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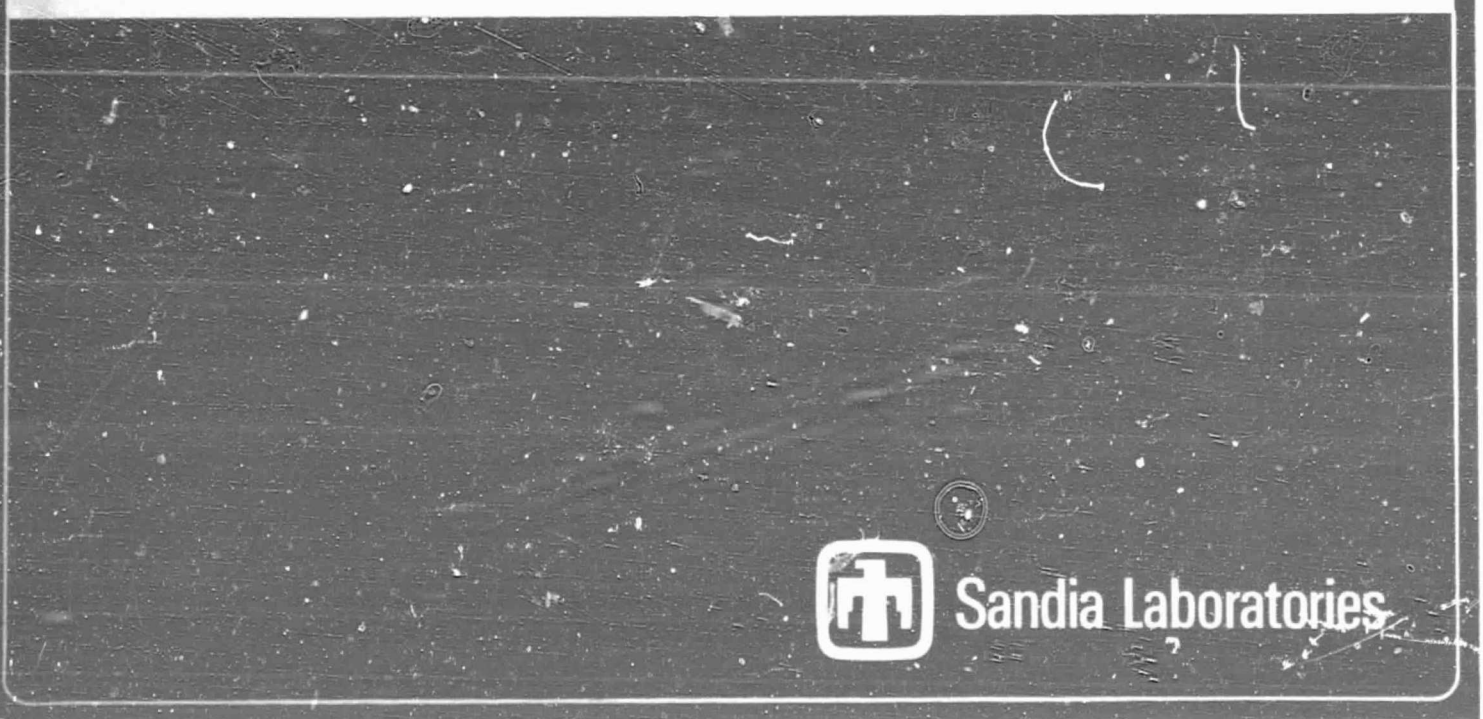
High-Temperature Ductility of Electrodeposited Nickel

(Report for Period Through May 31, 1977)

J. W. Dini, H. R. Johnson



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HIGH-TEMPERATURE DUCTILITY OF ELECTRODEPOSITED NICKEL

(Report for Period Through May 31, 1977)

Sponsored by NASA-Langley Research Center

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ABSTRACT

This report summarizes results of work done during the past several months on high-temperature ductility of electrodeposited nickel. Data are presented which show that earlier measurements made at NASA-Langley erred on the low side, that strain rate has a marked influence on high-temperature ductility, and that codeposition of a small amount of manganese helps to improve high-temperature ductility. Influences of a number of other factors on nickel properties were also investigated. They included plating solution temperature, current density, agitation, and elimination of the wetting agent from the plating solution. Work begun under an earlier contract to repair a large nozzle section by nickel plating is described in detail.

ACKNOWLEDGMENTS

The authors would particularly like to acknowledge the support and help of the following at Sandia: J. R. Helms and D. E. Brown, plating; M. J. Nash, plating and testing; P. J. Royval, testing; W. M. Clift, Auger analysis; and D. Spencer for technical editing. Boyd Wilfong of NASA Langley provided the essential link between our two organizations and was always available for helpful discussions.

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HIGH-TEMPERATURE DUCTILITY OF ELECTRODEPOSITED NICKEL

Introduction

This work is a continuation of earlier programs exploring the use of electroformed nickel on throat nozzle sections for the thermal protective system test facility at NASA Langley Research Center (LRC). In one study, special procedures were developed and then used to plate nickel over channels in a section of a throat nozzle.[1] When tested to failure hydrostatically at LRC, the part failed in the stainless steel substrate rather than at the interface between the plating and substrate.

Data on high-temperature properties had shown that the part could withstand long-term, high-temperature exposure without suffering degradation of the plated bond. However, some additional data revealed a loss of high-temperature ductility of the nickel at temperatures above 400°C. On the basis of this information and other data, some LRC personnel became concerned that perhaps the high-temperature ductility properties of nickel were inadequate for some nozzle applications. Therefore, one of the objectives of the present study was to obtain more information on high-temperature ductility loss in electrodeposited nickel in order to learn how to circumvent the problem or minimize it.

Another objective of the present study is to increase our understanding of the weldability of electrodeposited nickel. Past experience at LRC had shown that sometimes electroformed nickel was weldable, while at other times it was not; and the observation conformed well with industry-wide experience. In general, people who have worked with this material report that it is weldable; however, some have experienced difficulties such as cracking or excessive porosity at one time or another, presumably due to the presence of trace amounts of impurities occluded in the plated deposit. The objective of the study as it concerns weldability was to define the impurities that cause problems and to establish compositional limits.

A third objective of this study was to continue repair work on the nickel plating of the approach section of a 2.44 m (8 foot) HTST combustor. This part had been repaired[2] but had failed in a few places during pressure testing at LRC because of thinness of the repaired section. Therefore, we

decided to apply extra nickel in the form of a patch running the entire interior length of the part in the region where the previously plated nickel was too thin.

The work reported herein concerns objectives I and III, high-temperature ductility properties and nozzle repair. The work on weldability is just beginning and will be reported at a later date.

Revision of RA Data Obtained at LRC

Data for flat nickel specimens plated at SLL but tested at LRC showed very poor reduction-in-area (RA) properties at high temperatures (curve ● of Figure 1).[1] On the other hand, while recent work at SLL with round tensile specimens had shown a somewhat smaller RA above 400°C, the effect was not nearly so severe as that reported for the specimens tested at LRC (curve ● of Figure 1). Therefore, we obtained those specimens from LRC and re-measured them, using a filar eyepiece on a microscope at 10X magnification. The RA values we secured in this manner agree quite well with values for the round specimens (curves ▲ and ● in Figure 1), thus indicating that the original data taken at LRC were too low. Furthermore, recent data from another source (Rocketdyne[3]) also agree very well with data for both sets of samples measured at SLL (curve ■ in Figure 1).

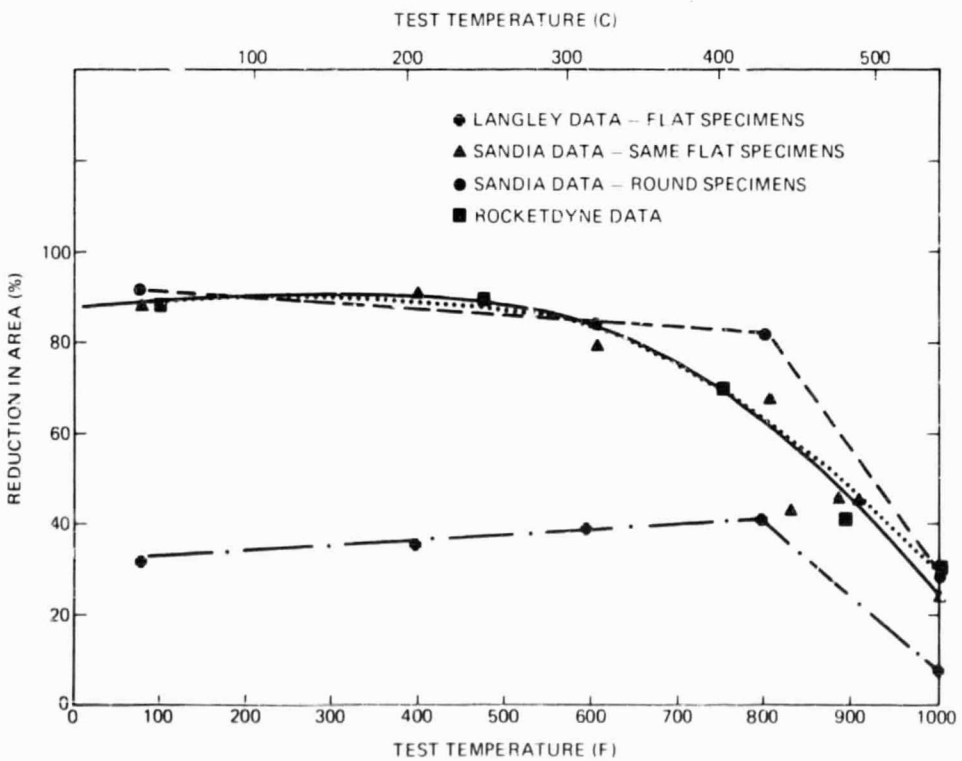

Figure 1. High-Temperature Ductility of Electrodeposited Nickel

Since we have obtained essentially the same values for both flat and round specimens produced at different times and find very close agreement with data obtained from another source, the conclusion is that the larger RA values are realistic. Therefore, the revised data should be used in any calculations directed at determining the suitability of nickel for nozzle applications.

Influence of Strain Rate on High-Temperature Ductility

Recent work at Sandia had shown that strain rate has a strong influence on high-temperature ductility properties of electrodeposited nickel. To examine this effect, we tested round tensile specimens, each with a reduced section 19 mm (0.75 in.) long and 3 mm (0.125 in.) in diameter. They were tested at varying strain rates, both at room temperature and at 538°C. The resulting data, included in Figure 2, show that RA at room temperature was not influenced by strain rate and was consistently around 90 percent. Analysis of the fracture surfaces by scanning electron microscopy (SEM) showed tensile failure to be by dimpled rupture, regardless of strain rate. By contrast, RA at 538°C was low (24 percent) at low strain rates (less than 10^{-1} sec^{-1}) but improved consistently as the rate increased, so that at a rate of 4×10^{-1} sec^{-1}, the high-temperature RA was approximately equal to that obtained at room temperature. Fractographic analysis revealed that, at the slowest strain rate, extensive creep cracking and intergranular failure occurs. As the strain rate was increased, the amount of intergranular fracture was reduced in favor of dimpled rupture, so that at a strain rate of $4 \times 10^{-1} sec^{-1}$, the fracture surface at 538°C was identical in appearance to that at room temperature. This shift in fracture mode with changes in strain rate is a common feature in creep of fine grain size materials. While sulfur at the grain boundaries can enhance creep cracking and thus lower RA, the exact correlation between sulfur diffusion and strain rate remains to be determined. Based on this information, when evaluating nickel for use in potential nozzle applications, NASA personnel should take a very careful look at strain rate that will be seen in the application. There is a good chance that ductility degradation does not occur because of the rate of the heating/cooling cycles involved.

High-Temperature Ductility of Electrodeposited Nickel

The high-temperature ductility loss for electrodeposited nickel discussed in an earlier section is of concern to some LRC personnel. They feel that this property (even with the adjusted higher values mentioned here)

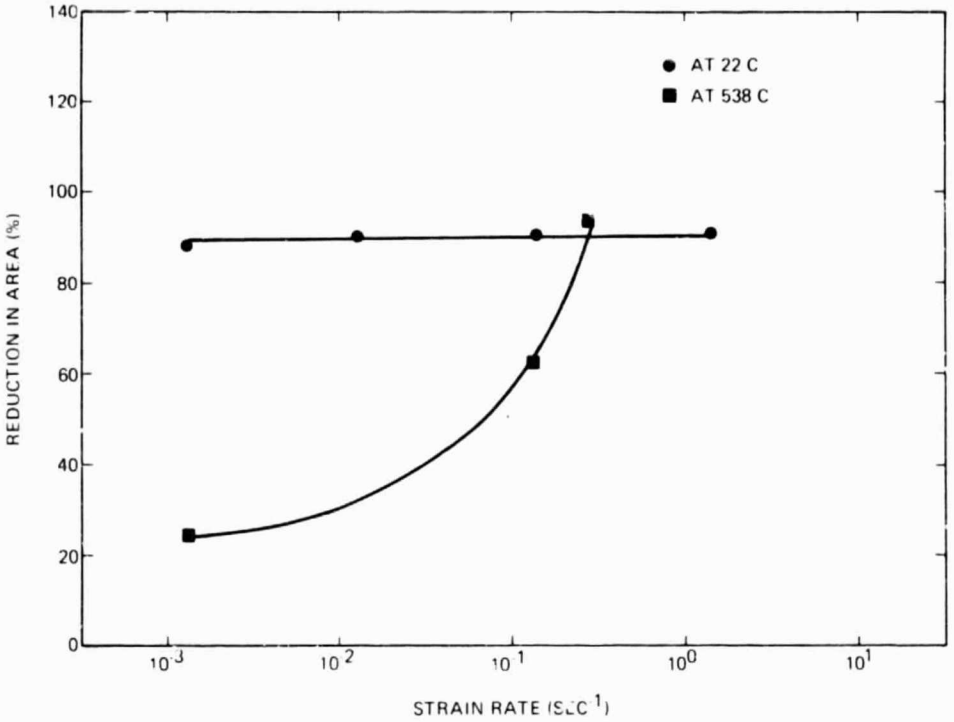

Figure 2. Influence of Strain Rate on the RA of Electrodeposited Nickel at 22 and 538°C

might be inadequate for some nozzle applications. Therefore, we devoted some work to determining the cause of the ductility loss and also to finding ways of minimizing it.

We are now convinced that the major cause is sulfur. Auger electron spectroscopy (AES) experiments were used to compare the mobility of sulfur in electrodeposited nickel and in 201 nickel, both with the compositions shown in Table I, which also includes details on the plating solution. Specimens were placed in a vacuum chamber, which was evacuated prior to ion-bombardment cleaning. They were then heated at various temperatures up to 1000°C for 1 minute, cooled to 20°C, and analyzed. Time to reach temperature in each experiment was about 1 minute.

While sulfur was not evident in the scans of either material not subjected to heating, as the temperature was increased, the sulfur peak became evident in both and started to grow. The influence was much greater for the electrodeposited than for the 201 nickel. Figure 3 includes AES scans for the two materials at 538°C and clearly shows the difference in sulfur peaks at this temperature for both. The data of all scans summarized in Table II reveal that the mobility of sulfur in electrodeposited nickel is about an order of magnitude greater than that in 201 nickel. For the electrodeposited material, there was some indication that the sulfur is most active around 550°C, since a smaller peak occurred at 1000°C. This observation

TABLE I

COMPOSITION OF 201 NICKEL AND ELECTRODEPOSITED SULFAMATE NICKEL

Element	201 Nickel (ppm)	Electrodeposited Nickel[1] (ppm)
Copper	250 max	<100
Iron	400 max	<100
Manganese	3500 max	< 5
Silicon	3500 max	< 10
Carbon	93	50
Cobalt	4700	1000
Hydrogen	2	8
Oxygen	17	20
Nitrogen	6	6
Sulfur	12	10

[1] Composition of the nickel sulfamate plating solution was 80 g/l nickel (as nickel sulfamate), <1.0 g/l nickel chloride, and 40 g/l boric acid. Wetting agent was used to reduce the surface tension to 35-40 dynes/cm. Current density was 268 A/m^2; pH, 3.8; and temperature, 49°C. Anodes were sulfur depolarized nickel.

is consistent with that of Kraai and Floreen,[4] who showed that the maximum influence of sulfur on ductility of high-purity cast nickel was encountered in the range of 550 to 650°C. The effect may be traceable to the fact that at 643°C nickel and sulfur form a eutectic, Ni-Ni_3S_2; and in this temperature range, the mobility of sulfur at nickel grain boundaries is sufficiently rapid so that sulfur wets new interfaces as rapidly as they are formed by deformation.

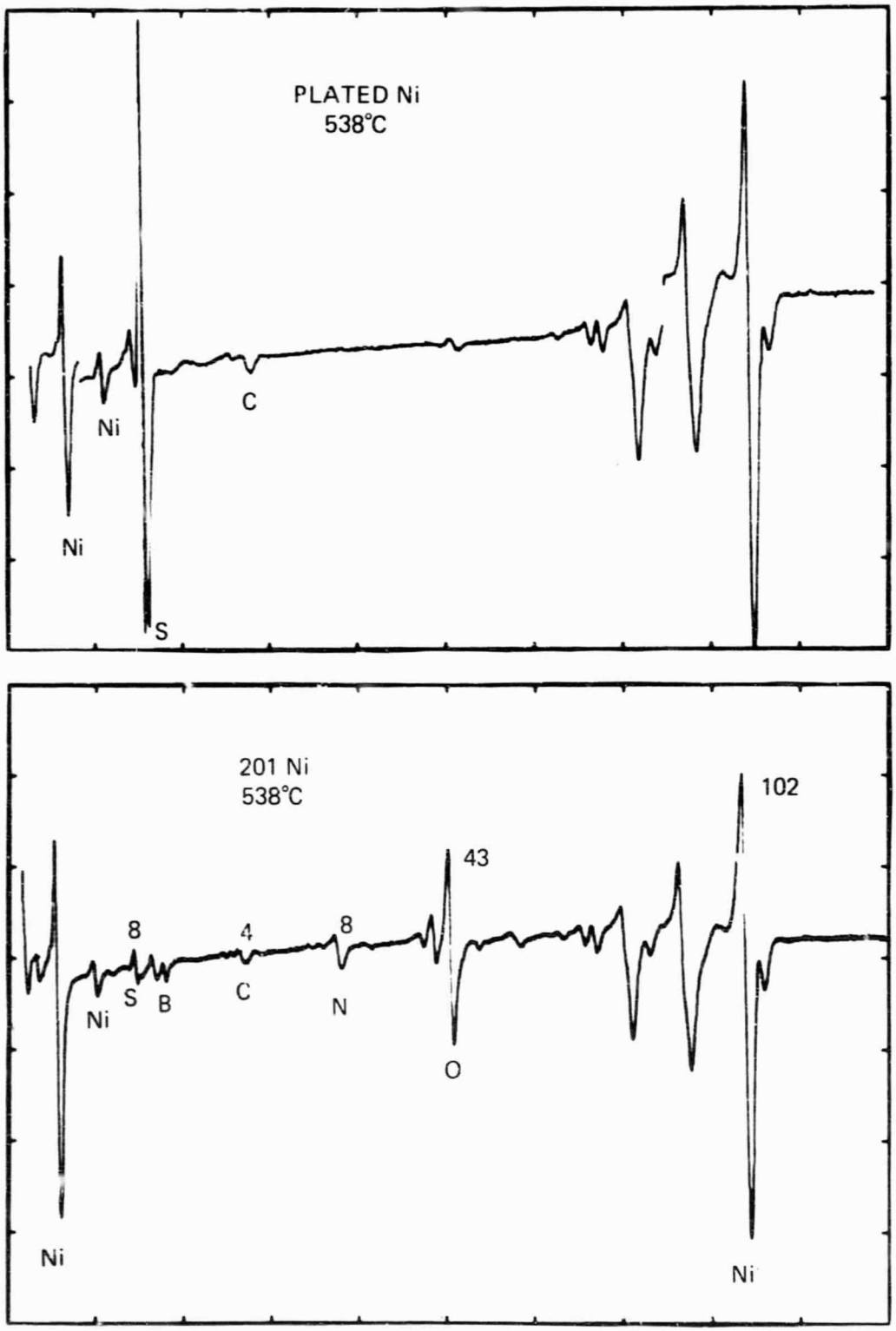

Figure 3. Auger Electron Spectroscopy Spectrum for 201 Nickel and Electrodeposited Nickel After Heating for One Minute at 538°C

TABLE II

TENSILE PROPERTIES OF ELECTRODEPOSITED Ni-0.5 Mn ALLOY
(From Stephenson, Reference 6)

Test Temp (°C)	Yield Strength		Tensile Strength[a]		Elongation (%)
	MN/m^2	psi	MN/m^2	psi	
22	1003	149 000	1240	179 000	5.4
93	990	143 000	1140	165 000	4.0
204	760	110 000	905	131 000	5.6
315	495	71 900	685	99 300	18.0
390	280	40 400	390	55 800	25.8

[a] 0.2 percent offset

The reason for the better performance of 201 nickel, in spite of the fact that it contained a little more sulfur than did the electrodeposited nickel (12 versus 10 ppm), is the large amount of manganese present in this alloy (3500 ppm compared to less than 5 ppm for electrodeposited nickel). Bieber and Decker[5] report that Mn, Mg, and Ca are strong metallic desulfurizers and earlier work of Kraai and Floreen[4] has shown that with as little as 230 ppm of Mg, no high-temperature ductility problems are encountered with cast nickel. Stephenson[6] has codeposited as much as 5000 ppm Mn with nickel and has obtained favorable ductility properties at temperatures as high as 400°C (Table II).

Some researchers have blamed the high-temperature ductility losses in nickel on the presence of grain boundary gas bubbles. Harris and Braddick[7] postulated that embrittlement is caused by carbon monoxide gas bubbles that form at grain boundaries during annealing at temperatures in excess of 600°C.

On the basis of findings and speculations such as the foregoing, we investigated the following areas:

1. Influence of codepositing manganese
2. Influence of plating solution temperature
3. Influence of agitation
4. Influence of current density
5. Influence of omitting a wetting agent

Codeposition of Manganese

To examine the effects of codepositing manganese, we used a 40-litre nickel sulfamate solution and added Mn in amounts varying from 1 to 5 g/l as manganese sulfamate. (See Table I for the compositions and operating conditions.) Round tensile specimens, each with a reduced section 2 mm (80 mils) in diameter and 20 mm (0.80 in.) long were machined from thick plated panels. The specimens were tested at 22 and 538°C, some in the as-deposited condition, others after heating at 538°C for one day, and a third set after heating at 538°C for one week. These latter sets were used in an attempt to determine if long-term use of nickel in nozzle applications might result in unforeseen degradation.

The temperature tests were performed by mounting the specimens in special jaws inside a sealed quartz tube of 2.9 cm (1-1/8 in.) diameter. Figure 4 shows a specimen and the jaws, and Figure 5, the test apparatus. A shielded chromel-alumel thermocouple was spot-welded to the center of the specimen. The quartz tube assembly was evacuated to a pressure of approximately 10 μm and the chamber was then purged with argon during the test and for at least 15 minutes before the tensile bar was heated. Heating was done with a Variac-controlled Dual Elliptical Radient Heater.* It took approximately 30 minutes to heat a specimen to the test temperature, and once at temperature, it was held for approximately 10 minutes before being tested.

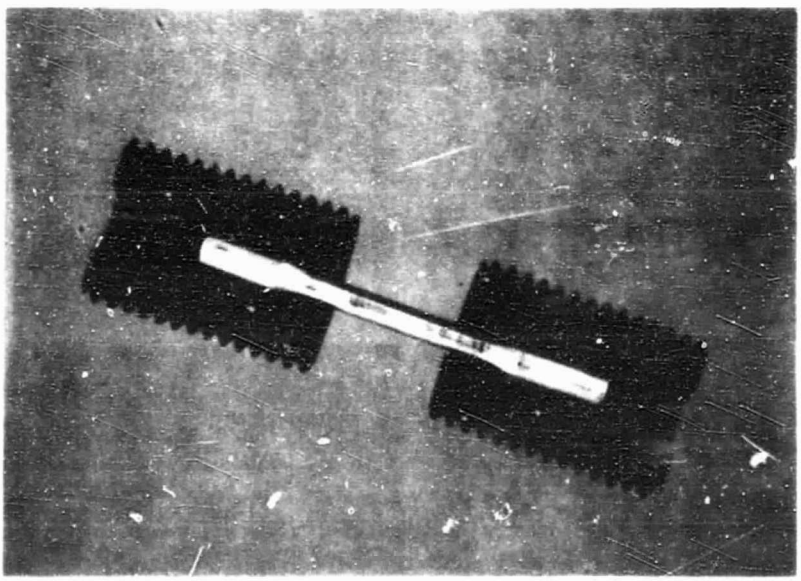

Figure 4. Nickel Specimen With Tensile Test Jaws

*Research Incorporated, Minneapolis, Minn.

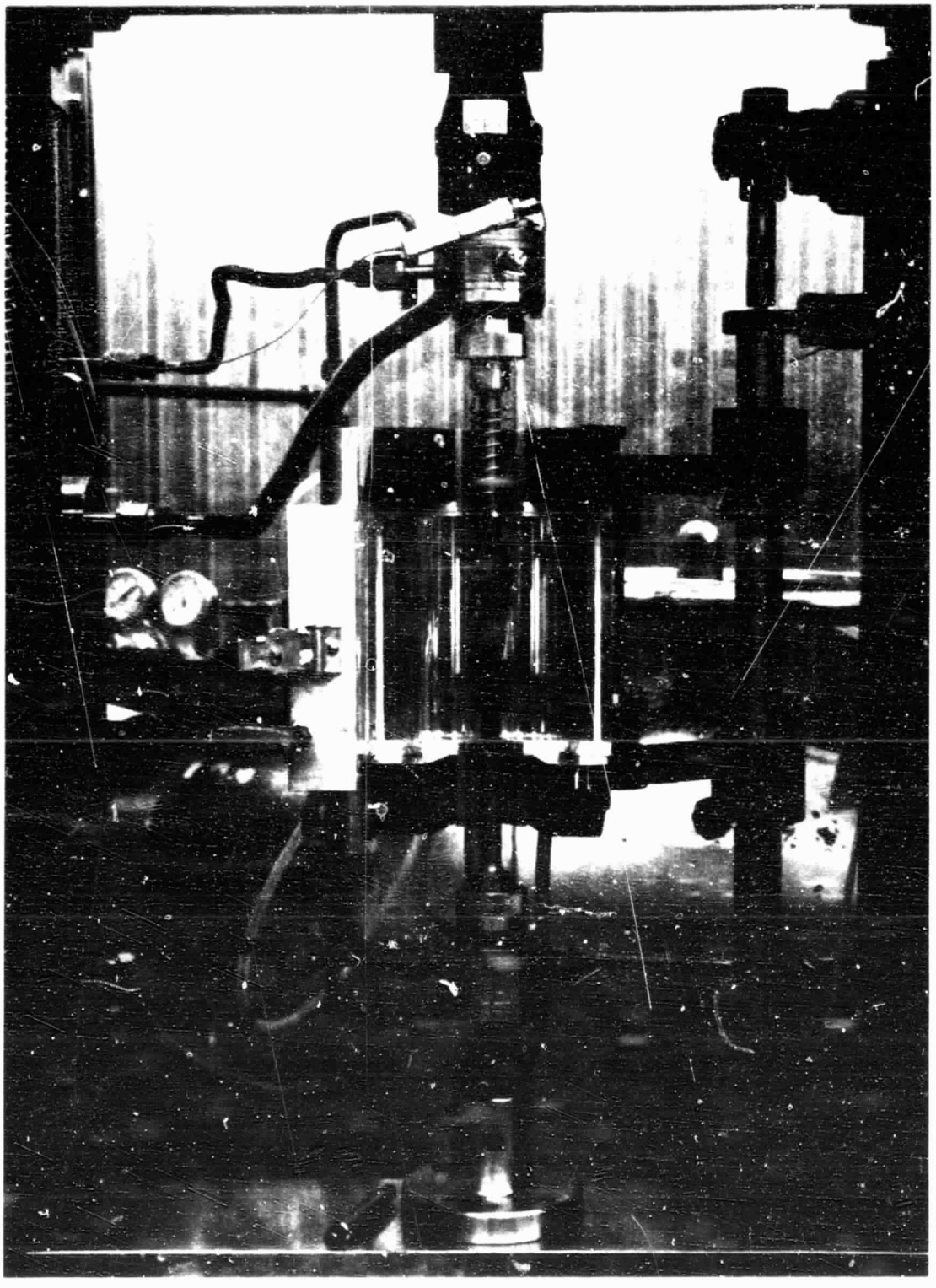

Figure 5. Apparatus for High-Temperature Testing of Nickel Deposits

The ductility results are summarized in Table III and yield and tensile strengths in Table IV. For the deposits containing no manganese, the typical decrease in RA values mentioned earlier occurred at 538°C. Heating these deposits for up to one week at 538°C before testing improved the room-temperature elongation and RA but did not influence these properties at 538°C. Once again, RA values at 22°C were in the high 80 percent range, while at 538°C they were around 24 percent.

Tensile properties of 538°C were considerably improved by codepositing manganese. Elongation and RA values were considerably higher than for specimens containing no manganese; quite often, the improvement was close to 100 percent. For example, after being heated for one day at 538°C, the deposits with manganese showed RA values of 43 and 38 percent at 538°C, whereas the RA for companion specimens with no manganese was 24 percent. A similar trend was noted for specimens heated at 538°C for one week prior to testing.

In the data, there are some inconsistencies which cannot be explained at this time. The 700 ppm Mn deposit exhibited an 83 percent RA at 538°C in the as-deposited condition. We do not understand why this value was so unusually high, especially when compared with the other data in Table III. Also, the data for deposits containing 1800 ppm Mn were not significantly better than those for deposits containing 700 ppm Mn, a result which may indicate that 700 ppm Mn is all that is needed to tie up the sulfur. It has been reported[8] that a Mn/S ratio of 25:1 is adequate to tie up sulfur in nickel; and since the sulfur content of the nickel of this experiment was ~10 ppm, 250 ppm of Mn should be adequate to account for our obtaining good results with 700 ppm.

Though adding 700 ppm Mn affected neither yield nor tensile strengths, adding 1800 ppm Mn increased both at high-temperatures.

Influence of Plating Solution Temperature

Bruch[9] reports that plating at lower-than-normal operating temperatures (27 versus 43°C or higher) resulted in material with higher room temperature ductility both in the as-deposited condition and after heating at 800 and 1000°C. He speculated that the cause of the embrittlement may have been hydrogen codeposition, which in turn was minimized by the reduced temperature of the plating solution.

Deposits were produced in two nickel sulfamate solutions. The volume of one solution, a formulation which had been used for general-purpose work for many years, was 95 litres (25 gallons), while the other solution, specifically formulated for NASA work, was 950 litres (225 gallons) in volume.

TABLE III

DUCTILITY PROPERTIES FOR Ni-Mn DEPOSITS [1]

System	Test Temperature (°C)	As-Deposited		538°C - 1 Day		538°C - 1 Week	
		Elong (%)	RA (%)	Elong (%)	RA (%)	Elong (%)	RA (%)
No Mn	22	21.1	76.7	33.7	88.1	41.1	88.4
	538	22.0	29.6	17.1	23.8	19.3	25.0
700 ppm Mn	22	15.0	65.0	39.3	94.1	49.8	92.1
	538	39.0	82.9	32.0	43.4	35.5	40.1
1800 ppm Mn	22	12.2	47.8	21.4	92.4	24.8	91.1
	538	22.0	47.6	26.1	37.8	34.6	47.3

[1] Round tensile specimens with a reduced section 2 mm (80 mils) in diameter and 20 mm (0.80 in.) long. Average of 2 specimens.

TABLE IV

TENSILE AND YIELD STRENGTH DATA FOR Ni-Mn DEPOSITS

System	Test Temperature (°C)	As-Deposited				538°C - 1 Day				538°C - 1 Week			
		Yield Strength (MN/m^2)	(psi)	Tensile Strength (MN/m^2)	(psi)	Yield Strength (MN/m^2)	(psi)	Tensile Strength (MN/m^2)	(psi)	Yield Strength (MN/m^2)	(psi)	Tensile Strength (MN/m^2)	(psi)
No Mn	22	566	82 100	790	114 500	280	40 500	503	82 900	297	43 100	535	77 500
	538	242	35 100	283	41 000	143	20 700	178	25 800	165	23 900	182	25 400
700 ppm Mn	22	714	103 600	856	124 300	224	32 400	447	64 700	212	30 800	448	64 900
	538	166	24 000	228	33 100	126	18 200	171	24 800	112	16 200	165	23 900
1800 ppm Mn	22	577	83 700	779	112 900	506	73 300	646	93 700	448	64 900	610	88 400
	538	177	25 700	245	35 500	200	29 000	249	36 100	222	32 200	243	35 200

[1] Round tensile specimens with a reduced section 2 mm (80 mils) in diameter and 20 mm (0.8 in.) long. Average of 2 specimens.

Results (Table V) clearly show that regardless of the plating solution, much higher gas content was occluded in the deposit at the lower temperature. Deposits were also much more highly stressed when plating was done at low temperature. For example, at a current density of 269 A/m^2 (25 A/ft^2) and a temperature of 27°C, deposit stress was 145 MN/m^2 (21 000 psi) compared to 17.2 MN/m^2 (2500 psi) at 49°C. Although none of these deposits was heated and checked for ductility properties at high temperature, we feel that there is no reason to expect the lower-temperature deposits to be lower in gas content after such an operation. Furthermore, because of the stresses created in deposits produced at lower temperatures, the likelihood of obtaining suitable deposits of the thicknesses required for nozzles would be remote because of the increased potential for cracking and warpage during deposition.

TABLE V

INFLUENCE OF PLATING TEMPERATURE AND CURRENT DENSITY ON GAS CONTENT OF DEPOSITS

Solution Volume	Current Density		Plating Temperature					
			27°C (80°F)			60°C (140°F)		
			Gas Content (ppm)					
	A/m^2	A/ft^2	H	O	N	H	O	N
Small (95 litres)	269	25[a]	115	105	47	8	37	5
	538	50[b]	175	139	97	4	11	5
Large (850 litres)	269	25[a]	25	49	12	10	50	3
	538	50[b]	72	48	31	3	16	1

[a]Deposit thickness was 0.2 mm (8 mils).

[b]Deposit thickness was 0.4 mm (16 mils).

Influence of Agitation

Ultrasonic agitation was evaluated as a means of reducing gas content of nickel deposits. Panels were plated in the same solution, some with ultrasonic agitation and others without, then analyzed for gas content. The data presented in Table VI show that no beneficial effects were derived from using the ultrasonic agitation.

TABLE VI

INFLUENCE OF ULTRASONIC AGITATION ON GAS CONTENT[a]

Condition	Gas Content (ppm)			
	C	H	O	N
No Ultrasonic Agitation	20	3	35	2
Ultrasonic Agitation	35	5	34	9

[a]The nickel sulfamate solution used for this work was 95 litres (25 gallons); current density was 215 A/m^2 (20 A/ft^2); and temperature, 54°C (130°F). The solution contained no wetting agent.

Influence of Current Density

Some data on the influence of current density (presented in Table V) show that at low plating temperatures (27°C) gas content was higher at the higher current density (538 versus 239 A/m^2), while at 60°C the situation was reversed. At this higher temperature, however, the gas content was within an acceptable range for deposits produced at either current density. Therefore, the conclusion drawn from the data in Table V is that the current density of around 269 A/m^2, which has been used for past nozzle work, appears best for minimizing gas content.

Additional data concerning the influence of current density on impurity content (Table VII) show that low current density results in noticeably higher sulfur content. For example, at 54 A/m^2 sulfur content was 30 ppm, whereas at the current densities more commonly used, sulfur was less than 10 ppm. Moreover, it has been our experience that the low-current-density deposits are the more highly stressed. In general, this information leads to the conclusion that current densities in the range of 250 to 350 A/m^2 are best for producing high-purity, low-stress nickel.

Plating Without a Wetting Agent

One means of improving the purity of electrodeposited nickel was considered to be the elimination of the wetting agent from the plating solution. Without sodium lauryl sulfate, the material typically used in sulfamate nickel plating solutions to reduce surface tension, there is one less material involved in the electrochemical processes which occur in solution. Malone,[10] of Bell Aerospace, reports good, consistent properties for nickel

TABLE VII

INFLUENCE OF CURRENT DENSITY ON IMPURITY CONTENT

Current Density		Impurities (ppm)				
A/m^2	A/ft^2	C	H	O	N	S
54	5	70	10	44	8	30
323	30	80	3	28	8	8
538	50	60	4	32	8	6

electroforming solutions operated without wetting agents; thus he shows that the approach is possible. Of course, eliminating the wetting agent, which is used to reduce surface tension, requires that an alternative means such as vigorous mechanical agitation be used if pitting is to be avoided.

A 100 litre solution with the same constituents and the same operating conditions as those given in Table I, but with wetting agent omitted, was used to obtain some properties and other data. Analysis of the data included in Table VIII shows that deposits plated in this solution contained less C and H than did deposits plated in solutions containing wetting agents. However, the most important data are shown in Table IX. In spite of reductions in the amounts of some impurities present in nickel produced in the solution containing no wetting agent, the high-temperature ductility properties were no better than those for nickel produced in solutions containing a wetting agent. Even without the agent, enough sulfur is included in the deposit to degrade its high-temperature ductility. This information, coupled with the fact that operation without wetting agent is difficult, and particularly so with complex shapes, leads to the obvious conclusion that this is not the step to take.

Repair of NASA Nozzle

Previously, NASA Langley Research Center (LRC) contracted with SLL to repair a portion of the approach section of the LRC 2.44 m (8 ft) HTST combustor nozzle by nickel plating.[2] The necessary procedures were developed and successfully demonstrated on small parts before we began repairing the large part. After plating, the repaired part was hydrostatically pressure tested to 500 psi before shipping to LRC for final machining and testing. After machining was completed at LRC, however, pressure tests revealed leaks in a few areas along the repaired joint. Attempts at LRC to repair these areas by electron beam welding were unsuccessful.

TABLE VIII

ANALYSIS OF DEPOSITS PRODUCED IN SOLUTION CONTAINING NO WETTING AGENT[a]

Code	Impurity Content (ppm)				
	C	H	O	N	S
2	30	3	13	3	40
4	36	3	32	2	-
5	26	1	33	3	12
8	30	2	20	2	15
10	-	2	32	4	9
With wetting agent	40-70	5-13	10-40	<10	<20

[a]See footnote of Table I for formulation and operating conditions.

TABLE IX

PROPERTY DATA FOR DEPOSITS PRODUCED IN SOLUTION CONTAINING NO WETTING AGENT[a]

Test Temperature	Yield Strength		Tensile Strength		Elongation	RA
(°C)	(MN/m^2)	(psi)	(MN/m^2)	(psi)	(%)	(%)
22	487	70 700	608	88 200	20.4	84.4
538	203	29 400	234	33 900	11.4	34.4

[a]This was sample Code 10 from Table VIII.

In approaching this second repair task, we were concerned not only with what could be done to achieve that end but also with determining the cause of the difficulty.

Cause of the Leaks

To explain the probable cause of the leaks it is necessary that we review briefly the reason for SLL involvement in the repair and the procedure used.

The A-216 steel combustor nozzle is a multi-channeled heat exchanger on which electroplated nickel was used as the closure material over the channels. The vendor who plated the part for LRC used a conductive wax to completely fill the channels and then plated over the top of the conductive wax. Unfortunately, after plating, the wax could not be completely removed; so two incisions 13 mm wide were made 180 degrees apart in the nickel skin (Figure 6) so that the wax could be removed by running a reamer on a flexible shaft through the channels. After the wax had been removed, the part was sent to SLL for repair. Here, the repair work was accomplished by wedging aluminum strips into the incisions in the channels; filling any voids between the steel, nickel, and aluminum with a silver conductive epoxy; plating with nickel to the desired thickness; and dissolving the aluminum strips. As furnished, the nickel skin thickness was ~1.5 mm (0.060 in.). Figure 7 shows the stages in the foregoing process.

To learn that the part had not passed the LRC pressure test because of some isolated failures at the nickel-to-nickel interface was indeed surprising, since ring shear specimens plated both before and after the NASA part in the same solution had shown excellent adhesion of nickel to nickel.

A review of the photographs taken of the incisions in the nickel skin, both as-received and after installation of the aluminum strips, has led us to believe that the leaks resulted from a thinning of the nickel in those areas during machining. An examination of both Figures 6 and 8 shows how unequal in length and shape the holes in the nickel skin were, whereas the machining operation should have left them approximately equal in length and in overall size. The only plausible cause for the irregular shape of the holes is the reaming operation performed at LRC. Figure 9 is a 3X magnification of a typical area where we believe the reaming operation reduced the thickness of the nickel enough to structurally weaken the repaired area. On the basis of the two complete holes (middle holes in Figure 10), we estimate the maximum amount of nickel in the minimum-thickness area (right side of holes) after the plating and machining operation to be between 0 and 0.2 mm (8 mils). It is not unreasonable to expect the minimally plated bond area to fail.

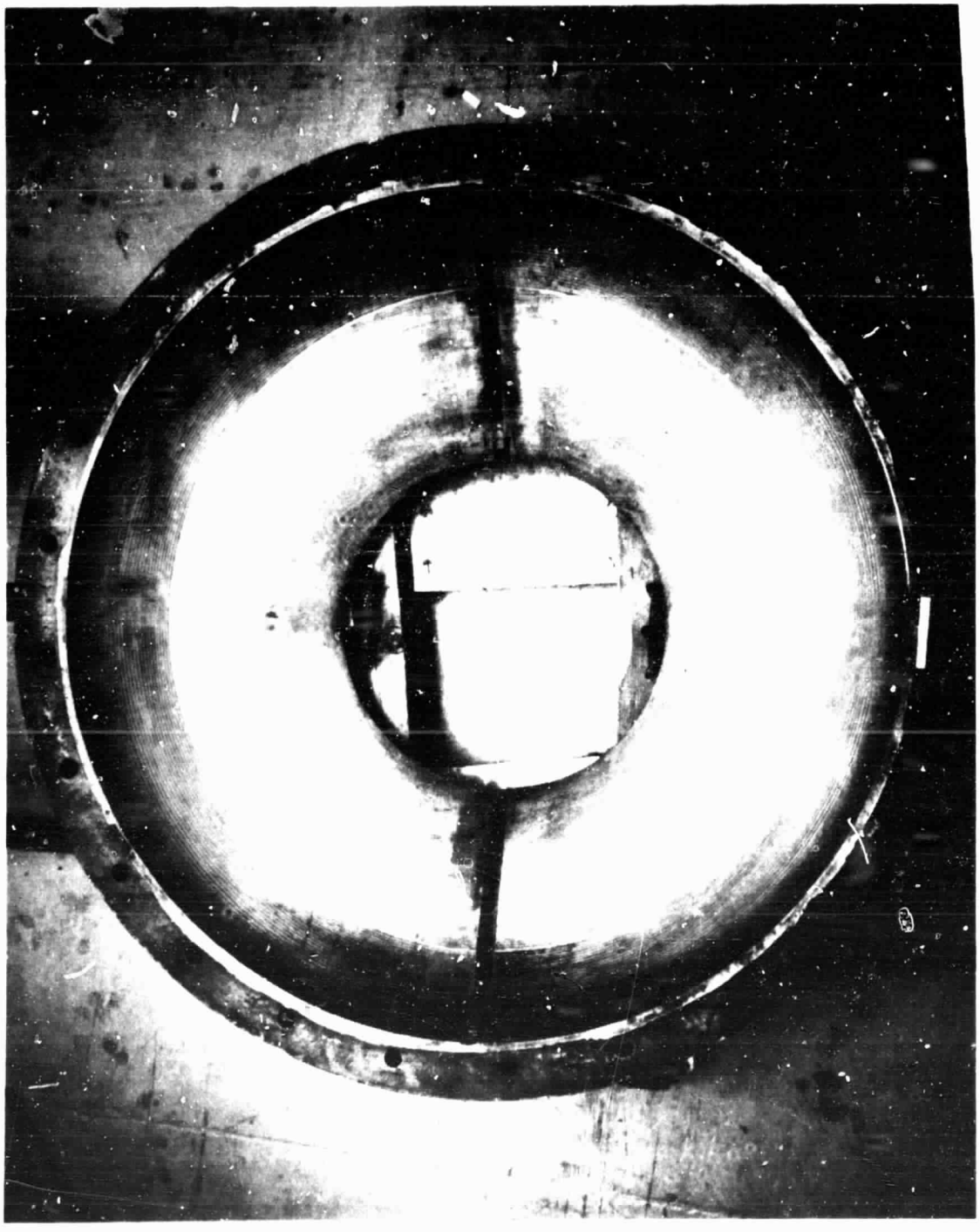

Figure 6. Combustion Nozzle With Incisions in Nickel Skin

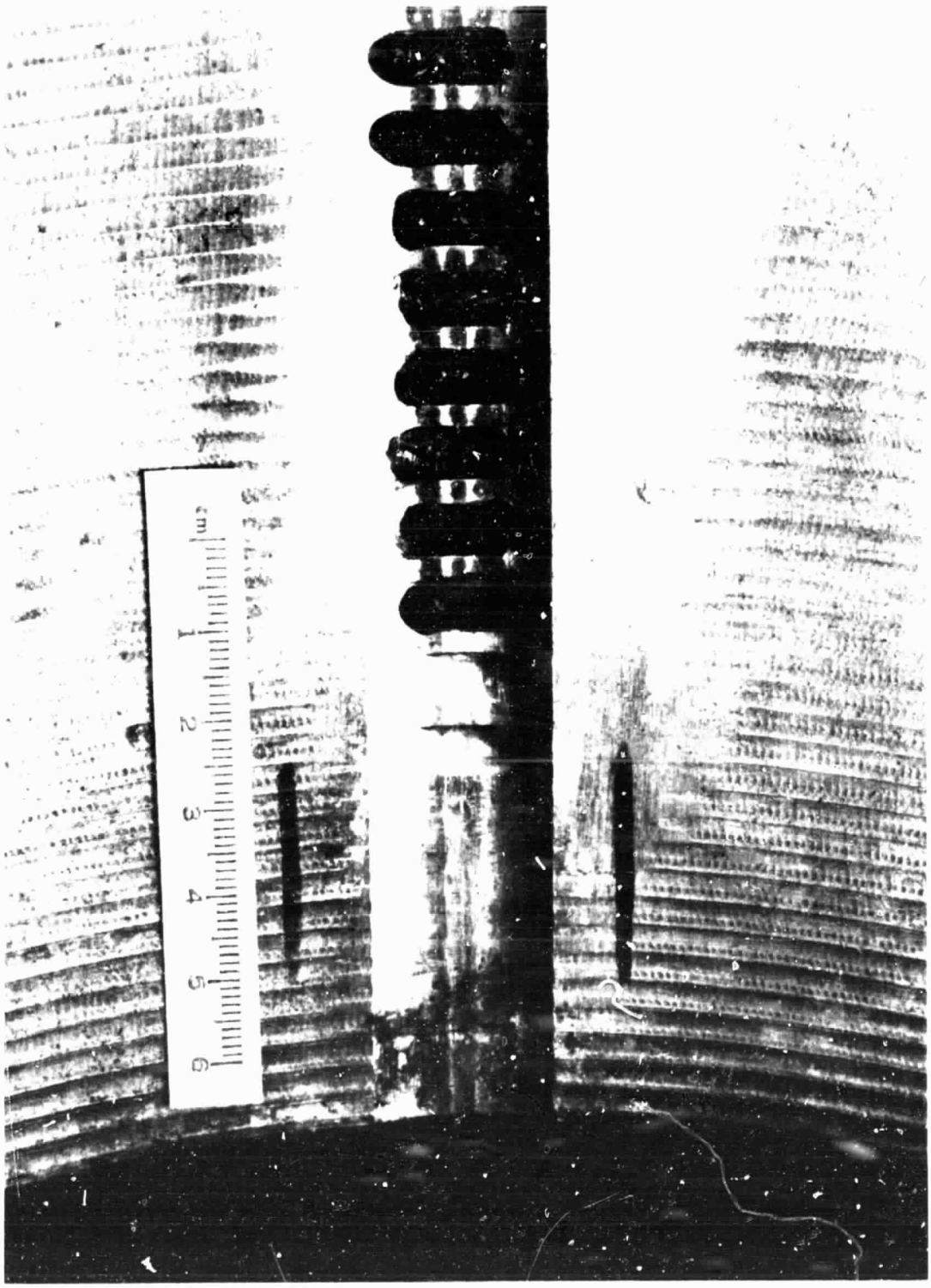

Figure 7. Photograph Showing Unevenness of Incision Holes in Nickel Skin

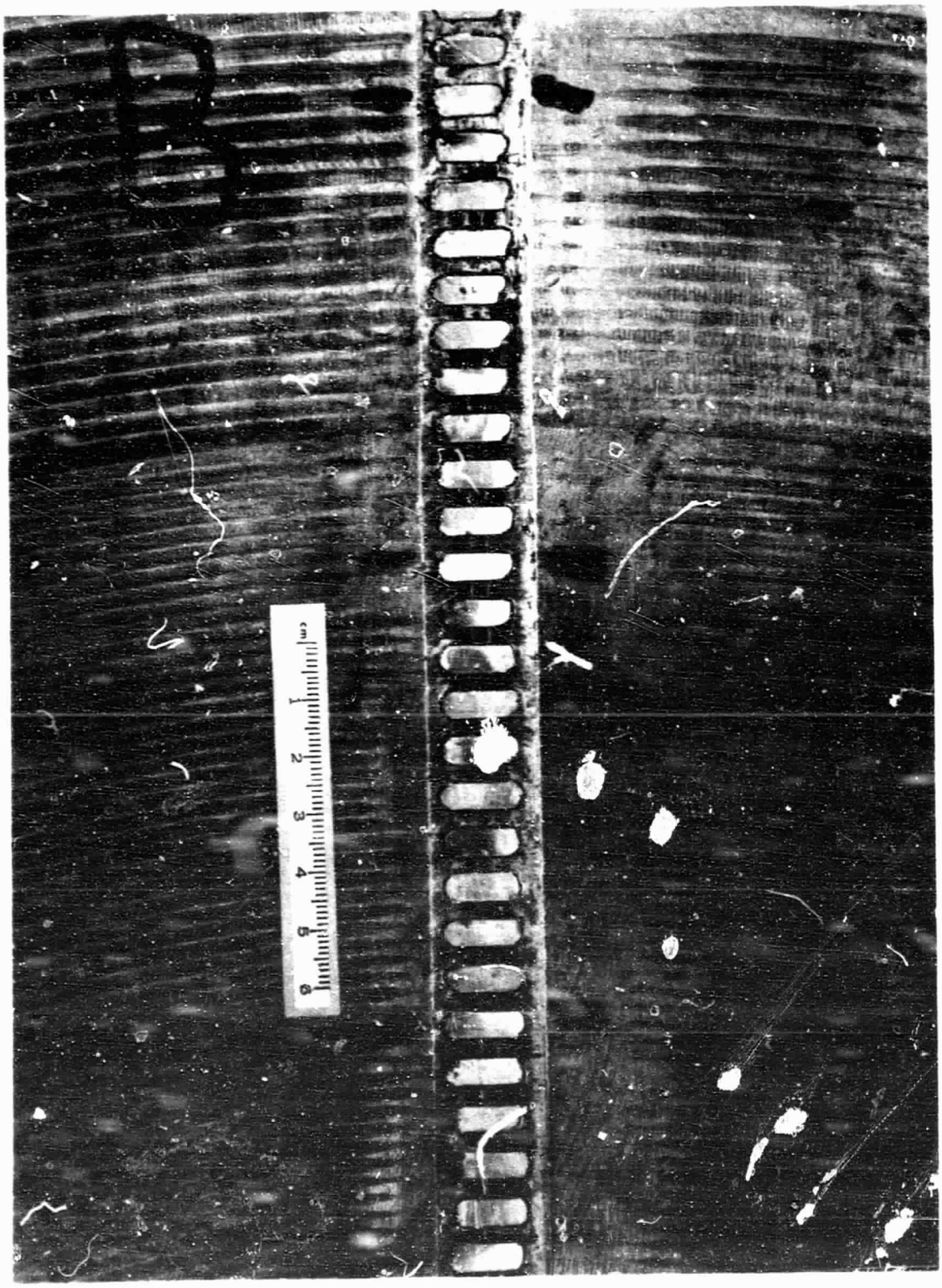

Figure 8. Another View of Incision Area, Again Showing Unevenness of Holes

(3X)

Figure 9. Close-Up View Showing Further Evidence of Unevenness of Incision Holes

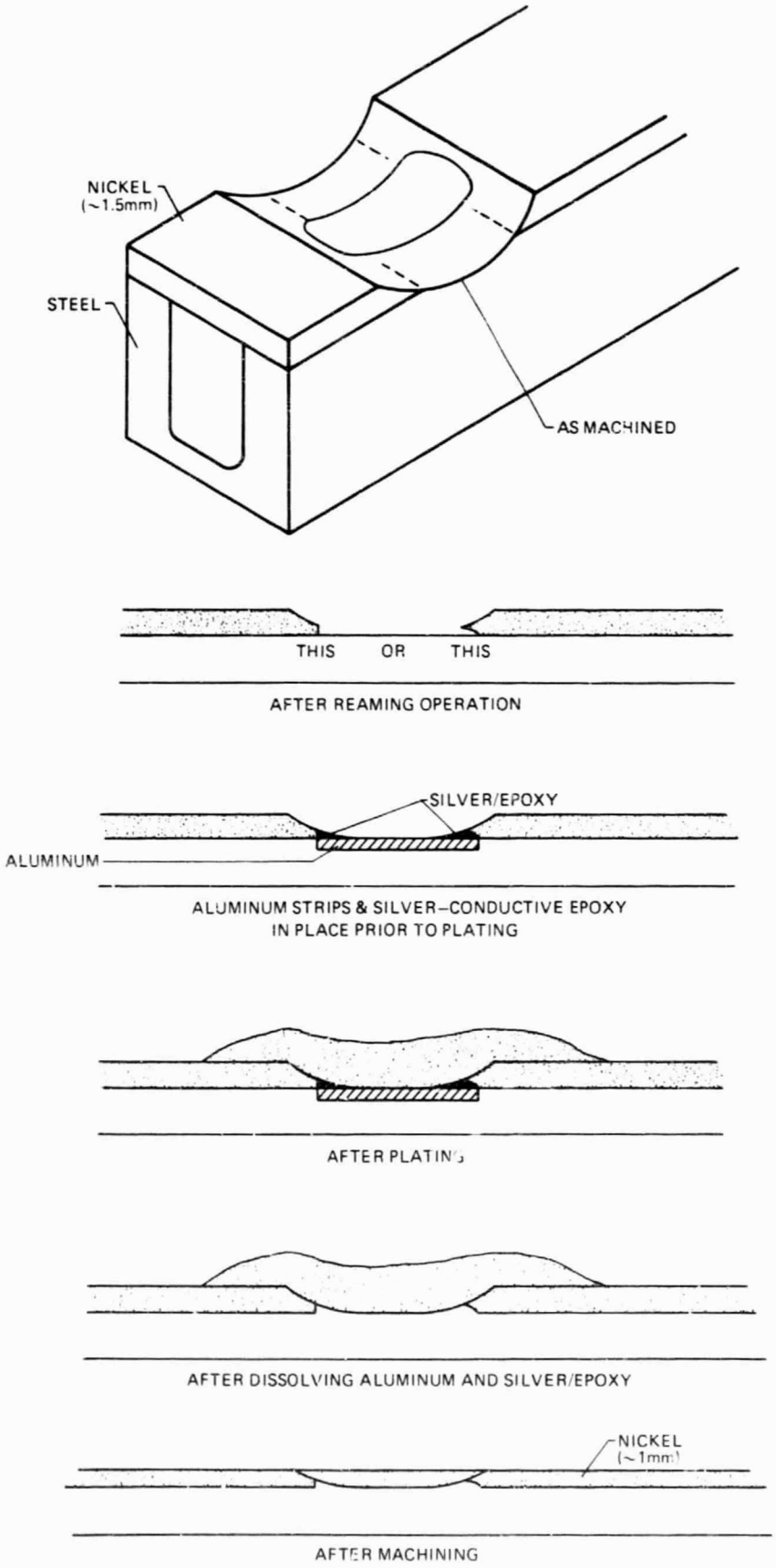

Figure 10. Cross Section Through Center of Channel

Method of Repair

Two methods were considered for repairing the part.

1. Machine an incision in the nickel skin at the same location, but slightly larger than the previous incision, and use the previous repair procedure.

2. Build up a 76 cm (3 in. wide), 1.0 mm thick nickel band over the present incision.

The latter procedure was chosen because of the shorter time available for repair and the simplicity of the plating operation. The part was again used as its own plating tank.

The part was cleaned and masked except for a strip approximately 76 cm wide over the incision area. Lucite shields approximately 1 cm high and machined to the contour of the nickel skin were used to define the area that was to be plated. The following operational sequence was used in plating the part.

1. Exposed nickel area was cleaned with xylene.

2. Surface was scrubbed with Alconox solution.

3. Surface was scrubbed with pumice and water rinse.

4. Steps 2 and 3 were repeated.

5. Part was heated with hot water.

6. Surface was activated by rinsing with 100 g/l solution of sulfamic acid.

7. Surface was anodically treated in a solution containing 450 g/l nickel sulfamate and 150 g/l sulfamic acid for 5 minutes at 540 A/m^2 and 55°C. Then the polarity was reversed and the part was given a nickel strike in the same solution for 7 minutes at 540 A/m^2 and 52°C.

8. Nickel strike solution was withdrawn from part and replaced with nickel sulfamate solution with no intermediate rinsing. The plating surfaces were kept wet by continuously pouring plating solution on them. The part was plated at 130 A/m^2, a pH of 4.0, a surface tension of 35 dyne/cm, and a temperature of 43°C. Sulfur depolarized anodes were used.

The part was plated for seven days. At the end of four days, roughness was noticed in the upper region of plating; so at the end of the plating cycle this roughness was removed by sanding. The roughness probably occurred because the solution had not been filtered. After plating, the thickness of the nickel at the upper end measured approximately 1.5 mm and at the lower end, 3 mm.

The part was hydrostatically tested at 500 psi and held for 10 minutes without any evidence of leaking. The part was then sent to NASA and machined to final dimension before retesting at 500 psi. It is now ready for service. Figure 11 shows one of the repaired sections.

Summary

Two major results of the work reported here are: (1) high-temperature ductility losses for electrodeposited nickel were found to be less severe than earlier work had led us to believe, and (2) means for minimizing those losses were found.

Work described herein showed that sulfur embrittlement causes the high-temperature problem. Codeposition of small amounts of manganese, which is a strong metallic desulfurizer, produced considerable improvement in high-temperature RA data. Another means of overcoming the high-temperature phenomenon is to use the nickel in applications subject to high strain rates. It was shown that as the strain rate increases, the high-temperature ductility problem disappears. Since the strain rates at which ductility improves may be in the regime seen in nozzle applications, this question should be evaluated by NASA personnel.

Influences on properties and impurity content were determined for a number of plating variables including solution temperature, current density, agitation, and elimination of a wetting agent. This work led to the conclusion that presently used operating ranges for these parameters are optimum and that no real benefits would be realized by changing them.

Finally, repair work on the approach section of the 2.44 m (8 ft) HTST combustor is described in detail.

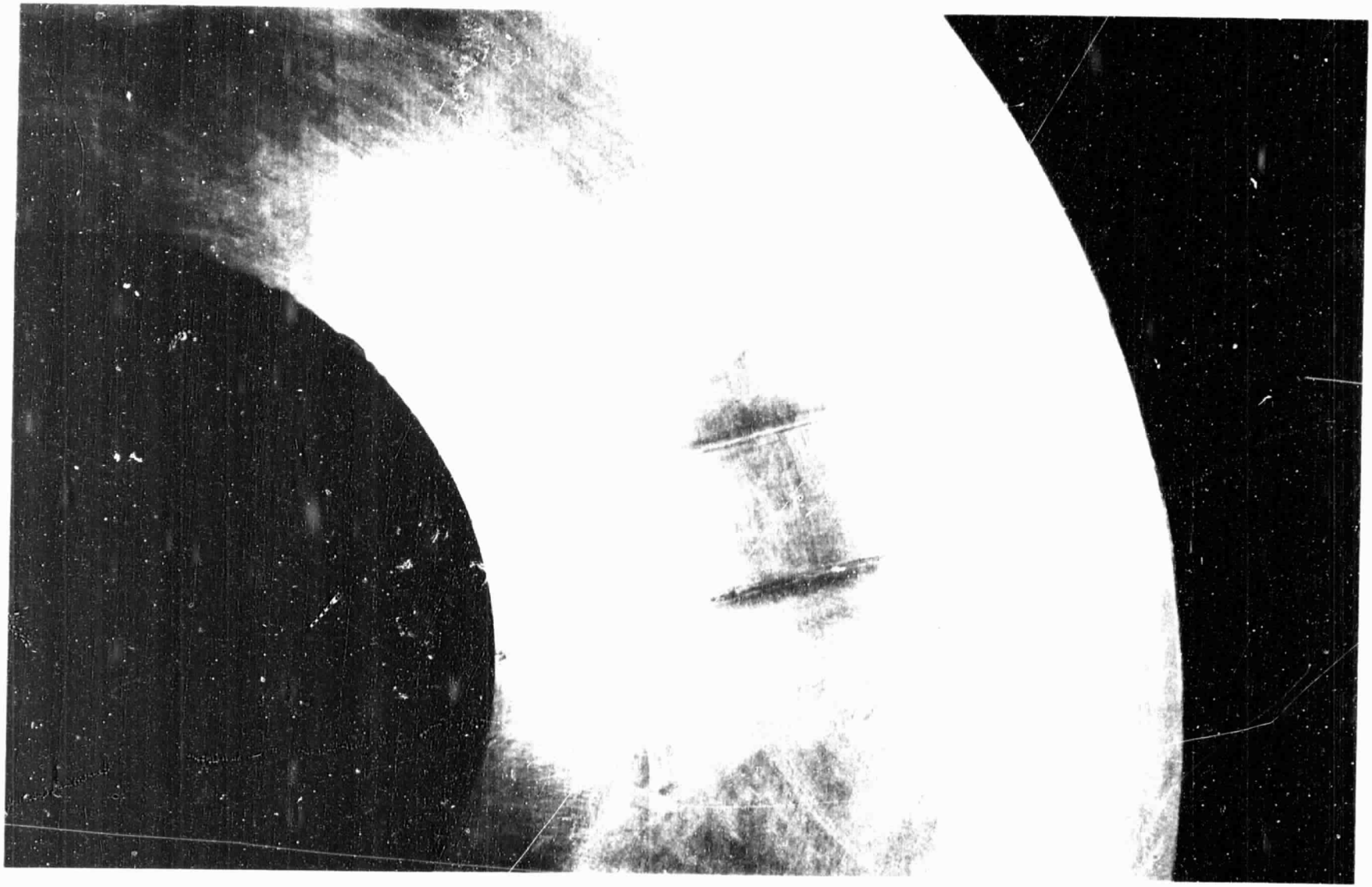

Figure 11. Repaired Section of NASA Nozzle

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