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# EFFECTS OF HIGH-TEMPERATURE BRAZING AND THERMAL CYCLING ON MECHANICAL PROPERTIES OF HASTELLOY X

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16. Abstract

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# EFFECTS OF HIGH-TEMPERATURE BRAZING AND THERMAL CYCLING ON MECHANICAL PROPERTIES OF HASTELLOY X

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## SUMMARY

Data are presented on the effects of brazing alloy, brazing operation, thermal cycling, and combinations of these on the yield strength, elongation, ultimate tensile strength, and fatigue life of thin-gage Hastelloy X. These data show that brazing with a Ni-Pd-Au alloy at 1461 K (2170° F) resulted in reductions of 35 percent in yield strength and elongation, 6 percent in ultimate tensile strength, and 18 percent in fatigue limit of Hastelloy X, as compared with as-received material. Subsequent exposure to 200 thermal cycles between 533 K and 1144 K (500° F and 1600° F) after brazing caused further losses of 4 percent in yield strength, 8 percent in ultimate tensile strength, and 6 percent in fatigue limit.

## INTRODUCTION

High-temperature brazing is a commonly used joining process in the aerospace industry. (See refs. 1 to 3.) Complex structures like heat exchangers are frequently built by use of a step-brazing technique that allows successive parts to be joined, one or two at a time, with successively lower melting point brazing alloys. (See refs. 2 and 4.)

A step-brazing process was employed in the fabrication of the regeneratively cooled hypersonic research engine. (See ref. 5.) Test models of the engine's cooling structure, which is made of Hastelloy X (a nickel-based superalloy), failed during burst tests at stress values much lower than predicted. In addition, other models of this same structure failed prematurely during thermal cycling under stress. (See ref. 6.) A research program was undertaken at Langley Research Center to assess the probable cause of the apparent degradation of the properties of Hastelloy X. The objective of this study was to determine the effects of (1) the brazing alloy, (2) the brazing operation, and (3) the simulated engine operating cycles (hereinafter referred to as service thermal cycles) on the fatigue and mechanical properties of Hastelloy X.

## MATERIALS AND SPECIMENS

Table I shows the chemical composition of the base metal and brazing alloys used in this study. Table II shows the melting temperature range of the two brazing alloys used, Palniro 1 and Palniro 4. The melting temperature range of Palniro 1 is lower than that of Palniro 4 and thus allows the use of these alloys in a step-brazing process.

Figure 1 shows the bare metal and as-brazed tensile-test specimen configurations. Bare metal and as-brazed fatigue-test specimen configurations are shown in figure 2. All specimens were cut from sheet material and machined to final size.

Figure 3 shows the specimen configuration used to study interactions between the base metal and the brazing alloy. Longitudinal sections were cut from specimens of this type after brazing and mounted for metallographic and electron microprobe analysis.

## EQUIPMENT AND PROCEDURES

Prior to brazing, specimens were deburred and cleaned by the following method: degreased with trichloroethylene, dipped in alkaline solution, rinsed with tap water, rubbed with pumice, descaled by immersion in an acid (30 percent nitric, 15 percent hydrofluoric, 55 percent water) bath for 15 minutes, rinsed with tap water, and then hot air dried. Tensile tests on cleaned specimens showed that this cleaning procedure had no detrimental effect on Hastelloy X. After cleaning, a 76.2-$\mu$m-thick (0.003 in.) strip of brazing alloy was tack spotwelded to the tensile and fatigue specimens. On the tensile specimens the brazing alloy strip covered the length of the test section, and on the fatigue specimens it covered a 5.1-cm (2 in.) section in the middle of the specimen. (See figs. 1(b) and 2(b).) Chromel-alumel thermocouples were tack welded to the specimens just outside of the test section for temperature control and monitoring during brazing. The specimens were laid flat on an alumina-coated carbon block and inserted into a vacuum furnace. The brazing heat cycles for both the Palniro 1 and Palniro 4 brazing alloys are described in table III and illustrated in figure 4. The two brazing operations are very similar, the Palniro 1 operation involving a lower maximum temperature and a shorter total time than that used for Palniro 4. After brazing, the tensile and fatigue specimens were hand sanded and hand polished to reduce the thickness of the brazing alloy to approximately 25.4 $\mu$m (0.001 in.), machined to final size, and then deburred.

Specimens to be exposed to service thermal cycling were again instrumented with thermocouples, mounted on an insulating block, and lightly clamped to prevent distortion during heating. Figure 5 illustrates the service thermal cycling procedure (simulating thermal exposure in the hypersonic research engine) which was accomplished by radiant heating in air with a tungsten filament quartz lamp bank coupled to a specimen temperature controller. Prior to the first cycle, the specimens were preheated to 977 K (1300$^{\circ}$ F) in 180 seconds (3 minutes) and cooled by an air blast to 533 K ($500^o$ F). These specimens were then cycled 200 times between 533 K and 1144 K ($500^o$ F and $1600^o$ F). Each cycle lasted 80 seconds and included a 20-second hold at 1144 K ($1600^o$ F). The specimens were cooled from 1144 K to 533 K ($1600^o$ F to $500^o$ F) by an air blast.

Two series of tensile tests were conducted in air at room temperature. An initial screening series covering the material conditions was conducted without strain measurement to determine ultimate tensile strength and to identify conditions that should be examined in the fatigue tests and another series of tensile tests. These initial screening tests were conducted at a strain rate of 0.02 per minute. The material conditions examined in the initial screening tensile tests were as follows:

(1) As-received

(2) As-brazed with Palniro 1

(3) As-brazed with Palniro 1 and service thermal cycled

(4) As-brazed with Palniro 4

(5) As-brazed with Palniro 4 and service thermal cycled

(6) As-exposed to Palniro 4 brazing heat cycle (no braze alloy used)

(7) As-exposed to Palniro 4 brazing heat cycle (no braze alloy used) and service thermal cycled

(8) As service thermal cycled (no braze alloy used).

The second series of tensile tests was conducted at strain rates of 0.002 per minute and 0.08 per minute before and after yielding, respectively. Tensile strain for this test series was measured by a strain-gage extensometer with a 5.1-cm (2 in.) gage length. Transverse and longitudinal sections were cut from tensile specimens after testing and mounted for metallographic examination. Fracture surfaces of the tensile specimens were analyzed by use of a scanning electron microscope.

Constant-amplitude fatigue tests were conducted in subresonant-type axial-load fatigue machines operated at a frequency of 30 Hz. (See ref. 7.) Load was sensed by a weigh-bar in series with the gripped specimen. A wire strain-gage bridge cemented to the weigh-bar supplied the load signal to an oscilloscope used to monitor the cyclic load. The machines were calibrated periodically to maintain a loading accuracy of ±89 N (±20 lb).

## DISCUSSION OF RESULTS

The results of the initial screening tensile tests are summarized in figure 6. Eight different parent metal conditions were examined with a minimum of five tests each. These data indicate that Palniro 1 brazing and/or the service thermal cycling had virtually no effect on the ultimate tensile strength of Hastelloy X. However, the four conditions involving the Palniro 4 brazing operation each showed about a 6-percent reduction in ultimate tensile strength. Since this reduction occurred in both brazed specimens and unbrazed specimens subjected to the Palniro 4 heat cycle, it appears that the brazing heat cycle rather than the interaction of the brazing alloy with the base metal was primarily responsible for the reduction in ultimate tensile strength.

Metallographic studies were conducted on sections cut from brazing alloy-base metal interaction specimens (fig. 3) to observe the effects of the brazing operation on Hastelloy X. Figures 7(a), 7(b), and 7(c), respectively, show the virgin base metal, the base metal after brazing with Palniro 1, and the base metal after brazing with Palniro 4. The Palniro 1 and Palniro 4 brazing operations resulted in grains which were 1.2 and 6 times larger, respectively, than the as-received grains.

Figure 8 illustrates the effects of the step-brazing procedure involving successive brazing operations with Palniro 4 and Palniro 1. The microstructure resulting from the first step, brazing with Palniro 4, is shown in figure 8(a). The result of step 2, brazing with the Palniro 1, is illustrated in figure 8(b). In figure 8(b) the base metal above the brazing alloy has experienced both brazing cycles and shows no further grain growth over that shown in figure 8(a). The base metal below the brazing alloy has experienced only the lower maximum temperature Palniro 1 brazing cycle.

Sections cut from these interaction specimens were subjected to electron microprobe analysis by W. Barry Lisagor of the Metals Section at Langley Research Center. Considerable dissolution of the nickel in the base metal adjacent to the brazing alloy interface occurred during the brazing operation. Migration of the nickel into the brazing alloy was indicated by the higher nickel content present in the brazing alloy. This dissolution and migration of the nickel, however, apparently had no effect on the ultimate tensile strength of the metal since the ultimate tensile strength of the brazed specimens and of the unbrazed specimens subjected to the Palniro 4 heat cycle was about the same. (See fig. 6.) Microsegregation of the gold in the brazing alloy was also indicated by the electron microprobe analysis.

Because of the results from the initial screening tests, additional tensile and fatigue tests were performed on Hastelloy X in the following conditions: as-received, as-brazed with Palniro 4, and as-brazed with Palniro 4 and service thermal cycled. Figure 9 shows typical stress-strain diagrams for the conditions just described. A summary of the tensile test results is shown in table IV. These data show that the Palniro 4 brazing operation results in a 6-percent decrease in ultimate tensile strength (as was also found in the screening tests), a 35-percent decrease in yield strength, and a 35-percent reduction in elongation to failure. Specimens subjected to service thermal cycling after brazing showed only small additional changes in these properties.

Because of the unusual finding that both the yield strength and the elongation to failure decreased as a result of the Palniro 4 brazing operation, sections were cut from the as-tested specimens for metallographic examination, and the fracture surfaces were studied with a scanning electron microscope. A photomicrograph of the as-received base metal before tensile testing is shown in figure 10(a). Figures 10(b) to 10(d) show photomicrographs typical of the as-received, as-brazed with Palniro 4, and as-brazed with Palniro 4 and service thermal cycled specimens after tensile testing. Figures 10(a) and 10(b) indicate no change in grain size as a result of tensile testing. Figures 10(a) and 10(c) indicate again the grain growth resulting from brazing with Palniro 4. Figures 10(c) and 10(d) indicate there was no additional grain growth as a result of service thermal cycling. Figure 11 shows typical fracture surfaces of the as-received, as-brazed with Palniro 4, and as-brazed and service thermal cycled specimens. Intergranular fracture occurred in the two specimens exposed to the brazing operation (figs. 11(b) and 11(c)). On the other hand, intragranular fracture always occurred in the as-received specimens (fig. 11(a)) and in unbrazed specimens subjected to the Palniro 4 heat cycle. Thus, it appears that selective embrittlement of the grain boundaries, promoted by the thermochemical reaction of the Hastelloy X with the brazing alloy, has occurred. In addition, the fracture surfaces within the grains of the specimens exposed to brazing had a ductile appearance.

The grain growth resulting from the Palniro 4 brazing operation normally would indicate an annealing phenomenon associated with the high brazing temperature. The annealing would account for the observed reduction in ultimate tensile strength and yield strength. The intergranular failure apparent in the fracture surfaces of the brazed specimens indicated embrittling, which would account for the reduction in elongation to failure.

Figure 12 presents the results of constant-amplitude fatigue tests on specimens exposed to the same brazing and service thermal cycling conditions as the tensile specimens. The specimens brazed with Palniro 4 exhibited a fatigue limit (the maximum stress that will not cause fracture in $10^7$ stress cycles) of 386 $MN/m^2$ (56 ksi) as compared with 469 $MN/m^2$ (68 ksi) for the as-received specimens, a reduction of 18 percent. Those specimens brazed and service thermal cycled had a fatigue limit of 345 $MN/m^2$ (50 ksi), a reduction of 26 percent from the as-received condition. These results are consistent with the tensile-test results in that the brazing operation caused most of the observed degradation of the mechanical properties of Hastelloy X and that service thermal cycling had only a small additional effect.

## SUMMARY OF RESULTS

The results of these studies into the effects of brazing alloy, brazing operation, and service thermal cycling on the mechanical properties of Hastelloy X are as follows:

1. The Palniro 1 brazing operation apparently had little or no effect on the ultimate tensile strength of Hastelloy X.

2. The Palniro 4 brazing operation, however, decreased the ultimate tensile strength by 6 percent, lowered the yield strength by 35 percent, and decreased the elongation to failure of Hastelloy X by 35 percent. These effects were probably caused by both the higher brazing temperature and a thermochemical reaction of the brazing alloy with the base metal. The lower ultimate tensile strength and yield strength were probably caused by the brazing temperature whereas the lower elongation to failure was apparently due to selective embrittlement of the grain boundaries resulting from a thermochemical reaction. In addition, the Palniro 4 brazing operation caused an 18-percent reduction in the fatigue limit of Hastelloy X.

3. The effect of service thermal cycling on the mechanical properties of Hastelloy X was much less significant than the effect of the Palniro 4 brazing operation. Two hundred thermal cycles between 533 K and 1144 K ($500^{\circ}$ F and $1600^{\circ}$ F) caused further reductions in ultimate tensile strength, yield strength, and fatigue limit of 8 percent, 4 percent, and 8 percent, respectively.

Langley Research Center,
National Aeronautics and Space Administration,
Hampton, Va., November 21, 1972.

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TABLE I.- MATERIAL COMPOSITION

| Material | Element | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Cr | W | Fe | C | Si | Co | Ni | Mn | Mo | P | S | Au | Pd |
| *Hastelloy X | 22.14 | 0.47 | 18.09 | 0.08 | 0.39 | 1.85 | Balance | 0.27 | 8.76 | 0.016 | 0.005 | | |
| **Palniro 1 | | | | | | | 25 | | | | | 50 | 25 |
| **Palniro 4 | | | | | | | 36 | | | | | 30 | 34 |

*Union Carbide Corporation.

**Western Gold and Platinum Company.

TABLE II.- MELTING TEMPERATURE RANGE OF BRAZING ALLOYS

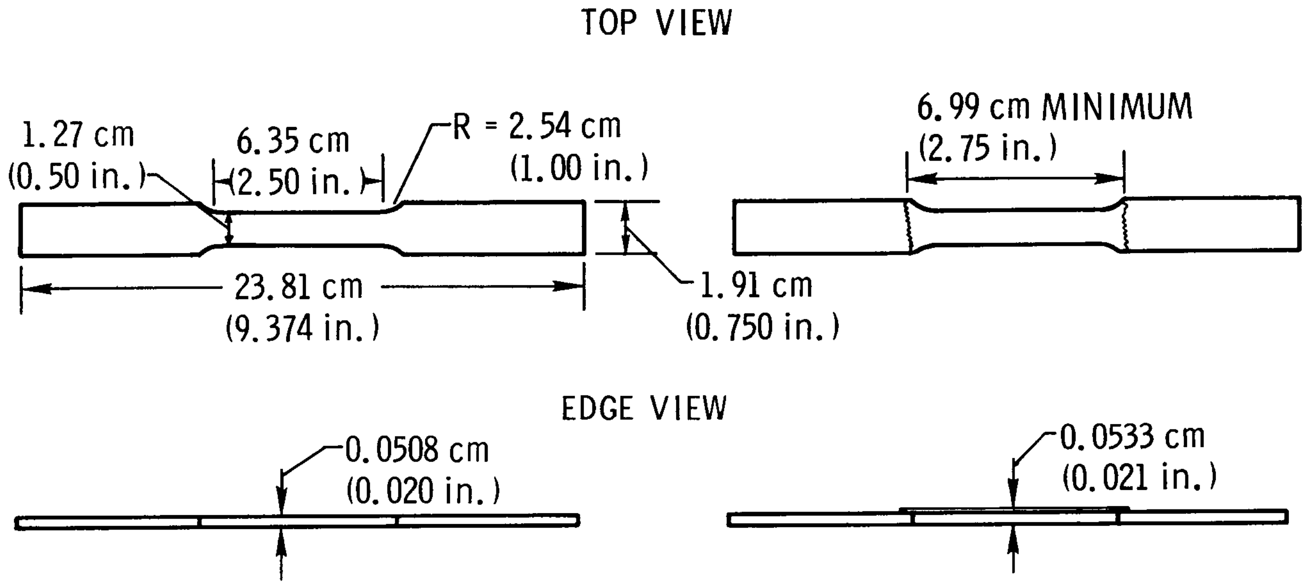
| Brazing alloy | Solidus | | Liquidus | |
|---|---|---|---|---|
| | K | $^{o}F$ | K | $^{o}F$ |
| Palniro 1 | 1375 | 2016 | 1394 | 2050 |
| Palniro 4 | 1408 | 2075 | 1442 | 2136 |

TABLE III.- BRAZING PROCEDURES

| Step | Palniro 1 | Palniro 4 |
|---|---|---|
| 1 | Evacuate furnace to 0.13 $N/m^2$ ($10^{-3}$ torr) | Evacuate furnace to 0.13 $N/m^2$ ($10^{-3}$ torr) |
| 2 | Raise specimen temperature to 1222 K (1740° F) in about 2 hours | Raise specimen temperature to 1255 K (1800° F) in about $2\frac{3}{4}$ hours |
| 3 | Hold specimen temperature at 1222 K (1740° F) for 15 minutes | Hold specimen temperature at 1255 K (1800° F) for 15 minutes |
| 4 | Raise specimen temperature to 1405 K (2070° F) and hold there for 5 minutes | Raise specimen temperature to 1461 K (2170° F) and hold there for 5 minutes |
| 5 | Cool specimen to 1033 K (1400° F) in 45 minutes | Cool specimen to 1033 K (1400° F) in 1 hour |
| 6 | Cut off furnace power and flood chamber with dry argon to a pressure of 20.7 $kN/m^2$ (3 psi) above ambient | Cut off furnace power and flood chamber with dry argon to a pressure of 20.7 $kN/m^2$ (3 psi) above ambient |
| 7 | Remove specimen from furnace after cooling to 366 K (200° F) | Remove specimen from furnace after cooling to 366 K (200° F) |

TABLE IV.- RESULTS OF TENSILE TESTS ON HASTELLOY X

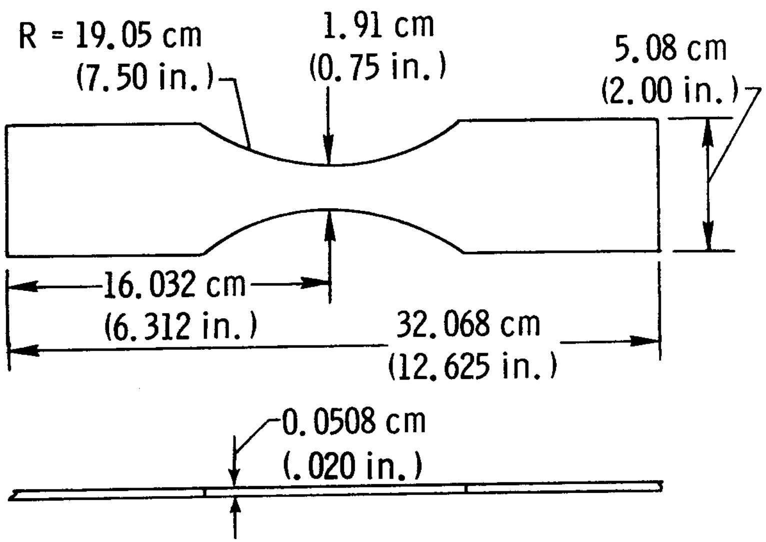
| Material condition | Yield strength (0.2-percent offset) | | Ultimate strength | | Elongation to failure, percent |
| --- | --- | --- | --- | --- | --- |
| | $MN/m^2$ | ksi | $MN/m^2$ | ksi | |
| As-received | 503.0 | 73.0 | 850.2 | 123.4 | 38.2 |
| Brazed with Palniro 4 | 325.9 | 47.3 | 795.8 | 115.5 | 24.8 |
| Brazed with Palniro 4 and service thermal cycled | 305.9 | 44.4 | 729.7 | 105.9 | 26.1 |



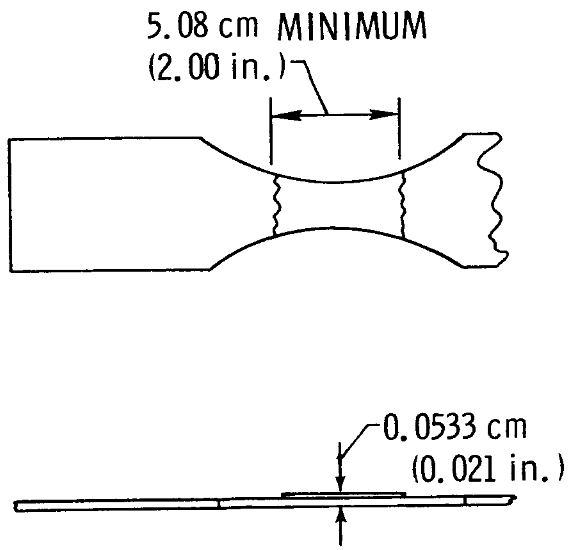

(a) Bare metal specimen.

(b) Brazed specimen with braze overlay. (Dimensions not shown are the same as those for bare metal specimen.)

Figure 1.- Tensile-test specimen configurations. R denotes radius.




(a) Bare metal specimen.




(b) Brazed specimen with braze overlay. (Dimensions not shown are the same as those for bare metal specimen.)

Figure 2.- Fatigue-test specimen configurations. R denotes radius.

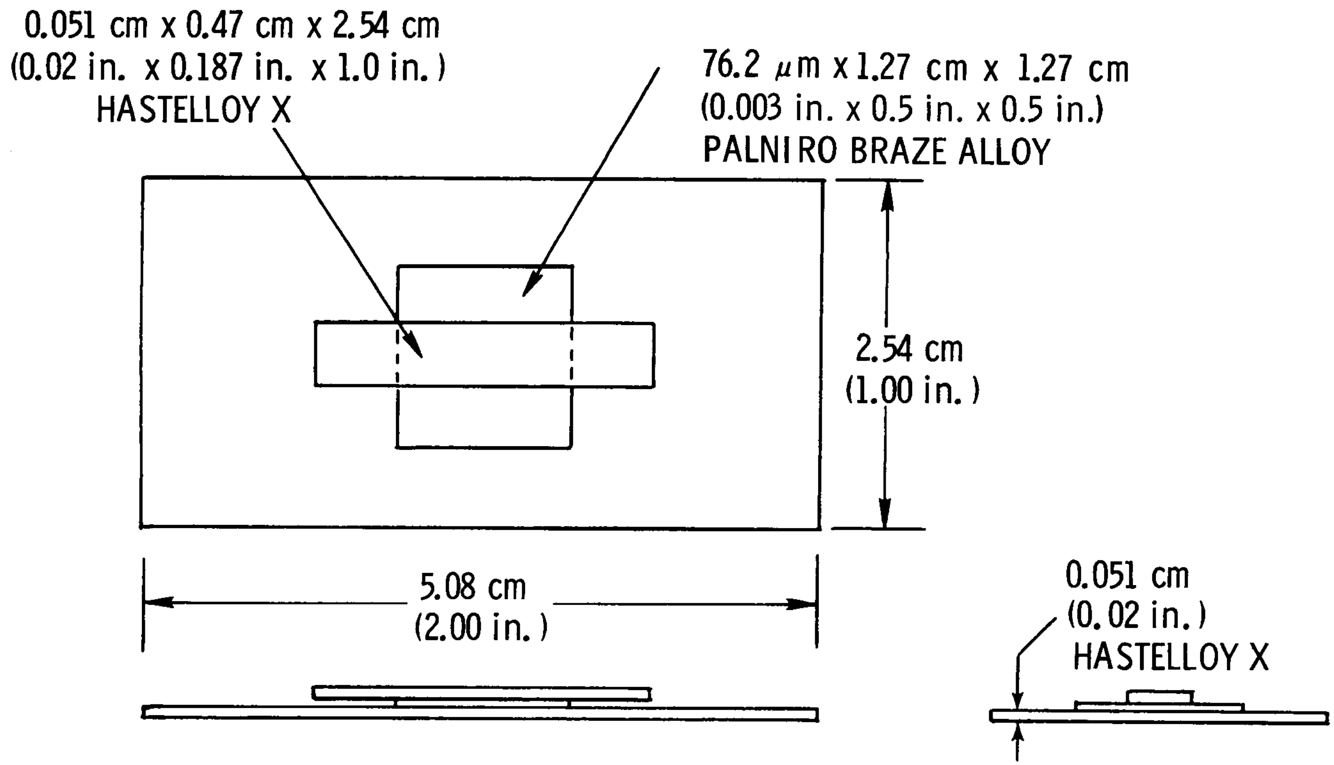



Figure 3.- Brazing alloy-base metal interaction specimen.

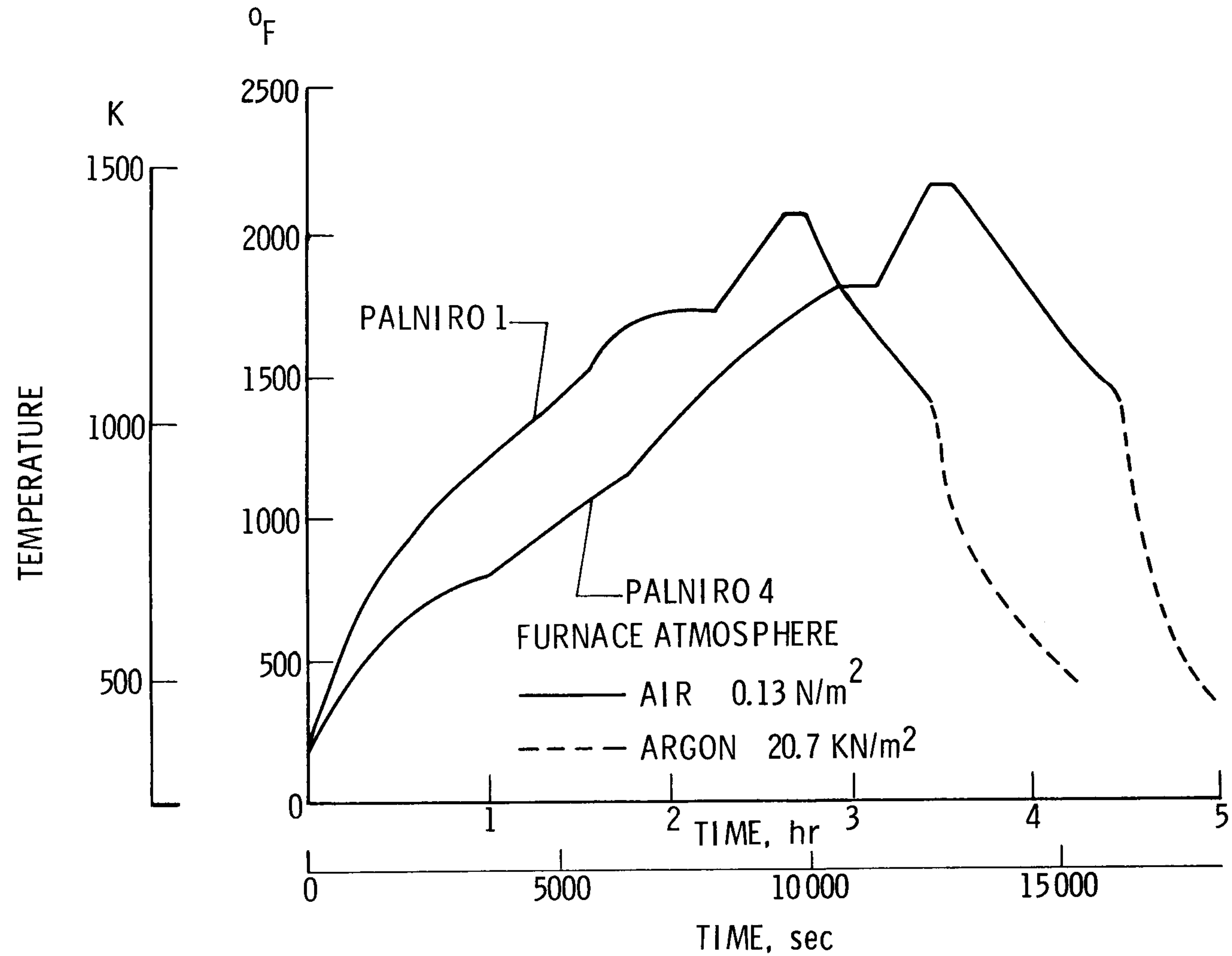



Figure 4.- Brazing heat cycles.

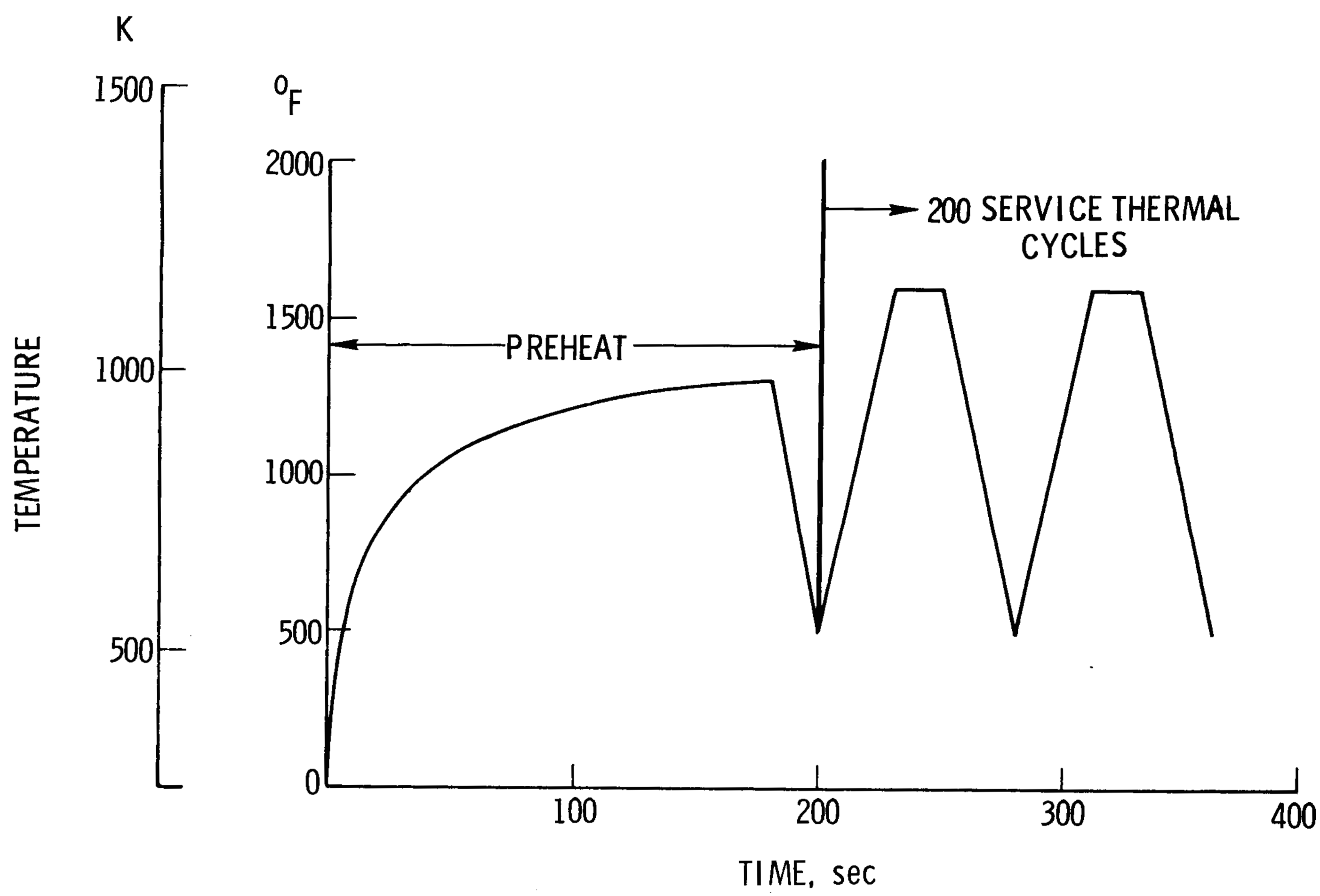



Figure 5.- Preheat and service thermal cycle.

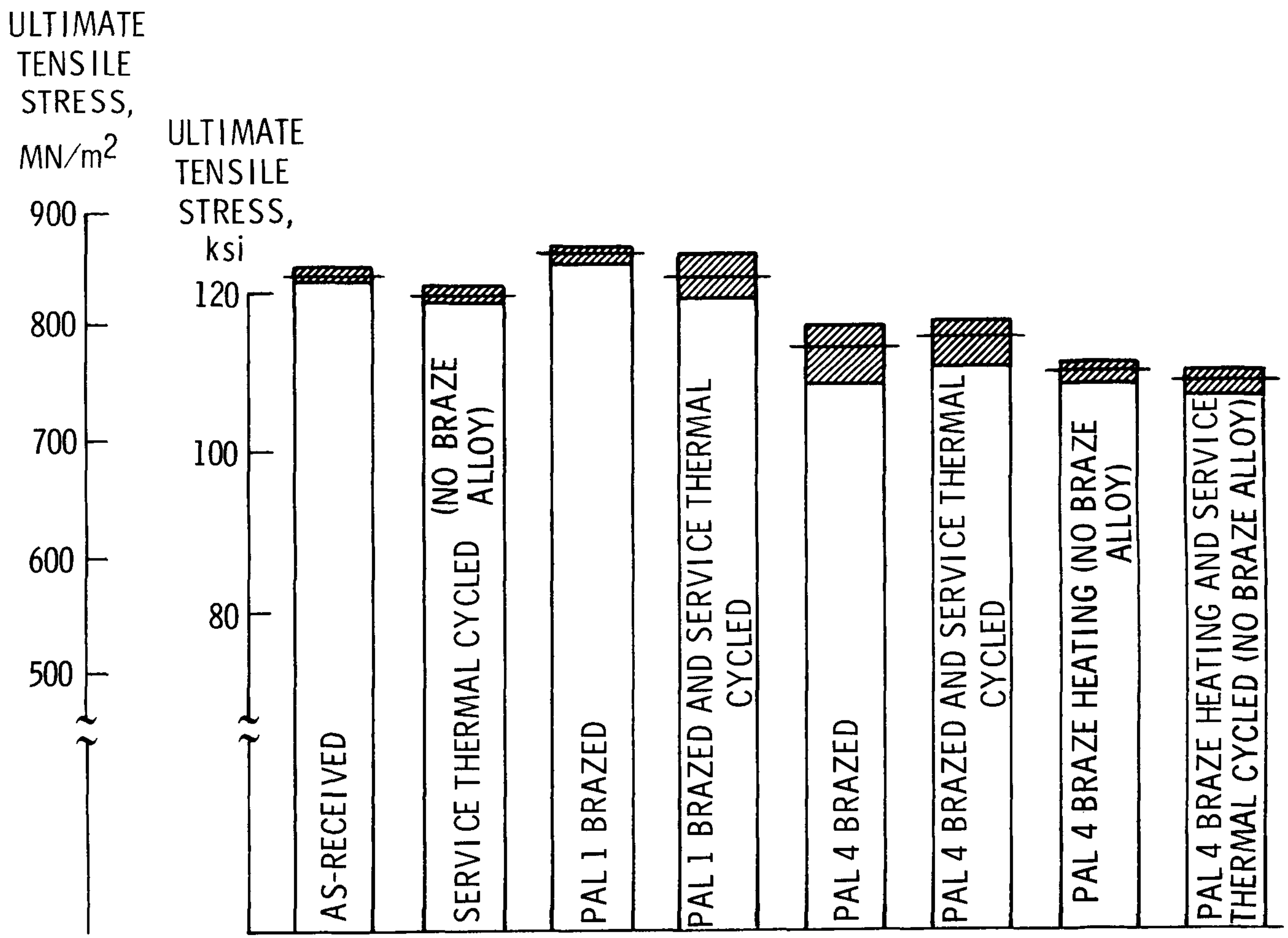
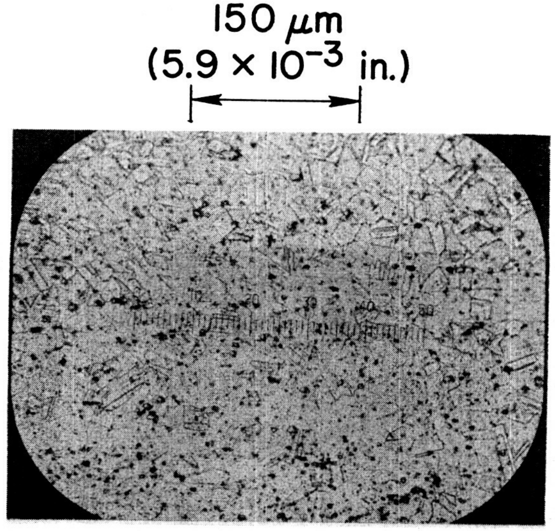
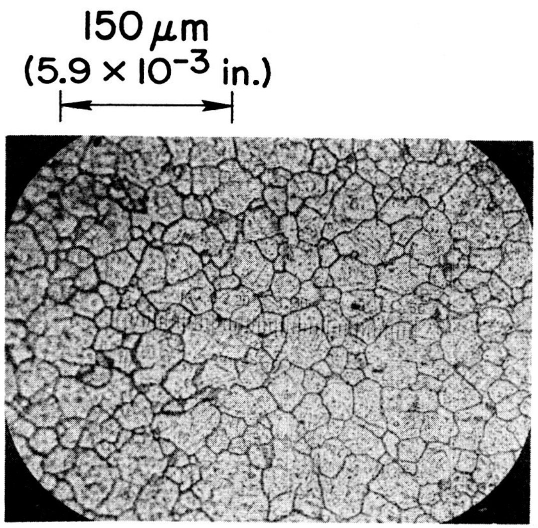


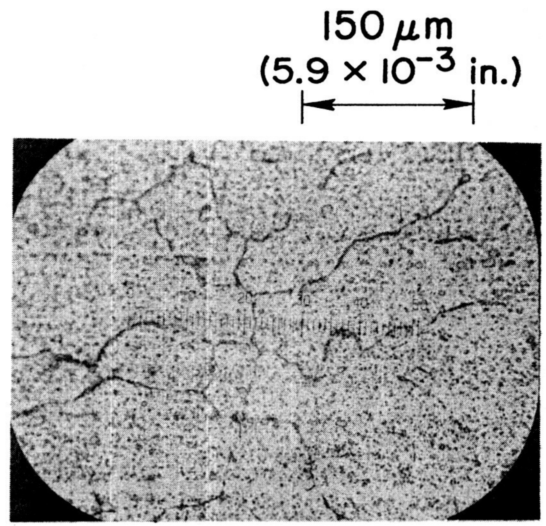

Figure 6.- Initial screening tensile tests on Hastelloy X. Pal 1 and Pal 4 are Palniro 1 and Palniro 4, respectively.




(a) Virgin Hastelloy X.

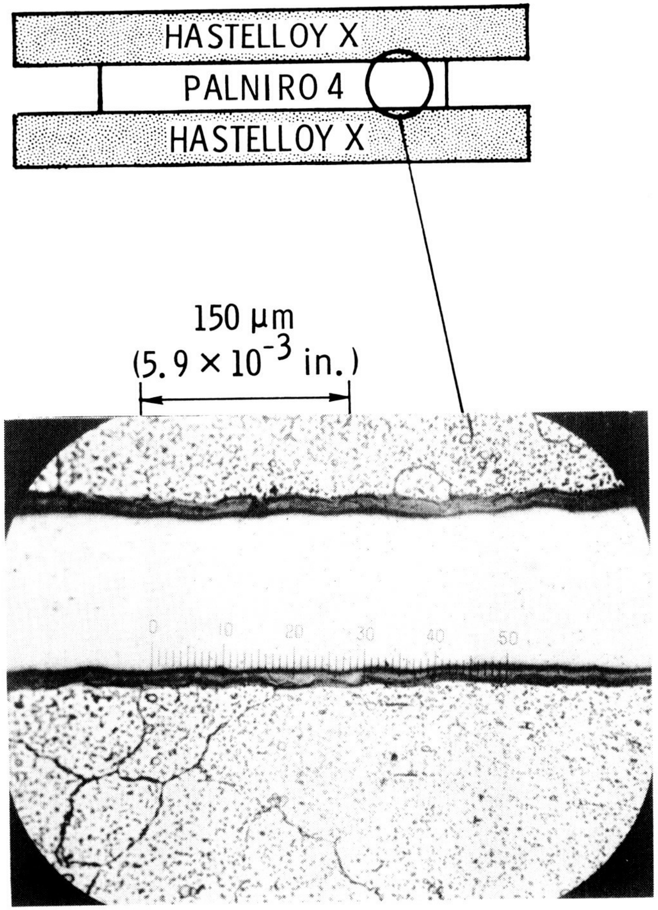



(b) After brazing with Palniro 1.

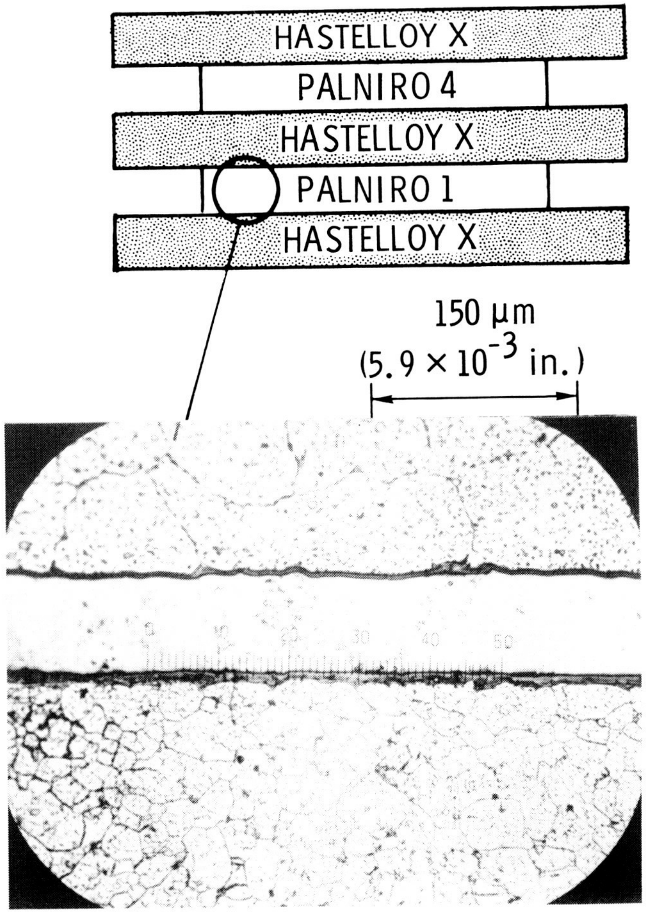



(c) After brazing with Palniro 4.

Figure 7.- Photomicrographs of virgin and as-brazed Hastelloy X.




(a) Step 1, brazed with Palniro 4.




(b) Step 2, brazed with Palniro 1.

Figure 8.- Photomicrographs of Hastelloy X – brazing alloy joints illustrating the effects of step brazing.

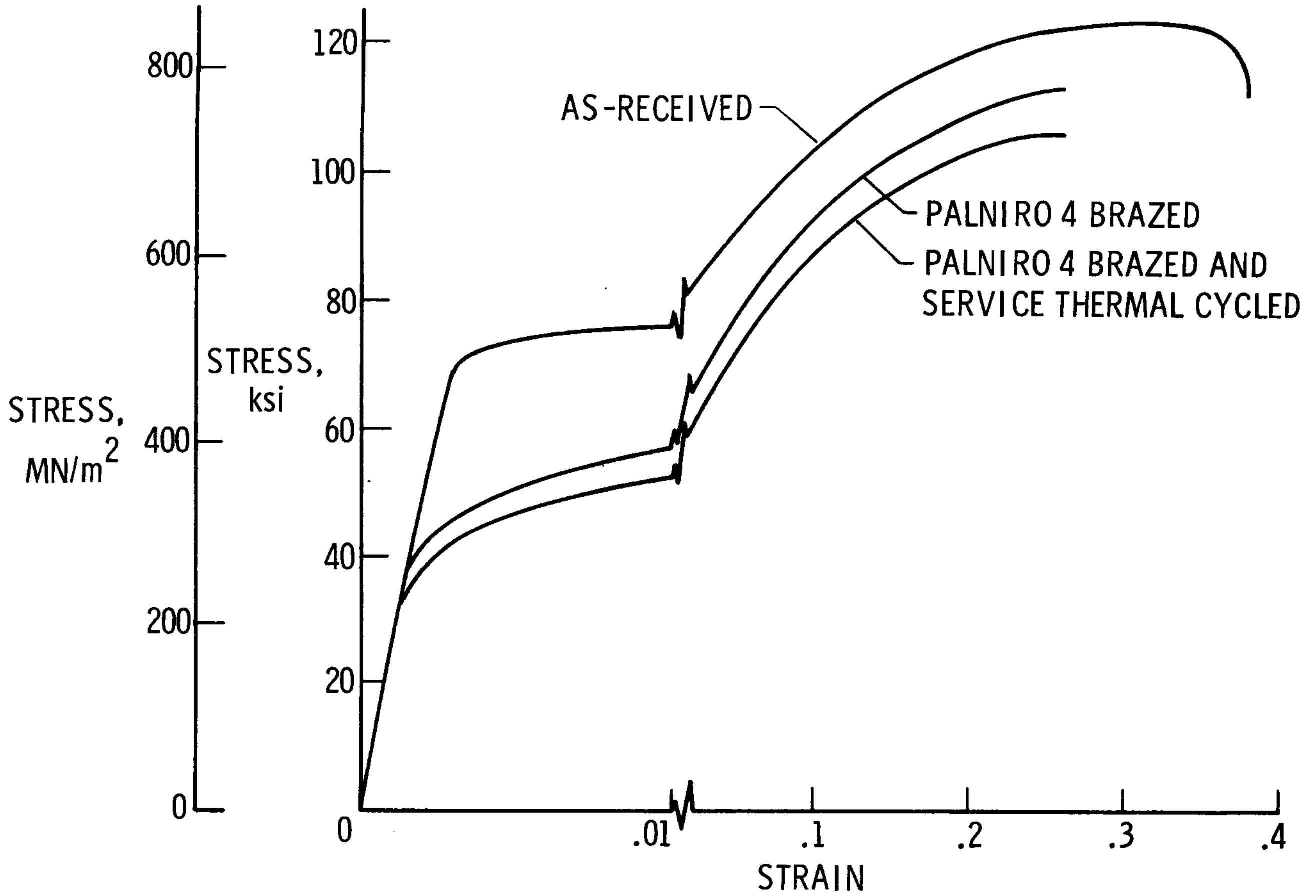
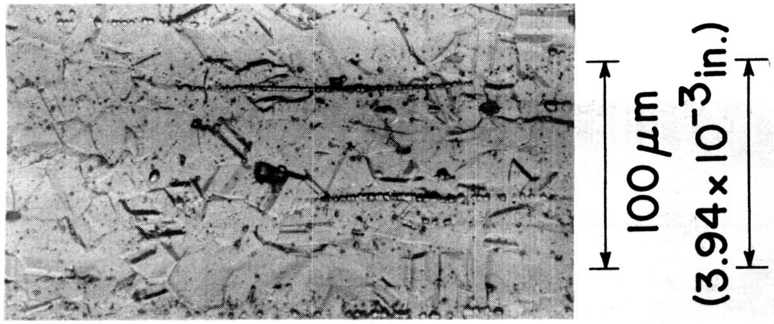
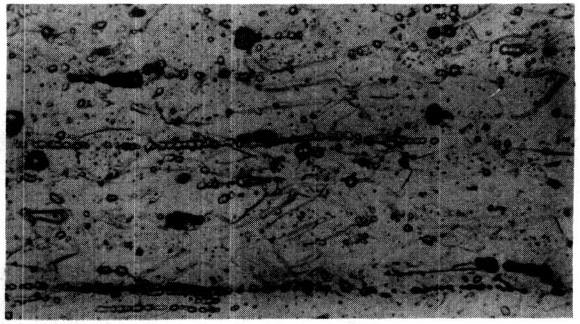


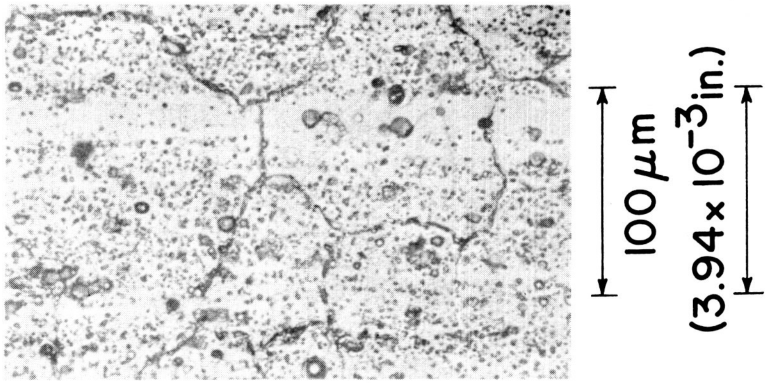

Figure 9.- Typical stress-strain diagrams for Hastelloy X.

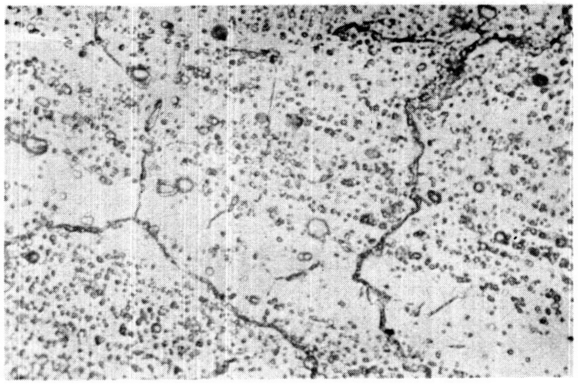


(a) As-received Hastelloy X; before tensile testing.

(b) As-received Hastelloy X; after tensile testing.

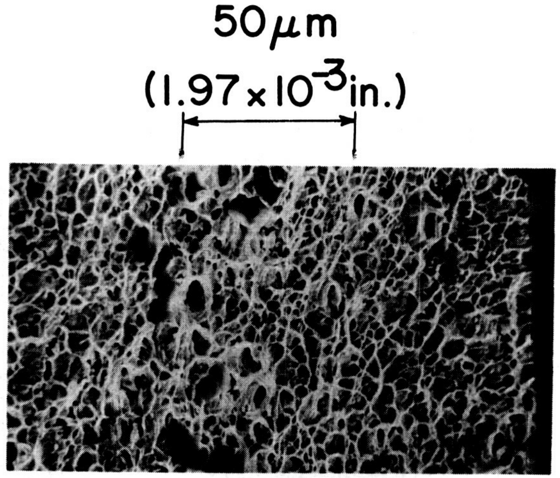


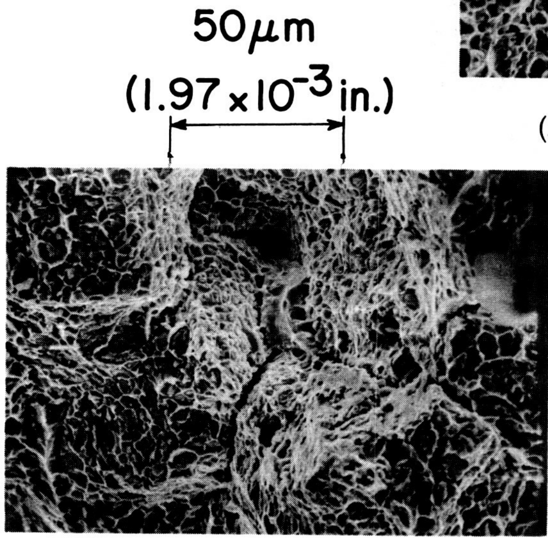
(c) Hastelloy X as-brazed with Palniro 4; after tensile testing.

(d) Hastelloy X as-brazed and service thermal cycled; after tensile testing.

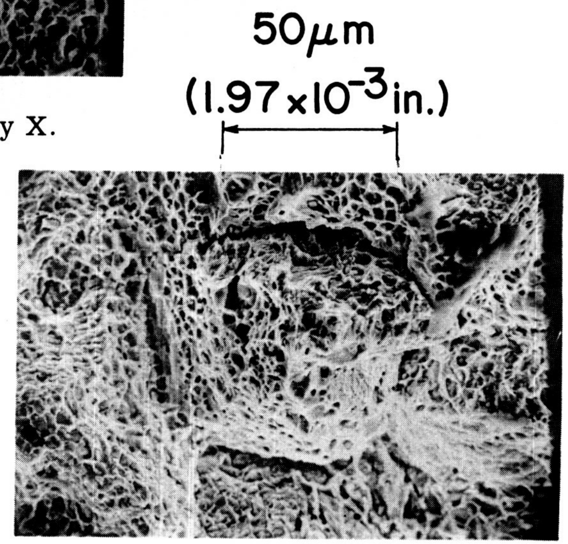
Figure 10.- Photomicrographs of Hastelloy X before and after tensile testing.




(a) As-received Hastelloy X.




(b) Hastelloy X as-brazed with Palniro 4.




(c) Hastelloy X as-brazed and service thermal cycled.

Figure 11.- Typical Hastelloy X fracture surfaces.

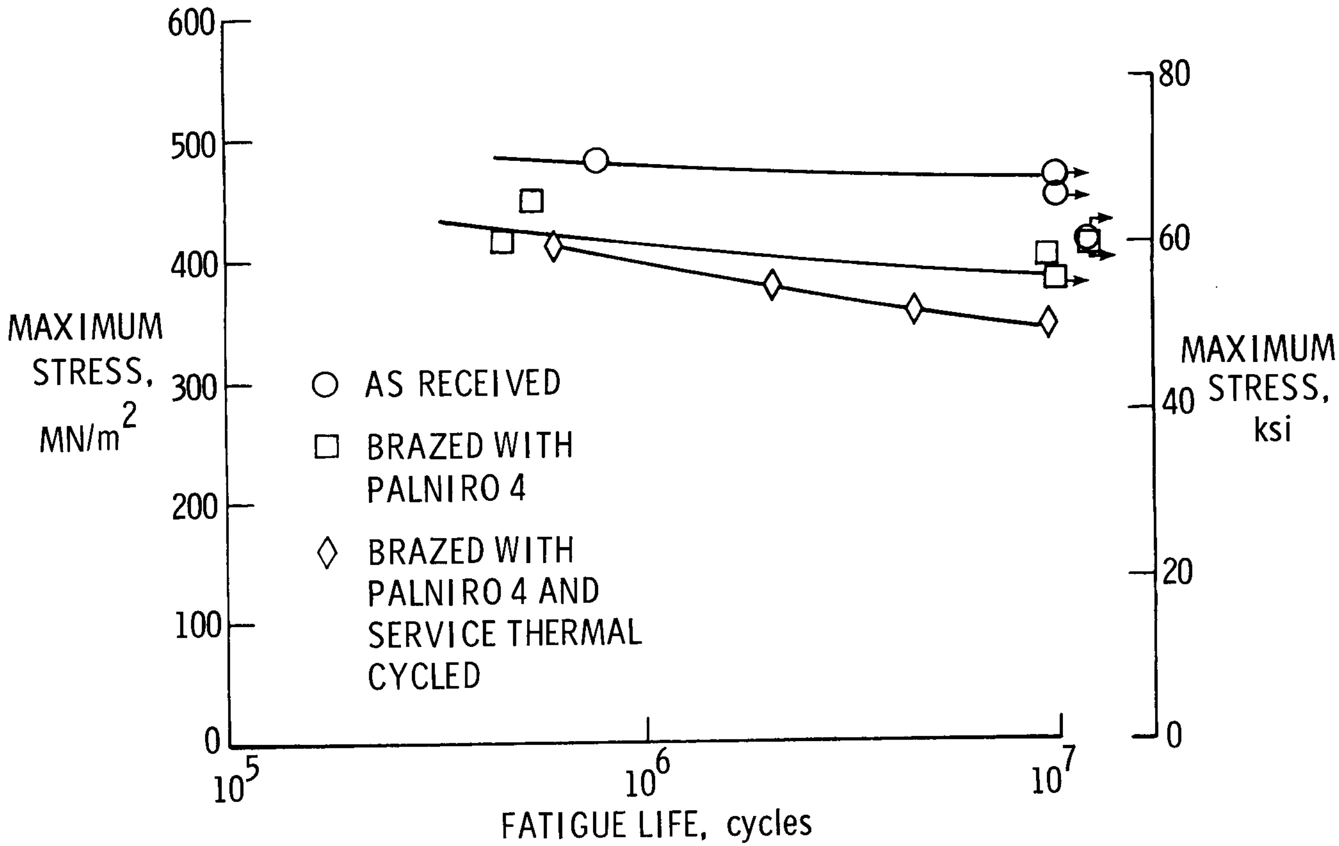



Figure 12.- Results of room-temperature fatigue tests on Hastelloy X. Ratio of minimum stress to maximum stress, 0.1.



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