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Phase 1, Task C and D

Contract DAAJ02-67-C-0048

March 1968

INVESTIGATION OF MATERIALS TO RESIST HOT CORROSION IN SMALL GAS TURBINE ENGINE RECUPERATORS

Third Quarterly Report

December 1, 1967 to February 29, 1968

AiResearch Report HT-67-2512(3)

for

US Army Aviation Materiel Laboratories
Fort Eustis, Virginia

60

U.S. Army Aviation Materiel Laboratories
Fort Eustis, Va. 23604

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Fort Eustis, Virginia

SUMMARY

This program has the primary objective to conduct theoretical and experimental investigations of hot corrosion in Type 347 Stainless Steel and three other candidate materials. The term "hot corrosion" is defined herein to include oxidation, sulphidation, and carbon deposition. The investigations are to be carried out on small diameter, thin wall tubing (0.125 inches OD x 0.0035 inches wall thickness).

The initial tasks of the program, now complete, were to select base metal materials for tubing and brazing alloys for joining. The selection to be based on a theoretical analysis and evaluation of the candidate materials ability to resist hot corrosion at 1500°F.

The preliminary brazing tests, now complete except for microprobe analysis, consisted of metallographical evaluation of the brazed joints and a 100 hour static hot corrosion test. Materials selected are as follows:

Tubing	Brazing Alloy
Hastelloy X	Palniro 1 and J-8100
Incoloy 800	Palniro 7 and Coast Metals 50B
Multimet N-155	Palniro 1 and Nicrobraz 200
347 CRES	Palniro 7 and Nicrobraz 135

The cyclic-temperature hot corrosion tests, now in progress, are designed to develop stress rupture data from which two brazed base metal combinations can be selected for further testing in Phase II of the program. The test procedure and equipment is described and the initial data points are tabulated.

Approved By: E. W. Gellersen

F. E. Carroll
Chief, Heat
Transfer Systems

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PRELIMINARY BRAZING TESTS

The objective of this task is to perform preliminary evaluation of candidate brazed tube-header specimens by metallography and static hot corrosion tests for the purpose of selecting two brazing filler metals for the four tubing materials to be further evaluated under Task D.

The preliminary brazing tests and evaluation were completed (except for the microprobe analysis) and the selection of brazing alloys for Task I-D was made based on the microstructures of the brazed joints and the results of the 100 hour hot corrosion test. The selection is as follows:

TUBING	BRAZING ALLOY
Hastelloy X	Palniro I J-8100
Incoloy 800	Palniro 7 Cost Metals 50B
N-155	Palniro I Nicrobraz 200
347 CRES	Palniro 7 Nicrobraz 135

Evaluation of Brazed Tube-Header Joints

Metallographical evaluation of the brazed joints was completed and the results are summarized in Tables I through V. Photomicrographs illustrating the brazing characteristics at each brazing temperature for all of the combinations of candidate brazing alloys and tubing materials are presented in Figures 1 through 20. N-155 and Hastelloy X had the best brazing characteristics for the brazing alloys used followed closely by Incoloy 800 and CRES 347. Although the Incoloy 800 had a dull surface appearance after brazing caused by oxidation, it still brazed fairly well. Inconel 625 had a surface appearance similar to that of Incoloy 800. Wetting and filleting, however, was poorer on Inconel 625.

Evaluation of Static Hot Corrosion Test Results

The one-inch long tube-header samples were sectioned longitudinally and their microstructures examined for the effects of the static hot corrosion test. Photomicrographs of the brazed joints in the unetched condition are shown in Figure 21 through 25 inclusive. These show the resistance of the various tube material/filler metal combinations to the corrosive atmospheres at 1500°F for 100 hours.

TABLE I. BRAZING CHARACTERISTICS OF HASTELLOY X TUBE/HEADER JOINTS
TASK I-C

BRAZING ALLOY	BRAZING TEMPERATURE °F	VISUAL OBSERVATIONS(1)			MICRO(2)	
		SURFACE CONDITION	WETTING (FLOW)	FILLETING	ALLOYING INTO TUBE WALL, %	PENETRATION MILS
Palniro 4	2200	C	E	E	80	1/4
	2175	C	E	E	60	1/2
	2150	C	E	E	20	1/4
J-8100	2175	C	E	E	100	—
	2150	C	E	E	40	1/2
	2125	C	E	E	40	1/2
Palniro 1	2100	C	E	E	75	1/4
	2075	C	E	E	10	1/4
	2050	C	E	E	10	1/4
Nicrobraz 135	2075	C	G	E	70	1
	2000	C	G	G-E	60	1
	1975	C	G	G	50	1

(1) C = Clean
G = Good
E = Excellent

(2) Example of measurements for brazed tube/header joint.

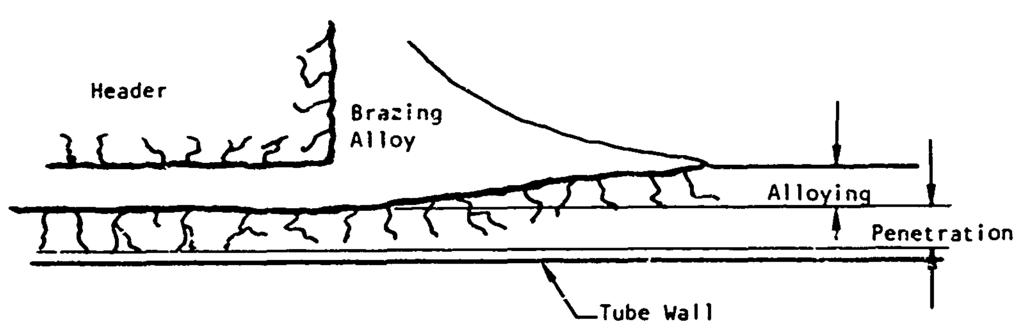


TABLE II. BRAZING CHARACTERISTICS OF INCONEL 625 TUBE/HEADER JOINTS TASK I-C

BRAZING ALLOY	BRAZING TEMPERATURE °F	VISUAL OBSERVATIONS (1)			MICRO	
		SURFACE CONDITION	WETTING (FLOW)	FILLETING	ALLOYING INTO TUBE WALL, %	PENETRATION MILS
J-8100	2175	D	G	F	100	None
	2150	D	G	P-F	70	"
	2125	D	G	P	60	"
Engelhard 135	2125	D	F	F-G	Slight	"
	2100	D	F	F	"	"
	2075	D	F	F	"	"
Nicrobraz 135	2025	D	P-F	P	(2)	(2)
	2000	D	P-F	P	None	1
	1975	D	P	P	None	1
Nicrobraz 65	1975	D	G	G	20	None
	1950	D	F	G	20	"
	1925	C	P	P	20	"

(1) C = Clean
D = Dull (oxide film)
E = Excellent
G = Good
F = Fair
P = Poor

(2) Inadequate brazing alloy in joint

TABLE III. BRAZING CHARACTERISTICS OF INCOLOY 800 TUBE/HEADER JOINTS
TASK I-C

BRAZING ALLOY	BRAZING TEMPERATURE °F	VISUAL OBSERVATIONS(1)			MICRO	
		SURFACE CONDITION	WETTING (FLOW)	FILLETING	ALLOYING INTO TUBE WALL, %	PENETRATION MILS
Nicrobraz 135	2025	D	G	E	50	None
	2000	C	G	E	20	"
	1975	D	F-G	G	20	"
Nicrobraz 65	1975	C	G	F	10	"
	1950	D	G	G	None	"
	1925	D	P	P	None	"
Palniro 7	1975	D	F	G	10	"
	1950	D	G	G	10	"
	1925	D	G	E	20	"
Coast Metals 50B	2075	D	G	G	50	"
	2050	D	G	G	40	"
	2025	D	G	G	30	"

(1) C = Clean
D = Dull (oxide film)
E = Excellent
G = Good
P = Poor

TABLE IV. BRAZING CHARACTERISTICS OF MULTIMET N155 TUBE/HEADER JOINTS
TASK I-C

BRAZING ALLOY	BRAZING TEMPERATURE °F	VISUAL OBSERVATIONS[1]			MICRO	
		SURFACE CONDITION	WETTING (FLOW)	FILLETING	ALLOYING INTO TUBE WALL, %	PENETRATION MILS
Palniro 4	2200	C	E	E	80	1/2
	2175	C	E	E	60	1/2
	2150	C	E	E	20	1/2
J-8100	2175	C	E	E	50	1/4
	2150	C	E	E	50	1/4
	2125	C	E	E	50	1/4
Palniro 1	2100	C	E	E	70	1/4
	2075	C	E	E	30	1/4
	2050	C	E	E	20	1/4
Nicrobraz 200	1975	C	G	G-E	15	Slight
	1950	C	G-E	G-E	10	Slight
	1925	C	G	G-E	5	Slight

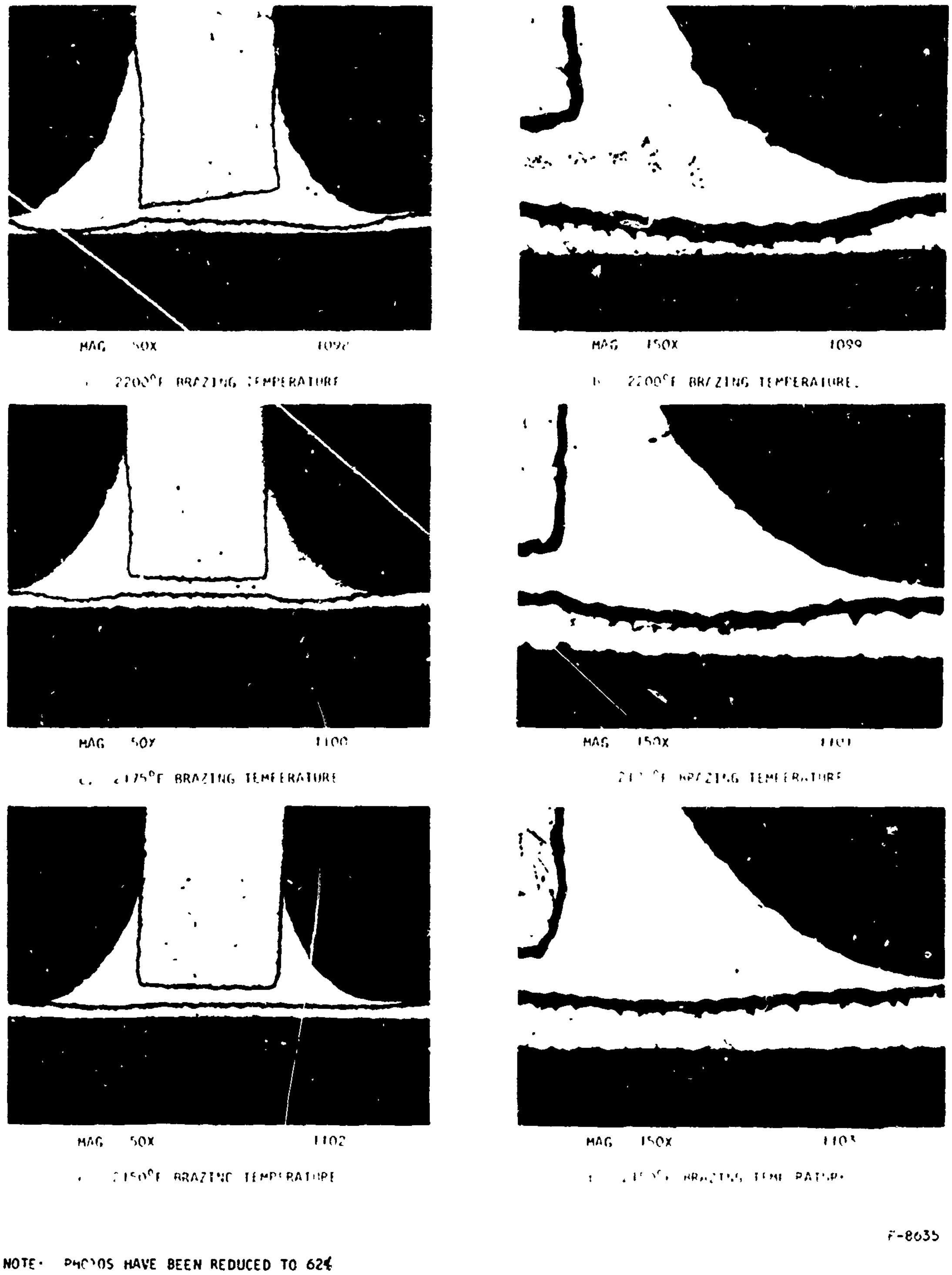
(1) C = Clean
G = Good
E = Excellent

TABLE V. BRAZING CHARACTERISTICS OF CRES 347 TUBE/HEADER JOINTS
TASK I-C

BRAZING ALLOY	BRAZING TEMPERATURE °F	VISUAL OBSERVATIONS[1]			MICRO	
		SURFACE CONDITION	WETTING (FLOW)	FILLETING	ALLOYING INTO TUBE WALL, %	PE[illegible]ATION [illegible]LS
Nicrobraz 135	2025	D	G	G	30	2-1/2 (2)
	2000	D	G	C	30	2-1/2 (2)
	1975	C	E	G	30	2-1/2 (2)
Nicrobraz 65 (AMI 930)	1975	C	E	G	10	None
	1950	C	G	G	Slight	None
	1925	Brazing alloy did not melt at this temperature				
Palniro 7	1975	C	E	E	25	None
	1950	C	G	G	20	1/2
	1925	C	G	G	25	None
Coast Metals 50B	2075	D	G	G	60	1-1/2 (2)
	2050	D	G	G	30	2-1/2 (2)
	2025	D	G	G	10	3 (2)

(1) C = Clean
D = Dull (oxide film)
E = Excellent
G = Good
P = Poor

(2) Penetrated through wall



NOTE: PHOTOS HAVE BEEN REDUCED TO 62%

Figure 1. Photomicrographs of Hastelloy X Tube-Header Joints Brazed with Palniro 4 Brazing Alloy, in Vacuum, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.

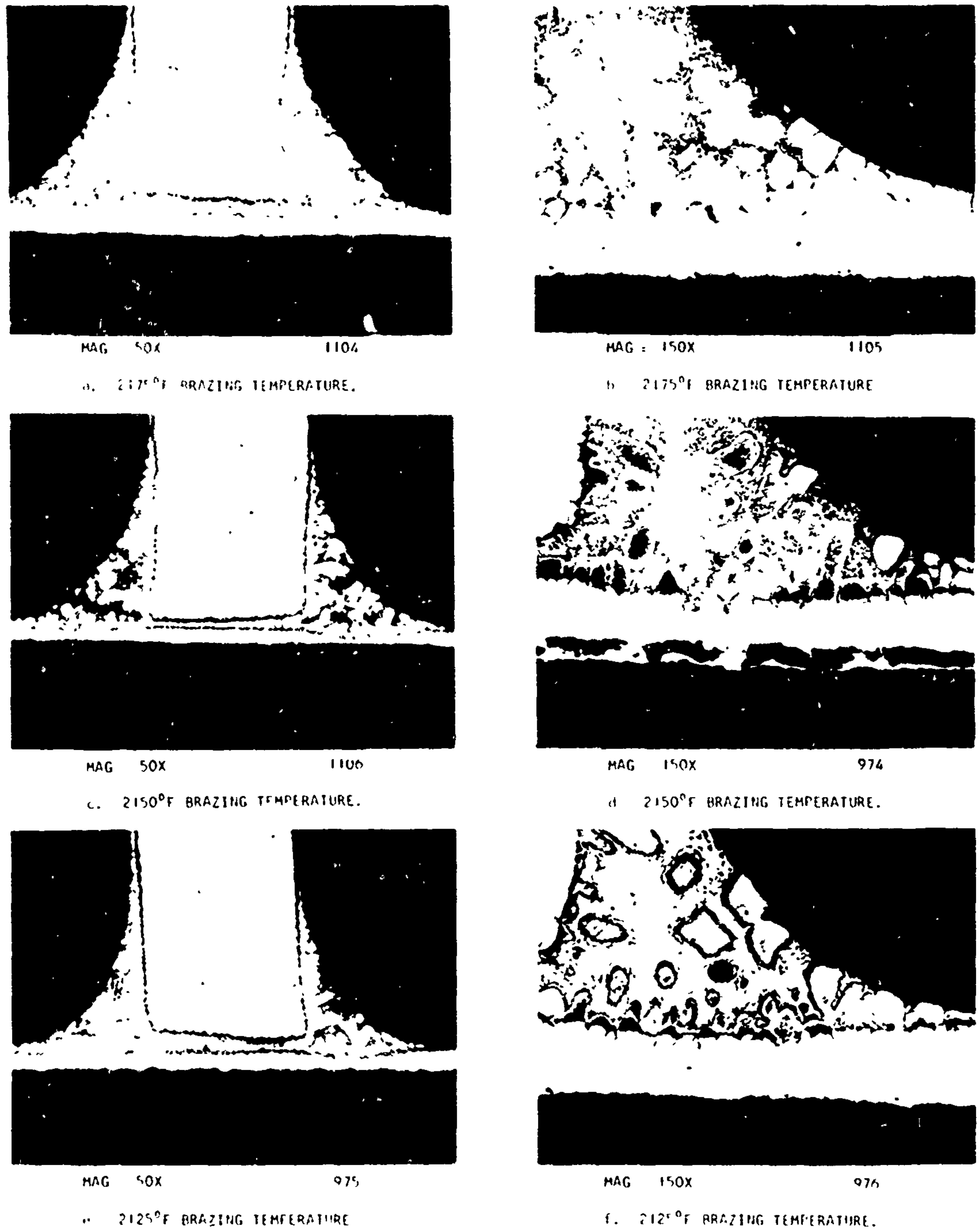
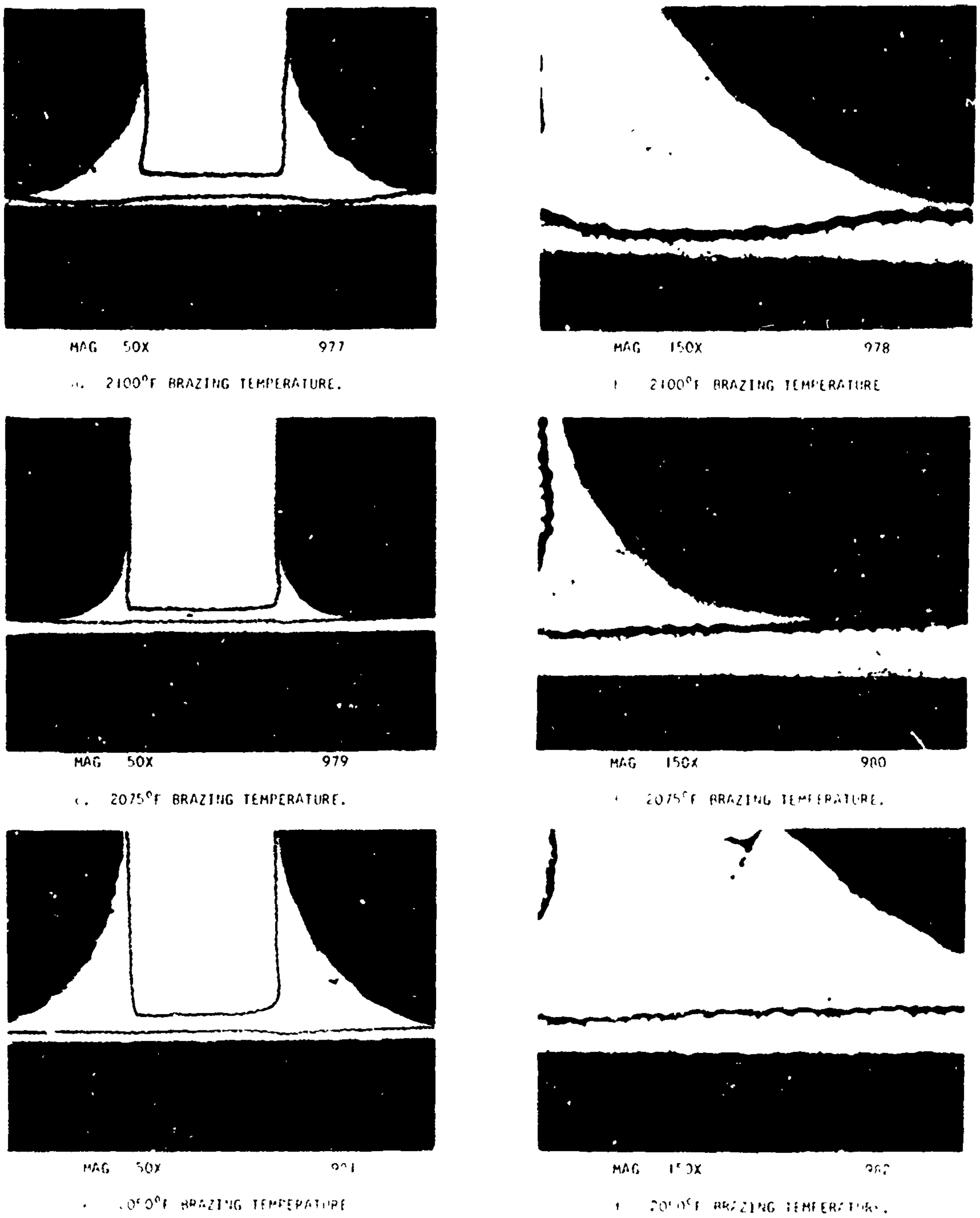


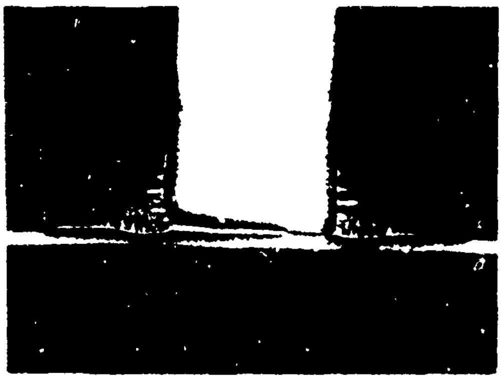
Figure 2. Photomicrographs of Hastelloy X Tube-Header Joints Brazed with J-8100 Brazing Alloy, in Vacuum, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.



F-8632

NOTE: PHOTOS HAVE BEEN REDUCED TO 65%

Figure 3. Photomicrographs of Hastelloy X Tube-Header Joints Brazed with Palniro I Brazing Alloy, in Vacuum, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.

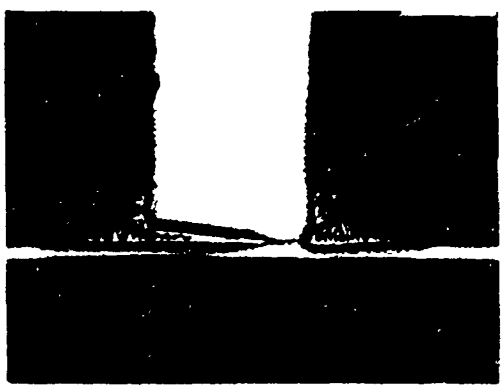


MAG = 50X 933

a. 2025°F BRAZING TEMPERATURE.

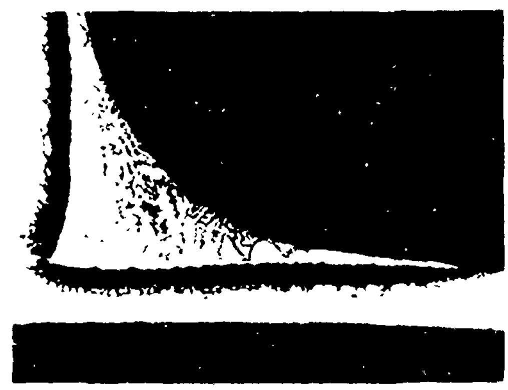
MAG - 150X 984

b. 2025°F BRAZING TEMPERATURE.



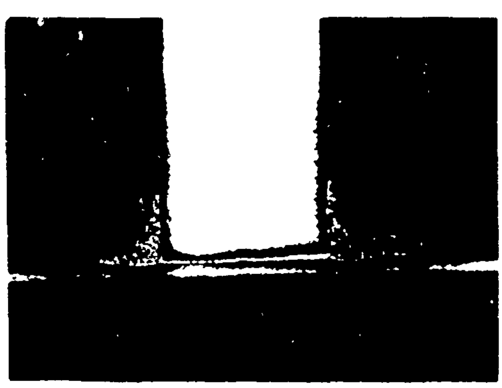
MAG = 50X 985

c. 2000°F BRAZING TEMPERATURE.



MAG = 150X 986

d. 2000°F BRAZING TEMPERATURE



MAG 50X 987

e. 1975°F BRAZING TEMPERATURE

MAG = 150X 988

f. 1975°F BRAZING TEMPERATURE.

F-8630

NOTE: PHOTOS HAVE BEEN REDUCED TO 63%

Figure 4. Photomicrographs of Hastelloy X Tube-Header Joints Brazed with Nicrobraz 135 Brazing Alloy, in Vacuum, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.

MAG = 50X 989

a. 2175°F BRAZING TEMPERATURE.

MAG = 50X 990

b. 2150°F BRAZING TEMPERATURE.

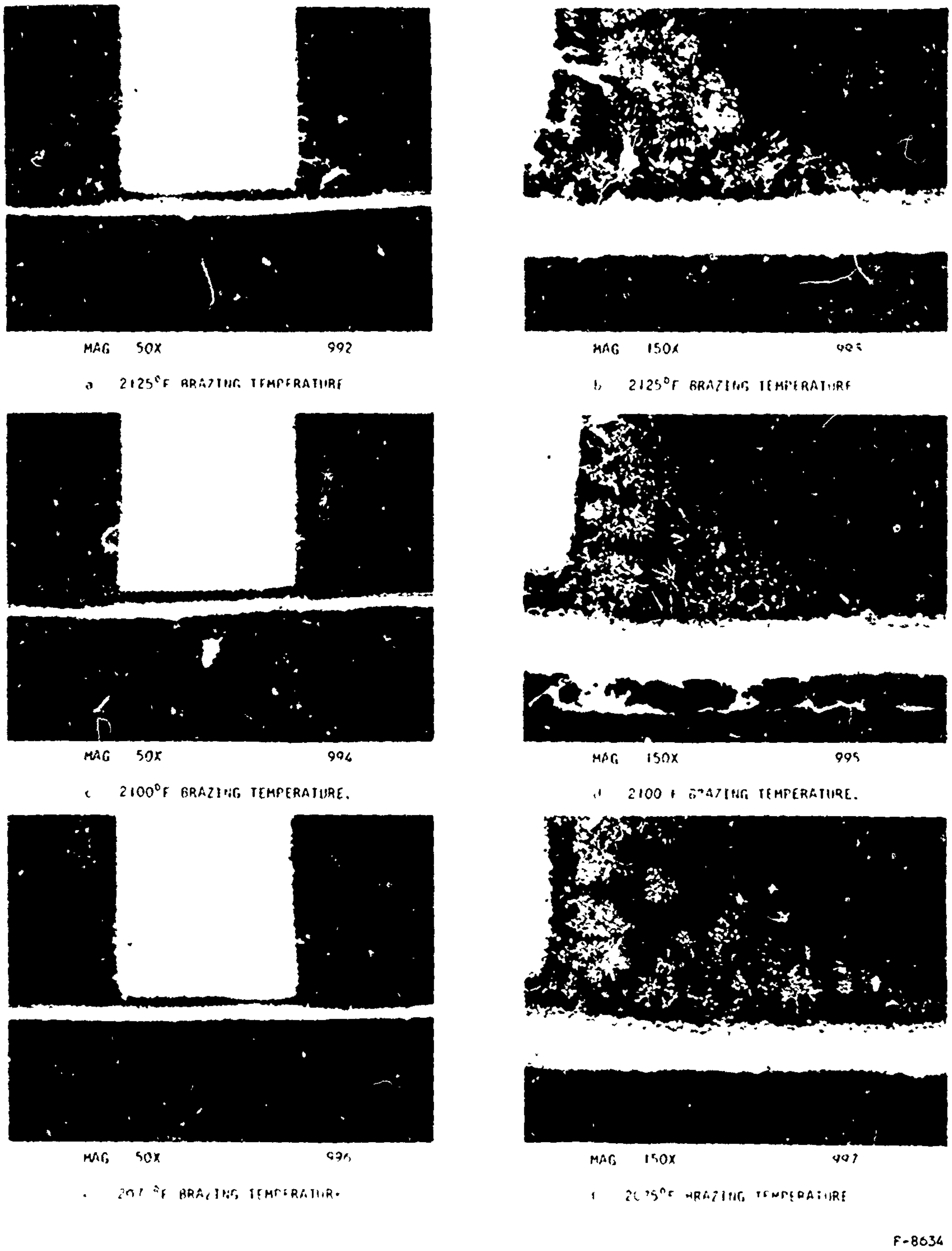
MAG 50X 991

c. 2125°F BRAZING TEMPERATURE.

NOTE: PHOTOS HAVE BEEN REDUCED TO 74%

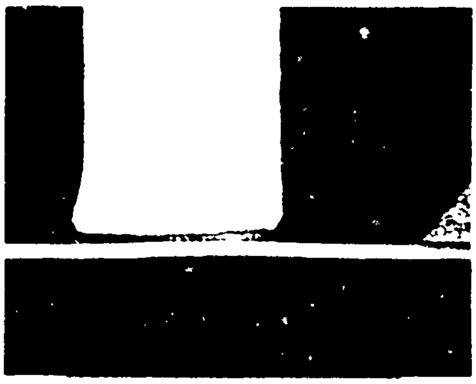
F-8629

Figure 5. Photomicrographs of Inconel 625 Tube-Header Joints Brazed with J-8100 Brazing Alloy, in Vacuum, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.



NOTE: PHOTOS HAVE BEEN REDUCED TO 62%

Figure 5. Photomicrographs of Inconel 625 Tube-Header Joints Brazed with Engelhard 135 Brazing Alloy, in Hydrogen, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.

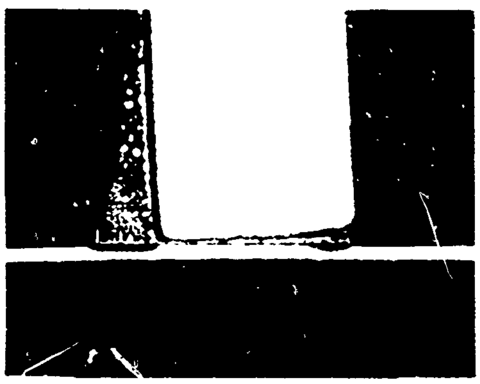


MAG 50X 998

a. 2025°F BRAZING TEMPERATURE

MAG 150X 999

b. 2025°F BRAZING TEMPERATURE.

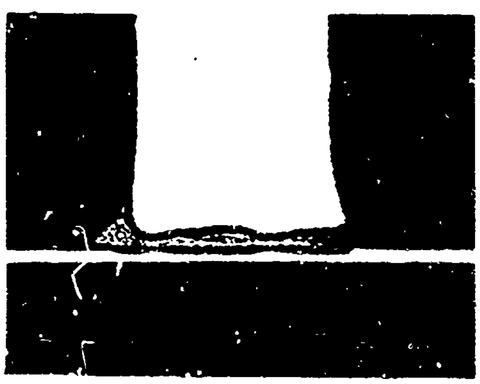


MAG 50X 1000

c. 2000°F BRAZING TEMPERATURE.

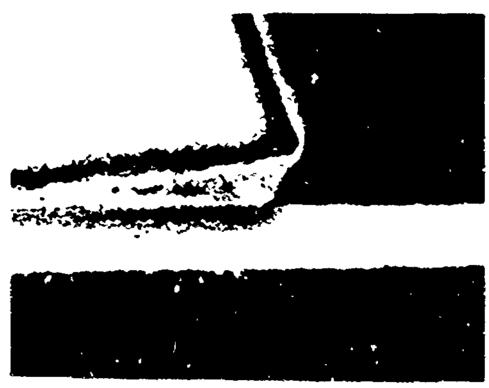
MAG 150X 1001

d. 2000°F BRAZING TEMPERATURE.



MAG 50X 1002

e. 19[illegible]5°F BRAZING TEMPERATURE



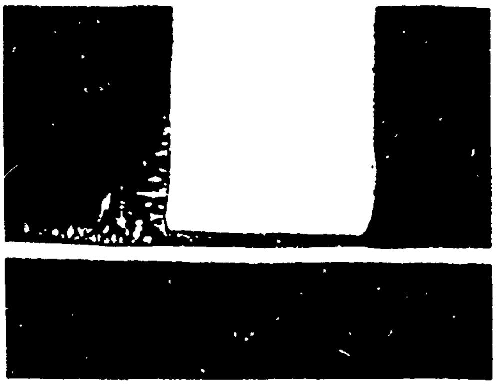
MAG 150X [illegible]

f. 19[illegible]5°F BRAZING TEMPERATURE

F-8636

NOTE: PHOTOS HAVE BEEN REDUCED TO 6[illegible]%

Figure 7. Photomicrographs of Inconel 625 Tube-Header Joints Brazed with Nicrobraz 135 Brazing Alloy, in Vacuum, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.

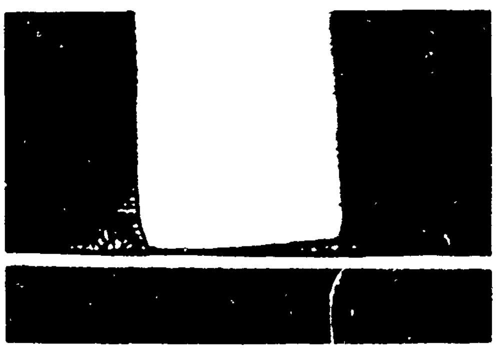


MAG - 50X 1004

a. 1975°F BRAZING TEMPERATURE.

MAG = 150X 1005

b. 1975°F BRAZING TEMPERATURE.

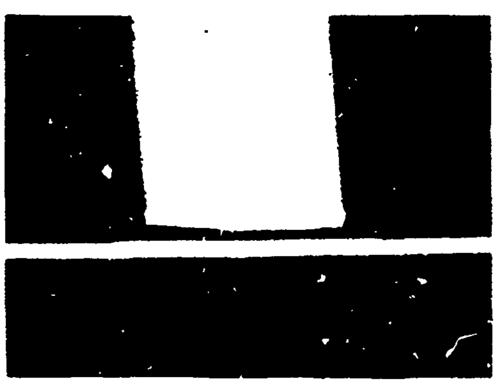


MAG = 50X 1006

c. 1950°F BRAZING TEMPERATURE

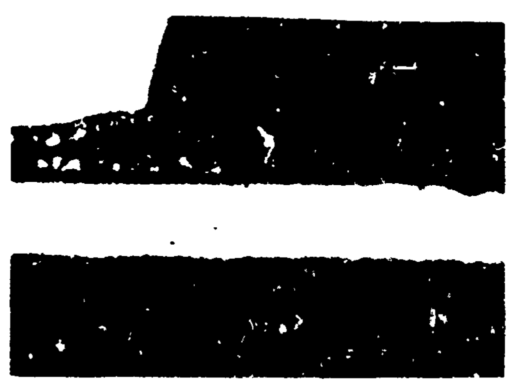
MAG = 150X 1007

d. 1950°F BRAZING TEMPERATURE.



MAG = 50X 1008

e. 1925°F BRAZING TEMPERATURE.



MAG = 150X 1009

f. 1925°F BRAZING TEMPERATURE.

F-8657

NOTE: PHOTOS HAVE BEEN REDUCED TO 63%

Figure 8. Photomicrographs of Inconel 625 Tube-Header Joints Brazed with Nicrobraz 65 Brazing Alloy, in Hydrogen, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.

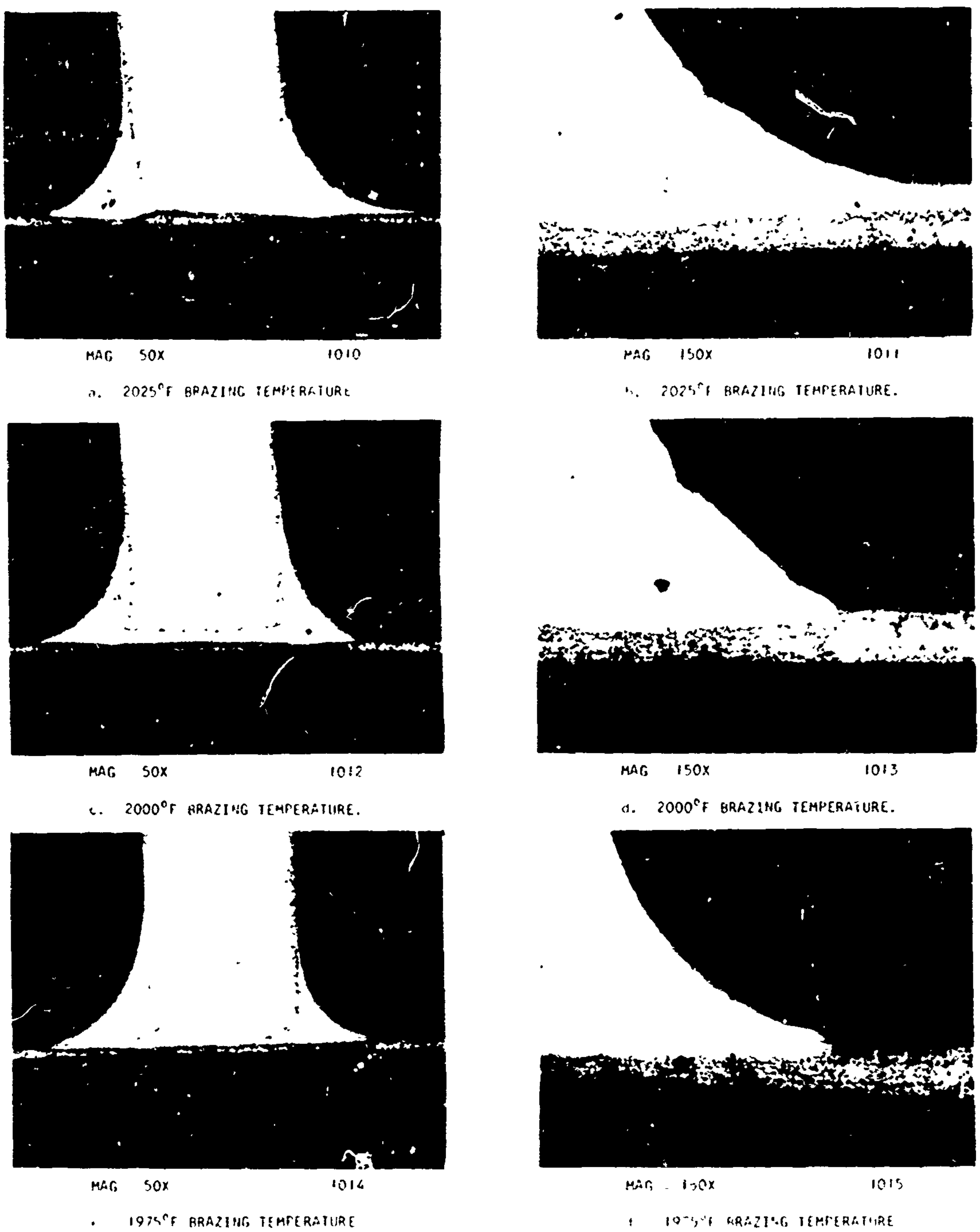
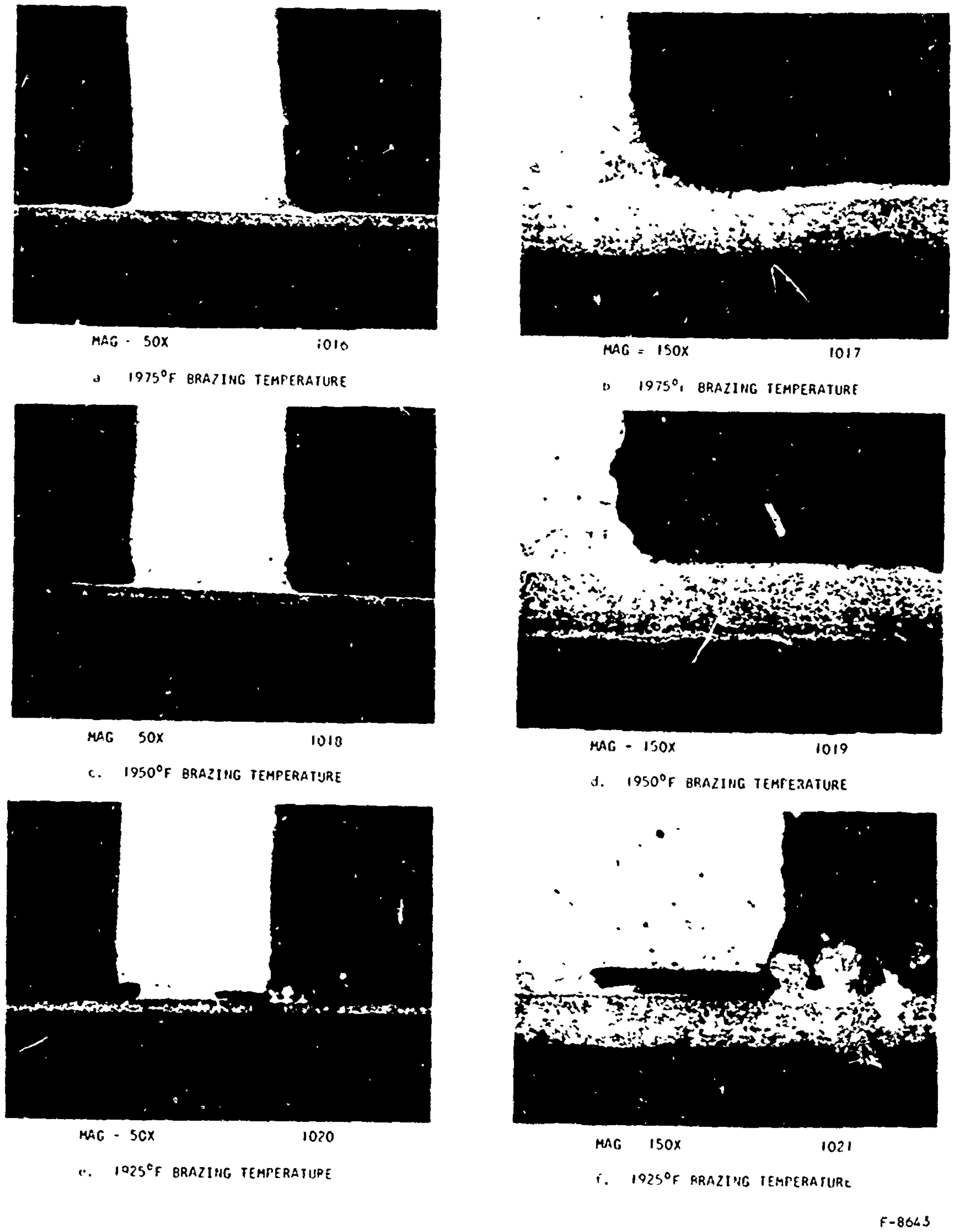
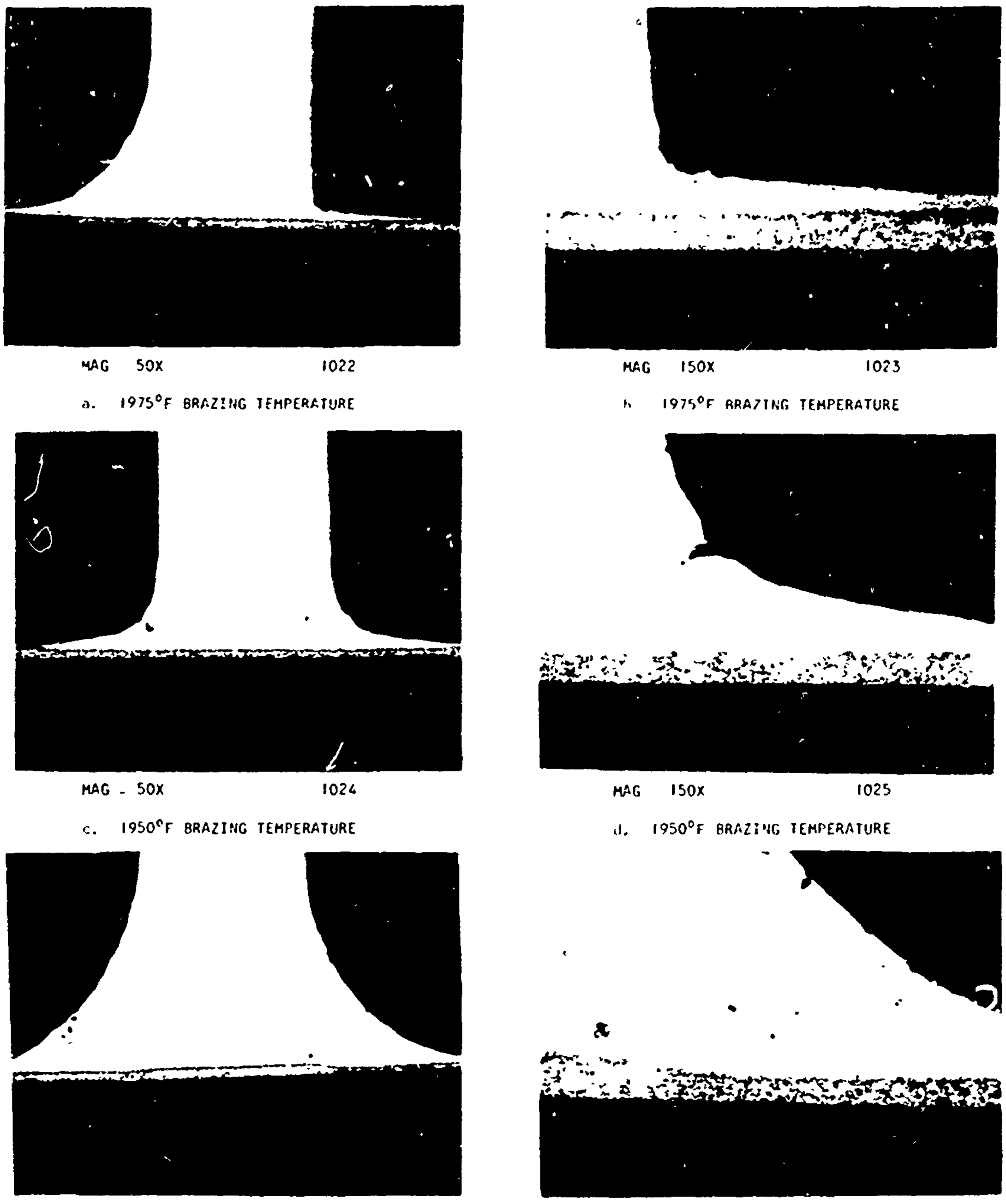


Figure 9. Photomicrographs of Incoloy 800 Tube-Header Joints Brazed with Nicrobraz 135 Brazing Alloy, in Vacuum, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.



NOTE: PHOTOS HAVE BEEN REDUCED TO 63%

Figure 10. Photomicrographs of Incoloy 800 Tube-Header Joints Brazed With Nicrobraz 65 Brazing Alloy, in Hydrogen, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.



MAG 50X 1022
a. 1975°F BRAZING TEMPERATURE

MAG 150X 1023
b. 1975°F BRAZING TEMPERATURE

MAG - 50X 1024
c. 1950°F BRAZING TEMPERATURE

MAG 150X 1025
d. 1950°F BRAZING TEMPERATURE

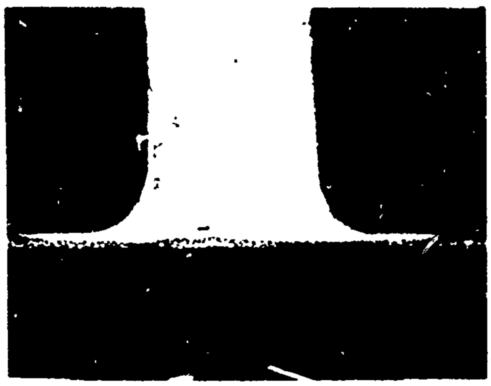
MAG 50X 1026
e. 1925°F BRAZING TEMPERATURE

MAG - 150X 1027
f. 1925°F BRAZING TEMPERATURE

F-8544

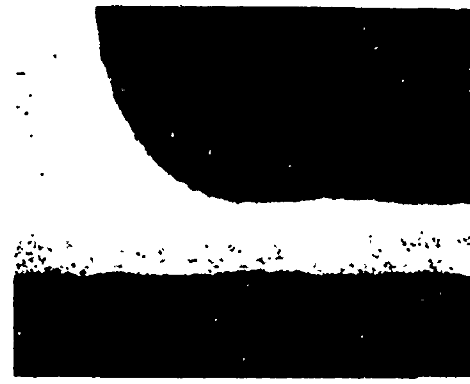
NOTE: PHOTOS HAVE BEEN REDUCED TO 61%

Figure 11. Photomicrographs of Incoloy 800 Tube-Header Joints Brazed with Palniro 7 Brazing Alloy, in Vacuum, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.



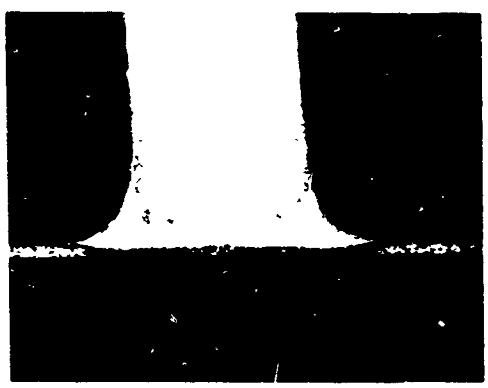
MAG = 50X 1028

a. 2075°F BRAZING TEMPERATURE.



MAG = 150X 1029

b. 2075°F BRAZING TEMPERATURE.

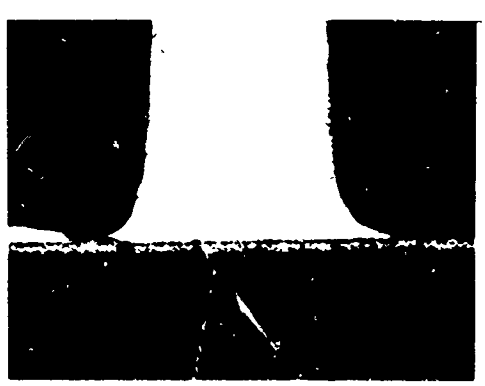


MAG = 50X 1030

c. 2050°F BRAZING TEMPERATURE.

MAG 150X 1031

d. 2050°F BRAZING TEMPERATURE.



MAG : 50X 1032

e. 2025°F BRAZING TEMPERATURE

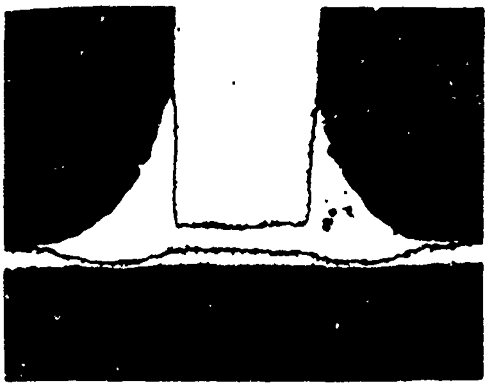
MAG 150X 1033

f. 2025°F BRAZING TEMPERATURE

F-8645

NOTE: PHOTOS HAVE BEEN REDUCED TO 61%

Figure 12. Photomicrographs of Incoloy 800 Tube-Header Joints Brazed with Coast Metals 50B Brazing Alloy in Vacuum, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.

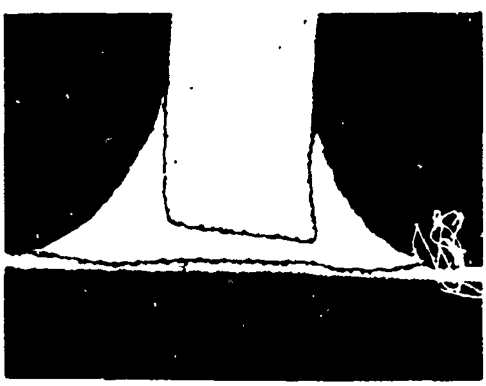


MAG = 50X 1034

a. 2200°F BRAZING TEMPERATURE.

MAG = 150X 1035

b. 2200°F BRAZING TEMPERATURE.

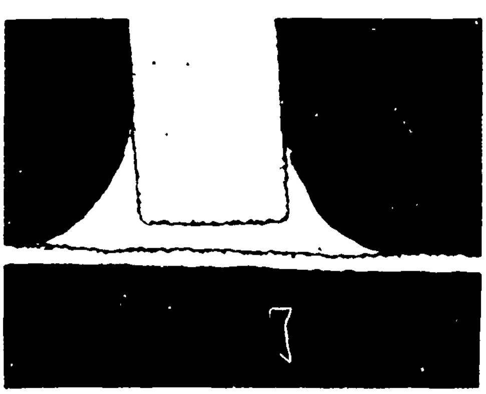


MAG = 50X 1036

c. 2175°F BRAZING TEMPERATURE.

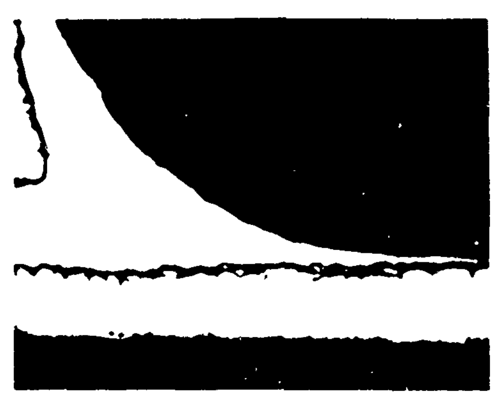
MAG = 150X 1037

d. 2175°F BRAZING TEMPERATURE



MAG 50X 1038

e. 2150°F BRAZING TEMPERATURE.



MAG 150X 1039

f. 2150°F BRAZING TEMPERATURE.

F-8646

NOTE: PHOTOS HAVE BEEN REDUCED TO 61%

Figure 13. Photomicrographs of Multimet N-155 Tube-Header Joints Brazed with Palniro 4 Brazing Alloy, in Vacuum, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.

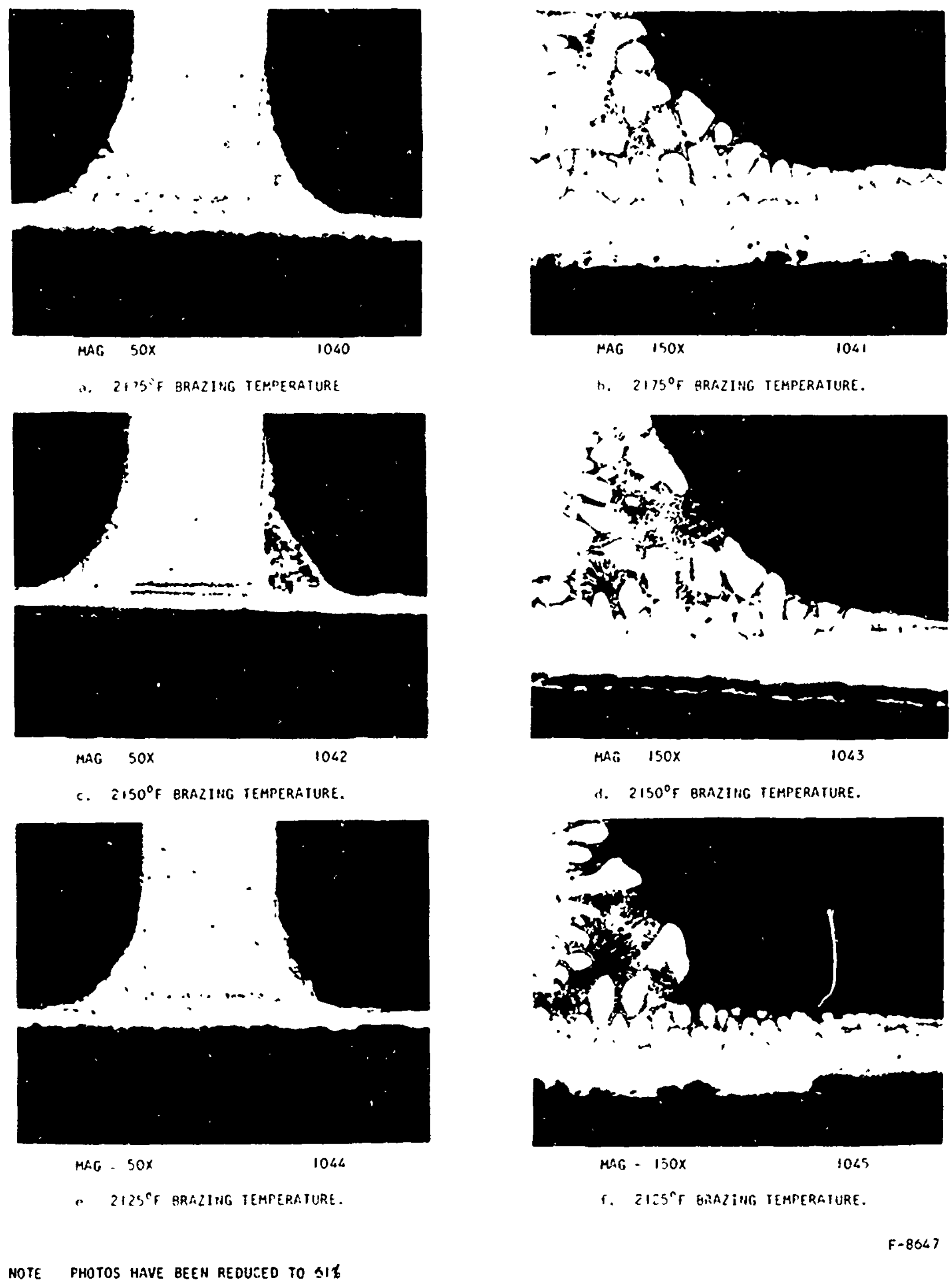
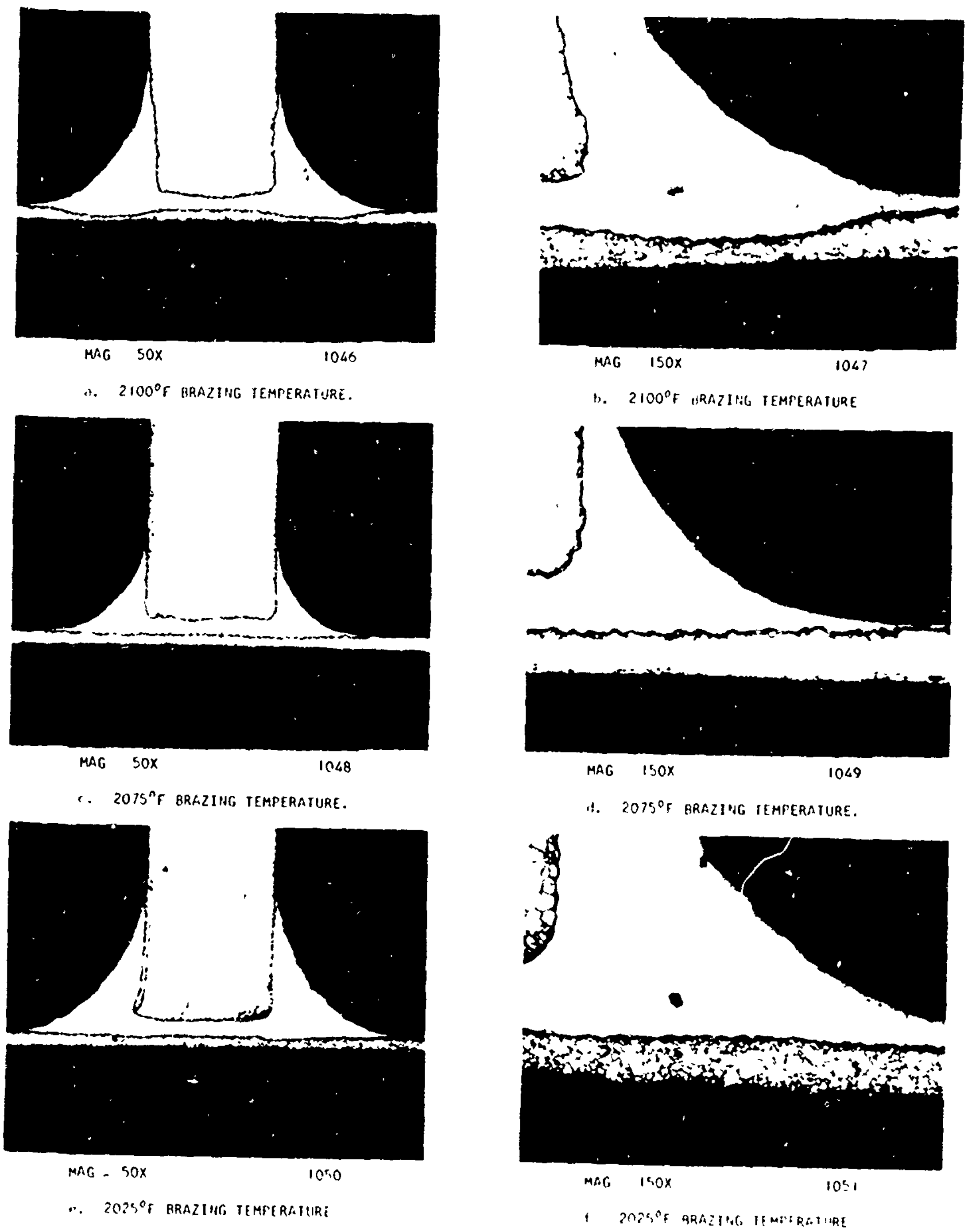


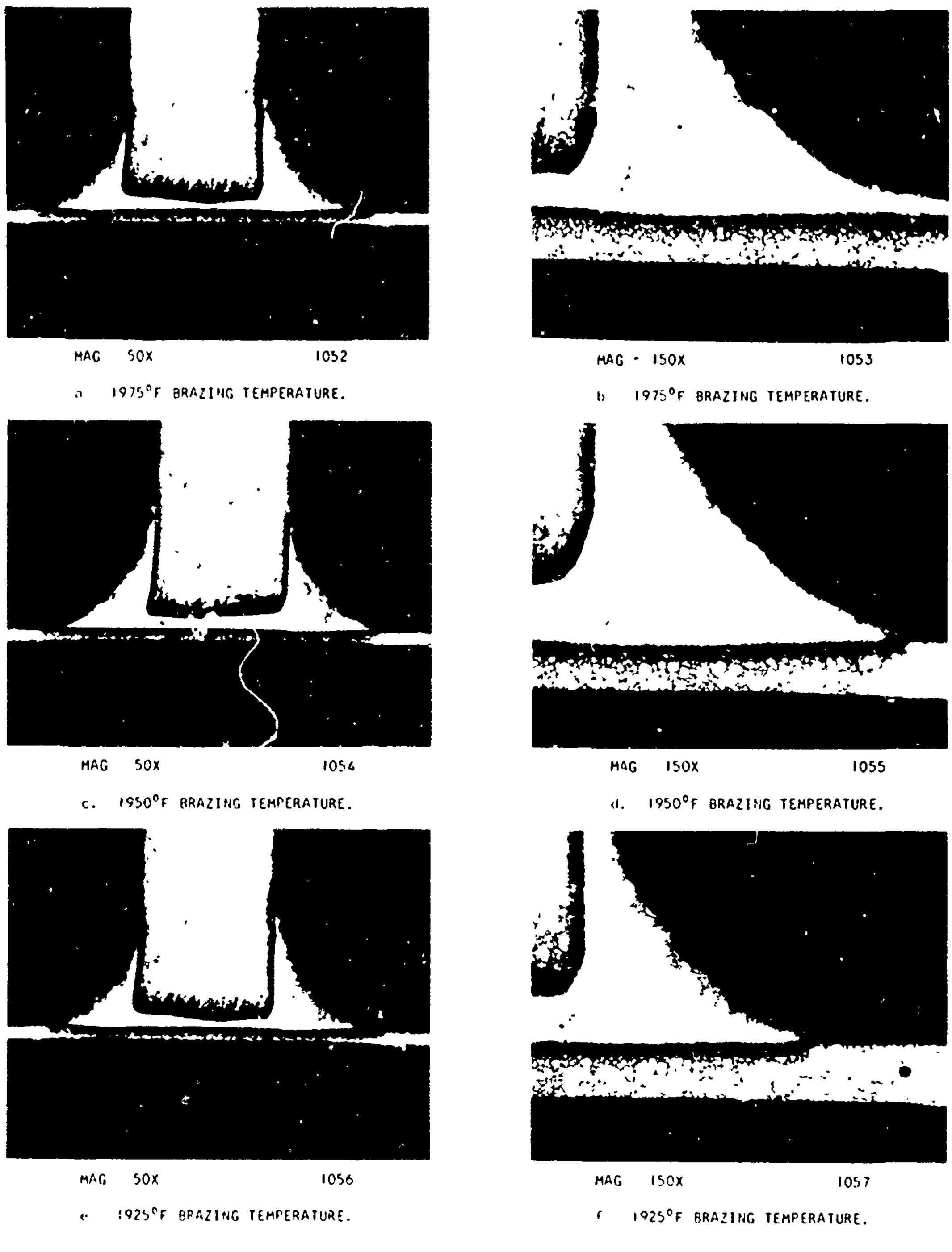
Figure 14. Photomicrographs of Multimet N-155 Tube-Header Joints Brazed with J-8100 Brazing Alloy, in Vacuum, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.



NOTE: PHOTOS HAVE BEEN REDUCED TO 61%

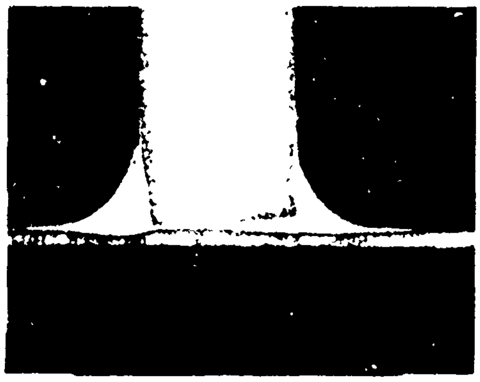
F-8648

Figure 15. Photomicrographs of Multimet N-155 Tube-Header Joints Brazed with Palniro I Brazing Alloy, in Vacuum, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.



NOTE: PHOTOS HAVE BEEN REDUCED TO 61%

Figure 16. Photomicrographs of Multimet N-155 Tube-Header Joints Brazed with Nicrobraz 200 Brazing Alloy, in Vacuum, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.

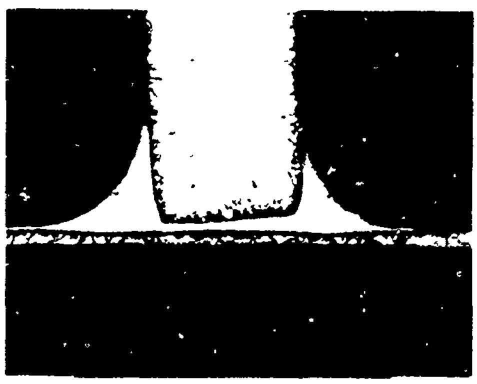


MAG 50X 1058

a. 2025°F BRAZING TEMPERATURE.

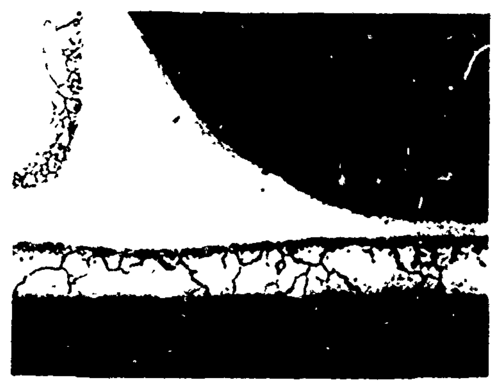
MAG - 150X 1059

b. 2025°F BRAZING TEMPERATURE.



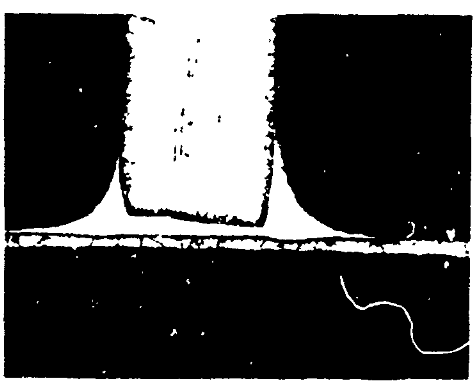
MAG - 50X 1060

c. 2000°F BRAZING TEMPERATURE.



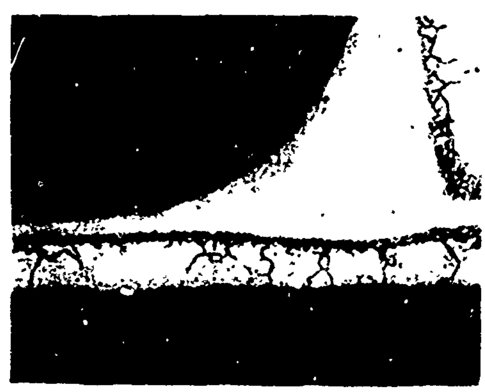
MAG 150X 1061

d. 2000°F BRAZING TEMPERATURE.



MAG 50X 1062

e. 1975°F BRAZING TEMPERATURE.



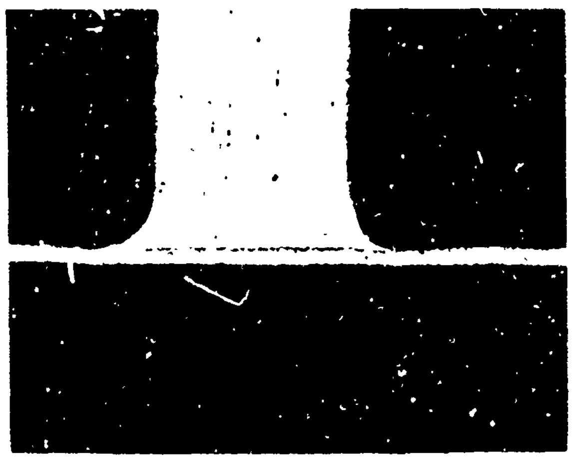
MAG 150X 1063

f. 1975°F BRAZING TEMPERATURE.

NOTE: PHOTOS HAVE BEEN REDUCED TO 61%

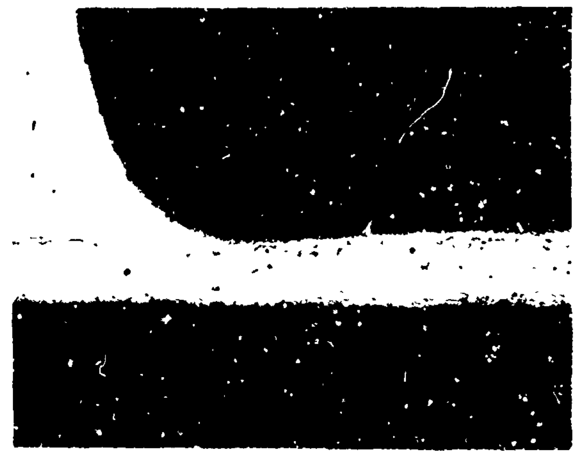
F-8649

Figure 17. Photomicrographs of 347 CRES Tube-Header Joints Brazed with Nicrobraz 135 Brazing Alloy, in Vacuum, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.



MAG 50X 1064

[illegible]°F BRAZING TEMPERATURE



MAG 150X 1064A

[illegible]°F BRAZING TEMPERATURE.

MAG 50X 1065

c. 1950°F BRAZING TEMPERATURE

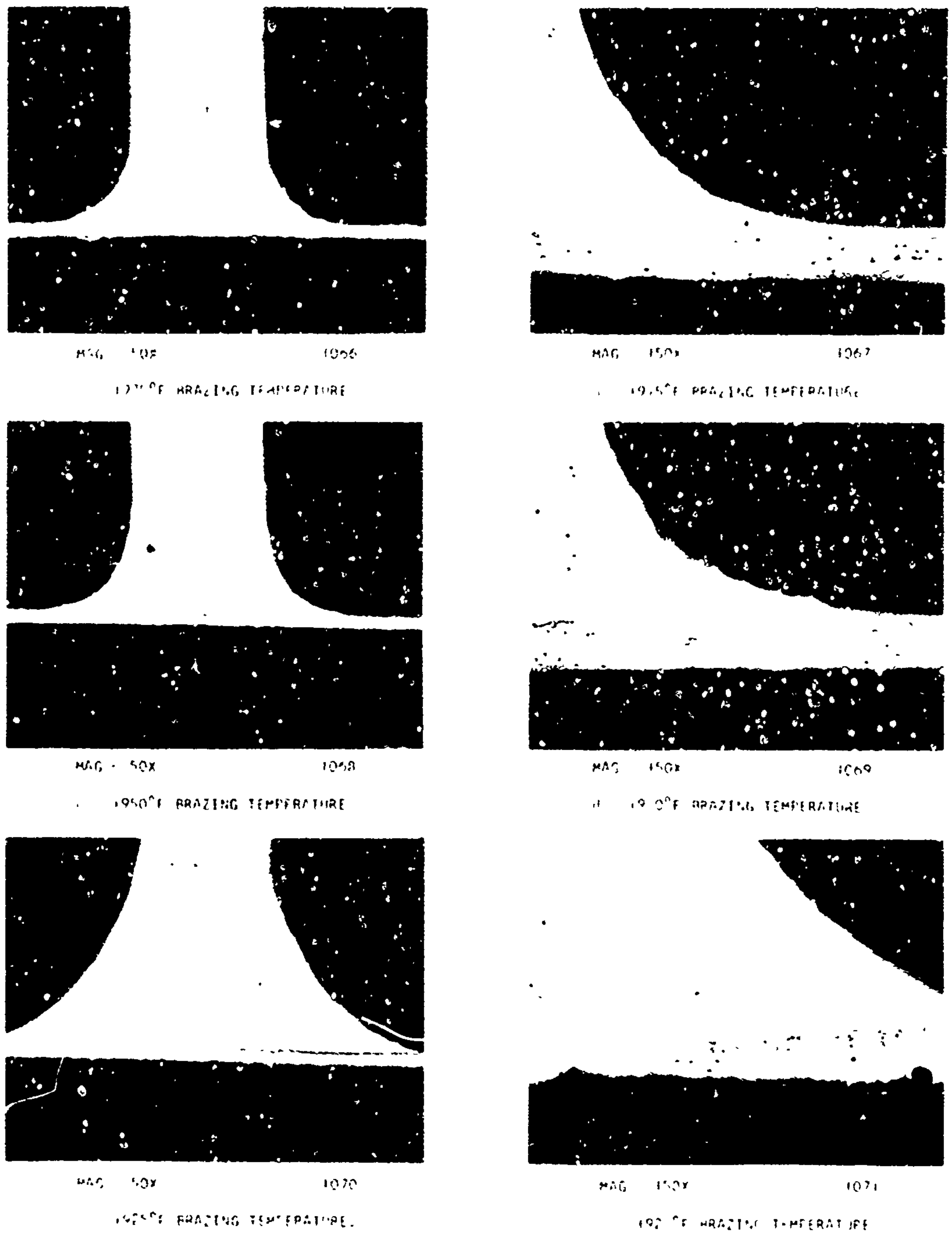
MAG 150X 1065A

d. 1950°F BRAZING TEMPERATURE.

NOTE: PHOTOS HAVE BEEN REDUCED TO 74%

F-8635

Figure 18. Photomicrographs of 347 CRES Tube-Header Joints Brazed with Nicrobraz 65 Brazing Alloy, in Hydrogen, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.



NOTE: PHOTOS HAVE BEEN REDUCED TO 59%

Figure 19. Photomicrographs of 347 CRES Tube-Header Joints Brazed with Palniro 7 Brazing Alloy, in Vacuum, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.

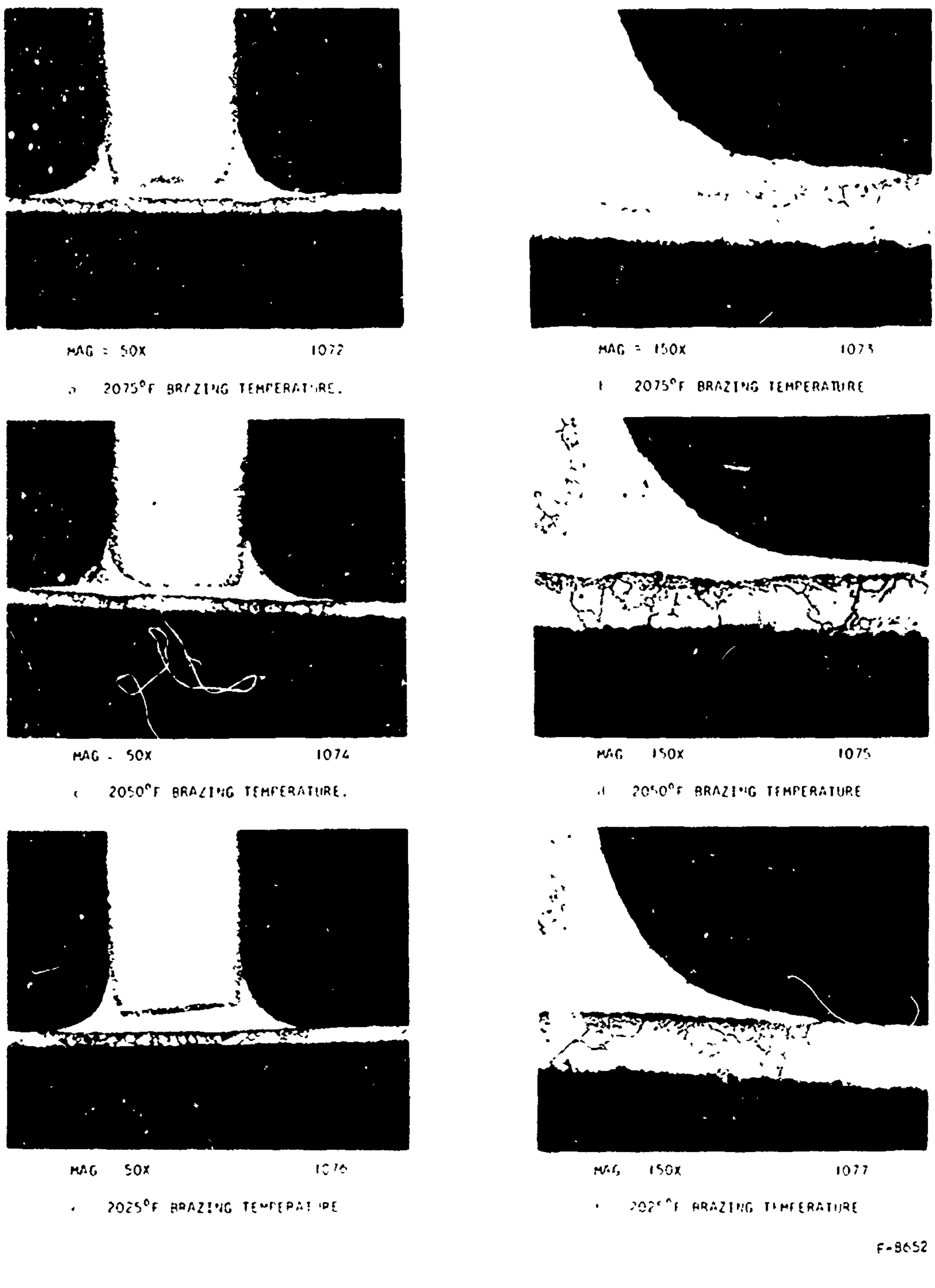


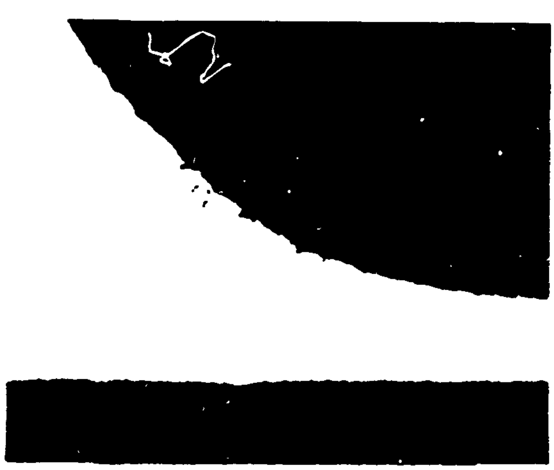
Figure 20. Photomicrographs of 347 CRES Tube-Header Joints Brazed with Coast Metals 50B Brazing Alloy, in Vacuum, with a 10 Minute Hold Time at Temperature. Etched with Kalling's Reagent.

MAG 150X 1078

PALNIRO 4 BRAZING ALLOY, VACUUM BRAZED AT 2170°F WITH HOLD TIME OF 10 MIN. NO CORROSION OF PALNIRO 4
UNETCHED

MAG 150X 1079

J-8100 BRAZING ALLOY VACUUM BRAZED AT 2150°F WITH HOLD TIME OF 10 MIN. NO CORROSION OF J-8100
UNETCHED



MAG 150X 1080

PALNIRO 1 BRAZING ALLOY VACUUM BRAZED AT 2070°F WITH HOLD TIME OF 10 MIN. NO CORROSION OF PALNIRO 1
UNETCHED

MAG 150X 1081

NICROBRAZ 135 BRAZING ALLOY VACUUM BRAZED AT 2000°F WITH HOLD TIME OF 10 MIN. SOME CORROSION OF NICROBRAZ 135 NOTED.
UNETCHED

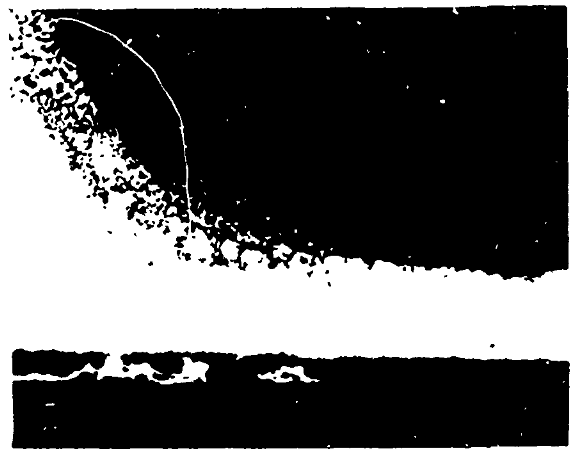
1-8-5

NOTE. PHOTOS HAVE BEEN REDUCED TO 70%

Figure 21. Photomicrographs of Hastelloy X Tube-Header Joints Following Hot Corrosion Test at 1500°F for 100 Hours. No Corrosion Noted on Hastelloy X Tubing (1/8-in. O.D. x 0.0035-in. Wall).

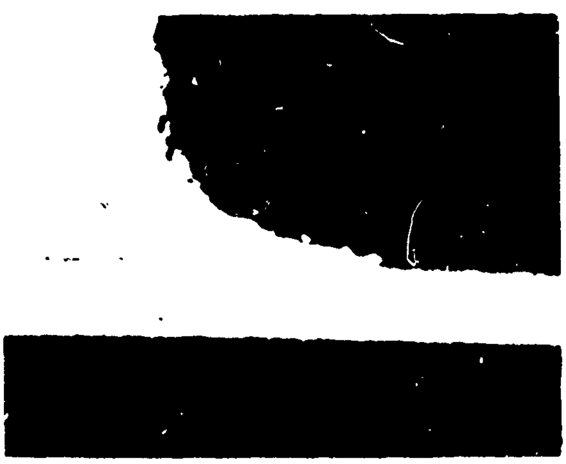
MAG 150X 1082

a. J-8100 BRAZING ALLOY. VACUUM BRAZED AT 2150°F WITH HOLD TIME OF 10 MIN NO CORROSION OF J-8100 NOTED UNETCHED



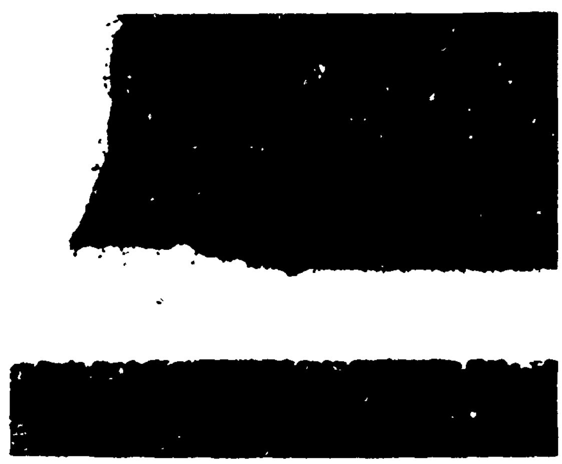
MAG 150X 1083

b. ENGELHARD 135 BRAZING ALLOY. BRAZED AT [illegible]100°F IN HYDROGEN ATMOSPHERE WITH 10 MIN HOLD TIME. EXTENSIVE CORROSION OF ENGELHARD 135 OCCURRED UNETCHED



MAG 150X 1084

c. NICROBRAZ 135 BRAZING ALLOY. VACUUM BRAZED AT 2000°F WITH HOLD TIME OF 10 MIN. SOME CORROSION OF NICROBRAZ 135 NOTED UNETCHED



MAG 150X 1085

d. NICROBRAZ 65 BRAZING ALLOY. BRAZED AT 1950°F IN HYDROGEN WITH HOLD TIME OF 10 MIN. HEAVY CORROSION OF NICROBRAZ 65. UNETCHED

F-8640

NOTE: PHOTOS HAVE BEEN REDUCED TO 71%

Figure 22. Photomicrographs of Inconel 625 Tube-Header Joint Following Hot Corrosion Test at 1500°F for 100 Hours. No Corrosion Noted on Inconel 625 Tubing (1/8-in. O.D. x 0.0035-in. Wall).

MAG 150X 1086

a. NICROBRAZ 135 BRAZING ALLOY. VACUUM BRAZED AT 2000°F WITH 10 MIN HOLD TIME. SOME CORROSION OF NICROBRAZ 135 NOTED
UNETCHED

MAG - 150X 1087

b. NICROBRAZ 65 BRAZING ALLOY. BRAZED AT 1950°F IN HYDROGEN WITH 10 MIN HOLD TIME. EXTENSIVE CORROSION OF NICROBRAZ 65.
UNETCHED

MAG . 150X 1088

c. PALNIRO 7 BRAZING ALLOY. VACUUM BRAZED AT 1950°F WITH 10 MIN HOLD TIME. NO CORROSION OF PALNIRO 7 NOTED.
UNETCHED

MAG 150X 1089

d. COAST METALS 50R BRAZING ALLOY. VACUUM BRAZED AT 2050°F WITH 10 MIN HOLD TIME. SOME CORROSION OF 50R NOTED
UNETCHED

F-8639

NOTE: PHOTOS HAVE BEEN REDUCED TO 71%

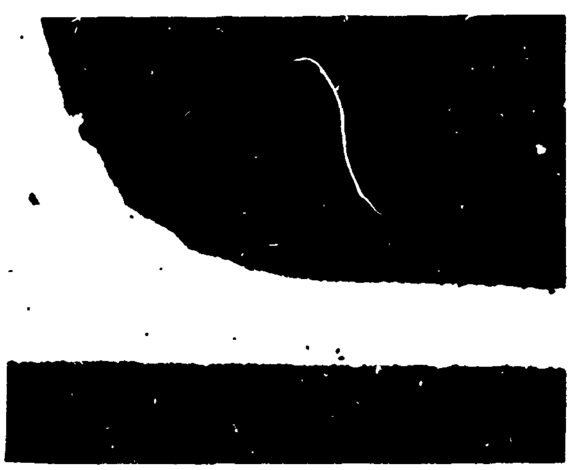
Figure 23. Photomicrographs of Incoloy 800 Tube-Header Joints Following Hot Corrosion Test at 1500°F for 100 Hours. No Corrosion Noted on Incoloy 800 Tubing (1/8-in. O.D. x 0.0035-in. Wall).

MAG 150X 1090

a PALNIRO 4 BRAZING ALLOY. VACUUM BRAZED AT 2175°F WITH 10 MIN HOLD TIME NEGLIGIBLE CORROSION OF PALNIRO 4 NOTED UNETCHED

MAG 150X 1091

b J-8100 BRAZING ALLOY VACUUM BRAZED AT 2150°F WITH 10 MIN HOLD TIME. NO CORROSION OF J-8100 NOTED. UNETCHED



MAG 150X 1092

c PALNIRO 1 BRAZING ALLOY. VACUUM BRAZED AT 2075°F WITH 10 MIN HOLD TIME. NO CORROSION OF PLANIRO 1 NOTED UNETCHED

MAG 150X 1093

d NICROBRAZ 200 BRAZING ALLOY. VACUUM BRAZED AT 1950°F WITH 10 MIN HOLD TIME SLIGHT CORROSION OF NICROBRAZ 200 NOTED UNETCHED

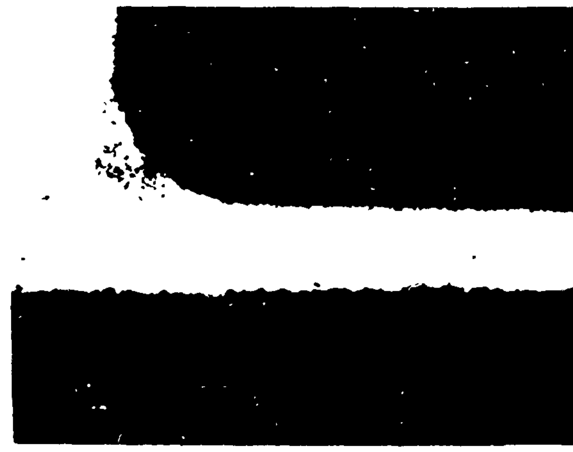
F-8041

NOTE PHOTOS HAVE BEEN REDUCED TO 74%

Figure 24. Photomicrographs of Multimet N-155 Tube-Header Joints Following Hot Corrosion Test at 1500°F for 100 Hours. No Corrosion Noted on Multimet N-155 Tubing (1/8 in. O.D. x 0.0035 in. Wall).

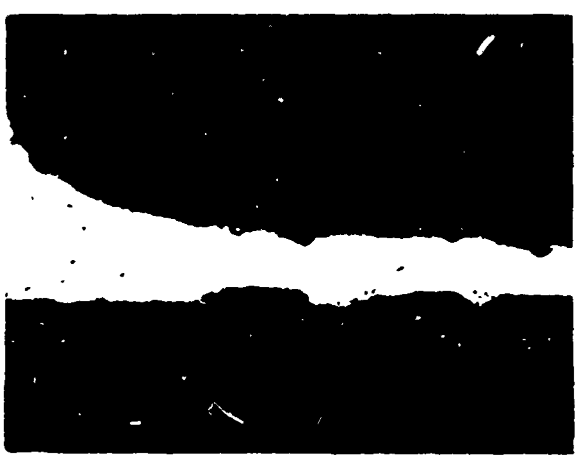
MAG 150X 1094

a. NICROBRAZ 135 BRAZING ALLOY. VACUUM BRAZED AT 2000°F WITH 10 MIN HOLD TIME SOME CORROSION OF NICROBRAZ 135 NOTED UNETCHED



MAG 150X 1095

b NICROBRAZ 65 BRAZING ALLOY. BRAZED AT 19?0°F IN HYDROGEN WITH 10 MIN HOLD TIME. CORROSION OF NICROBRAZ 65 NOTED. UNETCHED



MAG 150X 1096

c PALNIRO 7 BRAZING ALLOY. VACUUM BRAZED AT 1950°F WITH 10 MIN HOLD TIME NO CORROSION OF PALNIRO 7 NOTED. UNETCHED

MAG 150X 1097

d. COAST METALS 50B BRAZING ALLOY. VACUUM BRAZED AT 2050°F WITH 10 MIN HOLD TIME. SOME CORROSION OF 50B NOTED UNETCHED

F-8642

NOTE: PHOTOS HAVE BEEN REDUCED TO 71%

Figure 25. Photomicrographs of 347 CRES Tube-Header Joints Following Hot Corrosion Test at 1500°F for 100 Hours. The 347 CRES Tubing (1/8 in. O.D. x 0.0035 in. Wall) was Attacked to a Depth of Approximately 0 0015 in.

With the exception of the CRES 347, none of the other tube-header joints showed more than superficial corrosion. The stainless steel however was more heavily oxidized, as can be seen in Figure 25. Palniro 4, J-8100, Palniro I, Palniro 7 and Nicrobraz 200 filler metals were not attacked by the sulphurous atmosphere. Nicrobraz 135 and Coast Metals 50B suffered moderate attack, but Nicrobraz 65 and Englehard 135 were both severely corroded.

Selection of Brazing Alloys for Task I-D Tests

The selection of two brazing alloys for each of the four candidate tubing materials for the cyclic-temperature stress-rupture tests in Task I-D was based on the following:

1. Brazing characteristics for each brazing alloy-tubing material combinations at the three temperatures used in brazing;

2. Effect of brazing temperature on tubing material;

3. Results of the 1500°F, 100 hour static hot corrosion tests;

4. Comparison of the best of the inexpensive nickel-base alloys with a more expensive gold-base alloy serves as a standard for comparison.

On the basis of the hot corrosion tests, Englehard 135 and Nicrobraz 65 were eliminated because of poor corrosion resistance. Coast Metals 50B and Nicrobraz 135 had satisfactory hot corrosion resistance and brazing characteristics, warranting consideration for the cyclic temperature tests. All the Palniro brazing alloys, J-8100, and Nicrobraz 200 had excellent hot corrosion resistance and brazing characteristics.

Both Palniro 4 and J-8100 were brazed at high temperatures (2175° and 2150°F, respectively), and grain growth occurred in base metals brazed at these temperatures. Grain coarsening, however, was more severe with Palniro 4. Very large grains are considered undesirable because of the possibility of decreased low cycle fatigue (LCF) resistance. In addition, Palniro 4 has no advantage over Palniro I (brazed at 2070°F) with respect to joint strength at 1300°F to 1500°F. Palniro 4 was therefore eliminated as a candidate.

The final selection is shown in Table VI.

TABLE VI. SELECTION OF BRAZING ALLOYS FOR TASK I-D

Tubing	Brazing Alloy	Brazing Temperature, °F*
Hastelloy X	Palniro 1 J-8100	2075 2150
Incoloy 800	Palniro 7 Coast Metals 50B	1950 2050
N-155	Palniro 1 Nicrobraz 200	2075 1950
347 CRES	Palniro 7 Nicrobraz 135	1950 2000

*Hold time of 10 minutes, vacuum furnace brazing.

The brazing alloys selected brazed well at all three brazing temperatures and should be satisfactory for production brazing of recuperators. The middle brazing temperature was selected for brazing with each alloy with a hold time of 10 minutes at brazing temperature. The brazing alloys selected are further discussed for each of the tubing materials:

- Hastelloy X - Palniro 1 had excellent brazing characteristics as noted in Table I. It also had excellent hot corrosion resistance. The 2075°F brazing temperature will permit retention of a fine grained structure. The J-8100 had good brazing characteristics and excellent hot corrosion resistance. Alloying was greater with J-8100 than Palniro 1 but still satisfactory. Grain growth occurred in Hastelloy X when brazing with J-8100. A fine grained structure has better tensile and fatigue properties, but lower stress-rupture properties. Selection of these two brazing alloys will afford a comparison between fine and coarse grain material in Task I-D tests. These two brazing alloys have previously been evaluated for a similar application in a lightweight heat exchanger operating at 1540°F maximum temperature. Both alloys performed equally well in axial tensile and axial fatigue tests at room temperature and 1540°F. Failures occurred in the tubing (1/8 in. O.D. x 0.003 in. wall) away from the brazed joint.

- Incoloy 800 - The basis for selecting Palniro 7 for Incoloy 800 was excellent brazing characteristics, hot corrosion resistance, and retention of a fine grain structure. Coast Metals 50B permitted some grain growth in Incoloy 800, alloyed somewhat more than Nicrobraz 135, but penetrated less than Nicrobraz 135. Within the brazing temperature evaluated, Coast Metals 50B had less overall alloying and penetration with Incoloy 800 than Nicrobraz 135 and was selected as a second choice.

- N-155 - Palniro I had good brazing characteristics and excellent hot corrosion resistance. It was selected for brazing N-155 for the same reasons as for brazing Hastelloy X. Nicrobraz 200 also had good brazing characteristics and excellent hot corrosion resistance. Alloying and penetration of the N-155 by Nicrobraz 200 was only about 0.001 in. which should be satisfactory for this application.

- 347 CRES - Palniro 7 was an obvious first choice because of its excellent brazing characteristics and hot corrosion resistance. Nicrobraz 135 and Coast Metals 50B had similar brazing characteristics and hot corrosion resistance. Because of its lower brazing temperature, the Nicrobraz 135 (2000°F) would be more compatible with the annealing temperature of 347 CRES (1950°F maximum) than 50B (2050°F). Grain size of the 347 tubing was larger for the 50B brazing cycle than the Palniro 7 cycle. Nicrobraz 135 was selected as the second choice.

Evaluation of Inconel 625 Tubing

Inconel 625 was originally selected for the program because its chemical composition and mechanical strength is similar to Hastelloy X, indicating that its service life would be similar. However, the former material has important advantages in ductility and price. These are important criteria in recuperator design because ductility is reflected in good low cycle fatigue resistance (thermal fatigue resistance) and manufacturing economics. The low price of Inconel 625 (about 43 percent of the cost of Hastelloy X) makes it a particularly attractive high temperature recuperator material.

The first consignment of tubing to be received was found to contain numerous longitudinal cracks which rendered the material unsuitable for stress-rupture testing. During December a second batch of material was delivered and this too suffered from surface defects. Microstructural examination revealed what appeared to be foreign particles impressed into the surface of both the inner and outer wall of the tubing to a depth of about 0.0005 inch (Figure 26). These particles were not identified. It appeared that alloying occurred between the particles and the Inconel 625 during annealing. Because these areas could seriously affect the performance of the Inconel 625 in the stress-rupture tests, this second shipment of tubing was also considered unsatisfactory, and no additional brazing studies were carried out with this material.

The supplier was requested to analyze this tubing and his report confirmed that O.D. surface imperfections were widespread. His search for contamination was hampered by difficulty in etching without severe attack or staining and as a result none was identified. However, by a review of the manufacturing process, the test records, and micro examination, he concluded that the contamination occurred in finish processing. He also concluded that its unusual nature suggested an isolated occurrence and reoccurrence improbable. The program could not wait for a new supply of tubing, therefore Hastelloy X was made ready for test.

Figure 26. Inconel 625 Tube Wall Section

F-9001

CYCLIC-TEMPERATURE HOT-CORROSION TESTS

The object of this task is to develop stress-rupture data which, when analyzed with the engine and recuperator design data given in the contract schedule, will lead to a selection of two tube material candidates for test in a combustor exhaust environment under Phase II of the program.

Tubing made from four materials (Hastelloy X, Incoloy 800, Multimet N-155, CRES 347), each brazed with two different filler alloys, subject to a cyclic corrosive environment, will be pressurized at various levels to produce failure in approximately 75, 300 and 1000 hours. The tests are being conducted in three Marshall furnaces set-up as shown in Figure 27, maintaining metal temperatures of 1100, 1300 and 1500°F.

All samples of each material combination, required for testing, have been prepared in triplicate. Some difficulty was encountered in automating the test equipment to control the gas flow and thermal cycle so that an over-shoot in temperature would not occur. The electronic equipment initially selected to guard against an over temperature condition was found to interfere with normal control and had to be replaced. Testing began the last week in January after this condition was remedied.

Test Cycle

Schematic diagrams, Figures 28 through 32, show the test cycle plan and test hardware arrangement. The tube samples, shown fitted to a header plate in Figure 33, are confined within a stainless steel retort which itself is enclosed by a Marshall furnace. The test gas enters at the top of the retort, flows parallel with the tube bodies, and leaves at the bottom through the header plate to be vented to atmosphere.

A sea salt solution is injected into the gas stream, at the top of the retort, at a controlled rate to maintain a concentration of 5 ppm sea salts in the gases. The sea salt solution was prepared in accordance with Specification ASTM D665-60 Procedure B shown in Table VII. The temperature cycle consists of two and a quarter hours at temperature, cooling down 500 to 600°F in one minute, six minutes temperature recovery and repeat. Oxidizing gas (4 percent CO_2, 15 percent O_2, 81 percent N_2 and 150 ppm SO_2) is flowing for two hours up to the point of the cycle where cooling begins. Liquid nitrogen is used to effect the temperature drop. A reducing gas (3.5 percent CO_2, 19 percent CO, 77.5 percent N_2, and 350 ppm H_2S) is passed over the tubes during the temperature recovery period. Pure nitrogen is then passed through the retort for fifteen minutes to purge the system before the oxidizing gas is again admitted.

The tubes pressurized with nitrogen gas containing one percent oxygen. Air was not chosen for this purpose because of the potentially explosive situation which would arise if tube rupture occurred during the reducing cycle. Yet, some oxygen is required to cause oxidation on the inside surfaces of the tubes to simulate more closely the service conditions of recuperators. Pure nitrogen might result in formation of surface-nitrides,

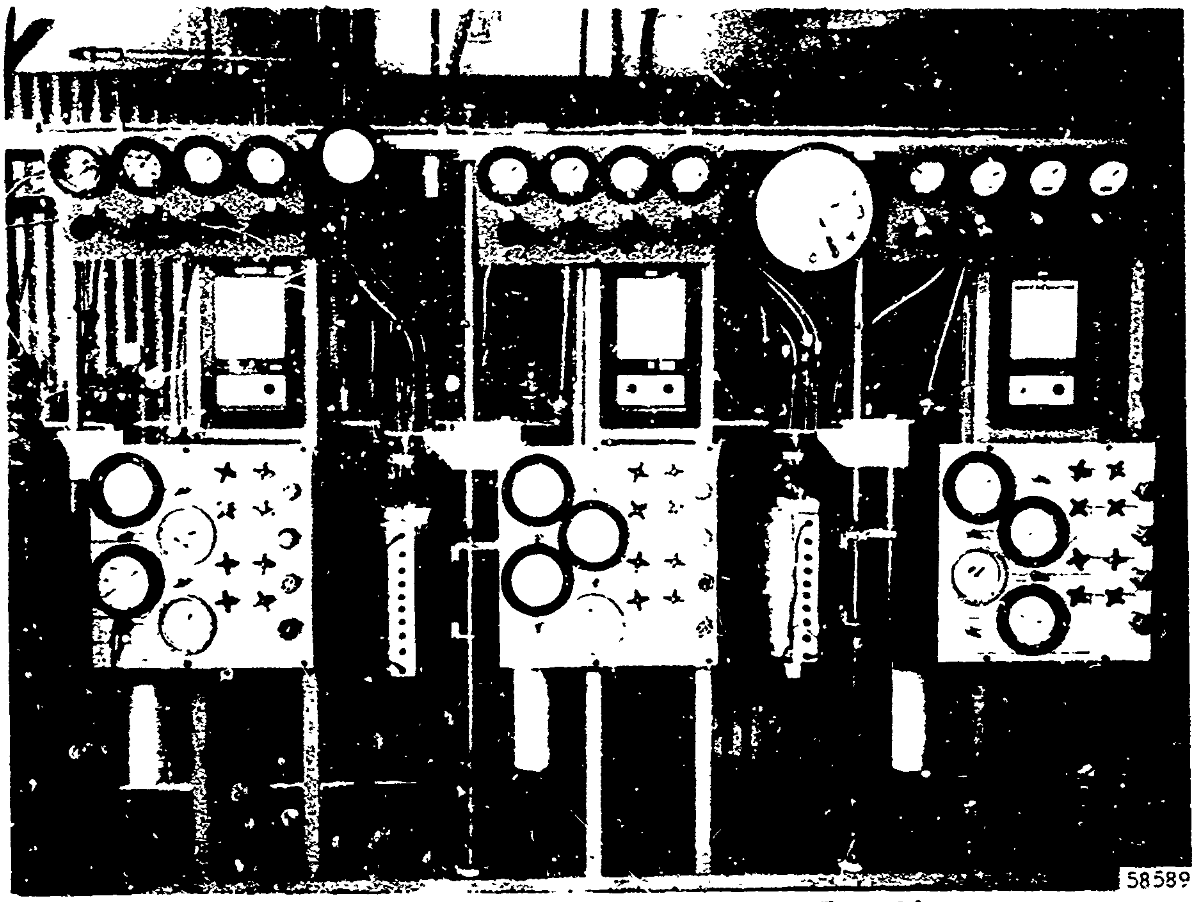


Figure 27 Tube Stress Rupture Test Rig

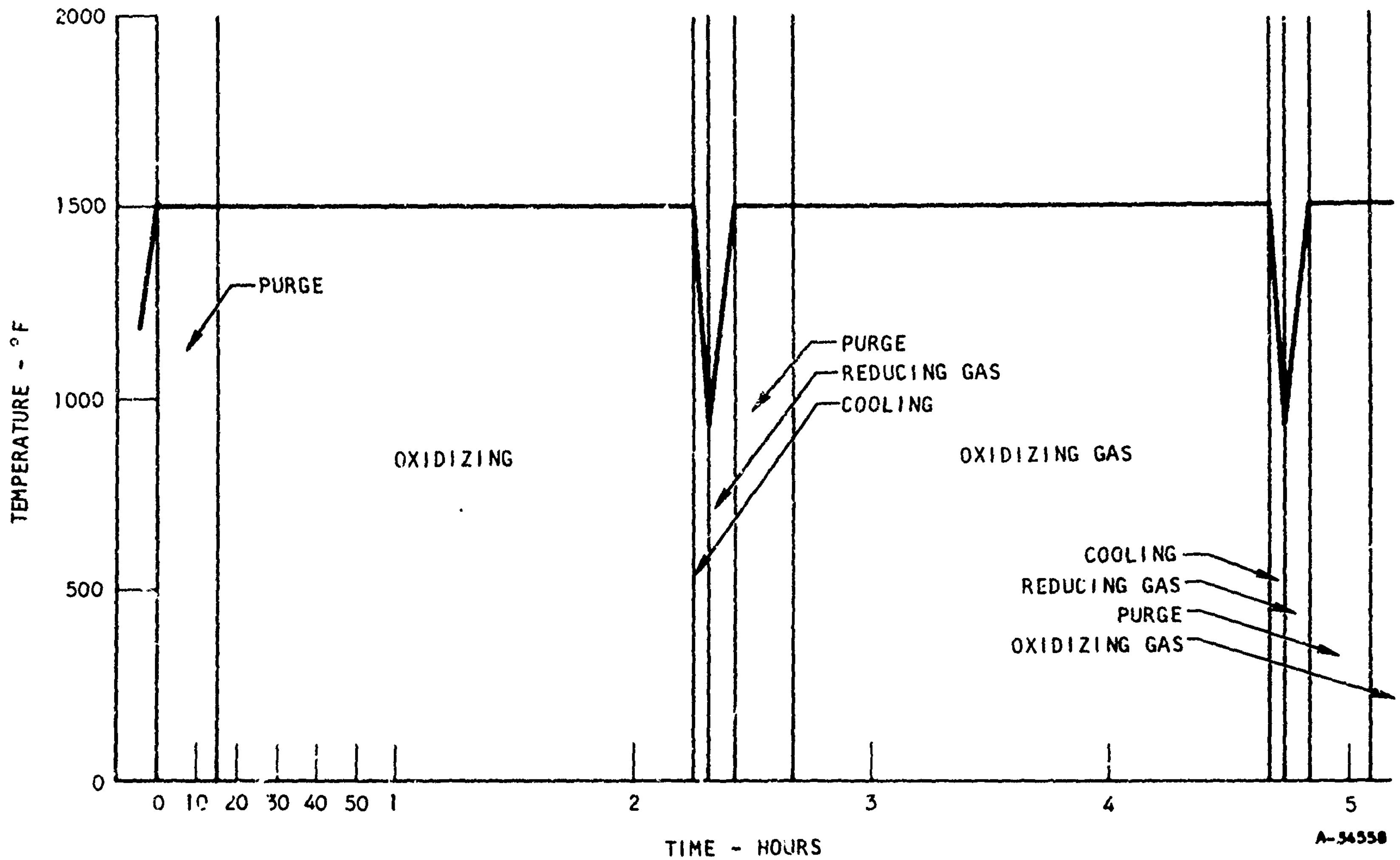


Figure 28. Typical Cyclic Test Program Plan

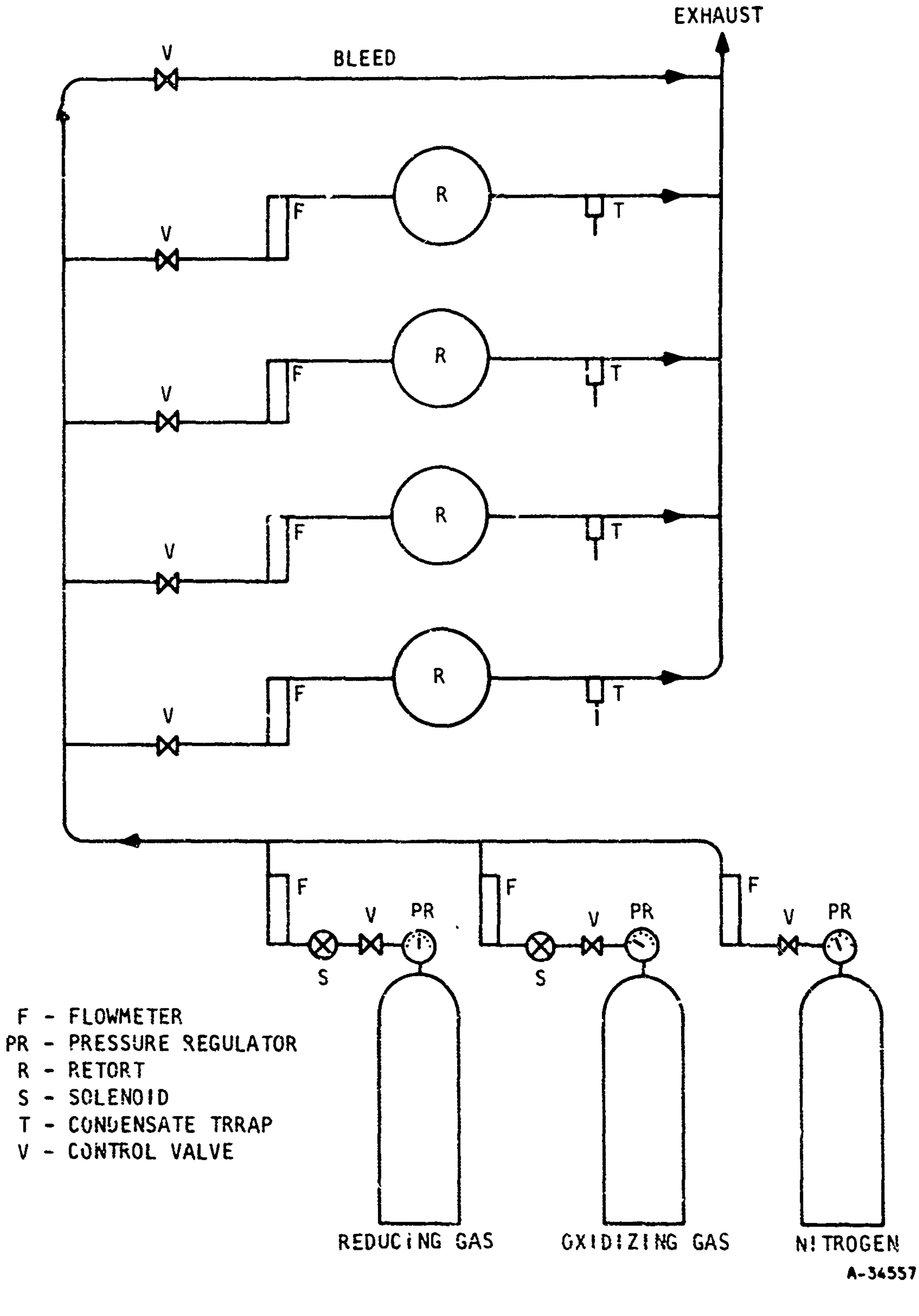


Figure 29. Test Atmosphere Control System

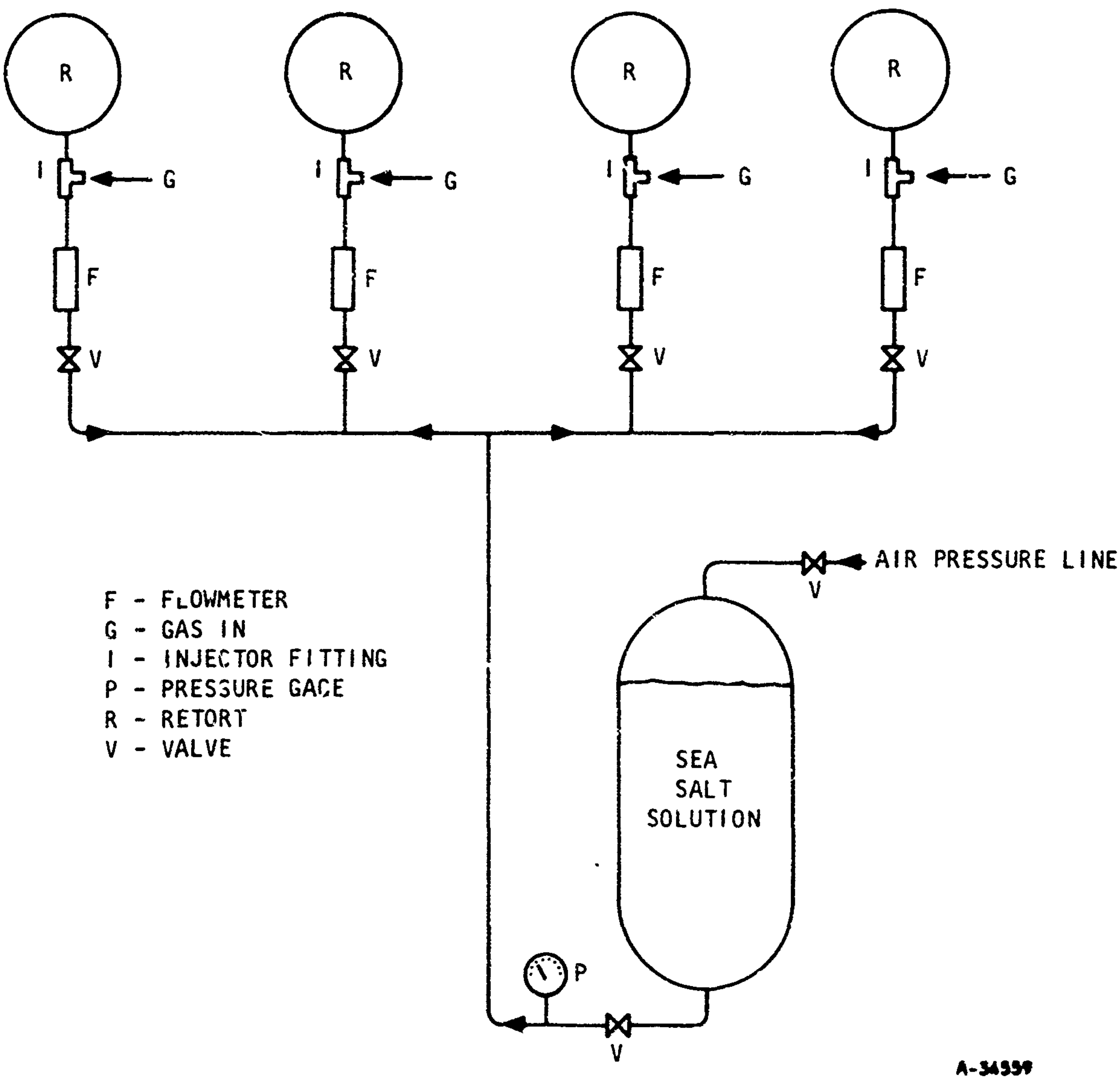


Figure 30. Sea Salt Solution Injection System

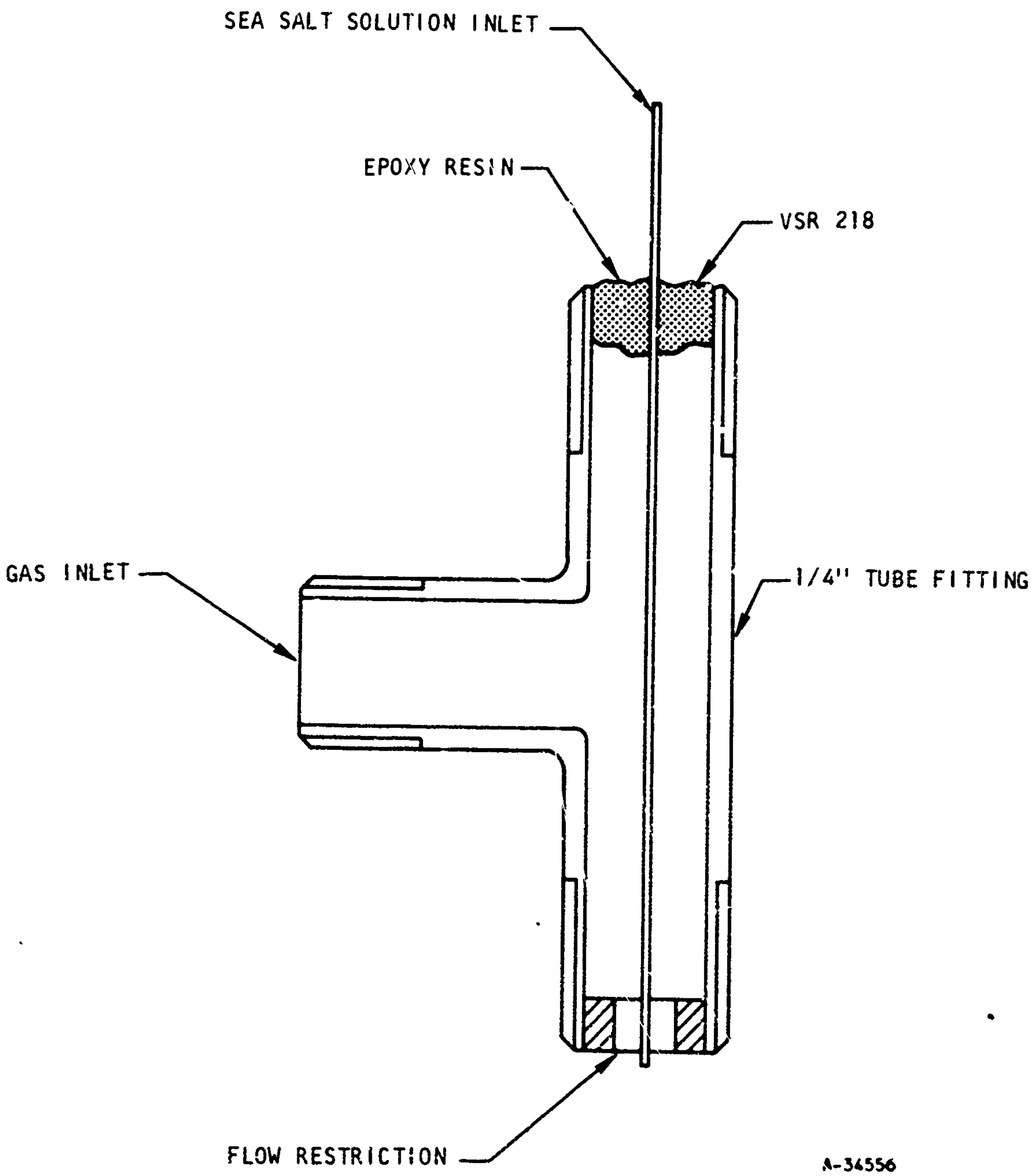


Figure 31. Sea Salt Solution Injector

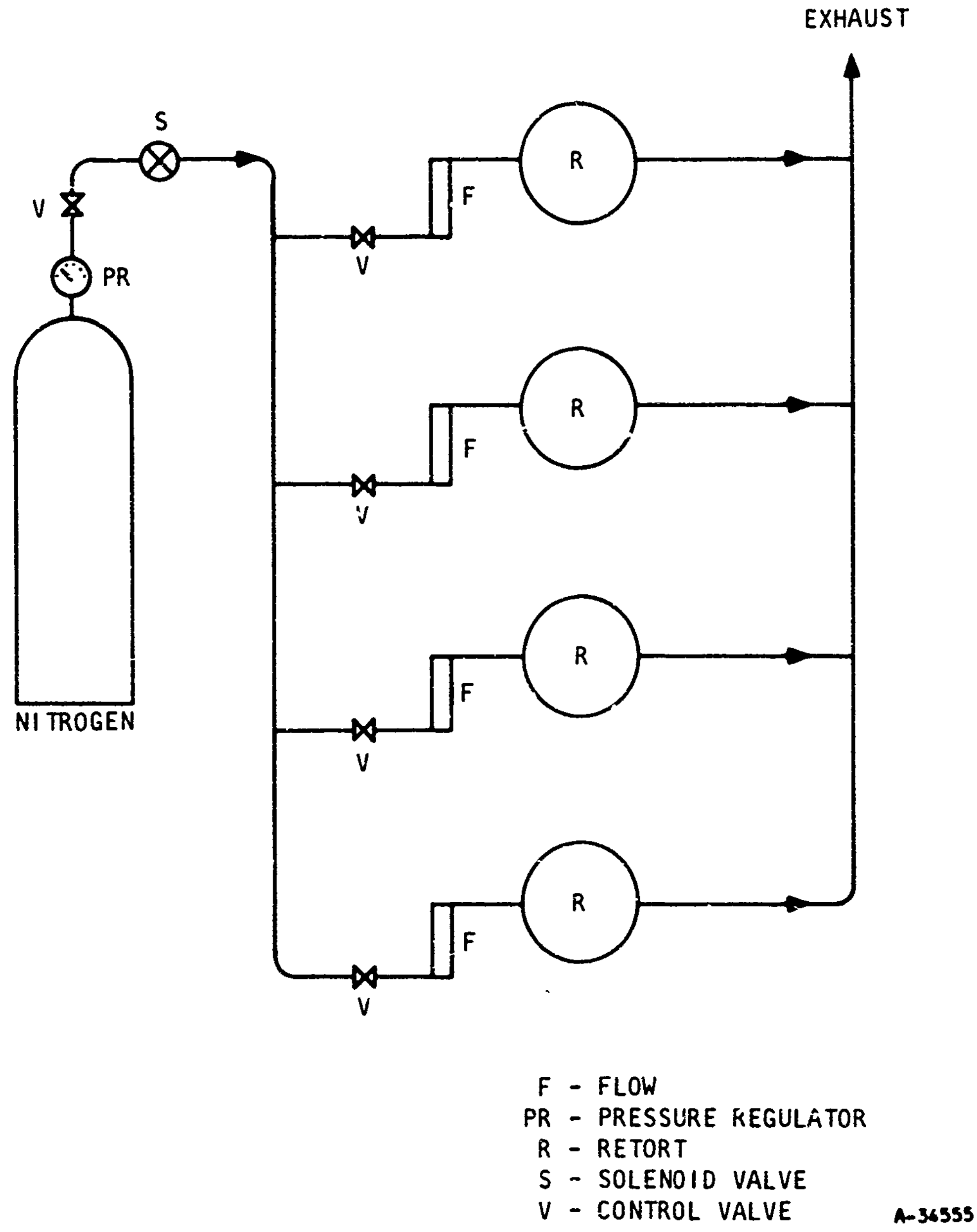


Figure 32. Cooling System

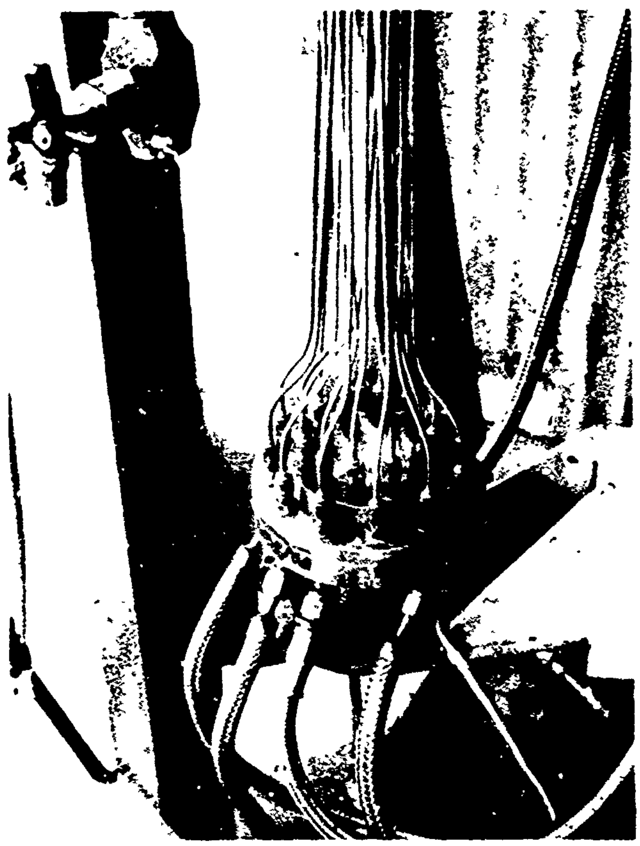


Figure 33. Test Header Plate F-9002

particulaly in aluminum and titanium bearing alloys. Previous experience at AiResearch has indicated that the chosen gas contains sufficient oxygen to produce the desired effect.

Test Results

A tabulation of results obtained to date can be found in Table VIII through XI. These initial points were plotted and compared against published sheet material stress rupture data so that pressure loadings for subsequent testing could be selected. An examination of these tables shows that, at 1500°F, most all samples tested at the high stress level failed earlier than planned, and that there is a wide scatter of results at the lower stress level. This is believed to be a result of a more severe corrosion condition existing at this temperature level than initially anticipated. Metallographic examination is being undertaken to conform this belief. Testing is continuing and is planned to be complete by mid June 1968.

TABLE VII. COMPOSITION OF ASTM D665 SYNTHETIC "SEA WATER"

Salt (a)	Formula	Grams per liter (b)
Sodium Chloride	$NaCl$	24.54
Magnesium Chloride	$MgCl_2 \cdot 6H_2O$	11.10
Sodium Sulfate	Na_2SO_4	4.09
Calcium Chloride	$CaCl_2$	1.16
Potassium Chloride	KCl	0.69
Sodium Bicarbonate	$NaHCO_3$	0.20
Potassium Bromide	KBr	0.10
Boric Acid	H_3BO_3	0.03
Strontium Chloride	$SrCl_2 \cdot 6H_2O$	0.04
Sodium Fluoride	NaF	0.003
	Total	41.953

(a) Use cp chemicals.

(b) Use distilled water.

TABLE VIII. HASTELLOY X TUBING STRESS RUPTURE DATA

Metal Temperature ^{0}F	Stress Level ksi	Time to Rupture - Hours Braze Alloys					
		Palniro I			J-8100		
	13.9	28.1	28.1	13.8	22.5	28.1	28.1
1500	6.5	35.4	341.3	cont.	218.7	141.8	cont.
	4.5		-			Continuing	
	20.8	87.9	175.9	225.2	225.4	207.4	270.2
1300	19		Continuing			Continuing	
	-		-			-	
	-		-			-	
1100	-		-			-	
	34.7		Continuing			Continuing	

TABLE IX. INCOLOY 800 TUBING STRESS RUPTURE DATA

Metal Temperature °F	Stress Level ksi	Time to Rupture - Hours, Braze Alloys: Palniro 7			Coast Metals 50B		
1500	8.3	28.1	10.6	16.4	29.8	10.6	11.8
	2.8	144.1	361.4	88.1	21.4	continuing	
	1.25		continuing				
1300	15.3	35.4	31.9	37.8	37.8	31.9	33.6
	9.4	166.9	201.7	249.5	217.5	251.0	cont.
	6.0		continuing				
1100	30.0	74.5	56.9	68.2	63.4	11.6	cont.
	28.0		continuing				
	-		-				

TABLE X. MULTIMET N-155 TUBING STRESS RUPTURE DATA

Metal Temperature °F	Stress Level ksi	Time to Rupture - Hours Braze Alloys					
		Palniro I			Nicrobraz 200		
1500	20	3.4	14.4	15.0	1.5	24.5	11.8
	10	101.4	83.6	101.4	126.6	91.8	81.0
	4.5		continuing			continuing	
1300	26.7	87.9	204.4	127.3	110.7	127.3	85
	22.5		continuing			continuing	
	-						
1100	-		-			-	
	-		-			-	
	40		continuing			continuing	

TABLE XI. CRES 347 TUBING STRESS RUPTURE DATA

Metal Temperature °F	Stress Level ksi	Time to Rupture - Hours Braze Alloys					
		Nicrobraz 135			Palniro 7		
1500	9.2	53.3	68.9	37.2	31.4	24.5	-
	3.0	77.4	86.1	18.7	6.1	17.8	6.8
	-		-			-	
1300	13.9	104.1	82.7	107.7	104.1	104.1	127.3
	10	continuing			continuing		
	-		-			-	
1100	26.9	continuing			continuing		