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AiResearch Manufacturing Division
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HYPERSONIC RESEARCH ENGINE PROJECT - PHASE IIA
HASTELLOY-X ANNEALING AND CHEMICAL MILLING EVALUATION
NASA CONTRACT NO. NAS1-6666

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## ABSTRACT

This report presents the results of an experimental evaluation as to the effects of multi-step braze cycles and chemical milling on the mechanical properties and metallurgical constituencies of Hastelloy-X. The results show little influence on the properties of Hastelloy-X when brazed at temperatures up to 2125 degrees F., but brazing at 2150 degrees F. and 2175 degrees F. result in large grain size and reduced ductility. Rapid cooldown is necessary to obtain maximum ductility, i.e. prevent carbide precipitation in Hastelloy-X when heated to 2175 degrees F. Chemical milling has little effect on the properties of Hastelloy-X if the chem milled surface is abrasively polished to remove 0.0005 inch of chem mill affected material. This amount of material removal will prevent an increase of integranular oxidation and resulting loss in ductility of the Hastelloy-X in an air operating environment at 1600 degrees F.

## FOREWORD

This Materials and Material Processing Report is submitted by the AiResearch Manufacturing Company to the NASA Langley Research Center in compliance with the provisions of Clause 42 of NASA Contract No. NASI-6666. The report covers the period of contract go-ahead, 3 February 1967, through 30 June 1968, and summarizes the technical effort relating to materials and their processes as conducted in fulfillment of contract requirements. It is expected that subsequent reports on this subject will be submitted at semi-annual intervals.

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## 1.0 HASTELLOY X ANNEALING EVALUATION

An evaluation program was conducted on the effect of cold work and heat treatment on Hastelloy X grain size and properties. It is recognized that in controlling the grain size and mechanical properties, particularly ductility, a relationship exists between three factors: previous cold work, annealing temperature, and rate of cooling after annealing. The objective of the evaluation was to determine this relationship, within the constraints of the design and manufacturing requirements imposed on the components.

### 1.1 PROCEDURE AND RESULTS

Published data show that Hastelloy X which has previously been cold worked in excess of 10 percent is relatively insensitive to variables in annealing conditions in reaching a relatively fine-grained ductile post-heat-treat condition. Nucleation and duplexing, which produce the large grains during heat treat, are primarily observed in material that has been previously cold-worked from 1 to 10 percent.

A series of Hastelloy X specimens was prepared from a 0.020-in.-thick sheet (all from the same heat). These were subjected to a three-step evaluation:

(a) Effect of cold work and heat treatment on grain size

(b) Effect of 2075° to 2175°F heat-treat temperatures and cooling rate on grain size and tensile properties

(c) Effect of Palniro 1 (2070°F) braze cycles on tensile properties

### 1.2 EFFECT OF COLD WORK AND HEAT TREATMENT ON GRAIN SIZE

For this evaluation, the specimens were subjected to the following conditions:

(a) Cold work induced by tensile stretching of 1, 2, 5, and 10 percent

(b) Anneal temperatures of 1875°, 1975°, and 2075°F for 25 min, followed by rapid air cool

(c) Exposure at 2170°F for 15 min followed by a slow cool (simulating the action of a Palniro 4 braze cycle)

Metallographic examination was used to measure grain size following each of the operations.

The results of this evaluation are as follows:

(a) No duplexing (large grains) or grain growth occurred on any material after exposure at 1875°, 1975°, and 2075°.

(b) Duplexing occurred on all materials subjected to exposure at 2170°F, regardless of prior exposures at 1875°, 1975°, and 2075°, except for specimens stretched 10 percent. Material stretched 10 percent had undergone uniform grain growth, with grain size only about three times larger than for as-received material.

## 1.3 EFFECT OF HEAT-TREAT TEMPERATURE AND COOLING RATE ON GRAIN SIZE AND TENSILE PROPERTIES

For this evaluation, specimens were heated to temperatures of 2075°, 2100°, 2125°, 2150°, and 2175°, followed by controlled cooling rates. One specimen, heated to 2175°, was pre-alloyed with Palniro 4. After this treatment, all specimens were subjected to metallographic examination and tensile testing.

The results are as follows:

(a) Small grain size was retained in as-received material after 15-min exposure at temperatures of 2050°, 2075°, 2100°, and 2125°. After exposure at 2150°F, large grains were beginning to form. Exposure at 2175° resulted in large grains. All material that was rapid air cooled (less than 1 min from brazing temperature to 1200°F) had a minimum of carbide precipitation in the grain boundaries. Material cooled in vacuum (30-min cooldown to 1200°F) had a continuous, fairly heavy grain boundary precipitate; material furnace cooled from brazing temperature (about 3-hr cooldown to 1200°F) had a somewhat heavier precipitate than that cooled in vacuum.

(b) Tensile and yield strength decreased with increasing temperature, but elongation increased for heat treatment at 25°F increments, from 2075° to 2175° and rapid air cooling.

(c) Both vacuum (30 min) and furnace cooling (3 hr) from 2175° only moderately decreased tensile and yield strength, but reduced elongation by almost 50 percent compared with specimens rapid air cooled.

(d) Tensile properties measured during this evaluation are listed in Table 1.

## 1.4 EFFECT OF PALNIRO 1 (2070°F) BRAZING CYCLES ON TENSILE PROPERTIES

For this evaluation, two specimens (one coated on one side with 0.001-in.-thick Palniro 1 braze-alloy foil) were subjected to a brazing cycle in a vacuum furnace. The specimens were then tensile tested.

TABLE I

EFFECT OF HEAT TREATMENT ON TENSILE PROPERTIES

| Heat-Treat Temperature, °F | Time at Temperature, Min | Cooldown Method | Cooldown Time, Min | Ultimate Strength, ksi | Yield Strength, ksi | Elongation, Percent | Grain Size Figure Ref |
|---|---|---|---|---|---|---|---|
| 2075 | 15 | R.A.C.** | 1 | 112 | 56 | 38 | 1b |
| 2100 | 15 | R.A.C. | 1 | 111 | 54 | 36 | 2a |
| 2125 | 15 | R.A.C. | 1 | 111 | 54 | 39 | 2b |
| 2150 | 15 | R.A.C. | 1 | 108 | 48 | 39 | 3a |
| 2175 | 15 | R.A.C. | 1 | 106 | 47 | 42 | 3b |
| 2175 | 15 | R.A.C. | 1 | 105 | 45 | 40 | |
| 2175 | 5 | Vacuum | 30 | 95 | 43 | 24 | 4b |
| 2175* | 5 | Vacuum | 30 | 96 | 44 | 24 | |
| 2175 | 15 | Furnace | 180 | 102 | 49 | 25 | 4a |
| 2150 | 15 | Hydrogen | 8 | 118 | 55 | 29 | |
| 2150 | 15 | Hydrogen | 8 | 90 | 44 | 24 | |

NOTES: *1 mil Palniro 4 on one side

**R.A.C. = Rapid Air Cool

The room temperature properties of these two specimens are tabulated below along with comparative properties extracted from the previous evaluation.

| Heat-Treat Temperature, $^{\circ}F$ | Condition | Cooldown Method | Ultimate Strength, ksi | Yield Strength, ksi | Elongation, Percent | Reduction in Area, Percent |
|---|---|---|---|---|---|---|
| 2070 | Uncoated | Vacuum | 112 | 49 | 40 | 35 |
| 2070 | 1 mil Palniro 1 foil on one side | Vacuum | 113 | 51 | 37 | 34 |
| 2075 | Uncoated | Rapid air cooled | 112 | 56 | 38 | 36 |
| 2170 | Uncoated | Vacuum | 95 | 43 | 24 | 23 |
| 2175 | Uncoated | Rapid air cooled | 106 | 47 | 42 | 30 |

From the above, it can be seen that Hastelloy X tensile properties (especially ductility) were significantly better after a simulated Palniro 1 vacuum brazing cycle (2070°F) than for a Palniro 4 vacuum brazing cycle (2170°F). Cooling rate was not as significant for exposure at 2070°F that as at 2170°F. Except for a 10-percent-lower yield strength, vacuum-cooled Hastelloy X from 2070°F had tensile properties equivalent to those of rapid-air-cooled Hastelloy X

## 1.5 CONCLUSIONS

The following conclusions were reached:

(a) Regardless of prior cold work or cooling rate, heat treatment or brazing of Hastelloy X at 2075°F to 2125°F does not increase grain size or significantly lower ductility.

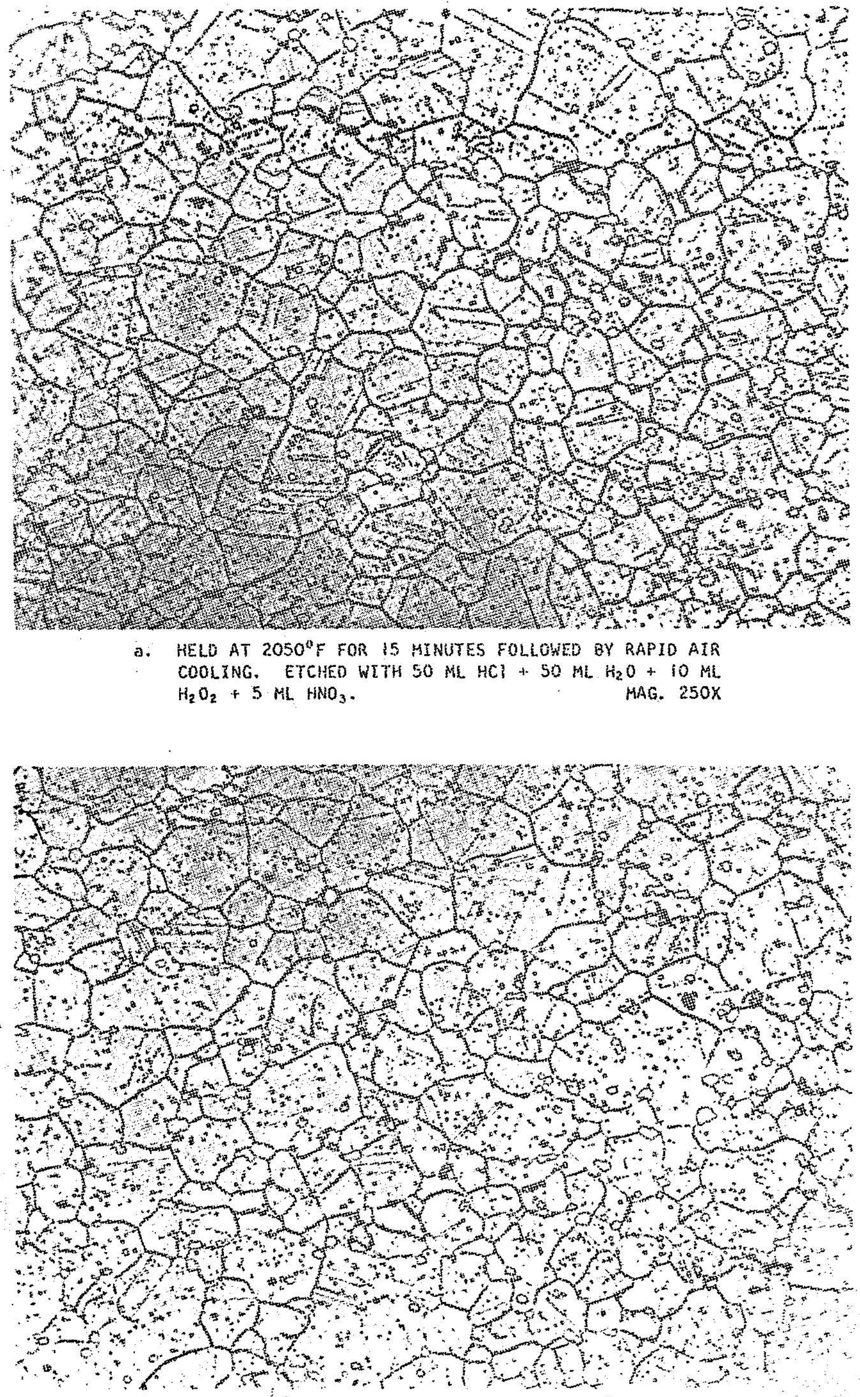
(b) Palniro 1 braze alloy on Hastelloy X tensile specimens only slightly influenced tensile properties.

(c) Exposure above 2150°F can result in large grain size. This temperature appears to be the critical one for growth: none occurs at 2125°F, some occurs at 2150°F, and large grains are formed at 2175°F.

(d) Rapid cooldown is necessary to obtain maximum ductility in Hastelloy X when heated to 2175°F. The slower the cooldown, the lower is the ductility. No further increase in ductility, however, occurred after 30 min, i.e., 30-min and 3-hr cooldown time to 1200°F produced the same results.

(e) Grain size affected yield strength, but not ductility, in rapidly cooled specimens where carbide precipitation did not occur.

(f) Palniro 4 brazing alloy (1-mil foil on one side of tensile specimen) did not decrease tensile properties of Hastelloy X in vacuum brazing.



a. HELD AT 2050°F FOR 15 MINUTES FOLLOWED BY RAPID AIR COOLING. ETCHED WITH 50 ML HCl + 50 ML $H_2O$ + 10 ML $H_2O_2$ + 5 ML $HNO_3$. MAG. 250X

b. SAME AS "a" BUT HELD AT 2075°F.

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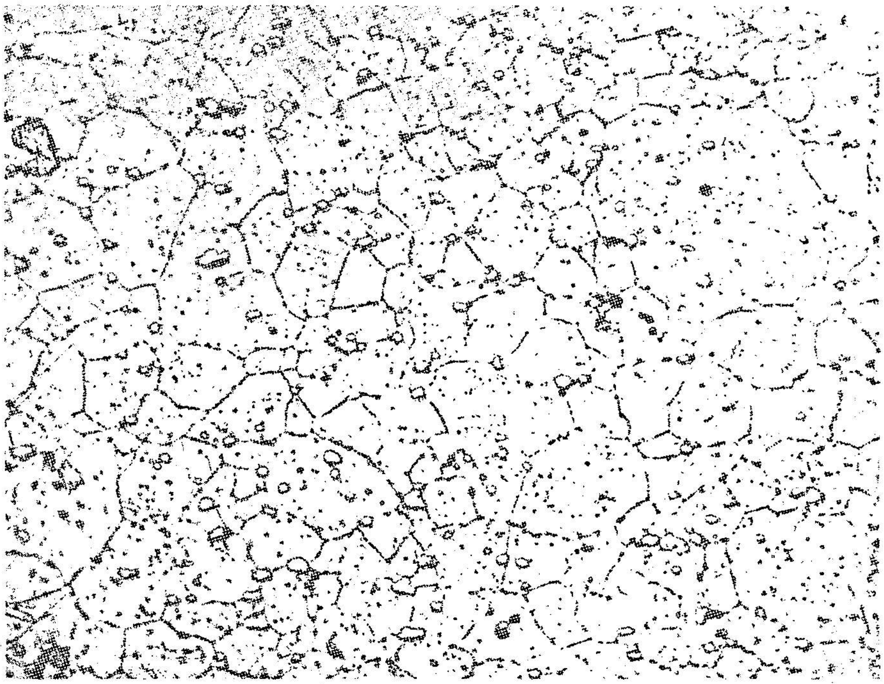
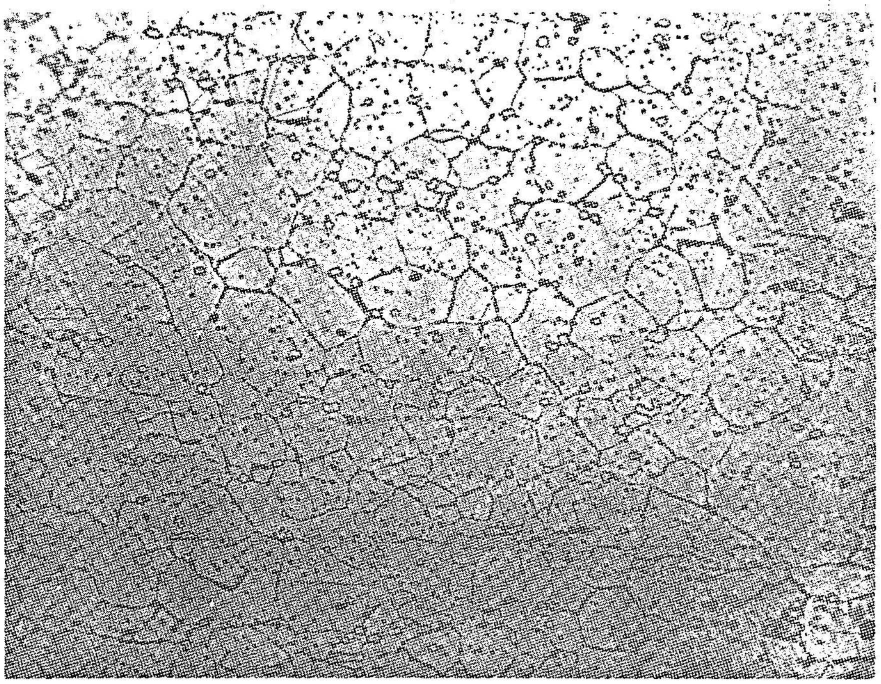
Figure 1. Effect of Heat-Treat Temperature on Microstructure of "As-Received" Hastelloy X Sheet (0.020-in. Thick)



a. HELD AT 2100°F FOR 15 MINUTES FOLLOWED BY RAPID AIR COOLING. ETCHED. MAG. = 250X



b. SAME AS "a" BUT HELD AT 2125°F

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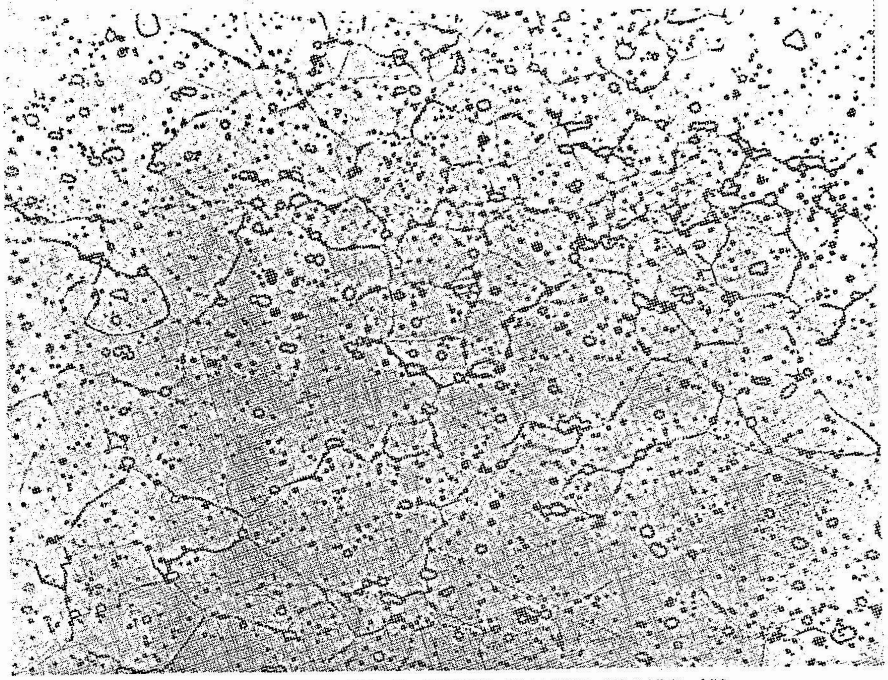
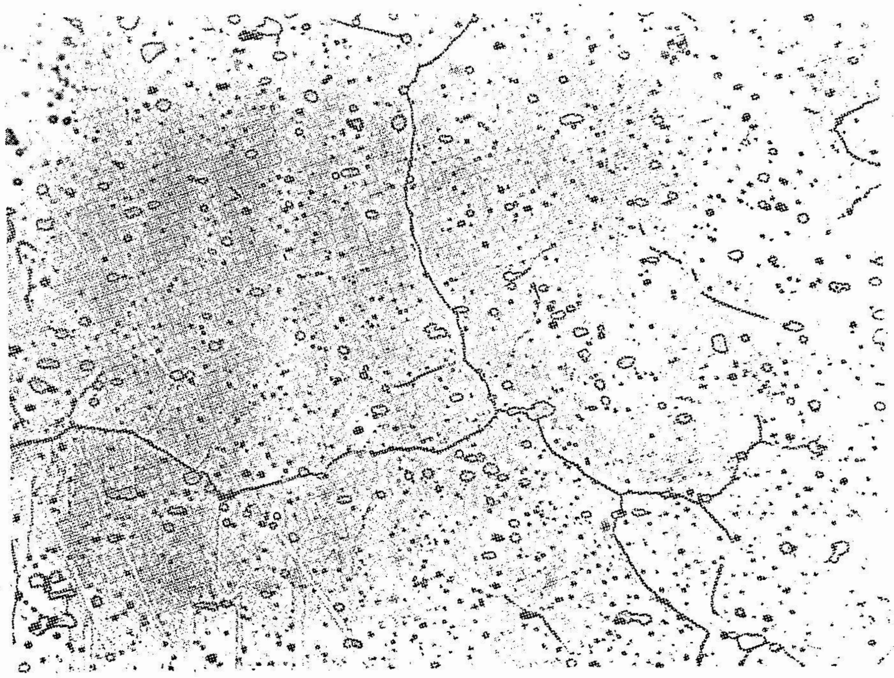
Figure 2. Effect of Heat-Treat Temperature on Microstructure of "As-Received" Hastelloy X Sheet (0.020-in. Thick)



a. HELD AT 2150°F FOR 15 MINUTES FOLLOWED BY RAPID AIR COOLING. ETCHED. MAG. = 250X



b. SAME AS "a" BUT HELD AT 2175°F

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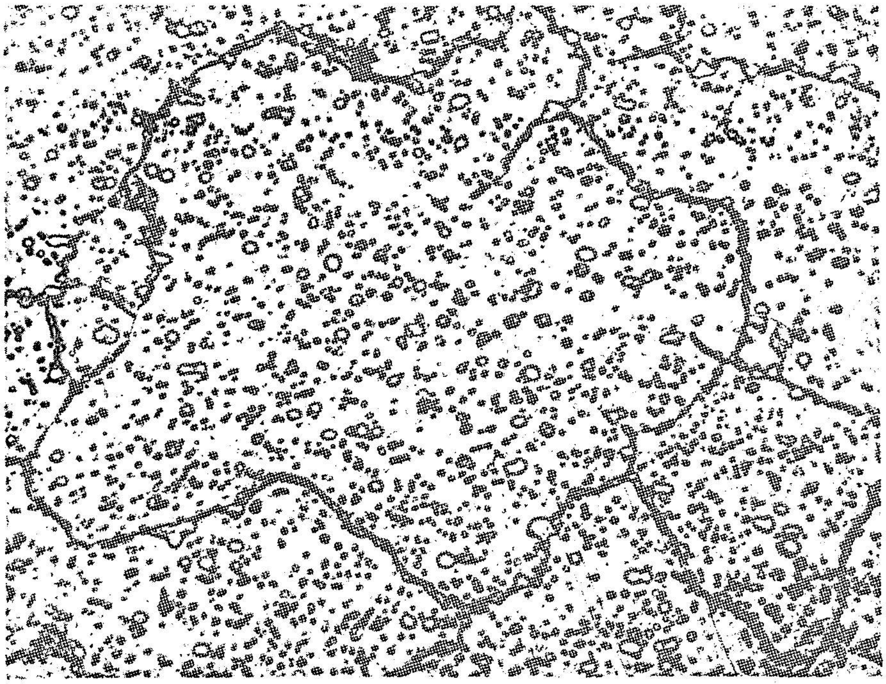
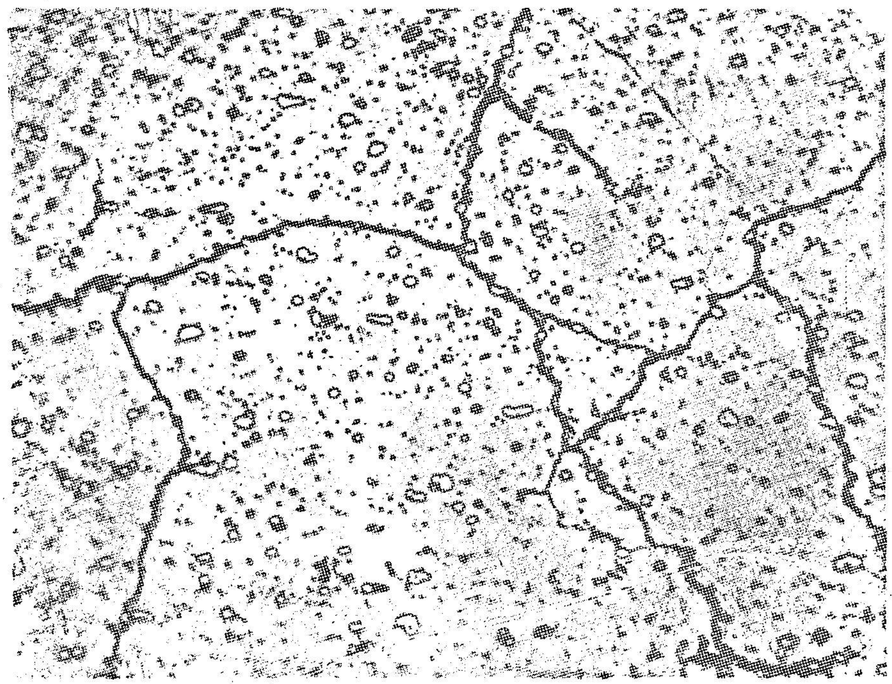
Figure 3. Effect of Heat-Treat Temperature on Microstructure of "As-Received" Hastelloy X Sheet (0.020-in. Thick)



a. HELD AT 2175°F FOR 15 MINUTES FOLLOWED BY FURNACE COOLING (3 HOURS TO 1200°F). ETCHED. MAG. = 250X



b. HELD AT 2170°F FOR 5 MINUTES FOLLOWED BY VACUUM COOLING (ABOUT 30 MINUTES TO 1200°F). ETCHED. MAG. = 250X

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Figure 4. Effect of Heat-Treat Temperature on Microstructure of "As-Received" Hastelloy X Sheet (0.020-in. Thick)

## 2.0 CHEMICAL MILLING OF HASTELLOY X MATERIAL

### 2.1 INTRODUCTION

An investigation was initiated into the effects of chem milling on Hastelloy X material and the possible methods of controlling the process to prevent excessive surface attack.

### 2.2 PRELIMINARY INVESTIGATION

Due to a lack of published data on the effects of chem milling on Hastelloy X, general statements and associated inferences had to be used for the preliminary data search.

One appropriate comment from Reference 1, which summarizes the current attitude toward chem milling, is: "The general feeling is that chemical milling does not adversely affect the mechanical properties of metals and alloys providing good uniform metal dissolution is achieved, i.e., no intergrannular attack, pitting or selective etching."

The following comments on Inconel X and Rene' 41 nickel-base superalloys would also be appropriate to Hastelloy X in considering possible effects of chem milling on properties:

> No effect due to chem milling on Inconel X was noted in tensile, stress-rupture, and fatigue tests carried out at $100^{\circ}F$ by North American Rockwell Corporation (Reference 2). No intergranular attack was noted by N.A.R. in production chem milling of Inconel X (Reference 3).
>
> Boeing chemically milled Rene' 41 welded and swaged tubing without getting intergranular attack of the tube metal, weld metal, or the weld zone (Reference 4). The etching rate was slightly less along the weld seam than in the welded zone, although no production problems were anticipated. In the above study by Boeing, Rene' 41 thermally cycled at $1400^{\circ}F$ and $1800^{\circ}F$, had a 15 percent loss in ductility at $80^{\circ}F$, 40 percent at $1400^{\circ}F$, and 20 percent at $1800^{\circ}F$. No loss in tensile or yield strength occurred. Part of the loss in ductility was attributed to intergranular attack by milling solution and surface sealing during thermal exposures. The proper combination of etchant composition and milling operating conditions are necessary to match the particular chemistry and microstructure of the alloy.
>
> The heat treat condition of the material affects surface finishes that can be used and chem milling solutions. For example, General Electric indicated fully hardened Rene' 41 was more difficult to chemically

mill than the annealed material because of its tendency to passivate after being in the etchant for two to three minutes (Reference 5). Rene' 41 and Hastelloy X lost about 15 percent of their fatigue life due to slight intergranular attack (0.0004 to 0.0006 in.) that occurred during chem milling. The Hastelloy X endurance limit decreased from about 38.8 ksi before chemical milling to 32.5 ksi after chem milling. With the A286 alloy where no intergranular attack occurred, no loss of fatigue strength was observed (Reference 5). Similar results with Rene' 41 and Hastelloy X would be expected if no intergranular attack had occurred.

## 2.3 EFFECTS OF CHEM MILLING ON PROPERTIES AND MICROSTRUCTURE OF HASTELLOY X

### 2.3.1 Evaluation Specimen

The part selected for evaluation was a compound-curved shell. The shell started out as a 0.020-in. sheet which was then welded into a cone. The weld bead was roll-plenished prior to the shell (cone) being stretch-formed to an approximate thickness of 0.018 in. The shell was then solution heat-treated at 2150°F for 15 minutes prior to the chem milling process, which reduced the material to a thickness of 0.012 to 0.014 in. The weld bead was ground down to blend in with the surface contour.

### 2.3.2 Evaluation of Properties

The specimens taken from the part (Figure 5) were evaluated from the results of the tensile test , bend test , and metallographic examination at the weld bead as well as the parent material.

#### 2.3.2.1 Tensile Properties

The room temperature tensile properties of the weld bead and the parent metal are shown below for the chem milled shell:

| Specimen Number | Location | Ftu (ksi) | Fty (ksi) | Elongation (% in 2 in.) | Comments |
|---|---|---|---|---|---|
| K454 | Parent Metal | 109.0 | 46.0 | 31 | |
| K453 | | 107.5 | 47.0 | 29 | |
| K454 | | 103.5 | 46.0 | 22 | |
| K455 | | 111.0 | 49.0 | 30 | |
| K456 | | 106.0 | 48.0 | 25 | |
| K457 | | 104.0 | 46.6 | 23 | |
| | Average = | 107.0 | 47.1 | 27 | |
| K449 | Weld Bead | 107.0 | 50.0 | 23 | Failed in Parent Metal |
| K450 | (Hastelloy W | 110.0 | 51.2 | 26 | Failed in Parent Metal |
| K451 | Rod) | 108.5 | 50.4 | 22 | Failed in Parent Metal |
| | Average = | 108.5 | 50.5 | 24 | |
| K458 | Weld Bead | 105.5 | 47.0 | 24 | Failed in Parent Metal |
| K459 | (Hastelloy W | 109.0 | 50.2 | 25 | Failed in Parent Metal |
| K460 | Rod) | 105.5 | 48.3 | 22 | Failed in Parent Metal |
| K461 | | 109.0 | 51.0 | 22 | Failed in Parent Metal |
| | Average = | 107.0 | 49.1 | 23 | |

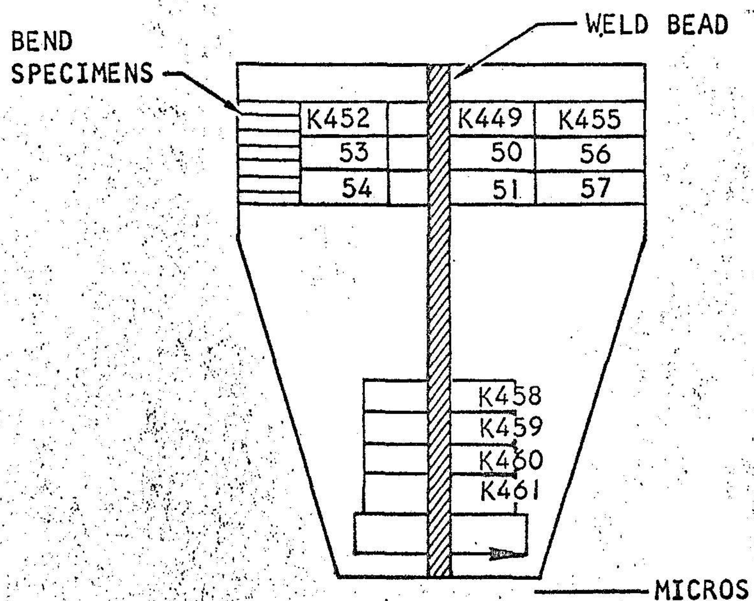


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Figure 5. Specimen Location from Shell (P/N 51085-1, S/N 1)

The tensile and yield strengths of the chem milled Hastelloy X material exceeded the minimum values of Specification AMS 5536D ($S_{yp}$ = 45,000 psi, $S_{ult}$ = 100,000 psi); the elongation values were below the Specification AMS 5536D minimum of 35 percent. However, the lower elongation was not the result of the chem milling, but of the hydrogen annealing.

2.3.2.2 Bend Test

The bend test around a diameter of 1.0 to 1.5t was satisfactory with no cracks occurring at the bend for either the parent metal or the weld bead.

2.3.2.3 Metallographic Examination

The metallographic examination revealed that some intergranular attack occurred in the Hastelloy X (Figure 6). Figure 7 shows that the Hastelloy X weld zone was not attacked as much by chem milling as the parent metal. The average depth of attack for the parent metal was 0.0002 to 0.0005 in. with a maximum attack depth of 0.001 in.

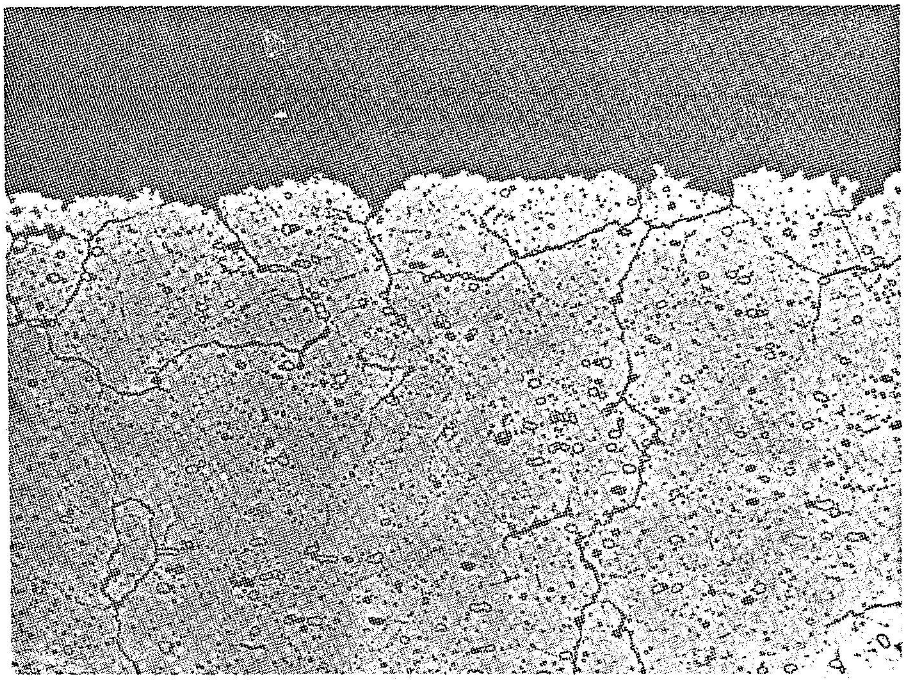
The exposure of the chem milled surface to an air environment at 1500-1600°F for 10 hours may result in increased intergranular oxidation of the Hastelloy X. The effects of the oxidation would be to reduce the ductility; but, because the ductility of the chem milled material measured at room temperature was not greatly reduced, no sharp decrease in ductility is expected. Polishing the chem milled surface to remove 0.0005 in. should be sufficient to remove most of the material affected by the chem milling process.

## 2.4 CONCLUSIONS

The oxidation resistance of the chem milled Hastelloy X material can be improved by abrasively removing (polishing) 0.0005 in. of the surface. In doing so, most of the material affected by the chem milling process can be removed.

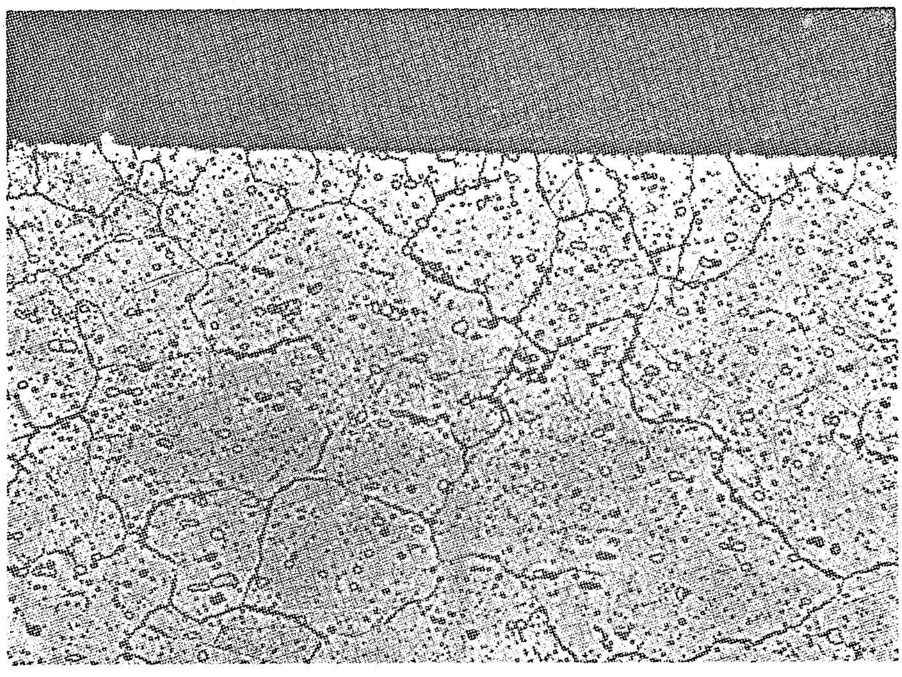
The weld joint efficiency after roll-plenishing, forming, annealing and chem milling was 100 percent using Hastelloy W weld rod. However, Hastelloy X weld rod is recommended due to its higher oxidation resistance and equivalent strength characteristics to the Hastelloy W weld rod.

Mag. = 250X



a. Chem Milled Outer Surface of Shell. Average Intergranular Attack of 0.0002 to 0.0005 in.; Maximum, 0.0010 in.

Mag. = 250X



b. Inner Surface Not Chem Milled

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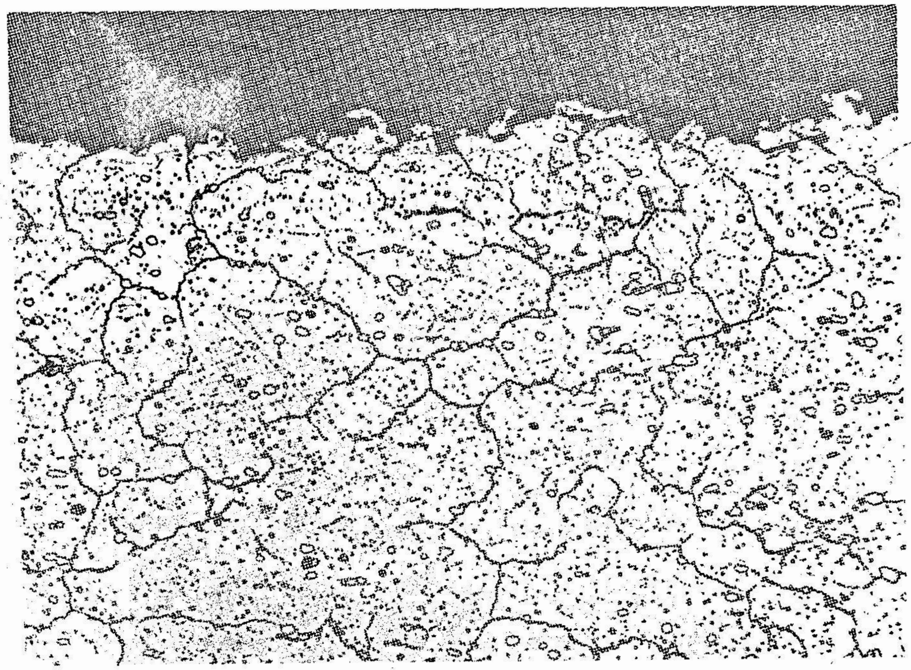
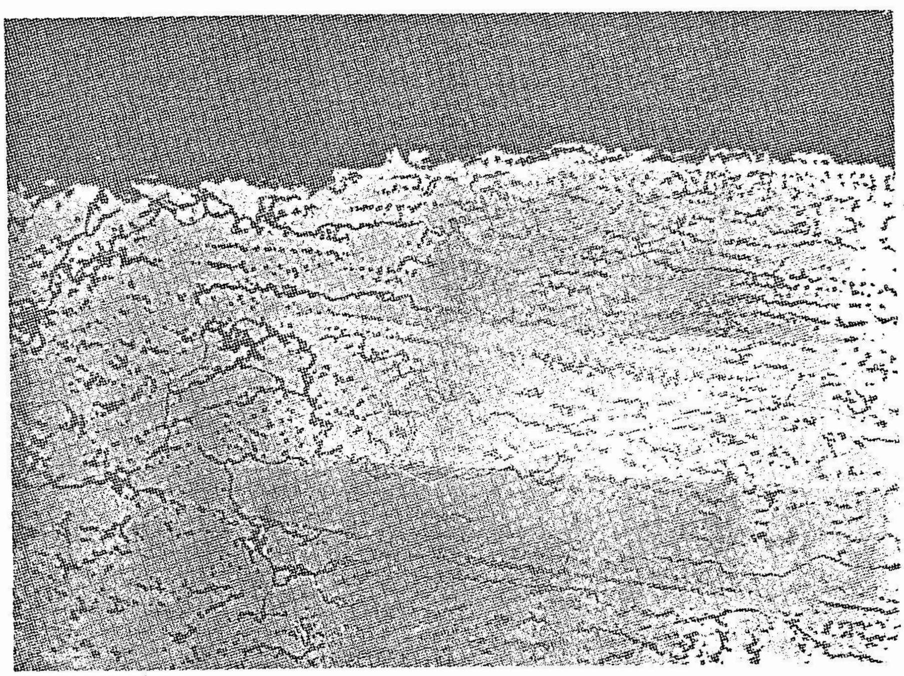
Figure 6. Photomicrographs Illustrating the Effect of Chem Milling on Hastelloy X Shell. (P/N 51085, S/N 1).

Mag. = 250X



a. Cross Section of Chem Milled Surface

Mag. - 250X



b. Cross Section of Chem Milled Surface at Weld Bead (Right Side) Indicating Less Attack Occurred at Weld Than in Parent Metal (Left Side). Hastelloy X Rod Used in Welding.

F-9569

Figure 7. Photomicrographs of Sections from Small Diameter of Hastelloy X Shell after Chem Milling (P/N 51085, S/N 1)

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