

N74 30927

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ON THE MECHANICAL PROPERTIES OF HASTELLOY X

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Presented at the Symposium on Welding, Bonding, and Fastening

Williamsburg, Virginia  
May 30-June 1, 1972

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ABSTRACT

Data are presented on the effects of brazing alloy, brazing operation, thermal cycling, and combinations of these on the yield strength, elongation, tensile strength, and fatigue life of thin gage Hastelloy X. These data show that brazing at 1461 K (2170° F) with a Ni-Pd-Au alloy and subsequent exposure to 200 service thermal cycles between 533 and 1144 K (500° and 1600° F) resulted in reduction of as much as 39 percent in yield strength, 33 percent in elongation, 14 percent in tensile strength, and 26 percent in fatigue limit of Hastelloy X, as compared to as-received materials. These property losses were primarily caused by the brazing operation rather than the subsequent service thermal cycles.

INTRODUCTION

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

A step-brazing process was employed in the fabrication of the regeneratively cooled Hypersonic Research Engine (ref. 5). Test models of the engine's cooling structure, which is made of Hastelloy X,\* failed during burst tests at stress values much lower than predicted by the known mechanical properties of the metal. In addition, other models of this same structure failed prematurely during thermal cycling under stress (ref. 6). A research program was undertaken at NASA 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) brazing alloy, (2) brazing operation, and (3) simulated engine operating cycles (hereinafter referred to as service thermal cycles) on the fatigue and mechanical properties of Hastelloy X.

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\*Nickel-based superalloy manufactured by Union Carbide Corporation.

## 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 allowing the use of these alloys in a step-brazing process.

Figures 1(a) and 1(b) show the bare metal and as-brazed tensile test specimen configurations. Bare metal and as-brazed fatigue test specimen configurations are shown in figures 2(a) and 2(b). 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. Transverse and longitudinal sections were cut from tensile specimens after testing and mounted for metallographic examination. Fracture surfaces of the tensile specimens were analyzed using a scanning electron microscope.

## 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 hot-air dried. Tensile tests on cleaned specimens showed this cleaning procedure had no detrimental effects on Hastelloy X. After cleaning, a 76.2  $\mu\text{m}$  (0.003 in.) thick strip of brazing alloy was tack spot welded 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 placed on an alumina-coated carbon block and inserted into a vacuum furnace in the horizontal plane. 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 quite similar with 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 polished to reduce the thickness of the brazing alloy to approximately 25.4  $\mu\text{m}$  (0.001 in.), final machined, and deburred.

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\*Manufactured by Western Gold and Platinum Company.

Specimens to be exposed to service thermal cycling were again instrumented with thermocouples, mounted on a transite block, and lightly clamped to prevent distortion during heating. Figure 5 illustrates the service thermal cycling procedure 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° F) in 180 seconds (3 minutes) and cooled by airblast to 533 K (500° F). These specimens were then cycled 200 times between 533 and 1144 K (500° and 1600° F). Each cycle lasted 80 seconds and included a 20-second hold at the high temperature. The specimens were cooled from 1144 to 533 K (1600° to 500° F) by airblast.

Two series of tensile tests were conducted in the air at room temperature. An initial screening series covering the material conditions shown in the table below 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.

Material Conditions Examined in Initial Screening Tensile Tests
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.

Constant amplitude fatigue tests were conducted in subresonant-type axial load fatigue machines operated at a frequency of 30 Hz (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  $\pm 88\text{N}$  ( $\pm 20\text{ lb}$ ).

## 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 caused 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 is 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 than as-received grains, respectively.

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 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 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. 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 higher nickel content present in the brazing alloy. In addition, microsegregation of the gold in the brazing alloy was also indicated.\* This dissolution and migration of the nickel apparently had no effect on the ultimate tensile strength of the metal, however, since the 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.)

Additional tensile and fatigue tests were performed on Hastelloy X in the following conditions: as-received, as-brazed with Palniro 4, and as-brazed and service thermal cycled. Figure 9 shows typical stress-strain diagrams for

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\*Analysis performed by W. Barry Lisagor, NASA Langley Research Center.

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, a 35 percent decrease in yield strength, and a greater than one-third reduction in elongation to failure. Specimens subjected to service thermal cycling after brazing showed only small changes in these properties as compared to the changes resulting from the brazing operation.

Due to 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) through 10(d) show photomicrographs typical of the as-received, as-brazed with Palniro 4, and as-brazed and service thermal cycled specimens after testing. These photomicrographs showed grain growth similar to that shown in figure 7. Figures 11(a) through 11(c) show 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, whereas intragranular fracture occurred in the as-received specimen. This intergranular cracking occurred only in specimens which were actually brazed with Palniro 4 (i.e., not in specimens subjected to the Palniro 4 heating cycle alone). Consequently, selective embrittlement of the grain boundaries promoted by the thermochemical reaction of the Hastelloy X with the brazing alloy appears to have 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 metal within the grains remained ductile. This 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 indicates 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) or  $386 \text{ MN/m}^2$  (56 KSI) as compared to  $469 \text{ MN/m}^2$  (68 KSI) for the as-received specimens. Those specimens brazed and service thermal cycled had a fatigue limit of  $345 \text{ MN/m}^2$  (50 KSI). 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.

## CONCLUDING REMARKS

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 mechanical properties 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 more than one-third. 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 while 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 and 1144 K (500° and 1600° F) caused further but smaller reductions in ultimate tensile strength, yield strength, and fatigue limit of 8 percent, 4 percent, and 8 percent, respectively.

## REFERENCES

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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	Bal	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	(F)	K	(F)
Palniro 1	1375	(2016)	1394	(2050)
Palniro 4	1408	(2075)	1442	(2136)

TABLE III  
BRAZING PROCEDURES

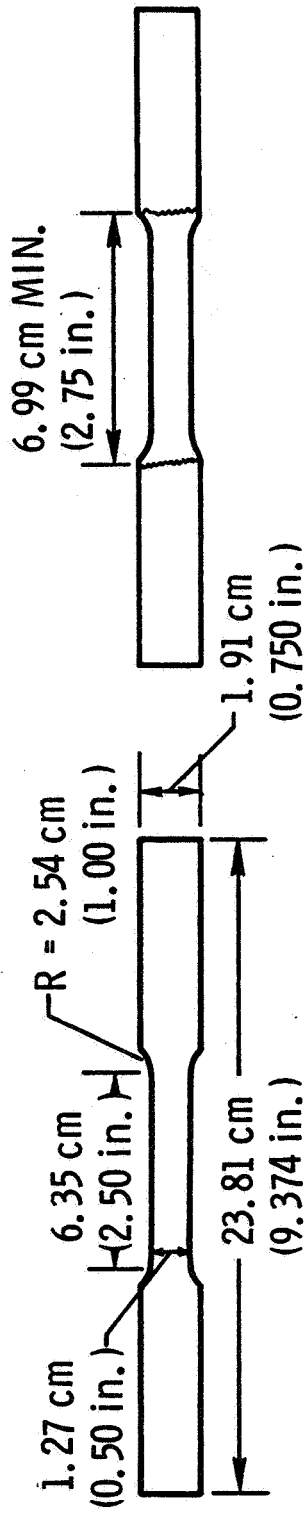
Step	Palniro 1	Palniro 4
1	Evacuate furnace to $0.13 \text{ N/m}^2$ ( $10^{-3}$ torr)	Evacuate furnace to $0.13 \text{ N/m}^2$ ( $10^{-3}$ torr)
2	Raise specimen temperature to $1222 \text{ K}$ ( $1740^\circ \text{ F}$ ) in about 2 hours	Raise specimen temperature to $1255 \text{ K}$ ( $1800^\circ \text{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

TABLE IV

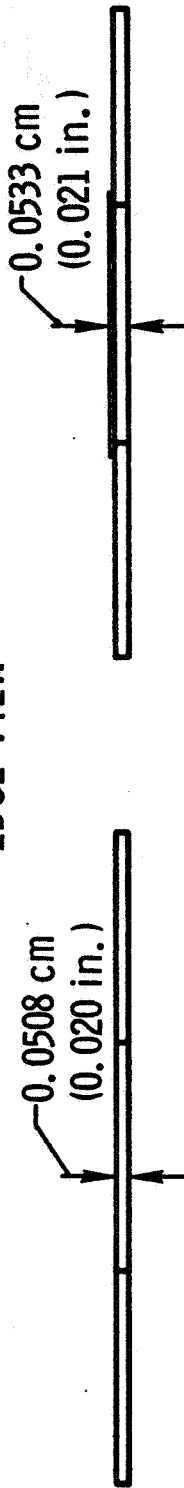
RESULTS OF TENSILE TESTS ON HASTELLOY X

Material condition	Yield strength (0.2 percent offset)		Ultimate strength		Elongation to failure, percent
	MN/m <sup>2</sup>	(KSI)	MN/m <sup>2</sup>	(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

TOP VIEW



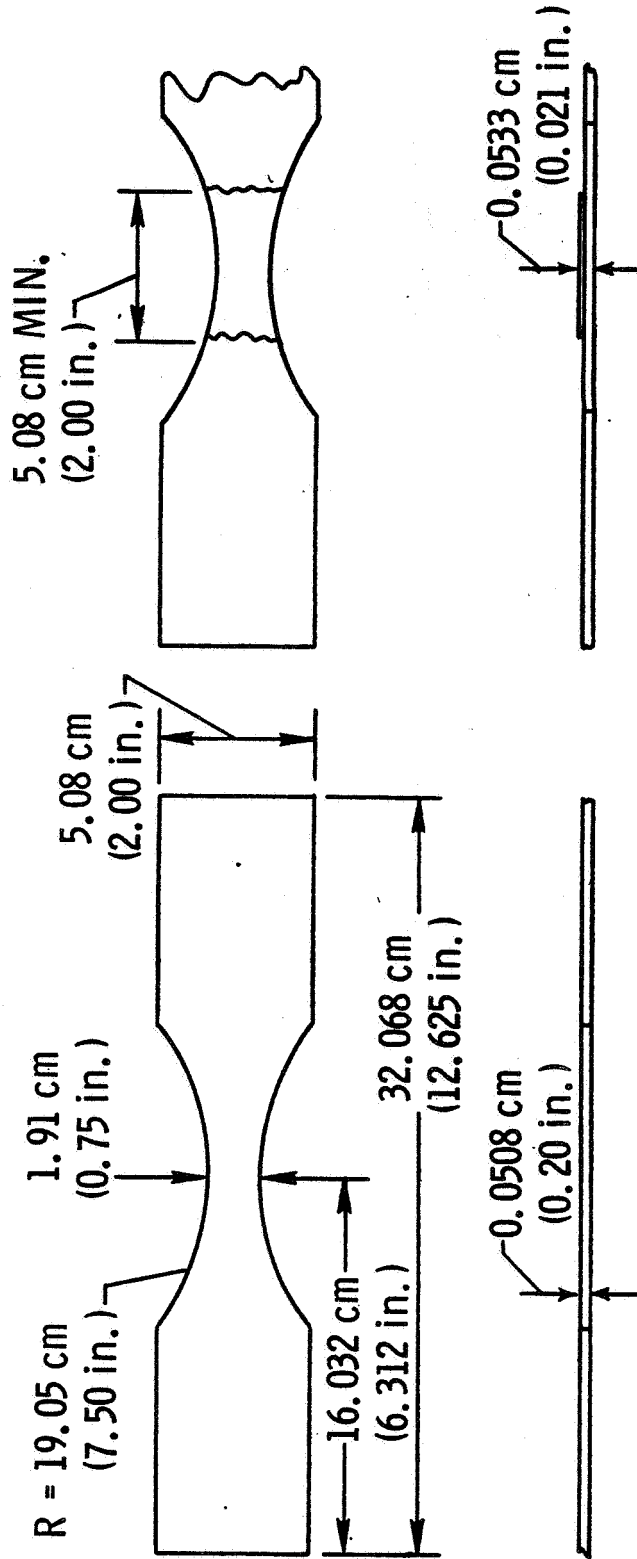
EDGE VIEW



(a) Bare metal specimen.

(b) Brazed specimen with braze overlay.  
(Dimensions not shown are same as figure (a).)

Figure 1.- Tensile test specimen configurations.



(a) Bare metal specimen.

(b) Brazed specimen with braze overlay.  
(Dimensions not shown are the same as for figure (a).)

Figure 2.- Fatigue test specimen configurations.

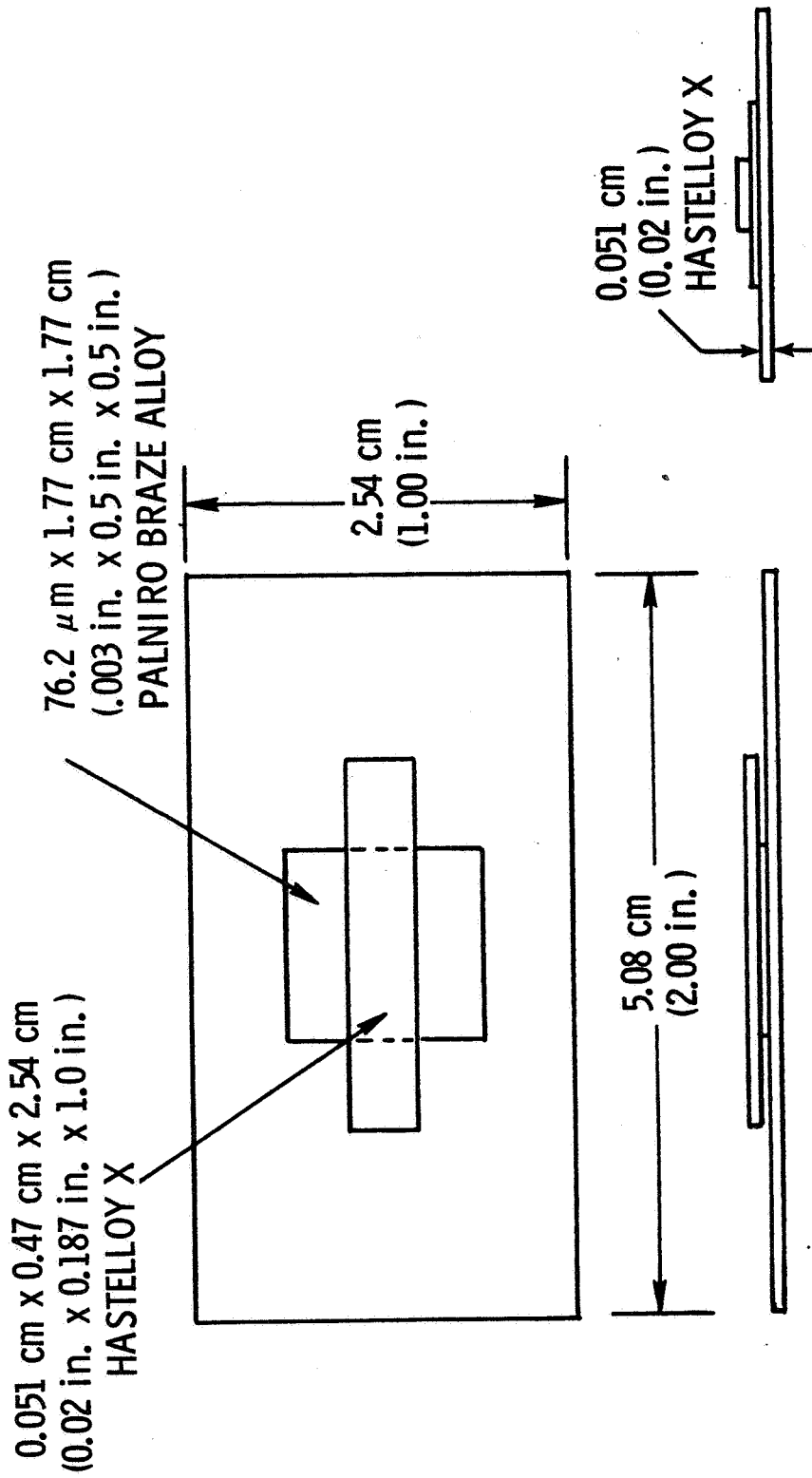


Figure 3.- Brazing alloy-base metal interaction specimen.

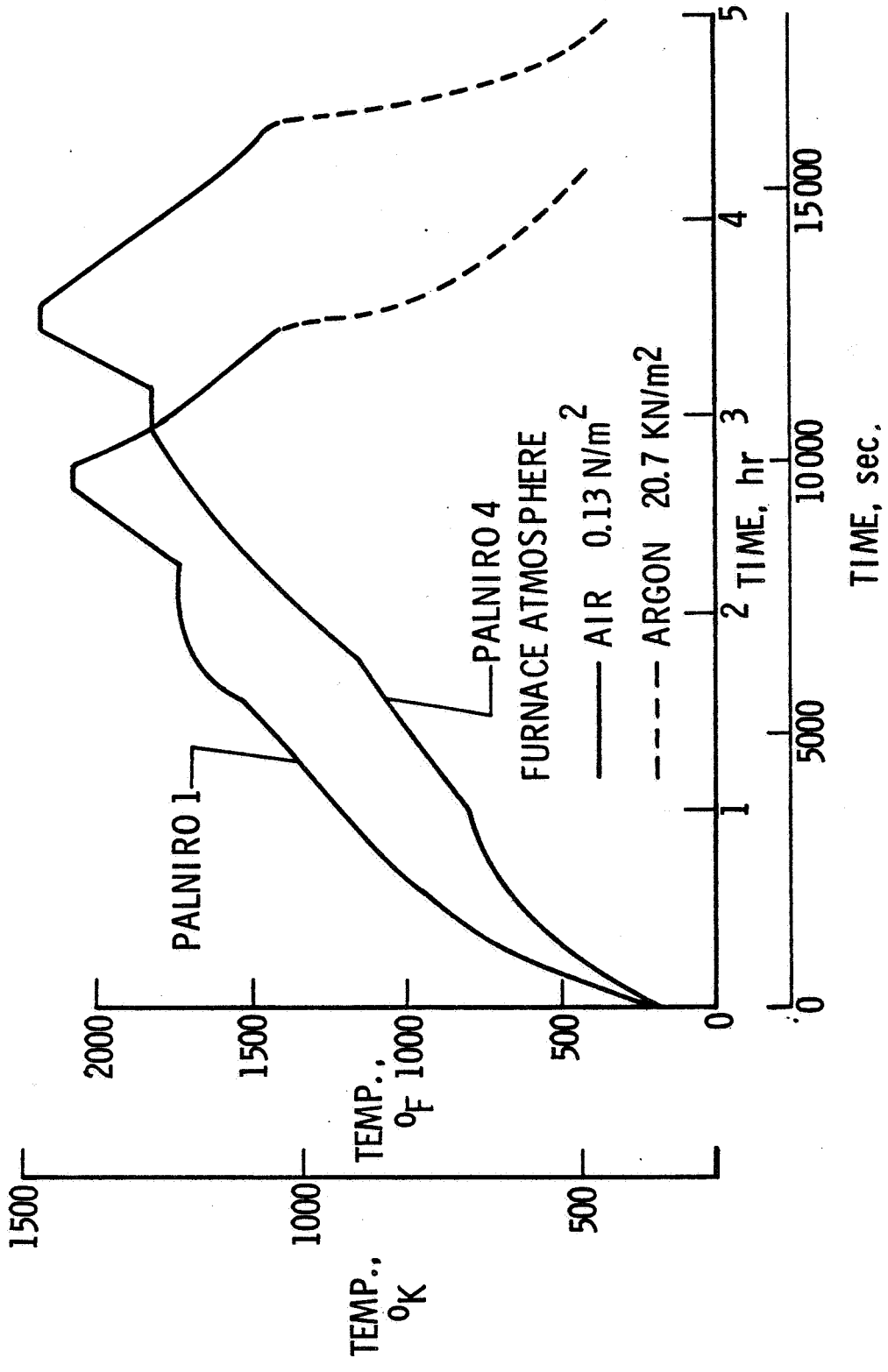


Figure 4.- Brazing heat cycles.

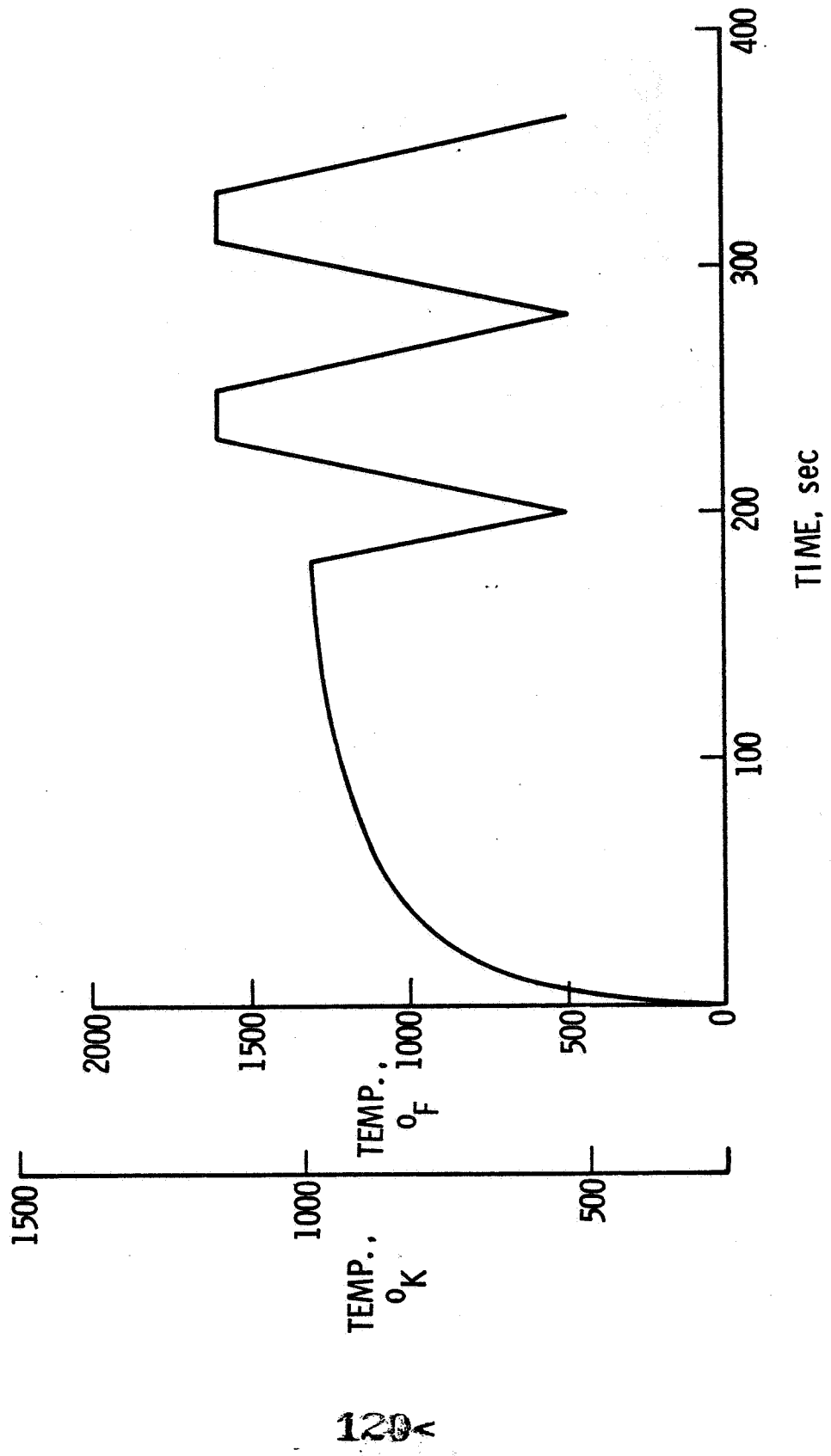


Figure 5.- Service thermal cycle.



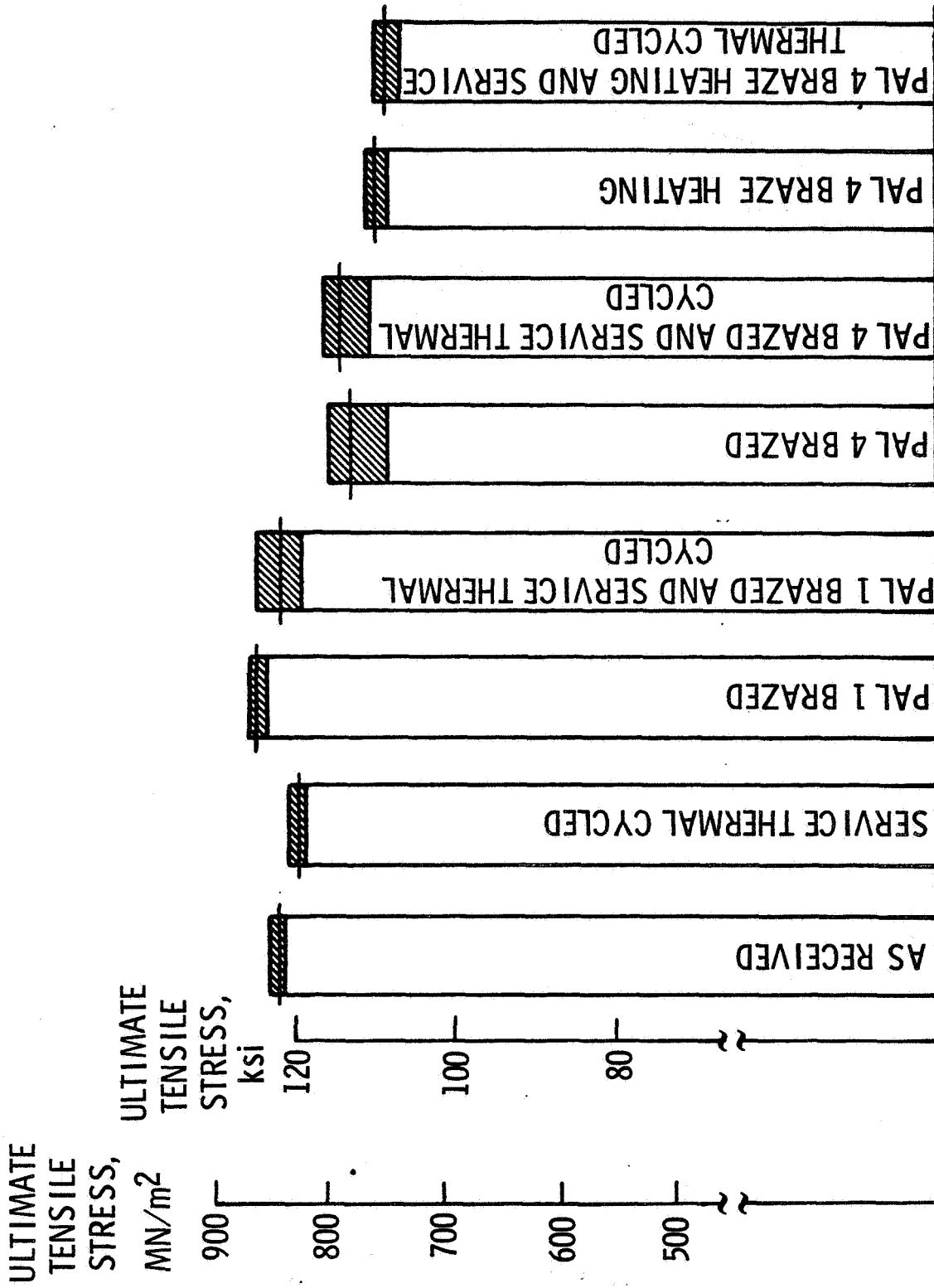
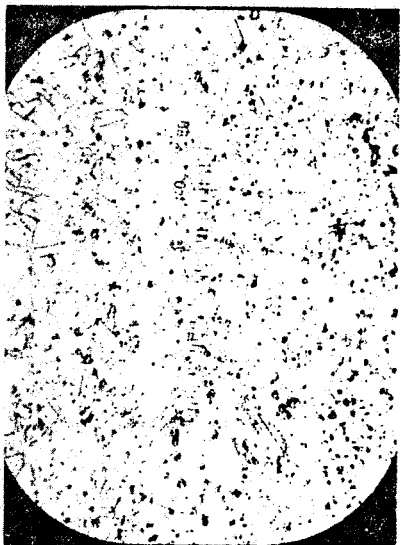


Figure 6.- Initial screening tensile tests on Hastelloy X.

150  $\mu\text{m}$   
(5.9 x 10<sup>-3</sup> in.)



(a) Virgin Hastelloy X.

150  $\mu\text{m}$   
(5.9 x 10<sup>-3</sup> in.)



(b) After brazing with Palniro 1.

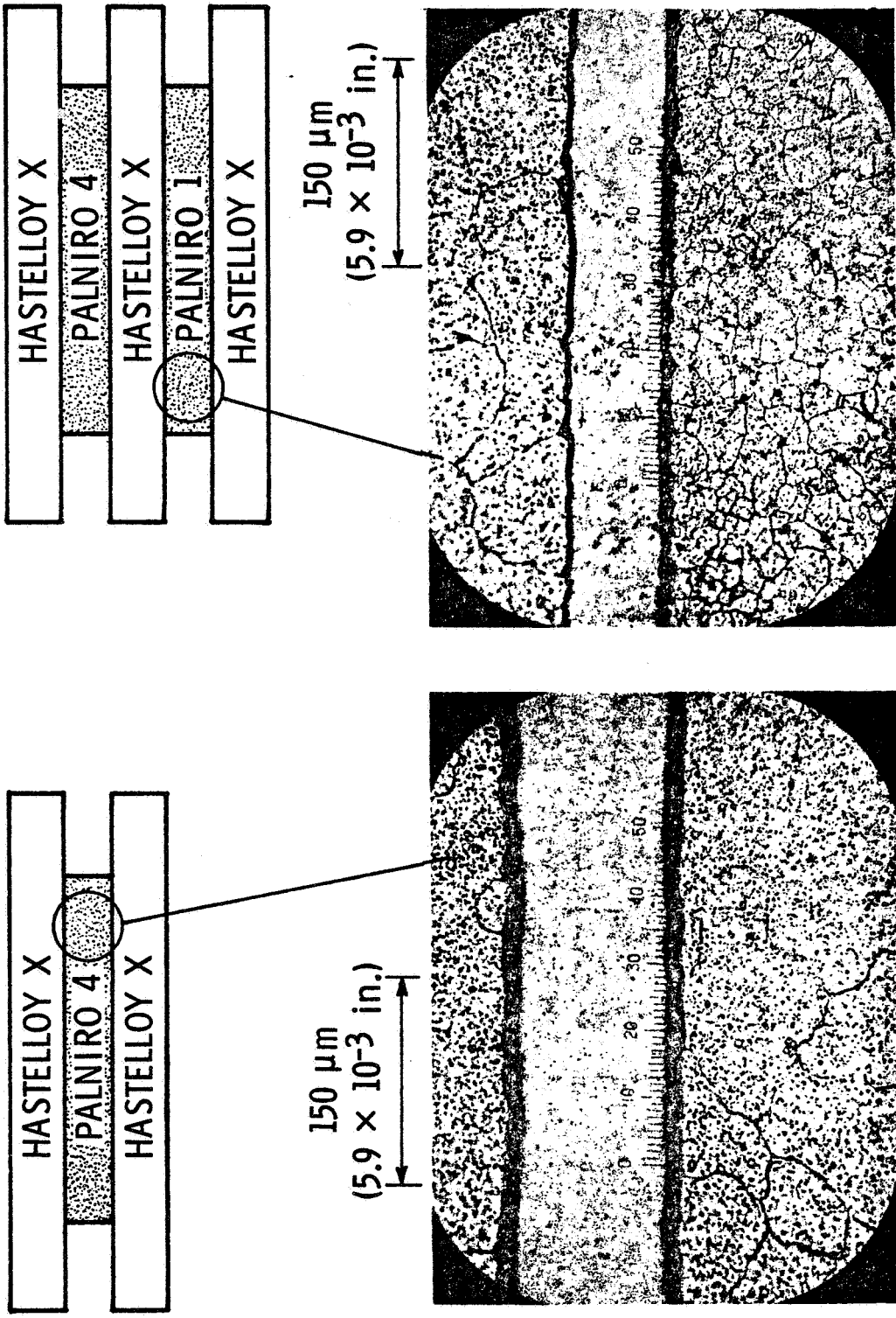
150  $\mu\text{m}$   
(5.9 x 10<sup>-3</sup> in.)



(c) After brazing with Palniro 4.

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

# HASTELLOY X - BRAZING ALLOY JOINTS ILLUSTRATING STEP BRAZING



(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.

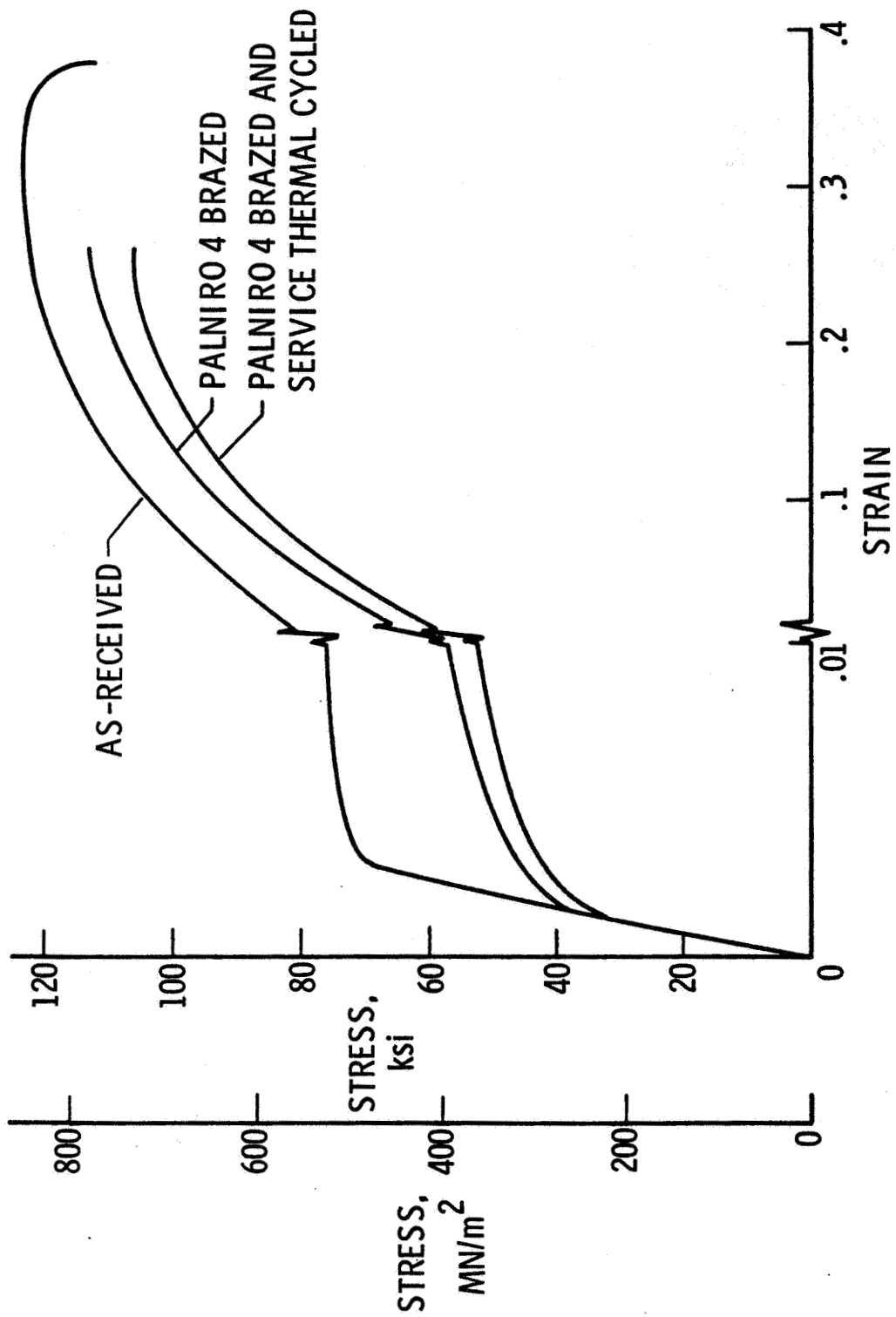
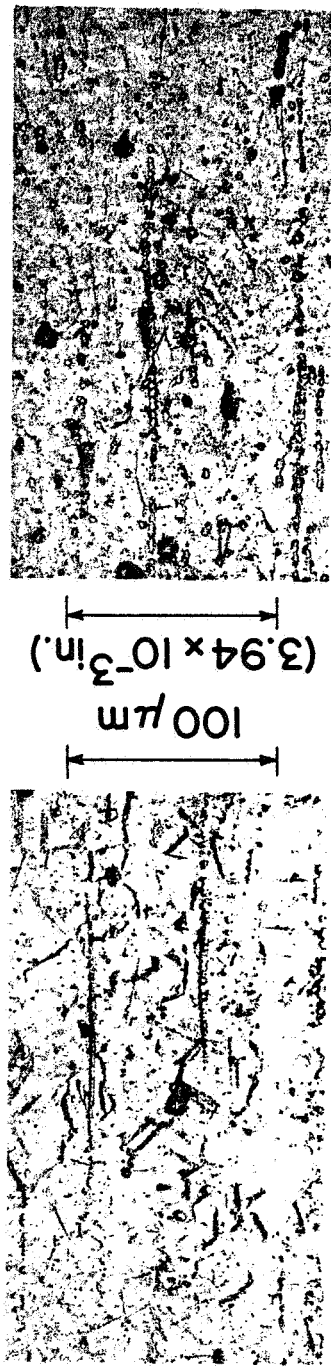


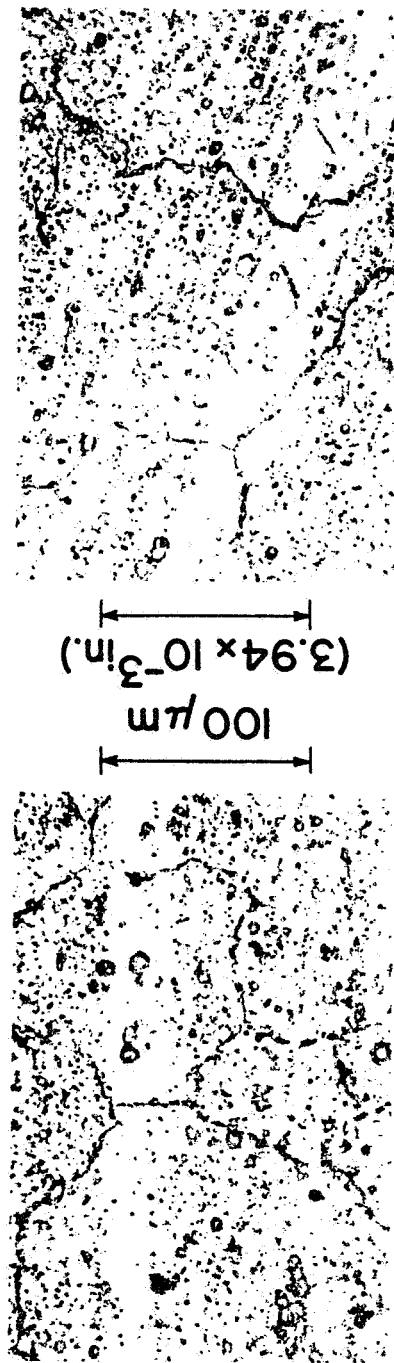
Figure 9.- Typical stress-strain diagrams for Hastelloy X.

# HASTELLOY X BEFORE AND AFTER TENSILE TESTING



(a) As-received Hastelloy X, before testing.

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



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

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

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

# TYPICAL HASTELLOY X FRACTURE SURFACES

50  $\mu$ m

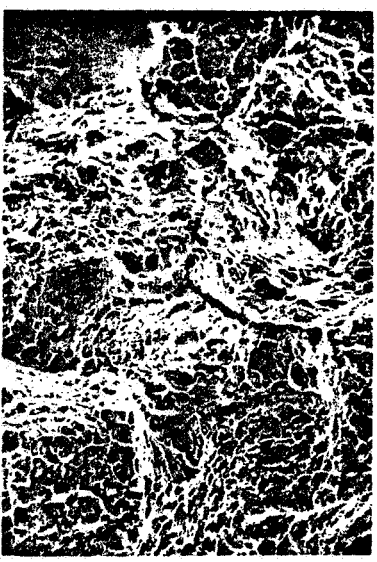
(1.97  $\times 10^{-3}$  in.)



(a) As-received Hastelloy X.

50  $\mu$ m

(1.97  $\times 10^{-3}$  in.)



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

50  $\mu$ m

(1.97  $\times 10^{-3}$  in.)



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

Figure 11.- Typical Hastelloy X fracture surfaces.

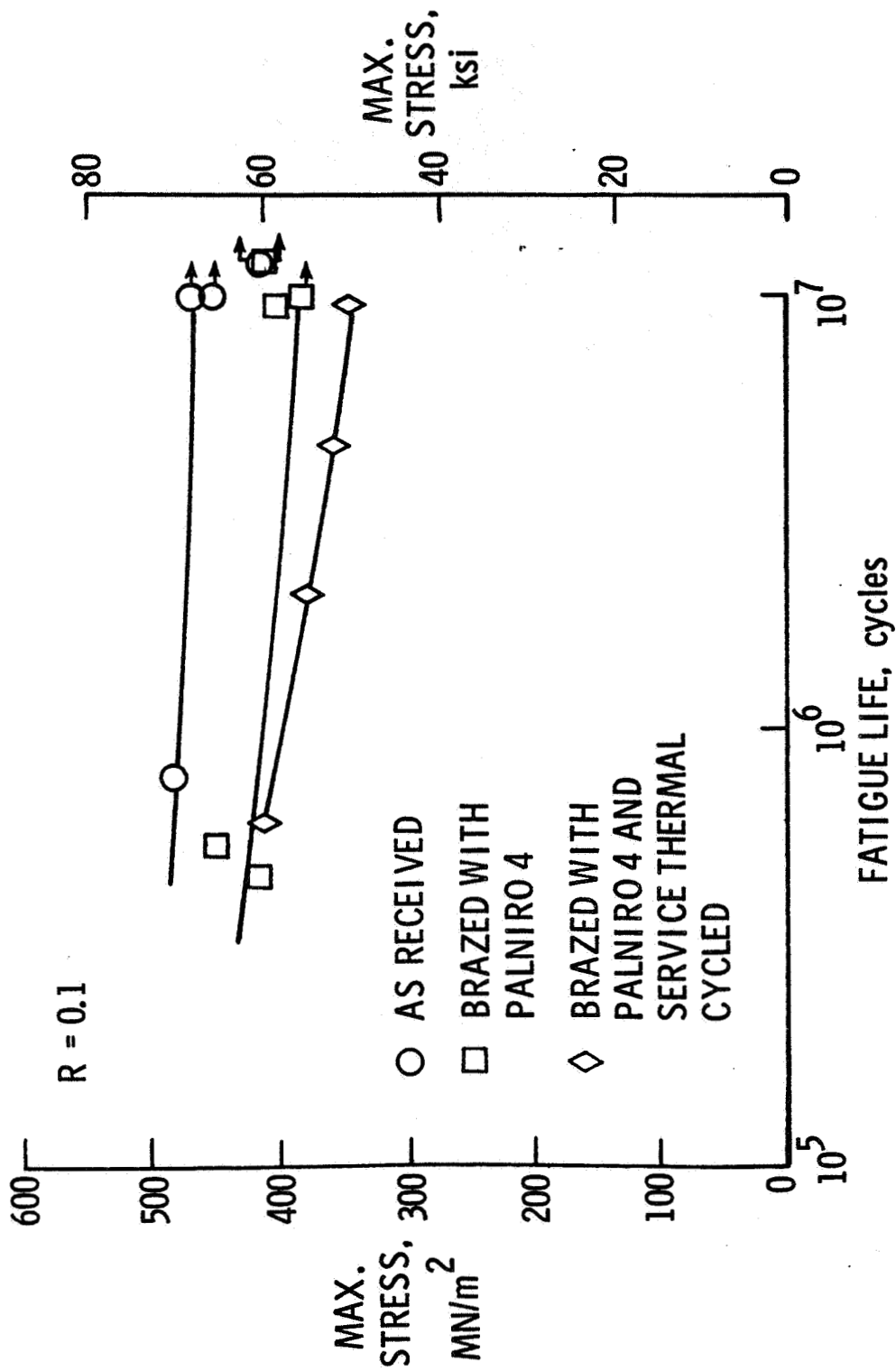


Figure 12.- Results of room temperature fatigue tests on Hastelloy X.