless steel for reception of an adherent electrodeposit without attacking the aluminum strips.

At LRC, the integrity of the plated bond was verified by ultrasonic "C" scan testing, and the channels in the stainless steel were shown to be free of obstructions by using temperature sensitive liquid crystals. Under hydrostatic load, the part failed at 24 MN/m^2 in the stainless steel. Planned operating pressure for this type of part is 3.5 MN/m^2, so procedures used for electroforming the nickel skin provide a significant factor of safety.

Long-term, high-temperature (2000 hours, 538°C) exposure of plated parts resulted in no room temperature degradation of the plated bond, although both nickel and nickel-cobalt deposits suffered a reduction in hardness. I-beam tensile tests showed that the elevated temperature (up to 649°C) strength of the bond between nickel and 405 stainless steel was at least as high as that of the substrate stainless material. Lastly, nickel tensile specimens tested at elevated temperature showed a reduction in ductility properties in the range of 400 to 500°C.

A brief review is included of extensive work undertaken by NASA-Lewis on NDE (nondestructive evaluation) of plated thrust chambers. Ultrasonic "C" scan, holography, and acoustic emission were evaluated in detail. The most discriminating results were obtained with a combination of holography and acoustic emission because these methods detected weak bonds that could not be distinguished by other methods.

ELECTROFORMING OF A THROAT NOZZLE FOR A COMBUSTION FACILITY

Introduction

The fabrication of the throat nozzle for the thermal protective system test facility (TPSTF) at NASA Langley Research Center (LRC) involves a somewhat unusual and difficult plating problem. An overall view of the TPSTF is shown in Figure 1, and a view of a nozzle throat in Figure 2. Figure 3 illustrates a section of a partially completed throat nozzle. As shown, channels ranging from 1.0 to 5.1 mm deep by 5.6 mm wide have been machined into a surface of varying curvature. The electroforming portion of the fabrication sequence consists of electroplating a thick nickel coating (0.9 to 4.0 mm) continuously across the lands to enclose the channels. The difficulty associated with the plating operation lies in deposition of nickel on the 405 stainless steel substrate without obstructing the channels.

Brazing has been the classical approach to the fabrication of this type of part. NASA is searching for a less expensive, more reliable process which yields consistently performing structures. In addition, brazing will not work for complicated designs.

The initial attempt at applying the nickel coating involved first filling the channels with wax, plating and machining the part, and them removing the wax. It was discovered, however, that all of the wax could not be removed from the channels after the nickel coating was applied. The following problems also arose: (1) poor adhesion between the nickel plating and the stainless steel, (2) laminations within the nickel deposit, and (3) poor weldability of the nickel deposit.

To better understand the electroforming technology and the problems NASA-LRS was encountering in producing nozzles, a contract was negotiated with Sandia Laboratories, Livermore (SLL). It was the opinion of SLL personnel that the key to solving the plating problems was to use a channel filler that did not contaminate the land surfaces. It was proposed that aluminum strips be wedged across the top of the channels to prevent plating in the channels. The process then included use of a nickel sulfamate-sulfamic acid solution to prepare the stainless steel for reception of an adherent electrodeposit without attacking the aluminum strips. The viability of this proposed technique had been previously demonstrated on small parts. Figure 4a shows such a part with some aluminum strips wedged in place. In some channels, aluminum spacers with pre-punched holes are shown ready to receiver the strips. * Figure 4b shows the same part after it has been plated and machined and the aluminum chemically removed.

*Subsequent development showed that these spacers were not necessary.

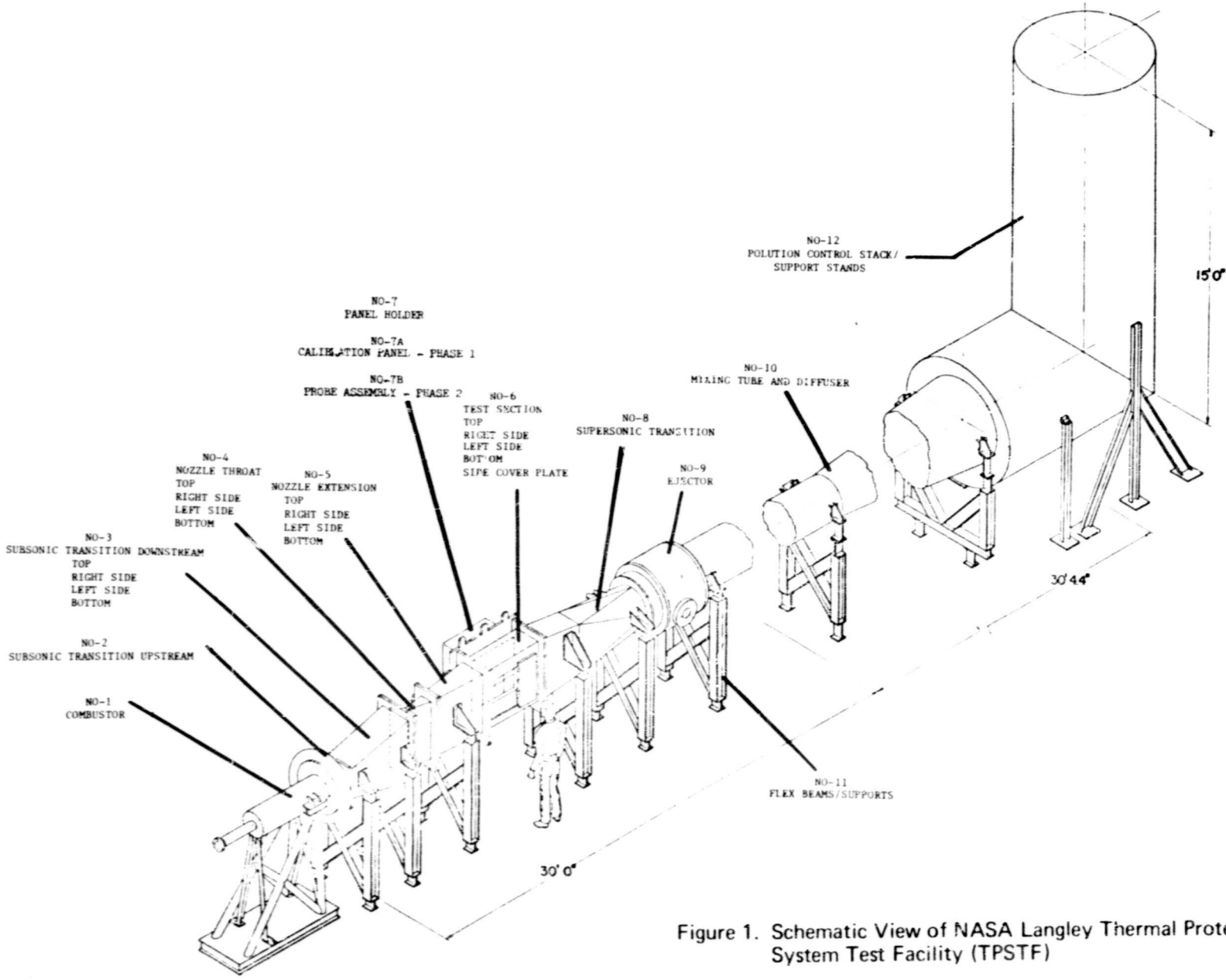


Figure 1. Schematic View of NASA Langley Thermal Protection System Test Facility (TPSTF)

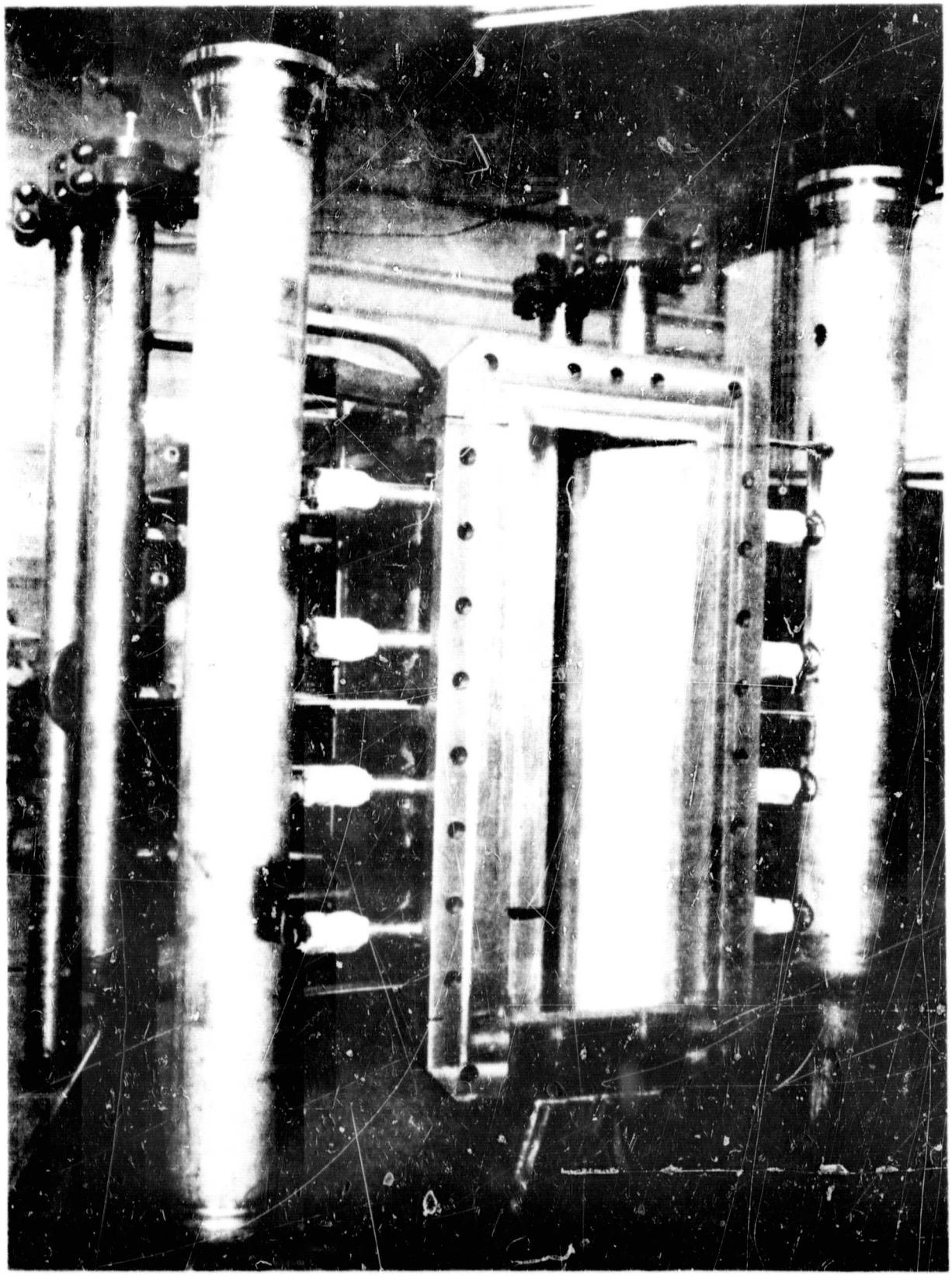


Figure 2. Nozzle Throat Section of NASA TPSTF

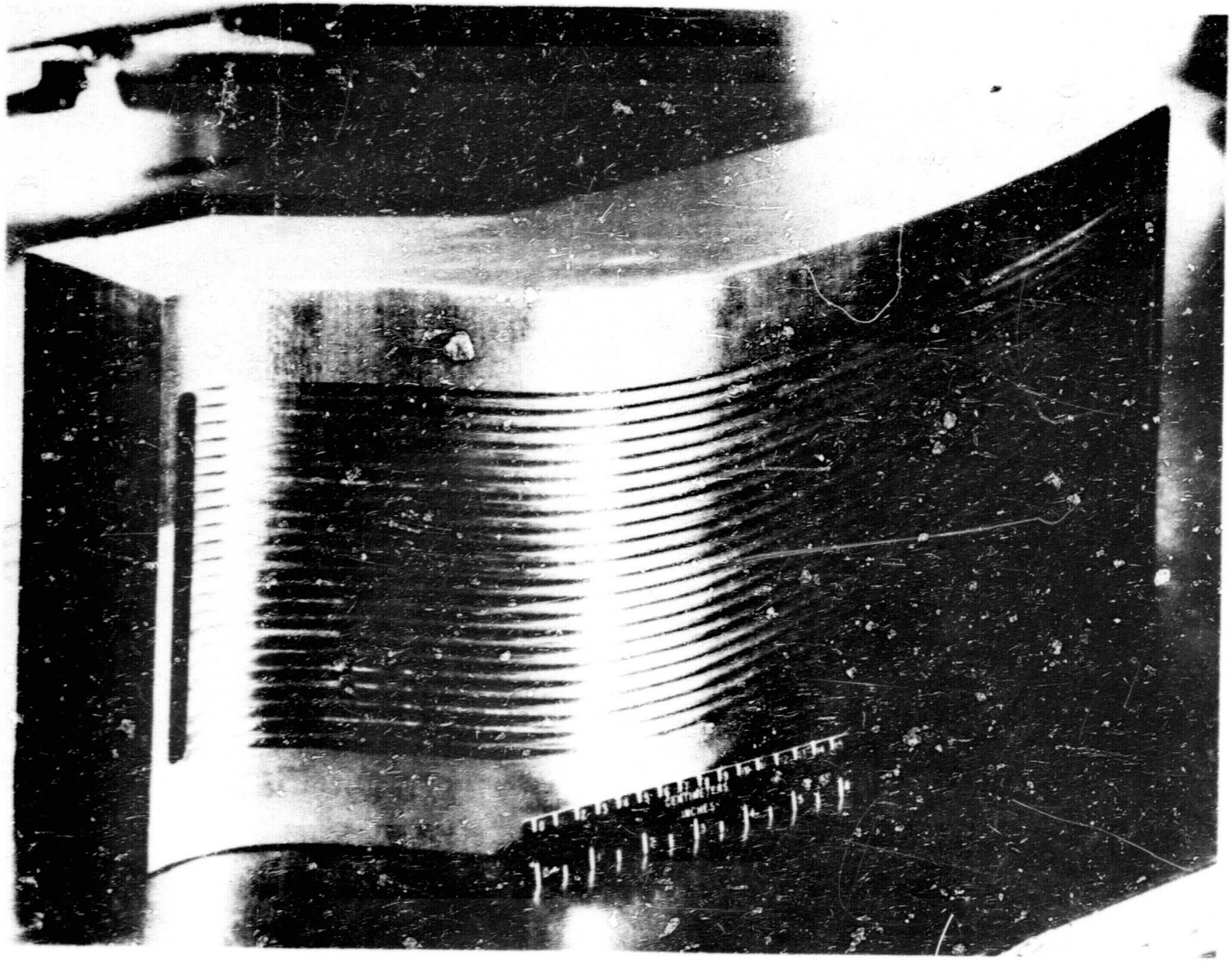
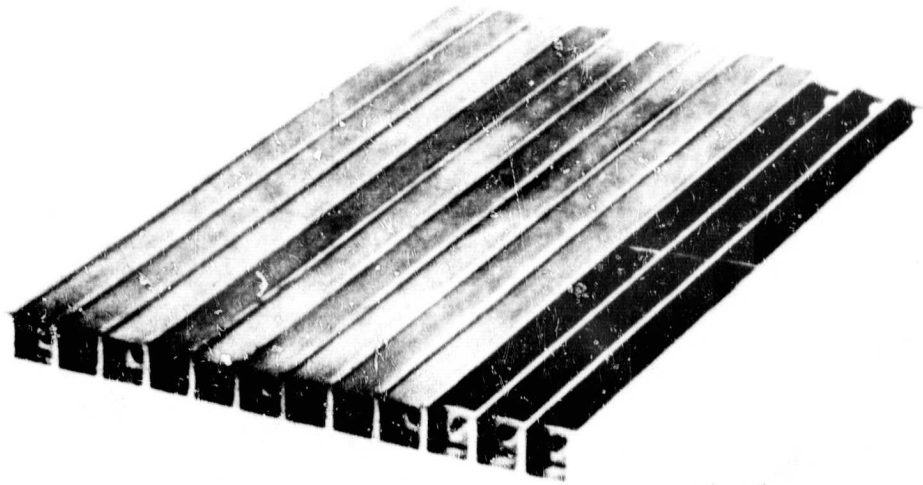
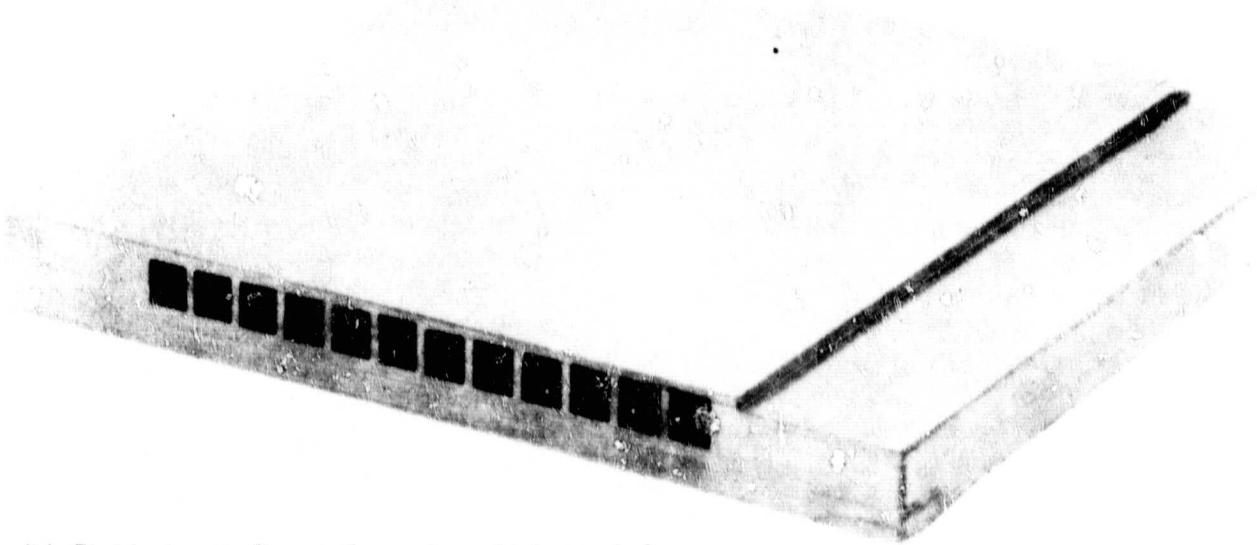


Figure 3. NASA Test Part Prior to Plating



(a) Aluminum strips and spacers in place prior to plating



(b) Finished part after plating and machining and after dissolving aluminum strips and spacers

Figure 4. Test Part Demonstrating Sandia's Proposed Fabricating Technique

Section 1 - Plating Nozzle Structure

A 15.2-cm wide by 66-cm long section of a full-scale throat nozzle (NASA-LRC drawings LD523892 and LD523893) was supplied for this program. The following sections describe (1) the trial runs in which nickel was plated nonadherently so that the dissolution of the aluminum could be evaluated; (2) quantitative adhesion tests used to determine the best cleaning/activating cycle for preparing the nozzle for its final plating; (3) the procedures used in the final plating operation, (4) test data for the finished part; and (5) the property data of the nickel deposited on the finished part.

Dissolution of Aluminum

For the first attempt at plating the nozzle, 0.81-mm thick aluminum strips were given a copper flash (about 0.013 mm) and wedged in the channels (Figure 5). The part was then cleaned, activated, and plated with enough nickel to meet LRC requirements. During dissolution of the aluminum, it became evident that adherence between the nickel plating and stainless steel was quite poor. In fact, as shown in Figure 6, it was possible to physically detach the nickel from the stainless steel.

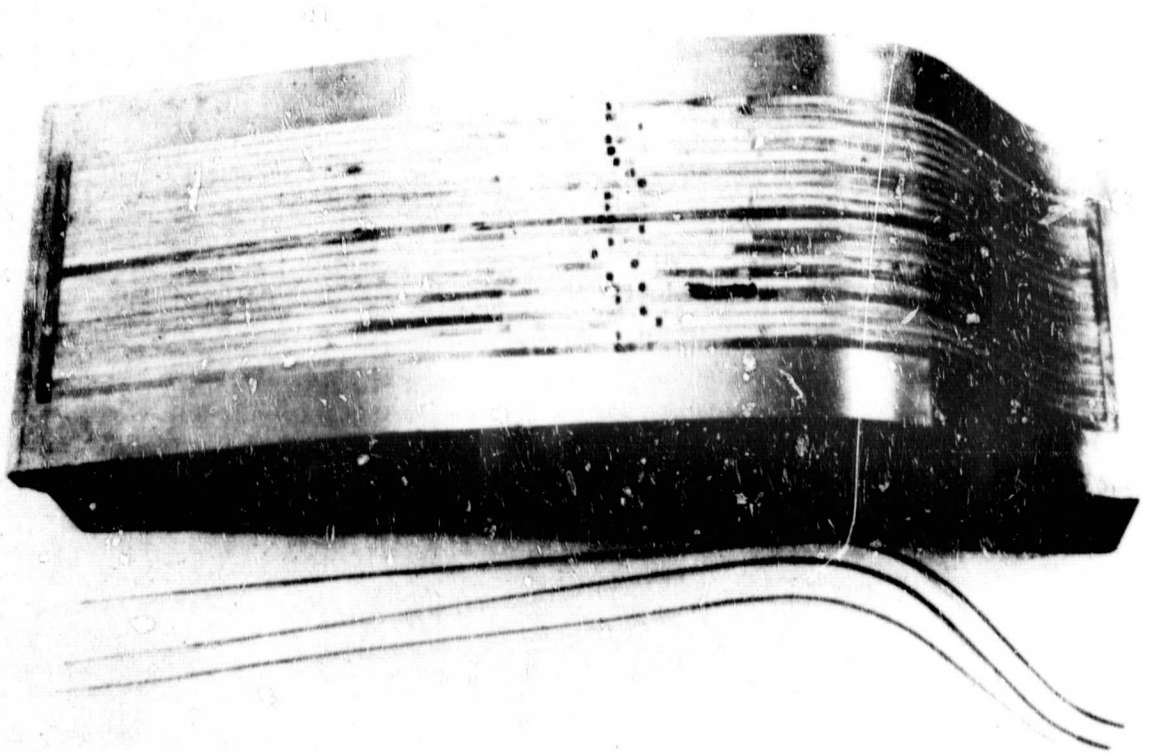


Figure 5. NASA Test Part With Copper Plated Aluminum Strips in Place
(Preformed aluminum strips are shown in foreground.)

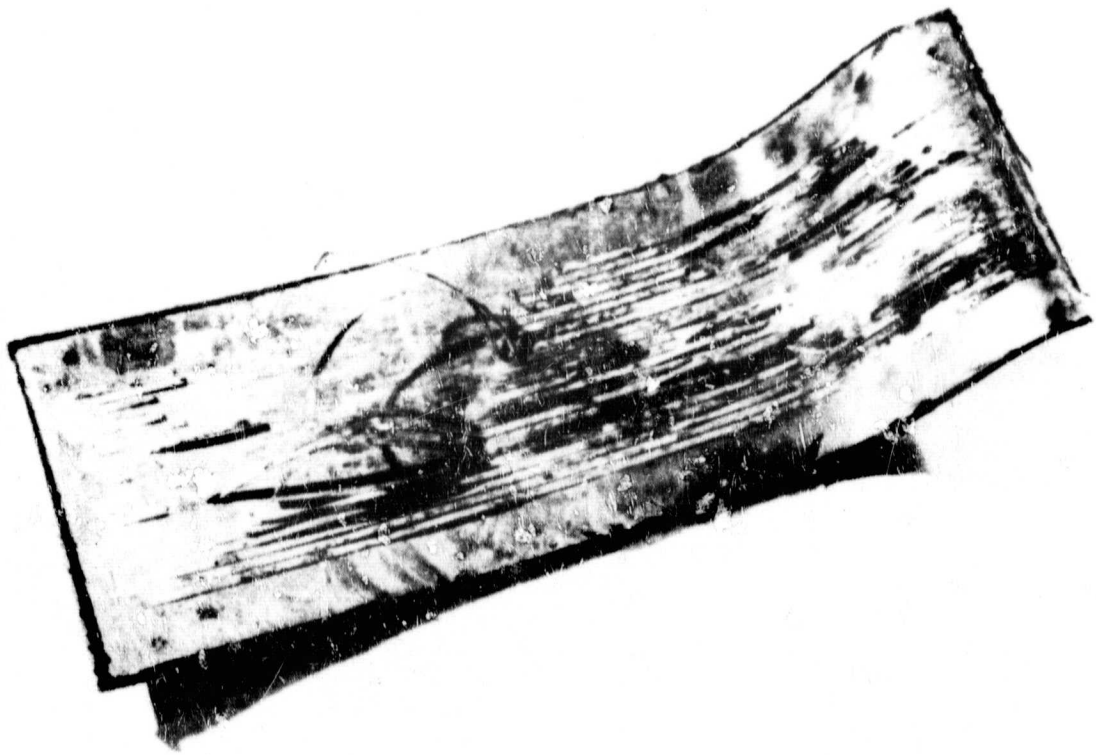


Figure 6. Electroformed Nickel Cover After Separation From NASA Test Part Showing Aluminum Strips Which Were Not Dissolved

Removal of the nickel revealed that the dissolution of the aluminum was not complete in that heavy precipitates, formed by alternate immersion in caustic and acid solutions, clogged the channels and prevented flow (Figure 7). Thus the poor adherence of the nickel actually proved to be beneficial in that it revealed the need for further development of the dissolution technique.

The source of the poor adherence was found to be the combination of sulfur depolarized (SD) anodes and wetting agent used in the nickel strike solutions. These anodes provided a convenient means of nonadherently plating the nickel in the two subsequent runs that were made to modify the dissolution technique for the aluminum. The poor adherence produced by the anodes was completely unexpected because SD anodes help remove copper from nickel sulfamate plating solutions by tying it up as an insoluble sulfide.[1] It was felt that this same purification process would work in the highly acidified nickel strike solution (Wood's strike solution). However, in this solution the SD anodes cause SO_2 to form which also reacts with the available wetting agent (sodium lauryl sulfate) to form gummy, insoluble, oil-like products that cause poor adhesion. (The wetting agent had been

Figure 7. NASA Test Part After Separation of Electroformed Nickel Cover Showing Precipitates and Aluminum Strips Plugging the Channels

introduced into the strike solution via the bagged anodes which had previously been used in a nickel sulfamate solution containing the wetting agent.)

In a second trial, the aluminum was not flashed with copper; thus acid was not needed during dissolution. The aluminum strips were wedged in place and the channels backfilled with water. The backfilling was done by welding fittings in place on the inlet and outlet sections of the nozzle and then using a finger pump to move water through the channels. Approximately 0.64 mm of nickel was plated. Caustic solution was then pumped through the channels to dissolve the aluminum strips. Upon cessation of the reaction, the nickel was removed from the stainless steel and the channels inspected. Initially, the channels were filled with 205 grams of aluminum; after completion of the above steps, 25 grams remained in the channels. This residual aluminum was attributed to formation of insoluble aluminum compounds which precipitated on the part walls. It was felt that these aluminum compounds could be completely removed by using a caustic solution with additives to sequester these compounds and thereby prevent their precipitation on the walls of the channels.[2]

The third trial run resulted in another improvement in that a source* was discovered for producing 0.51-mm thick, 5052-H34 aluminum strips considerably thinner than the 0.81-mm thick strips used on the previous trials. With the 0.51-mm thick strips, much less aluminum had to be removed, and therefore the flow of the caustic stripping solution was less impeded. After plating 0.64 mm of nickel on the part, dissolving the aluminum, and then separating the nickel from the stainless steel, visual inspection indicated that all the aluminum was removed from the channels.

Quantitative Adhesion Tests

Concurrent with the aluminum dissolution studies, quantitative adhesion tests were run to determine the best cleaning/activating cycle for preparing the part for final plating. Activation of both nickel and 405 stainless steel was desired because some of the LRC nozzles contained nickel inserts. Although the nozzle section plated at SLL contained no nickel inserts, the procedures developed do perform equally well with nickel.

Adhesion of the plating to the substrate was quantitatively measured with ring shear and conical head tensile tests. The ring shear tests were performed by plating thick nickel deposits on 12.7 mm diameter rods of the substrate material and then machining several separate rings. The machined samples were then forced through a hardened steel die with a hole larger in diameter than the rod but smaller than the plated rings. The technique, shown in Figure 8, is described in detail in References 3 and 4. A drawing of a conical-head tensile test specimen is shown in Figure 9. In this test, developed by Moeller and Schuler,[5] specimens machined from 76 x 76 mm plated panels are tested in the short transverse direction with the bond normal to the loading direction, whereas in the shear test the bond is parallel to the loading direction.

The ring shear data for nickel-plated 405 stainless steel, presented in Table I, show that the best results were obtained when the sulfamate strike was used anodically and immediately followed by a cathodic treatment (code 5, 428 MN/m^2). Therefore, this treatment was chosen for the nozzle section. Simple cathodic treatment in this solution was also satisfactory but adhesion values were slightly lower (code 4, 373 MN/m^2; code 1, 345 MN/m^2; and code 2, 338 MN/m^2). When no acid pickle was used prior to cathodic treatment at 108 A/m^2, adhesion was noticeably reduced (code 3, 221 MN/m^2). Simple immersion in sulfamic acid solution or anodic treatment in this solution resulted in extremely poor adhesion that was not improved by heating at 220°C (codes 6, 7, 9, and 10, all less than 100 MN/m^2).

*Guardian Metals, Morton Grove, IL

TABLE I

RING SHEAR DATA FOR NICKEL-PLATED 405 STAINLESS STEEL

Code	Cleaning/Activating Cycle[a]	Ring Shear Strength[b] MN/m^2	Ring Shear Strength[b] (psi)
1	Clean, pickle, sulfamate nickel strike at 108 A/m^2 - 5 min, nickel plate[c]	345	50,000
2	Clean, pickle, sulfamate nickel strike at 270 A/m^2 - 5 min, nickel plate	338	49,000
3	Clean, sulfamate nickel strike at 108 A/m^2 - 5 min, nickel plate	221	32,000
4	Clean, sulfamate nickel strike, 270 A/m^2 - 5 min, nickel plate	373	54,000
5	Clean, sulfamate nickel strike anodic at 540 A/m^2 - 1 min, then cathodic at 540 A/m^2 - 5 min, nickel plate	428	62,000
6	Clean, strike in 150 g/l sulfamic acid anodic at 1080 A/m^2 - 3 min, nickel plate	45	6,500
7	Clean, immerse in 150 g/l sulfamic acid for 5 min, nickel plate	90	13,000
8	Same as 5, but heated at 220°C for 16 hours before testing at room temperature	428	62,000
9	Same as 6, but heated at 220°C for 16 hours before testing at room temperature	66	9,500
10	Same as 7, but heated at 220°C for 16 hours before testing at room temperature	83	12,000

[a] Unless otherwise specified, the composition of the sulfamate nickel strike was 80 g/l nickel (as nickel sulfamate) and 150 g/l sulfamic acid, and the temperature was 49°C.

[b] All reported values are the average of at least two tests.

[c] The final nickel plating was done in sulfamate solution of the composition and conditions described in Table III.

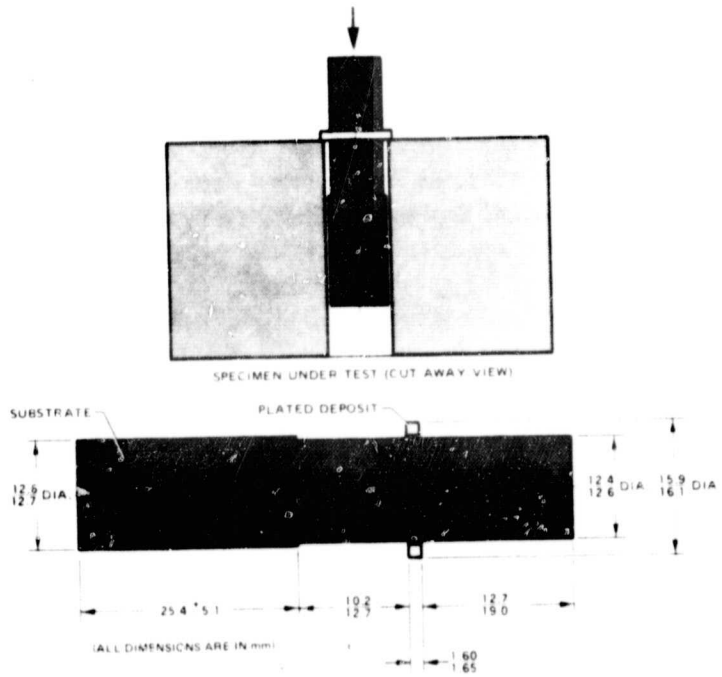


Figure 8. Ring Shear Test Specimen and Die

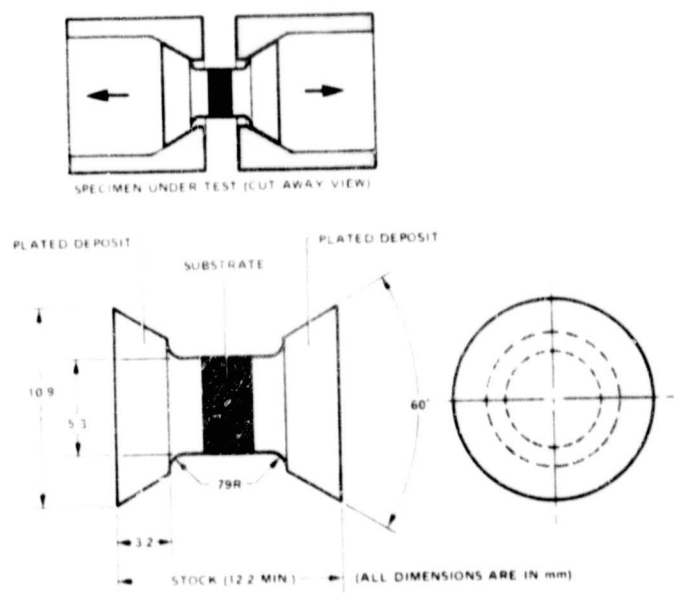


Figure 9. Conical Head Test Specimen

The code 5 cleaning/activating cycle was next evaluated with nickel substrate material. First, stainless steel rods were plated with 0.13 mm of nickel. Approximately 12 hours later, the rods were cleaned, activated, and plated with a thick coating (1.5 mm) of nickel. Ring shear tests on machined specimens showed failure at 386 MN/m^2 (Table II) at the stainless steel-original nickel plate interface. These results revealed that the bond between the two nickel deposits was stronger than the bond between the original nickel deposit and stainless steel, which, incidentally, caused tearing in the stainless steel.

TABLE II

RING SHEAR AND CONICAL HEAD TENSILE DATA FOR NICKEL PLATED ON NICKEL

Cleaning/Activating Cycle	Test Type	Strength (MN/m^2)	Strength (psi)
Clean, sulfamate nickel strike[a] anodic at 540 A/m^2 for 1 min, then cathodic at 540 A/m^2 for 5 min, and then nickel plate	Ring Shear	386	56,000[b]
Same as above	Conical Head	815	118,000[c] (RA = 80%)

[a] The composition of the sulfamate nickel strike was 80 g/l nickel (as nickel sulfamate) and 150 g/l sulfamic acid, and the temperature was 49°C.

[b] Average of three tests.

[c] Average of two tests.

Conical head tensile tests of nickel plated on nickel using the code 5 cleaning/activating cycle resulted in failure in the original nickel deposit at 815 MN/m^2 (Table II). This data, together with a recorded reduction in area of 80%, provided further proof of the suitability of this procedure for plating on nickel.

Final Plating Sequence

The procedures used in the final plating of the nozzle are outlined in Table III. The channels were filled with 0.51 mm thick strips of 5052-H34 aluminum, together with special machined aluminum inserts for the ends

of the channels. The sanding operation (step 2) reduced the depth of the channels an average of about 0.076 mm each time the part was sanded. Therefore, the channels should be cut 0.076 to 0.13 mm deeper than required so that the parts will be within tolerance after sanding. Any obvious holidays due either to the aluminum not being wedged totally in place or to original machining defects were filled with conductive epoxy. The conductive epoxy worked quite well in plugging up these defects so that nickel could be plated over the damaged areas.

TABLE III

PROCEDURES USED FOR CLEANING AND PLATING NOZZLE SECTION

1.	Clean nozzle and aluminum strips
2.	Wedge aluminum strips and inserts in place
3.	Repair holidays with silver conductive epoxy
4.	Sand to smooth the surface
5.	Scrub with Alconox detergent and then with pumice
6.	Rinse
7.	Spray with solution containing 25 g/l sulfamic acid
8.	Rinse
9.	Mount plastic shields in place*
10.	Scrub with Alconox detergent and then with pumice
11.	Rinse
12.	Backfill channels with water
13.	Spray with solution containing 25 g/l sulfamic acid
14.	Nickel strike in a solution containing 450 g/l nickel sulfamate and 150 g/l sulfamic acid, anodic for 2 minutes at 540 A/m^2 and then cathodic for 5 minutes at 540 A/m^2. Temperature of the solution was 50°C.
15.	Transfer directly (no rinsing) to nickel sulfamate solution and plate at 108-162 A/m^2. Composition of this solution was 450 g/l nickel sulfamate and 40 g/l boric acid. The operating conditions were 38 dyne/cm surface tension, 3.8-4.0 pH, and 43-50°C. Sulfur depolarized anodes were used. Total plating time was 3 weeks.

*These shields essentially created a box around the part which extended approximately 25 mm beyond the surface to be plated. The purpose of the shields was to help minimize noduling during plating.

The cleaning and plating portions of the sequence are fairly simple and straightforward; however, a few comments are in order. The nickel strike solution (step 13) was developed specifically for this part. With this solution, adherent electrodeposits can be applied to 405 stainless steel and nickel without attacking any aluminum exposed to the solution. The nickel is deposited nonadherently on the aluminum.

During the early stages of the nickel sulfamate plating operation (step 14) the channels were flushed every 60 minutes with fresh water to keep them free of any plating solution that leaked through pinholes between the aluminum and stainless steel. This flushing was continued for the first 4 hours, after which it was no longer necessary. However, the channels were kept filled with water for the entire duration of the three-week plating cycle. Upon completion of plating, the part was removed from the solution and the aluminum channels were dissolved by circulating caustic solution (Oakite 160)* through the channels. This solution, which was maintained at about 60°C, dissolved all of the aluminum in about 4 hours. This dissolution was confirmed by chemical analysis using atomic absorption analysis. Of the 130 to 137 grams of aluminum used to fill the channels, chemical analysis revealed that 128 grams were dissolved. Because an undetermined amount of material is removed during the sanding operation (step 2, Table I), it was felt that this finding was a very good indication that all aluminum had been removed from the part.

The part was baked for 18 hours at 200°C and then hydrostatically tested at 5 MN/m^2 for 30 minutes at room temperature. No evidence of degradation was noted as a result of this test; therefore the plated nozzle was shipped to LRC for machining to final dimension and further testing. Figure 10 shows the part before shipment to LRC.

Tests on Finished Part

After the part was machined to final dimensions at LRC, the electroformed skin was nondestructively evaluated with an ultrasonic technique, and the channels in the stainless steel were checked with a temperature-sensitive liquid crystal. Following these operations, a stress-indicating coating was applied and the part hydrostatically tested to failure.

Ultrasonic "C" scan data, which, in the opinion of LRC personnel, provides better resolution than holography, indicated a high-strength bond with no significant disbonds. A few small discrete nonbonds, each about 1.5 mm in diameter, were found but none of these were in the area that subsequently failed first during hydrostatic testing. Liquid crystal testing

*Oakite Products, Inc., Berkeley Heights, NJ

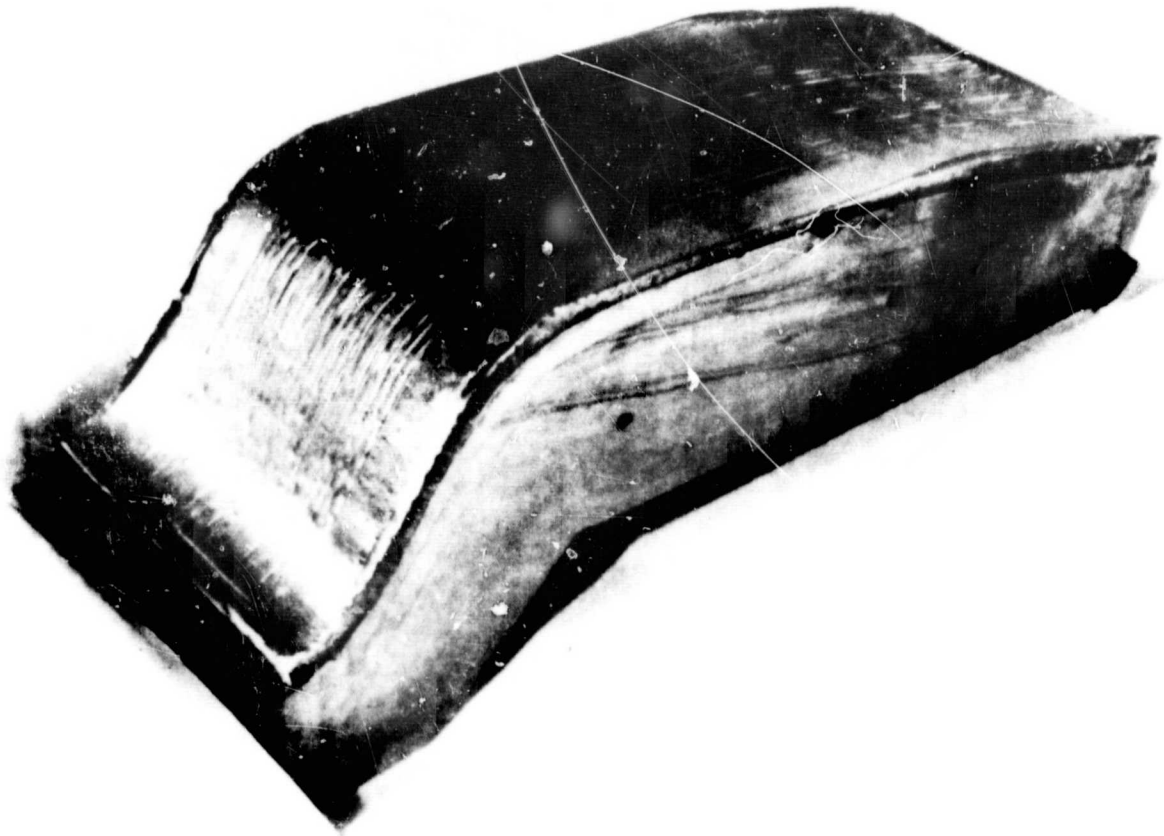


Figure 10. NASA Test Part After Nickel Plating

performed in conjunction with temperature cycling of the part showed that each channel in the stainless steel was free of obstructions. This result was confirmed by visual inspection of the part after the skin was removed.

Prior to filling the part with water and hydrostatically testing it in 0.7 MN/m^2 increments, a stress-indicating coating was applied to the part to aid in observing failure (see Figure 11). At 18.6 MN/m^2 a strain indication was observed in the coating in one section, and at 24.1 MN/m^2 a large section of the electroformed skin became unbonded in this same region (Figure 12). A pressure of 2.8 MN/m^2 was then used to propagate the separation to one edge. The part is shown in Figure 13 with the skin removed. Except for isolated areas of nonbonding, all failure was 0.7 to 1.5 mm deep in the stainless steel. Figure 14 shows the underside of the electroformed nickel skin, confirming that failure occurred in the stainless and not at the nickel or interface between nickel and stainless steel. Operating pressure for the part in actual service will be 3.5 MN/m^2, considerably less than the 24.1 MN/m^2 required for failure.

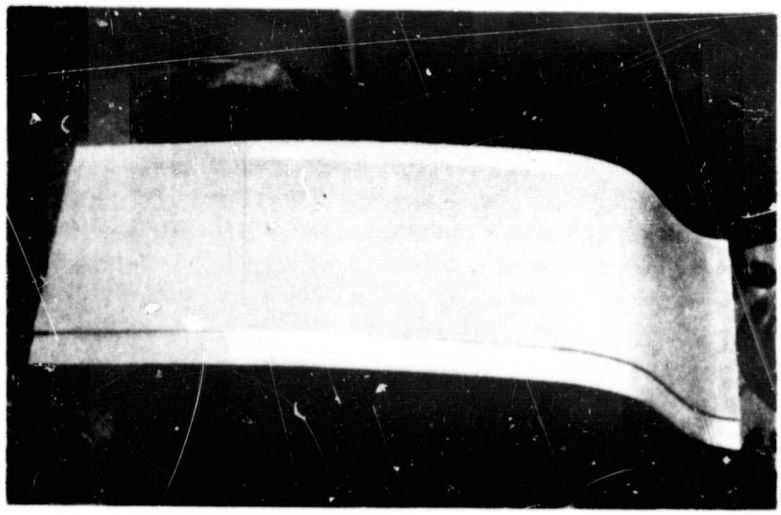


Figure 11. Stress Coat on Nozzle Prior to Hydrostatic Testing

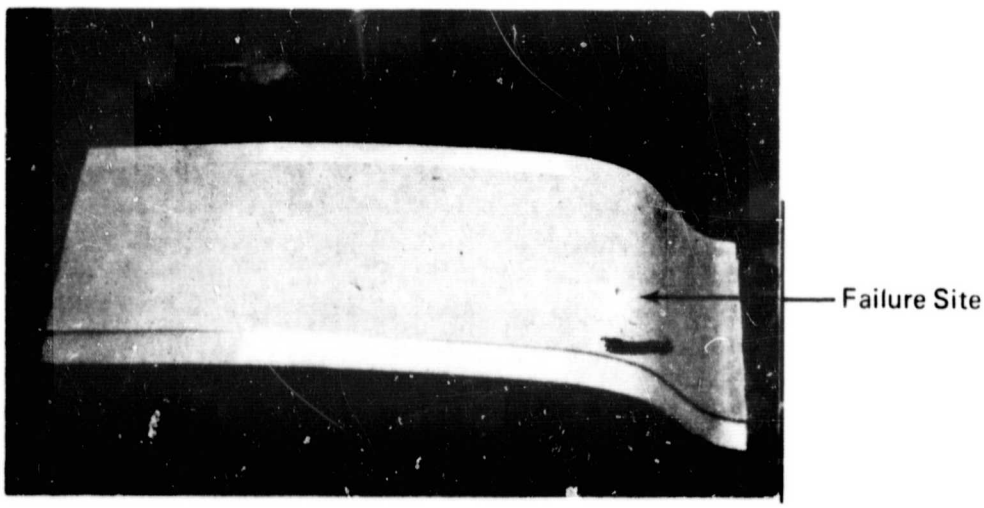


Figure 12. Failure Site at 24.1 MN/m^2

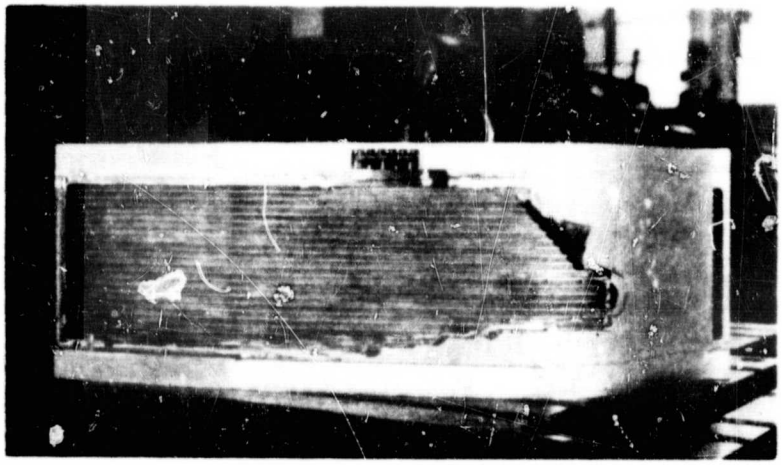


Figure 13. Electroformed Skin Removed After Hydrostatic Testing

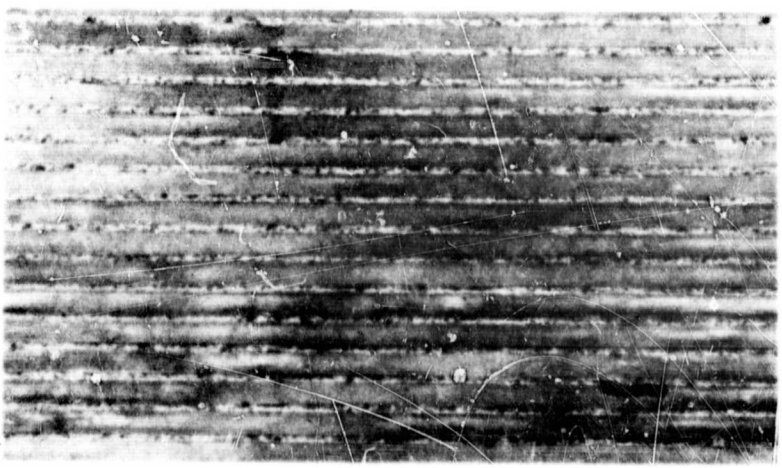


Figure 14. Underside of Electroformed Nickel Skin

Property Data for Nickel Deposits

To determine the properties of the finished nickel, round tensile bars of nickel electrodeposits with a reduced section (19.1 cm long and 0.32 cm diameter) were machined to the specifications outlined in Reference 6 and tested on an Instron machine. These specimens were taken from a flat panel plated at 216 A/m^2 in the same solution used for plating the nozzle section. This plating current density was chosen so that the properties of this panel would closely approximate those of the nickel in the center section of the nozzle, e.g., the area exposed to highest temperature during testing. The resulting data are included in Table IV.

TABLE IV

ROOM TEMPERATURE PROPERTY DATA FOR THICK ELECTROFORMED NICKEL[a]

Specimen	Flat Panel[b]	
Yield Strength (MN/m^2)	517	483
Ultimate Strength (MN/m^2)	631	635
Elongation (%)	--	12.5
Reduction in Area (%)	91.3	91.3

(a) Specimens were round tensile bars with a reduced section 19.1 cm long and 3.2 cm in diameter (see Reference 6).

(b) These samples were plated in the same solution used for the nozzle but on flat plates of aluminum which were subsequently dissolved. Current density was 216 A/m^2, similar to what would be experienced in the midsection of the nozzle (the area where the stainless steel channels are the shallowest).

Section 2 - Influence of Temperature on the Properties of Nickel Used on the Plated Nozzle

Influence of Long-Term Heating on Room Temperature Strength of Plated Bond

I-beam tests, a method for testing substrate-electrodeposit combinations in a tensile fashion, were used to evaluate nickel and nickel-cobalt deposits plated on 405 stainless steel. The nickel-cobalt deposit was included in this program because earlier work showed it to be superior in strength to electrodeposited nickel at both room temperature and after exposure to elevated temperature.[7,8] The samples were prepared so that they would duplicate as closely as possible the plated nozzle section discussed in Section 1 of this report. Parallel grooves 6.35 mm by 6.35 mm deep spaced 1.58 mm apart were machined on one side of 15.2 x 15.2 x 1.3 cm stainless steel plates, and 0.81 mm thick aluminum strips were then wedged into the grooves. The parts were then cleaned and plated using the same procedures described in Table I for the nozzle section. The formulation and operating conditions for the nickel plating solution are included in Table III; those for the nickel-cobalt solution are given in Table V. Three panels were plated with nickel and one with nickel-cobalt. After plating, the parts were cut into the desired specimen shape and the aluminum dissolved. Specimens were then heated at 427 or 538°C and tested at room temperature. Figure 15 shows approximate dimensions for I-beam specimens and Figure 16 some machined I-beam specimens with the grips used for tensile testing. A minimum of 50 I-beam specimens were obtained from each 15.2 x 15.2 x 1.3 cm panel.

The data were very encouraging inasmuch as all samples showed good room temperature strength in spite of long-term exposure at high temperature. Rather than segregate the data according to the individual panels, they are presented together in Table VI because of the similarity of the results. For example, nickel deposits showed no degradation in strength after 1510 hours at 538°C nor did nickel-cobalt deposits after 1770 hours at 538°C. The data from these tests represent essentially a measure of the tensile strength of the 405 stainless steel substrate material. Tested samples failed in the stainless steel as shown in Figure 17 or at the interface between the stainless steel and plating. In practically all specimens, some failure was noted in the stainless steel. The data in Table VI agree well with the handbook value of 414 MN/m^2 for ultimate strength of 405 stainless.[9]

TABLE V

NICKEL-COBALT SOLUTION FORMULATION AND OPERATING CONDITIONS[a]

Nickel (as nickel sulfamate)	77.0 g/l
Cobalt (as cobalt sulfamate)	8.0 g/l
Boric Acid	30.0 g/l
Surface Tension	26-31 dynes/cm
Temperature	49°C
Current Density	270 A/m^2

[a] Composition of the deposits produced in this solution was 40-50% Co. For more details on this solution and the properties obtainable, see References 7 and 8.

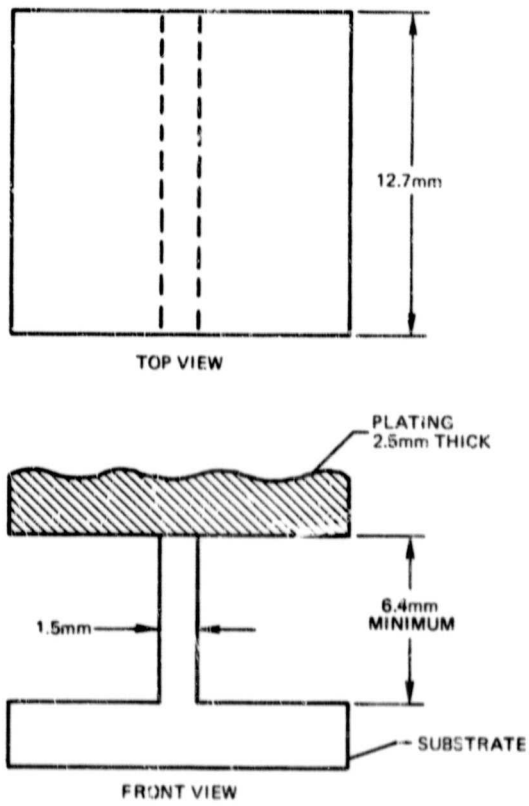


Figure 15. Dimensions of I-Beam Test Specimens

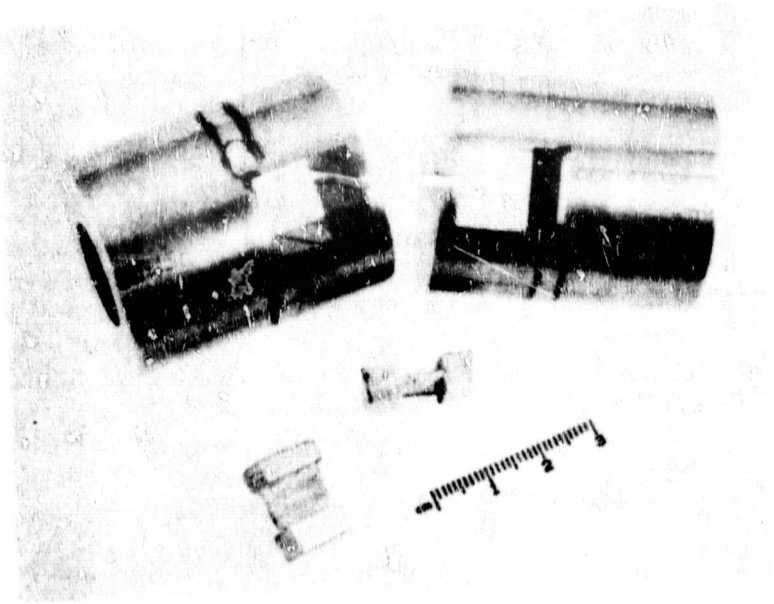


Figure 16. I-Beam Test Specimens and Dies

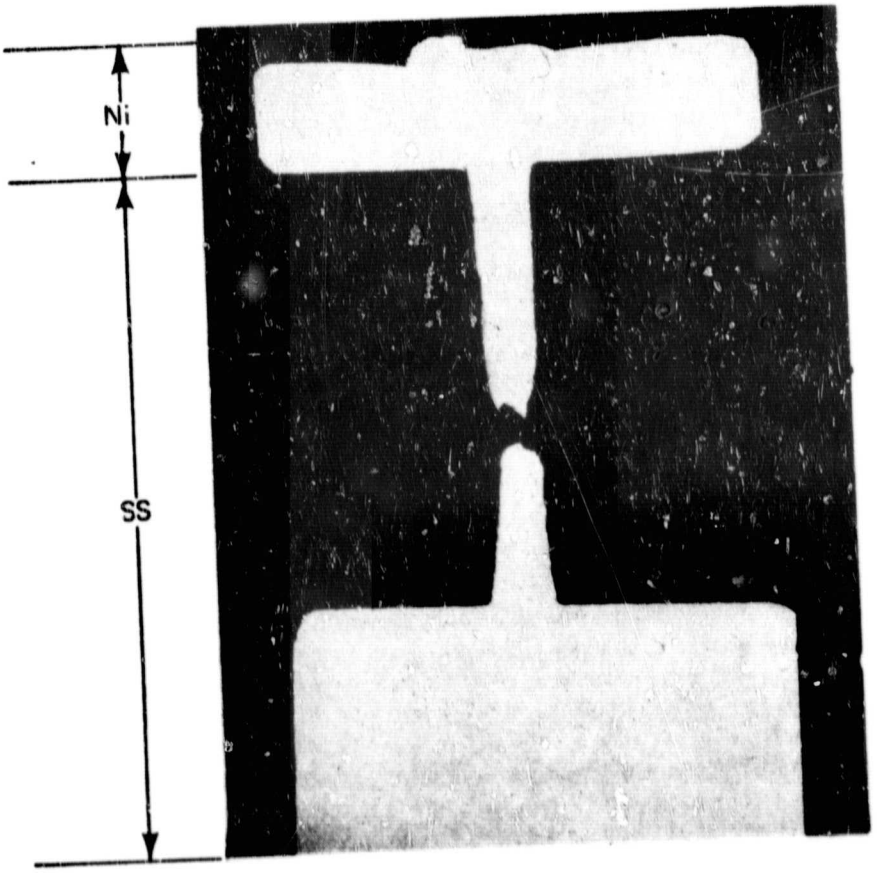


Figure 17. Tested Nickel Plated I-Beam Specimen After 1510 Hours at 1000°F

TABLE VI

INFLUENCE OF HEATING ON THE BOND BETWEEN PLATED NICKEL AND NICKEL-COBALT AND 405 STAINLESS STEEL

Deposit	Time (hours)	Temperature (°C)	Temperature (°F)	I-Beam Tensile Strength[a] (MN/m^2)	I-Beam Tensile Strength[a] (psi)
Nickel	Control		--	414	60,000
"	65	538	1000	380	55,000
"	90	538	1000	452	65,500
"	720	538	1000	452	65,500
"	1510	538	1000	454	65,900
Nickel-Cobalt	Control		--	359	52,000
"	76	427	800	457	66,400
"	145	427	800	408	59,200
"	242	427	800	450	65,200
"	408	427	800	436	63,200
"	72	538	1000	444	64,400
"	190	538	1000	442	64,000
"	270	538	1000	442	64,000
"	1000	538	1000	418	60,700
"	1770	538	1000	452	65,500

[a] All samples were tested at room temperature. Each reported value is the average of three tests.

Influence of Long-Term Heating on Hardness

Hardness of the deposits was also measured at various stages in this program. The data for both nickel and nickel-cobalt, presented in Figure 18 show some drop in hardness as a function of time at 538°C. Of particular interest is the fact that the nickel-cobalt deposits were still harder after 2000 hours at 538°C than nickel in the as-deposited condition.

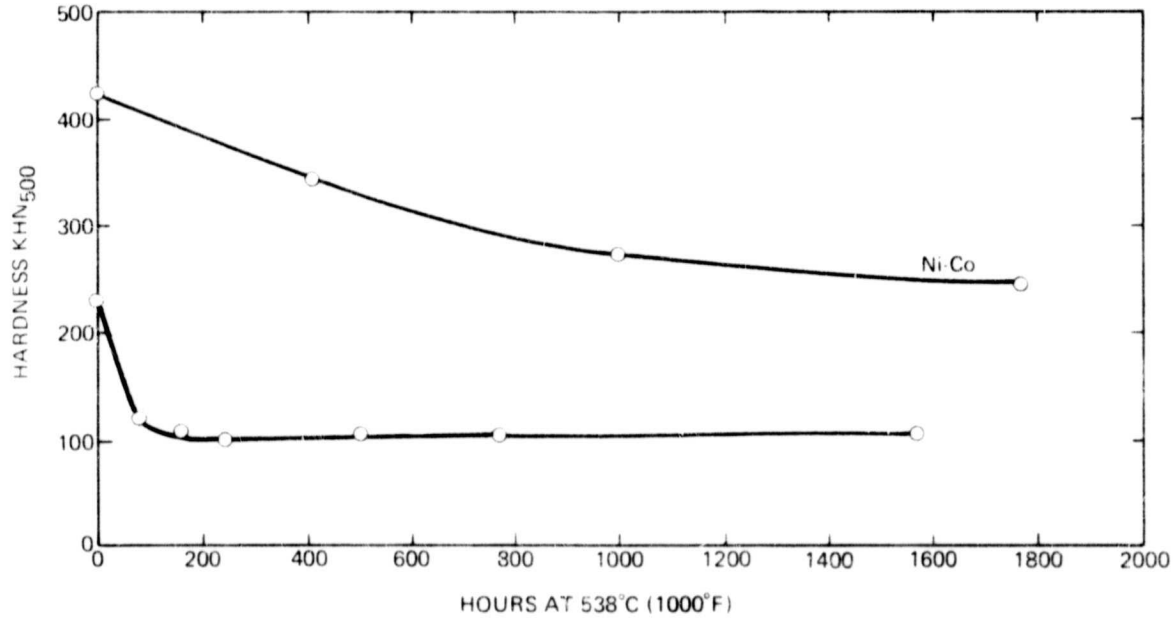


Figure 18. Hardness of Nickel and Nickel Cobalt Alloy at Various Temperatures

Influence of Temperature on Bond Strength

Some I-beam specimens were tested at LRC at temperatures up to 649°C. The samples were resistance heated and pulled within approximately one minute after reaching temperature. Therefore, there was very little dwell time at temperature. Specimens tested at room temperature and 204°C failed in the stainless steel (Table VII). The rest of the samples failed partly in the stainless and partly at the interface between the stainless and nickel plating. However, in all cases the strength was equal to or better than that for 405 stainless steel at temperature. The specimens tested at 538°C and 649°C were considerably stronger than the reported strength for 405 stainless at these temperatures. The conclusion drawn from this work is that the high-temperature bond strength of nickel-plated 405 stainless steel is at least equal to the high-temperature strength of 405 stainless steel.

Influence of Temperature on Ductility

Tensile specimens 1.5 mm thick and approximately 23 cm long, with a 1.27 cm reduced section 10 cm long, were machined from a 30 by 30 cm panel electroformed in the same solution under the same conditions used for the part described earlier in this report. They were tested at temperatures ranging from 25 to 538°C at LRC to determine the influence of temperature on mechanical properties, especially ductility. The time required to reach testing temperature was between 10 and 20 minutes; specimens were tested

TABLE VII

INFLUENCE OF TEMPERATURE ON THE BOND STRENGTH OF NICKEL-PLATED 405 STAINLESS STEEL*

Test Temperature (°C)	(°F)	I-Beam Joint Strength (MN/m^2)	(psi)	Tensile Strength of 405 Stainless Steel (MN/m^2)	(psi)
22	72	433	62,800	443	64,200
204	400	372	53,900	384	55,700
316	600	346	50,200	378	54,900
427	800	308	44,700	320	46,400
538	1000	268	38,900	199	28,800
649	1200	159	23,100	106	15,400

*Allegheny Stainless Type 405 Blue Sheet, Allegheny Ludlum Steel Corp., Pittsburgh, PA

within a few minutes of reaching temperature. One set of specimens was tested in the as-received condition, and another set was baked at 538°C for 6 hours prior to testing. Three samples were included for each test condition.

The data are summarized in Table VIII. The most notable observation is that the ductility properties suffered a drastic reduction between 400 and 500°C. As shown in Figure 19, other researchers have noted a similar trend. By contrast, no noticeable ductility reduction is obtained with annealed 201 nickel (Figure 19). The reasons for the performance differences between electrodeposited nickel and annealed nickel are not known. Harris and Braddick[12] postulated that the presence of grain boundary gas bubbles may in some way be connected with the observed behavior for electrodeposited nickel at elevated temperatures. Earlier work of theirs showed that embrittlement was caused by carbon monoxide gas bubbles that form at grain boundaries during annealing at temperatures in excess of 600°C.[14] By contrast, Kraai and Floreen[15] showed that sulfur exerted a noticeable influence on the ductility of cast nickel in the range of 550 to 600°C. Additional work is needed to determine which impurities are predominant in reducing the high-temperature ductility of electrodeposited nickel.

TABLE VIII

INFLUENCE OF TEMPERATURE ON THE
MECHANICAL PROPERTIES OF NICKEL ELECTRODEPOSITS*

Test Temperature (C°)	25		204		316		427		440**	470**	482**	538	
	A	B	A	B	A	B	A	B	A	A	A	A	B
Tensile Strength (MN/m^2)	531	359	421	290	324	256	231	221	234	172	186	141	148
Yield Strength (MN/m^2)	294	152	283	145	228	138	166	124	186	103	145	107	114
Elongation (%)	24	47	25	43	24	47	38	45	9	26	23	13	12
Reduction in Area (%)	32	38	35	45	40	46	42	44	13.3	27	23	8	7
Modulus of Elasticity ($MN/m^2 \times 10^4$)	166	124	173	152	138	145	97	111	90	124	159	76	89

*Specimens were 1.5 mm thick, approximately 23 cm long with a 1.27 cm reduced section 10 cm long. See Table III, step 15, for plating details. Three specimens per each data point unless otherwise specified.

A Tested in the as-plated condition.

B Tested after heating at 538°C for 6 hours.

**Data for only one specimen.

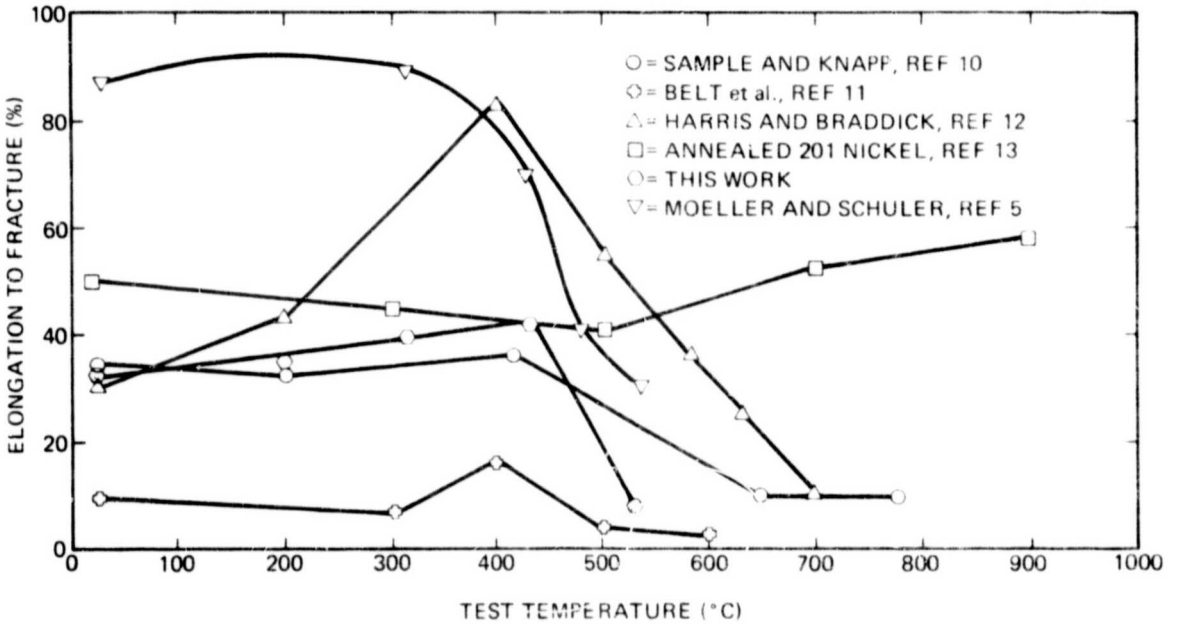


Figure 19. Influence of Temperature on Ductility Properties of 201 Nickel and Nickel Electrodeposits

Some comments should be made on the wide range of ductility values for electrodeposited nickel shown in Figure 19. Moeller and Schuler[5] obtained values of 90% reduction in area at room temperature while all the other researchers, including ourselves, reported values of less than 35% at room temperature. It is believed that this difference is due to specimen geometry rather than material differences. Moeller and Schuler used round conical head specimens which allow for precise determination of RA values. All other data for electrodeposited nickel included in Figure 19 was obtained on flat sheet specimens. Table IV in this report shows that when we used round tensile specimens, RA values were greater than 90%, which is in good agreement with the data of Moeller and Schuler. However, flat specimens prepared in the same solution and under the same operating conditions exhibited RA values of only 32% thereby showing the effect of specimen geometry. The flat specimens were used for the high-temperature studies because they required considerably less thickness, and therefore less plating time. Any future work on high-temperature properties should be done with round tensile specimens.

One last item worthy of note is the observation that although Moeller and Schuler[5] also show a reduction in RA at elevated temperature, they still obtained values of 40% at 500°C. This RA could be adequate for some high-temperature applications.

Section 3 - Nondestructive Evaluation (NDE)

A review of other work on nondestructive evaluation (NDE) of plated thrust chambers revealed that considerable time and effort had been expanded in this area by NASA-Lewis. The Lewis work covered two programs and was sponsored at Bell Aerospace Company.[16-19] It is very concisely summarized in Reference 16, from which the following information was extracted. The first program (NAS3-14376) established ultrasonic "C" scan, holography, and acoustic emission as the most promising NDE methods. These methods were used for testing metal-to-metal bonds involving nickel and nickel alloys in structures simulating regeneratively cooled thrust chamber walls. The objective of the follow on program (NAS3-16800) was to develop standards, determine limitations, and gain operational experience for the nondestructive methods.

For initial inspection, ultrasonic pulse echo was shown to be capable of detecting nonbonds adequately. However, ultrasonics could not distinguish a weak bond from a full strength bond. Holography, using low pressure [0.35 to 3.1 MN/m^2 (50 to 450 psi)] as a stressing means, could identify nonbonds and under the proper conditions of pressure, flaw size, and cover plate thickness give an indication of a weak bond. An advantage of holography over ultrasonics is that a complex three-dimensional part could be examined without special and expensive tooling such as would be required for ultrasonic inspection. Acoustic emission monitoring during pressurization was able to distinguish weak bonds and propagating nonbonds. The combination of holography and acoustic emission could identify weak bonds that could not be distinguished by any other method.

The work done on the two programs was felt to provide a good starting point for developing the NDE methods for inspecting specific hardware. R. A. Duscha and John Kazaroff of NASA-Lewis were quite helpful in providing us with this information.

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