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Period Ending July 28, 1956.
TO: E. G. Bohlmann
FROM: R. E. Wacker and E. L. Compere

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SUMMARY OF CORROSION SAMPLE DATA FOR HRT MOCKUP PERIOD ENDING JULY 28, 1956.

The HRT mockup was shut down on July 28, 1956 to repair a leak in the Fulton-Sylphon block valve used to isolate a hydroclone by-pass loop from the main loop. At this time it was decided to remove and replace the corrosion samples in the loop and examine the loop for any evidence of corrosion attack.

I. GENERAL

During the reported period of mockup operation, March 26, 1956 to July 28, 1956, operation was on 0.042 m HNO_3SO_4 , 0.024 m H_2SO_4 , and 0.005 m CuSO_4 at 300°C and 2000 psi pressure. High pressure oxygen injection was employed. The pressurizer was operated at a temperature of 340°C. Total operating time during this period was 2453 hours. The over-all corrosion rate for this period, as determined by chemical analysis of the solution, was 1.5 mpy. Chloride concentration remained at < 3 ppm throughout the run. Several shut-downs were made during this period due to leaks and mechanical failures¹. On two occasions, the system was filled with new solution before resuming operation. The uranium concentration varied from 9.0 to 11.5 g U/l during this period.

Three 7-day runs were made to test the removal of rare-earth and corrosion product solids with a hydroclone². In each of these tests, approximately 360 g of solids in the form of iron, zirconium, copper, and rare-earth sulfates or oxides were injected into the mockup fuel system. Most of these solids were deposited in the system before reaching the hydroclone. The horizontal header between the main loop and the pressurizer contained a heavy deposit of solids on the bottom. After removing the solids, the bottom of this line had a red, rusty film and the top a black film separated from the bottom by a small area of shiny metal. No evidence of pitting or localized corrosion was found in the header. A sample of the solids from the header sent for chemical analysis gave the following result in weight percent:

Fe, 34.1; Cr, 3.88; Zr, 20.44; Ni, 0.26; Cu, 0.49; F⁻, 0.2; Cl⁻, 0.025; SO_4^{2-} , 1.56; and U, 1.2.

The uranium and sulfate were present in the soup; nickel and chromium are corrosion products; copper was both in the soup and solids injected; iron and zirconium were injected in the solids; and chloride and fluoride were impurities in the solids injected into the system. An analysis of an original solids batch showed concentrations (based on solids) of chloride and fluoride of 170 and 60 ppm, respectively.

The pipe cap at the pressurizer end of the horizontal header was cut from the system in order to examine this part of the system. In machining a "J" bevel on this pipe cap prior to welding it back into the system, pits and cracks were found on the inner surface about 0.2 in. through the 0.469-in. wall. This cap had been machined from 4-in. type 347 stainless steel barstock. Chemical analysis, along with Huey test results, of this stock are shown in Table I. The Huey tests were conducted in accordance with ASTM Specification

A-262-52T and Specification HRP-240a on specimens sensitized for one hour at 1200°F. The results presented are average values expressed in mils per month of corrosion for five separate successive forty-eight hour periods.

TABLE I
CHEMICAL AND HUEY TESTS ON CAP MATERIAL

Cr	Ni	Mn	C	Si	S	P	Nb
19.1	11.4	1.58	0.049	0.56	0.013	0.022	0.84
<u>Huey Test Values</u>				<u>mpm - avg.</u>			
1st Test				1.0			
2nd Test				0.7			

This cap had been in the system for a total of 3662 hr of operation. It had been removed from the loop on two previous occasions and welded back in place. The cracks were approximately one-half inch from the weld area. The bottom of the header leg was gamma-graphed and no evidence of further cracking was detected.

A type 347 stainless steel O-ring used in the main circulating loop flange where the sample holder is located had a black film indicating some leakage into one of the Elange grooves. A dye check of this ring showed several pin-point holes in the seating surface. Both the pressurizer header cap and the O-ring have been submitted to metallography for examination and a separate report will be issued by that group on their findings.

II. SAMPLES

The corrosion coupons were installed in the mockup March 26, 1956. Coupons made of titanium 75A, Zircaloy-2 and types 309 SCb and 347 stainless steel in a titanium holder were exposed in the downstream side of the main circulating line. The titanium samples had a gold colored film with patches of blue; the stainless steel samples were rusty colored; and the Zircaloy-2 samples had a grey-black, tightly adherent film. Type 347 stainless steel coupons mounted on a stainless steel bar extended through the vertical leg of the pressurizer. These samples had a heavy rust colored scale on the bottom samples and thin, black films on the top samples. Complete data and photographs of the main line samples and holder before exposure are given in a previous report³. Corrosion data for all samples are presented in Tables II and III.

Samples of the film or scale from several of the samples and holders, both main circulating line and pressurizer leg holders, were submitted for x-ray and spectrographic analysis. Results of these analyses are shown in Table IV. It will be seen that all scales had essentially the same composition except the scale from sample MK-25 had a higher uranium, nickel, and copper content. No reason is known for this scale being different from the scale from the holder which was in the same vicinity.

Photographs of the sample holders as removed, as well as photographs of the individual coupons both before and after defilming, are shown in the accompanying photographs. Figures 1 through 4 show the holder in the circulating line as removed. Figures 5 through 7 show the bottom half of the pressurizer leg holder as removed. The large deposit of solids on the bottom of the holder and bottom coupon are easily seen. Figures 8 through 15 show the top and bottom of the circulating line samples as removed. Figures 16 through 31 show all surfaces of these coupons after defilming. Figures 32 through 35 show the pressurizer leg coupons after defilming. Figures 36 through 41 are enlarged views of the pressurizer leg coupons, showing the localized attack which occurred.

The appendix contains a tabulation of the negative plate numbers of all photographs.

A similar array of new samples has been inserted in the mockup for the next period of operation.

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1. HRP Quarterly Progress Report for July 31, 1956. ORNL-2148, p 17-18.
 2. HRP Quarterly Progress Report for July 31, 1956. ORNL-2148, p 118-119.
 3. Corrosion Sample Data and Fabrication. ORNL-CF-56-6-83.

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TABLE II

MAIN CIRCULATING LINE SAMPLES

Form	Sample		Coupon Position from Flange End of Holder	Sample Area cm ²	Weight Change, mg/cm ²		Corrosion Rate, mpy Defilmed	Appearance
	No.	Type			As Removed	Defilmed		
Coupon	H-108	Ti-75A	1	20.7	+ 3.90	- 0.12	0.04	Gold-colored film. Patches of blue.
Coupon	H-109	Ti-75A	8	20.7	+ 4.07	- 0.23	0.07	" " " " " "
Coupon	H-99	Zircaloy-2	2	20.7	+ 8.12	Gain	*	Gray-black, tightly adherent film.
Coupon	H-100	Zircaloy-2	7	20.7	+10.97	Gain	*	" " " " "
Coupon	H-102	347 SS	3	20.7	+ 6.78	- 0.86	0.15	Rust-colored scale. Uniform attack.
Coupon	H-103	347 SS	6	20.7	+ 5.94	- 0.84	0.15	" " " " "
Coupon	H-81	309 SCb	4	20.7	+ 7.85	- 1.08	0.19	" " " " "
Coupon	H-82	309 SCb	5	20.7	+ 8.73	- 1.05	0.18	" " " " "
Screw	MJA	Ti RC-55	--	4.3	--	0.51	0.16	
Screw	MJC	Ti RC-55	--	4.3	--	- 0.88	0.27	

Samples exposed for 2452.5 hr in uranyl sulfate solution.

* Tightly adherent film could not be removed by standard defilming methods.

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TABLE IIIPRESSURIZER VERTICAL LEG SAMPLES

Coupon No.	Position* from Top of Pressurizer	Weight Change, mg/cm ²		Corrosion Rate, mpy	Appearance
		As Removed	Defilmed	Defilmed	
MK-20	1	+0.04	-0.24	0.04	Dark gray stain. Moderate pit attack near drilled hole where fastened.
MK-2	2	+0.07	-0.45	0.08	Dark gray stain. Few pits. Heavy attack at drilled hole where fastened.
MK-21	3	+0.04	-0.25	0.04	Dark gray stain. Moderate pit attack near drilled hole where fastened.
MK-4	4	+0.06	-0.25	0.04	Dark gray stain. Few pits. Heavy attack at drilled hole where fastened.
MK-22	5	-0.01	-0.35	0.06	Dark gray stain. Moderate pit attack near drilled hole where fastened.
MK-23	6	-0.16	-0.95	0.17	Dark gray stain. Heavy pit attack over-all.
MK-7	7	+0.37	-0.62	0.11	Dark gray stain. Few pits. Heavy attack at drilled hole where fastened.
MK-24	8	+0.33	-0.35	0.06	Dark gray stain. Moderate pit attack over-all.
MK-25	9	+2.33	-1.38	0.24	Dark gray stain. Heavy pit attack over-all. Few large deep pits. Cracks on ends and edges.

All coupons exposed for 2452.5 hr in uranyl sulfate solution. Exposed area of coupon = 17.7 cm².

* Positions 1 through 3 in pressurizer vapor phase. Position 9 in water-soup interface in horizontal header to pressurizer. Others in water region of vertical leg.

TABLE IV
ANALYSIS OF SAMPLE SCALES

Analysis	Weight Percent					
	Sample A ^a		Sample B ^b		Sample C ^c	
	Spec.	X-Ray	Spec.	X-Ray	Spec.	X-Ray
Fe	> 10		> 10		> 10	
Cr	3		2		3	
U	2		> 10		2	
Zr	> 5		> 5		> 5	
Ni	0.2		1		0.5	
Cu	0.1		1		0.5	
Other ^d	(e)		(e)		(e)	
Fe ₂ O ₃		75-90		75-90		75-90
ZrO ₂		10-40		10-40		10-40
UO ₂		1-5		1-5		1-5
NiO				1-5		
CuO				1-5		

- Scale removed from the bottom of the pressurizer leg sample holder. Also contained 170 ppm chloride.
- Scale from pressurizer leg sample MK-25. No analysis for chloride.
- Scale removed from the circulating line sample holder. No analysis for chloride.
- Solid solution of Cr₂O₃ with Fe₂O₃.
- Small amounts of rare-earths and corrosion products.

APPENDIX

SAMPLE PHOTOGRAPHS¹

<u>Sample Number</u>	<u>Holder</u>	<u>Corrosion Lab Plate No.</u>
H-108	Main Line	1646, 1662, 1666, 1668
H-109	Main Line	1646, 1662, 1667, 1669
H- 99	Main Line	1647, 1663, 1666, 1668
H-100	Main Line	1647, 1663, 1667, 1669
H-102	Main Line	1648, 1664, 1666, 1668
H-103	Main Line	1648, 1664, 1667, 1669
H-81	Main Line	1649, 1665, 1666, 1668
H-82	Main Line	1649, 1665, 1667, 1669
MK-2	Pressurizer	1644, 1645, 1670
MK-23	Pressurizer	1671
MK-7	Pressurizer	1644, 1645
MK-25	Pressurizer	1644, 1645, 1672

SAMPLE HOLDER PHOTOGRAPHS²

<u>Figure No.</u>	<u>Holder</u>	<u>Y-12 Plate No.</u>
1	Main Line	26881
2	Main Line	26882
3	Main Line	26883
4	Main Line	26884
5	Pressurizer	26878
6	Pressurizer	26879
7	Pressurizer	26880

SAMPLE PHOTOGRAPH FIGURES

<u>Figure No.</u>	<u>Corrosion Lab Plate No.</u>
8 and 9	1646
10 and 11	1647
12 and 13	1648
14 and 15	1649
16 and 17	1662
18 and 19	1663
20 and 21	1664
22 and 23	1665
24 and 25	1666
26 and 27	1667
28 and 29	1668
30 and 31	1669
32 and 33	1644
34 and 35	1645
36 and 37	1670
38 and 39	1671
40 and 41	1672

¹ Sample photographic plates are on file with J. L. English of the Materials Research Section, Reactor Experimental Engineering Division, Oak Ridge National Laboratory.

² Sample photographic plates are on file with the Y-12 Photographic Section.

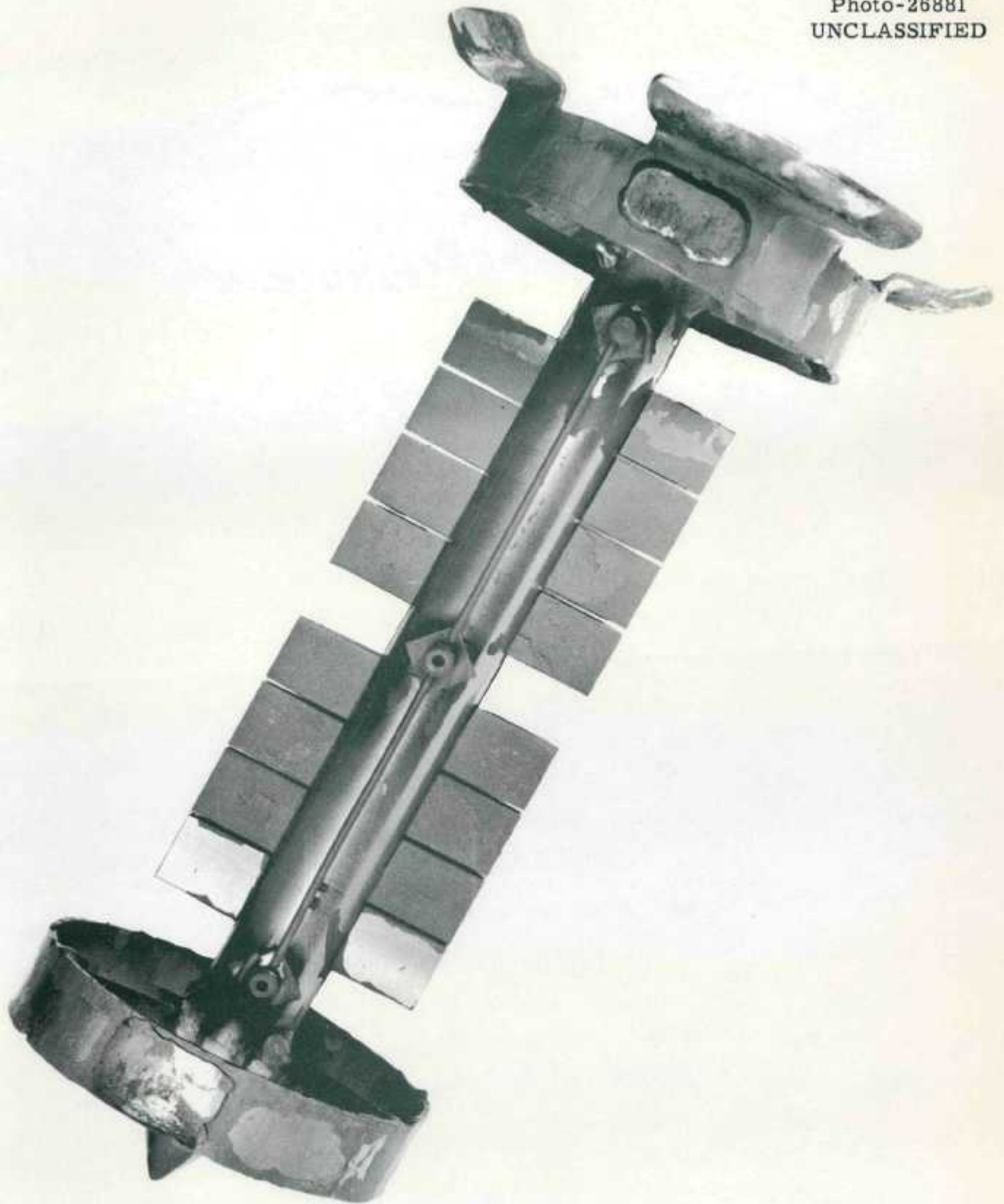


Figure 1

-10-
Photo-26882
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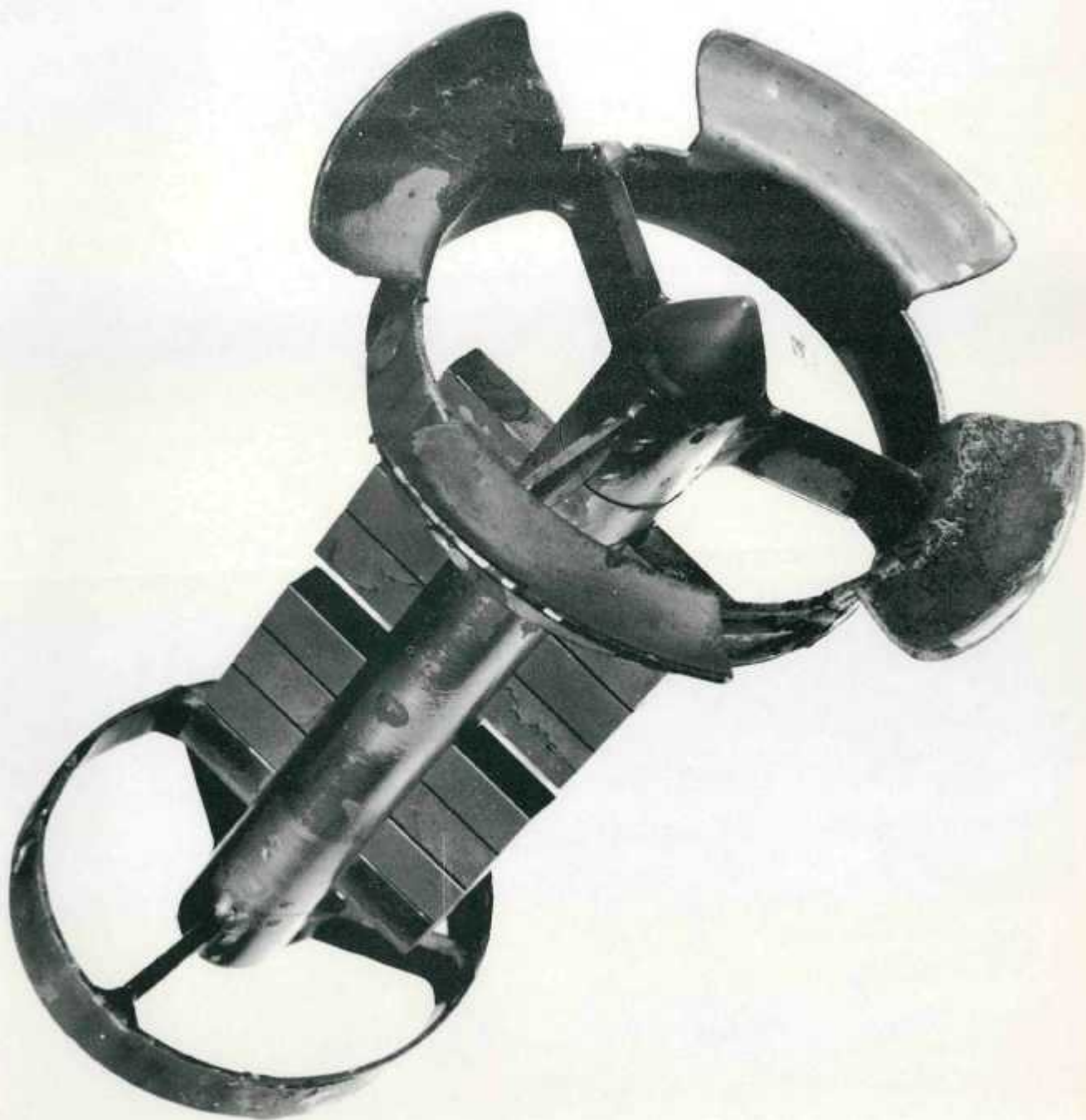


Figure 2



Figure 3

-12-
Photo-26884
UNCLASSIFIED

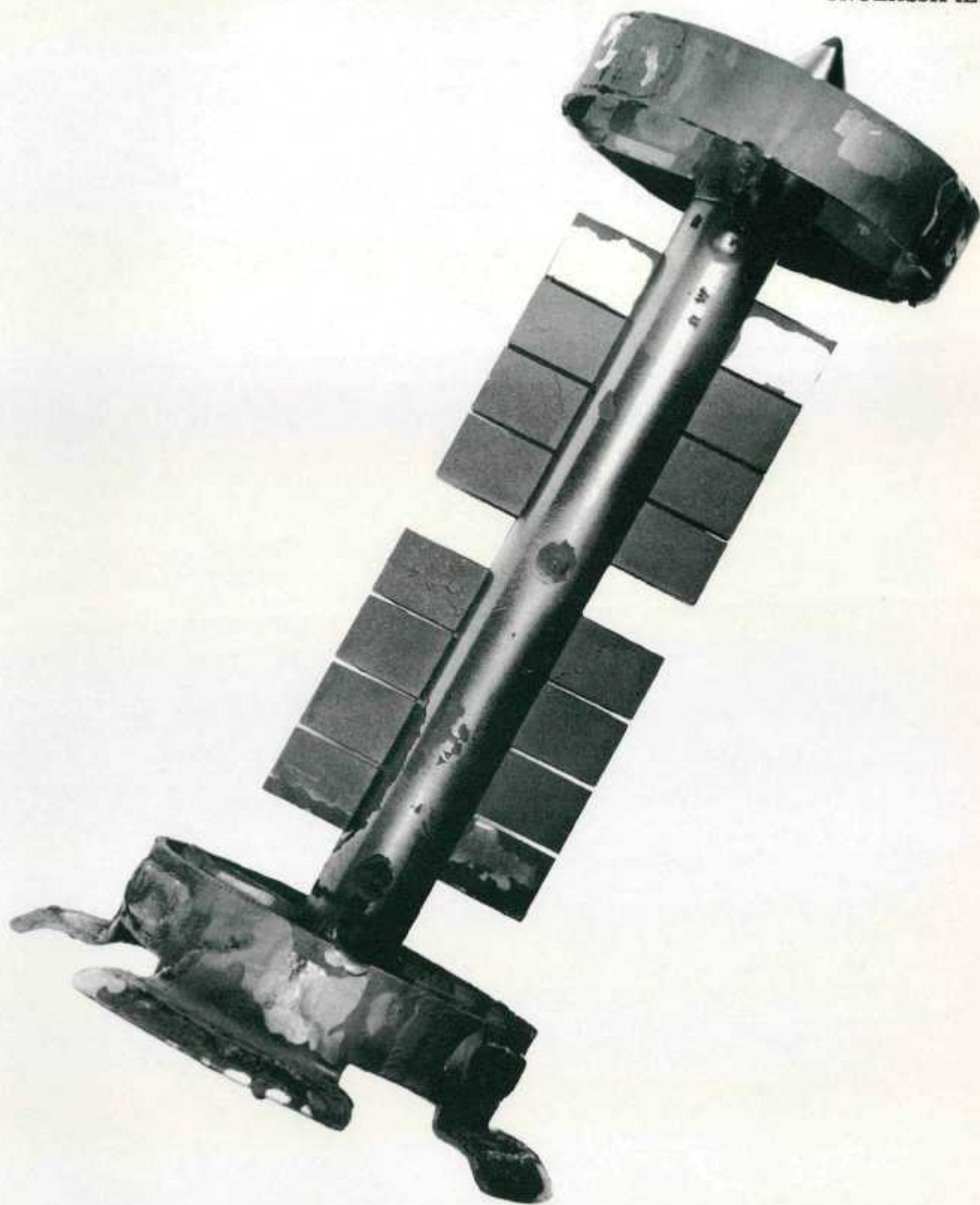


Figure 4

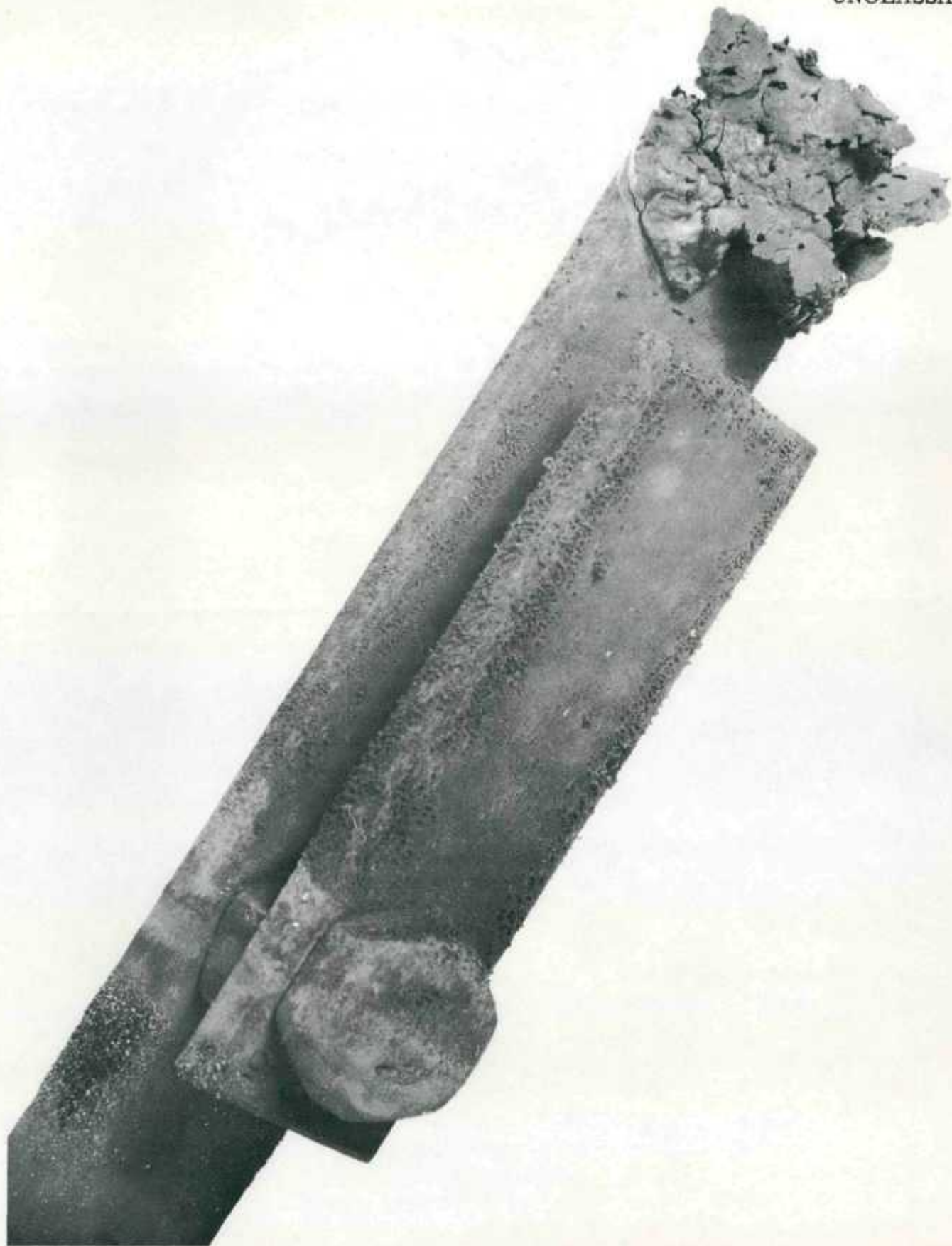


Figure 5



Figure 6

-15-
Photo-26880
UNCLASSIFIED



Figure 7



108



109



108



109

Fig. 8 - Coupons As Removed. (2.6X)

Fig. 9 - Coupons As Removed. (2.6X)



99



100



99

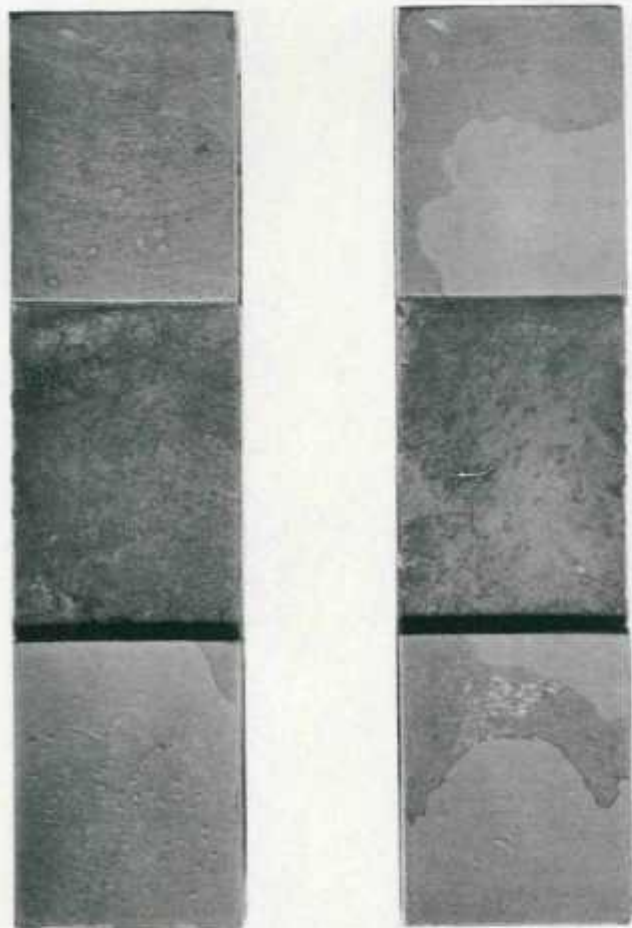


100

Fig. 10 - Coupons as Removed. (2.6X)

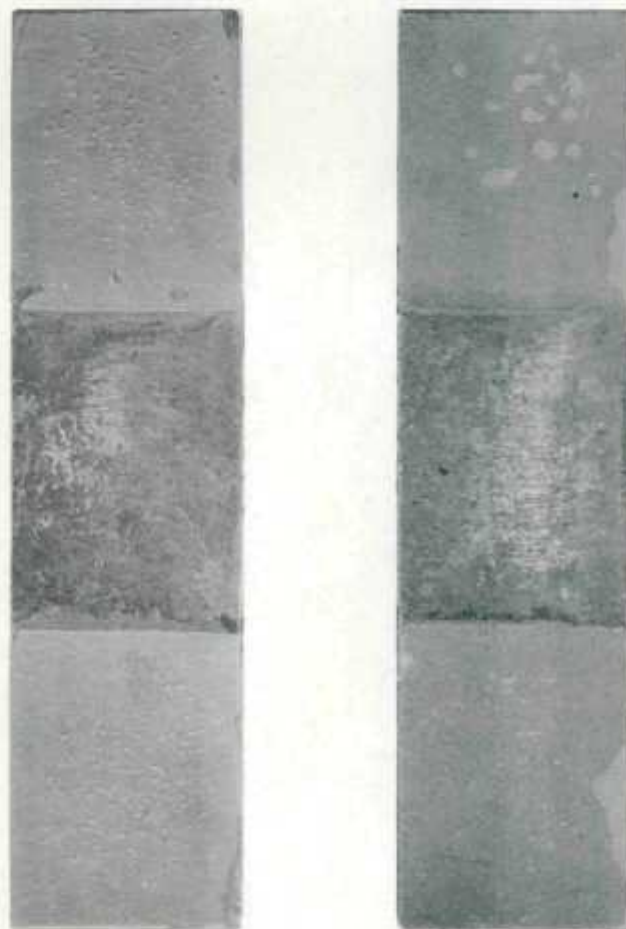
Fig. 11 - Coupons as Removed. (2.6X)

18



102

103



102

103

Fig. 12 - Coupons as Removed. (2.6X)

Fig. 13 - Coupons as Removed. (2.6X)



81



82



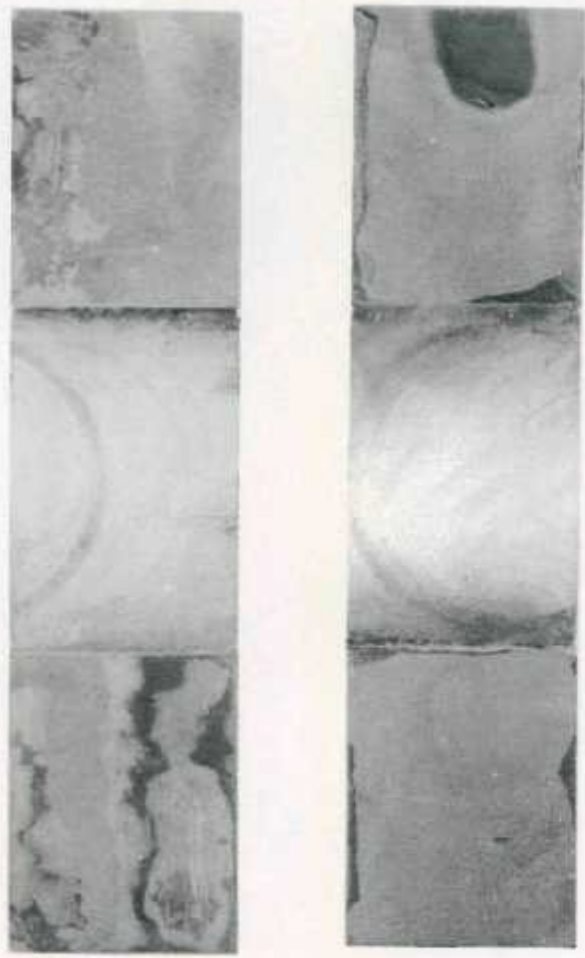
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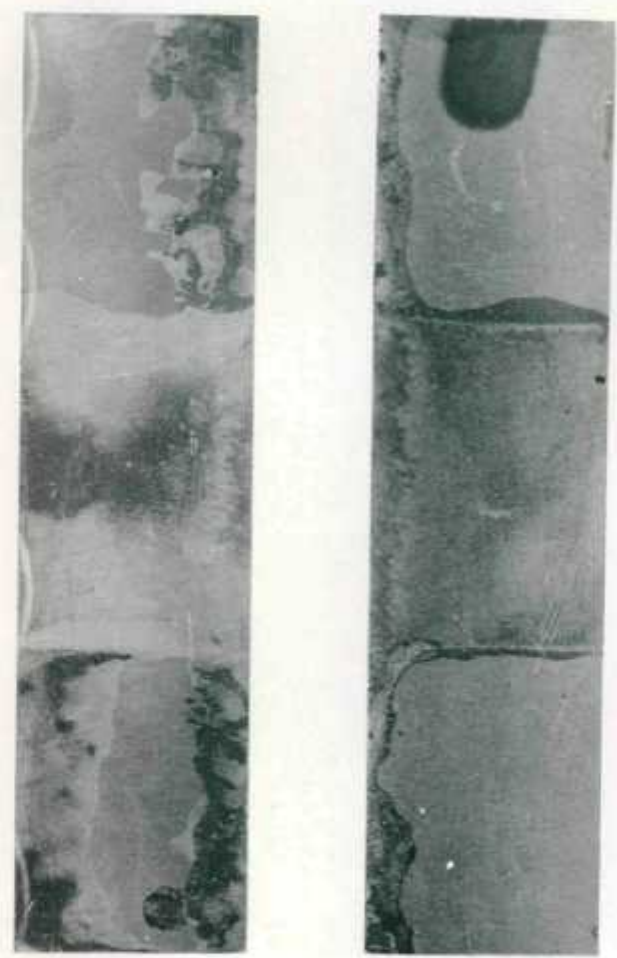
82

Fig. 14 - Coupons as Removed (2.6X)

Fig. 15 - Coupons as Removed. (2.6X)



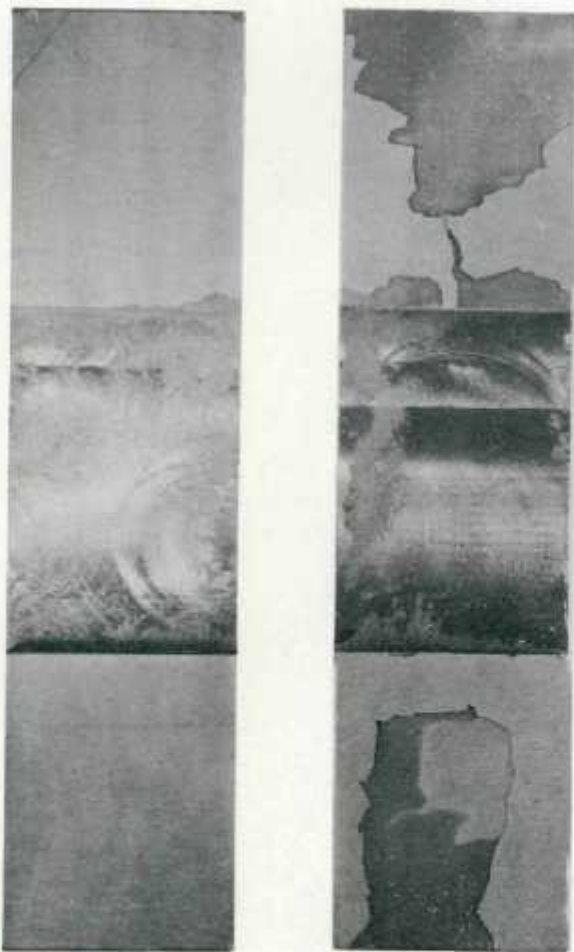
H108 H109



H108 H109

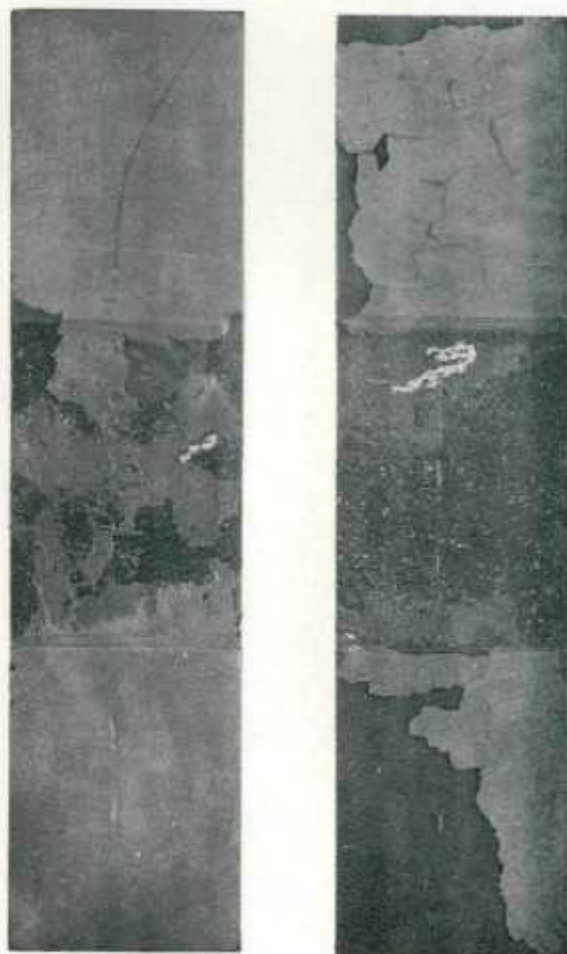
Fig. 16 - Coupons Defilmed. (2.5X)

Fig. 17 - Coupons Defilmed. (2.5X)



H99

H100



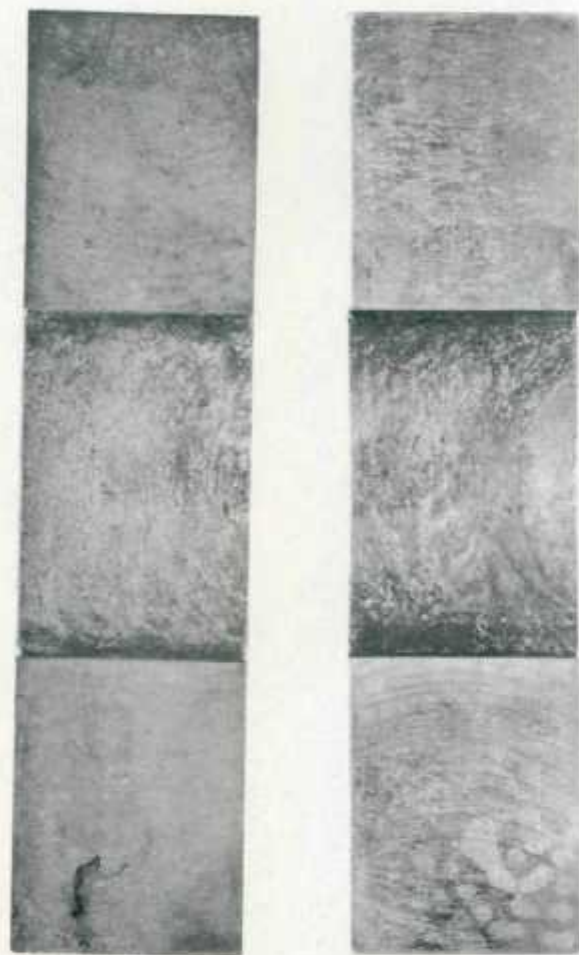
H99

H100

Fig. 18 - Coupons Partially Defilmed. (2.5X)

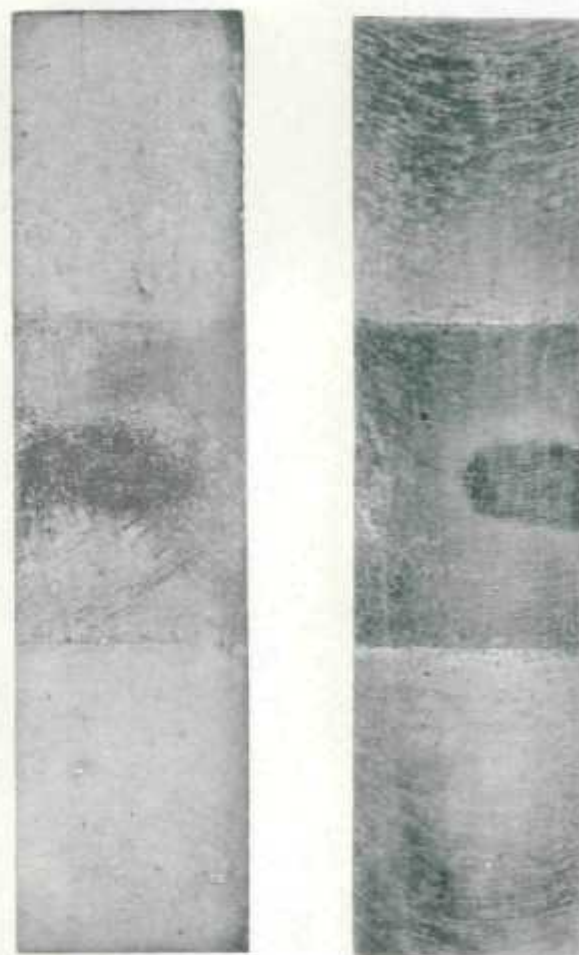
Fig. 19 - Coupons Partially Defilmed. (2.5X)

22



H102 H103

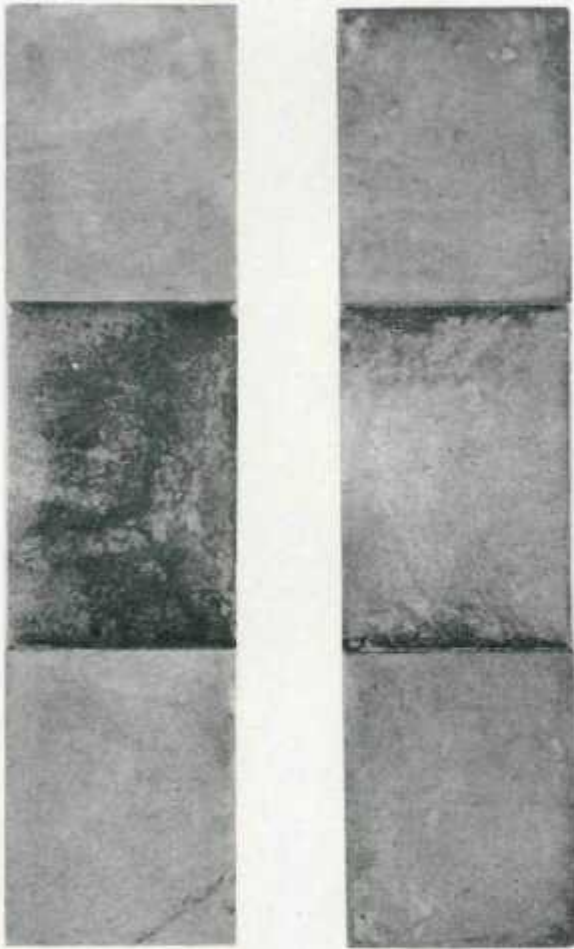
Fig. 20 Coupons Defilmed. (2.5X)



H102 H103

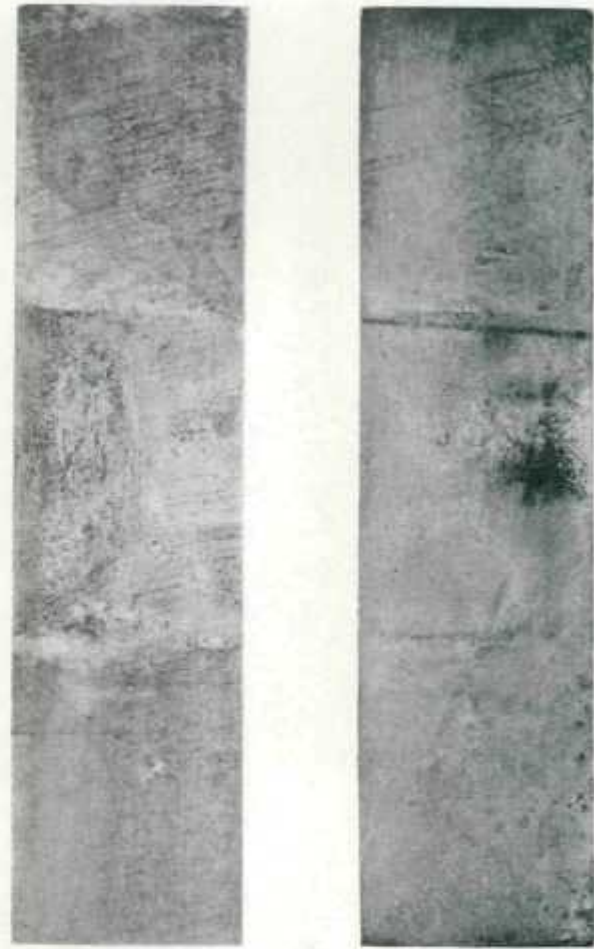
Fig. 21 Coupons Defilmed. (2.5X).

23



H81

H82



H81

H82

Fig. 22 Coupons Defilmed. (2.5X)

Fig. 23 - Coupons Defilmed. (2.5X)

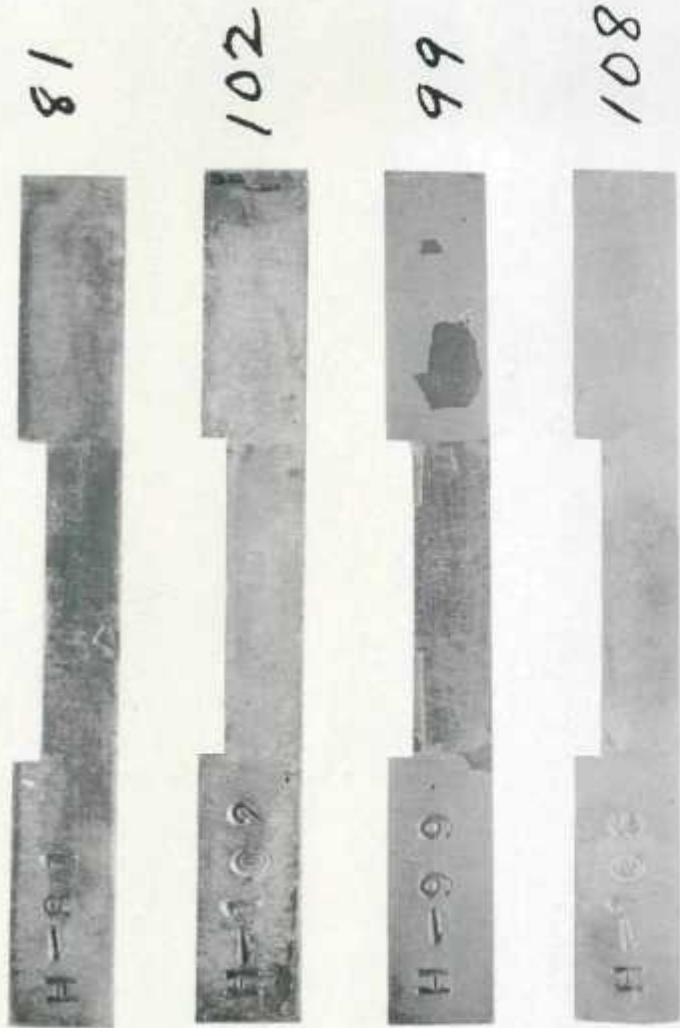


Fig. 24 Coupon Sides Defilmed. (2.5X)

24

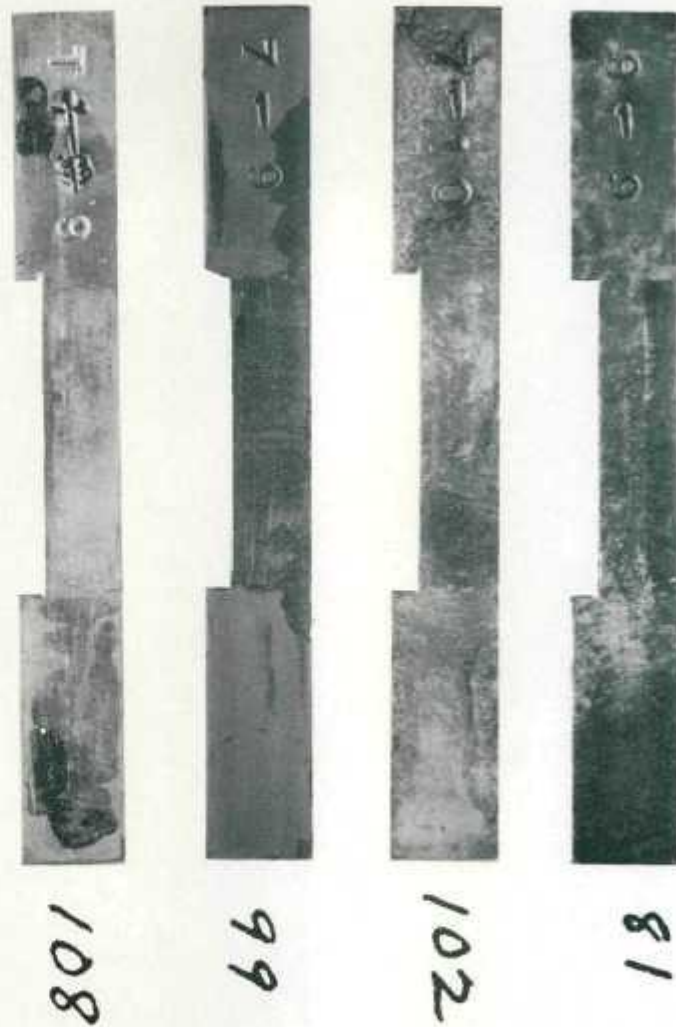
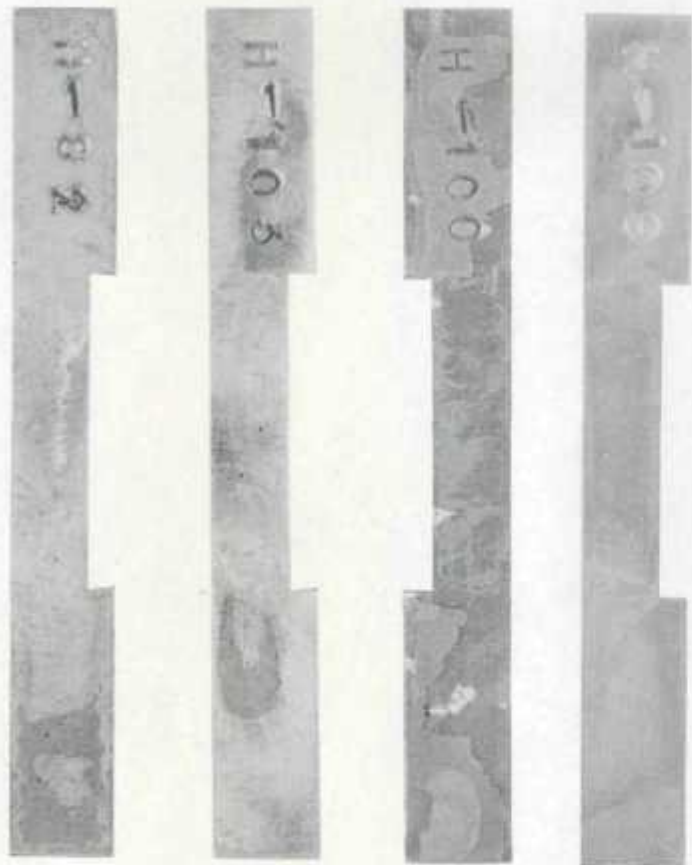
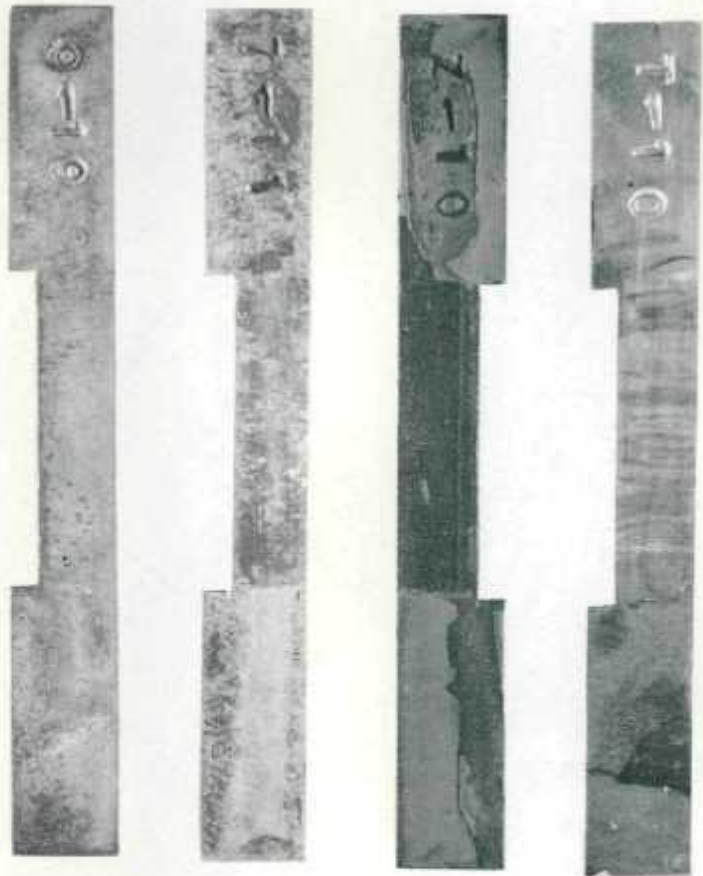


Fig. 25 Coupon Sides Defilmed. (2.5X)



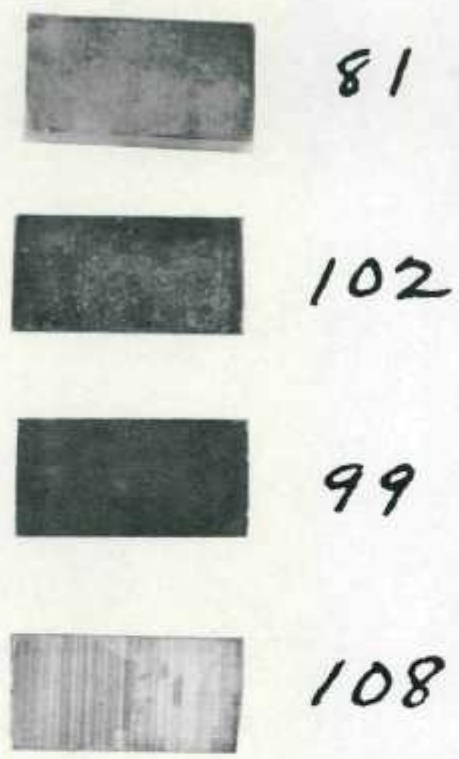
82
103
100
109

Fig. 26 Coupon Sides Defilmed. (2.5X)



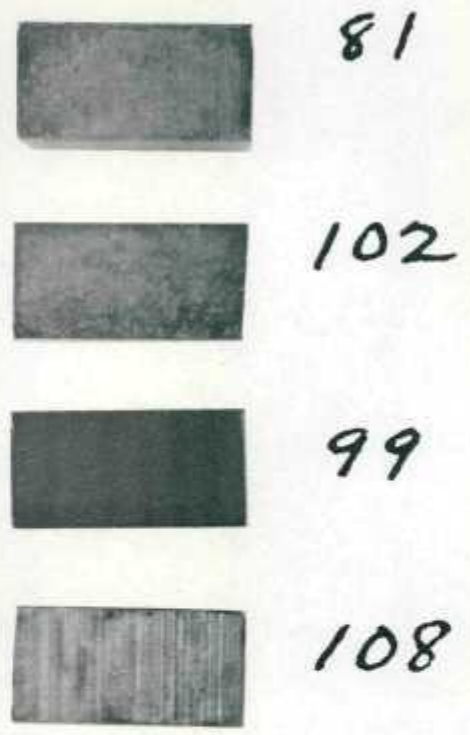
82
103
100
109

Fig. 27 Coupon Sides Defilmed. (2.5X)



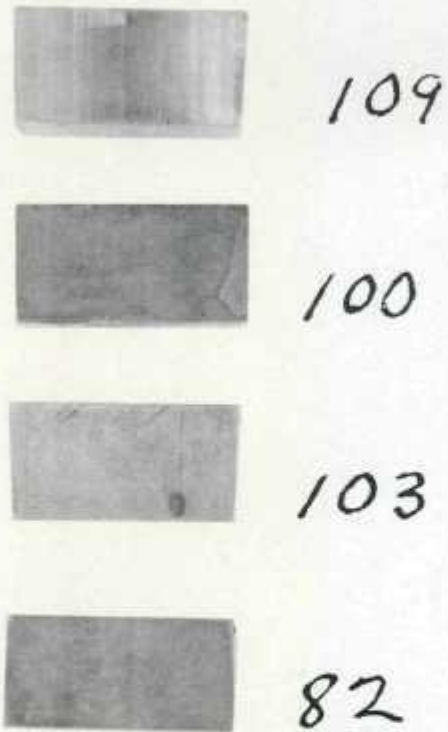
END NEAR NUMBER

Fig. 28 Coupon Ends Defilmed. (2.5X)



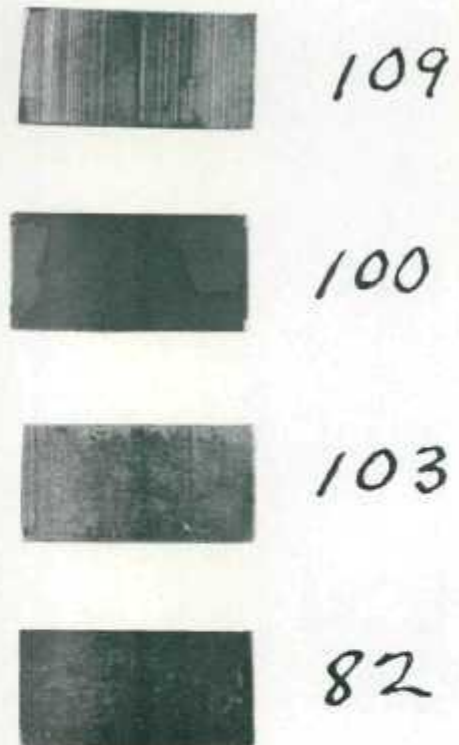
END AWAY FROM NUMBER

Fig. 29 Coupon Ends Defilmed. (2.5X)



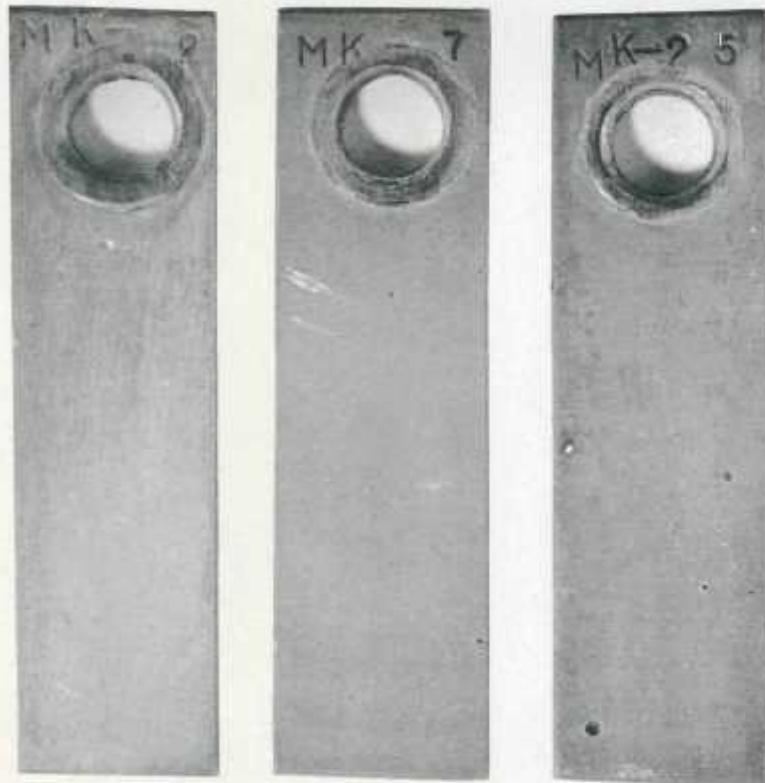
END NEAR NUMBER

Fig. 30 Coupon Ends Defilmed. (2.5X)



END AWAY FROM NUMBER

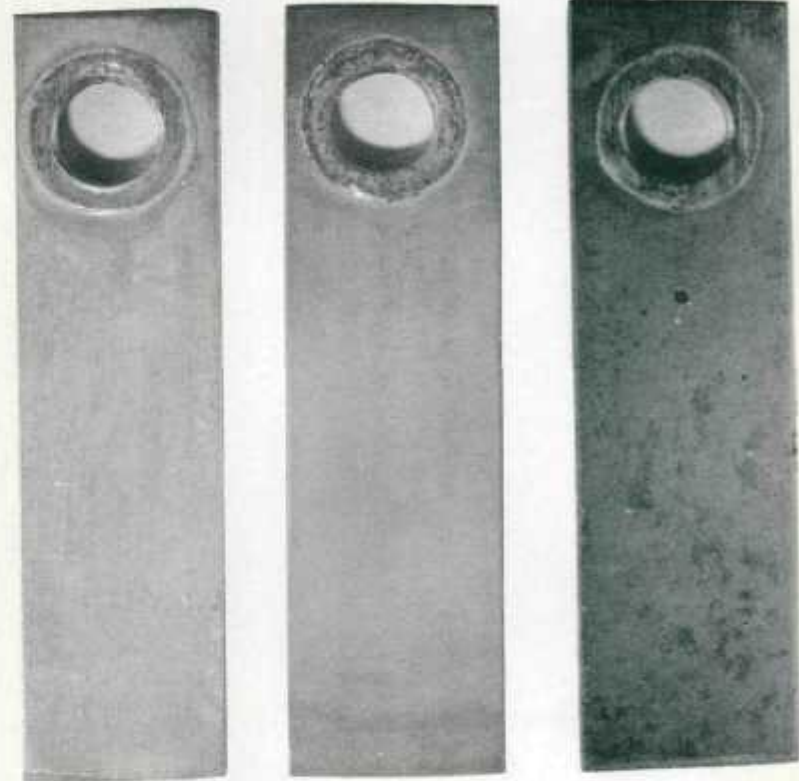
Fig. 31 Coupon Ends Defilmed. (2.5X)



MK-2

MK-7

MK-25



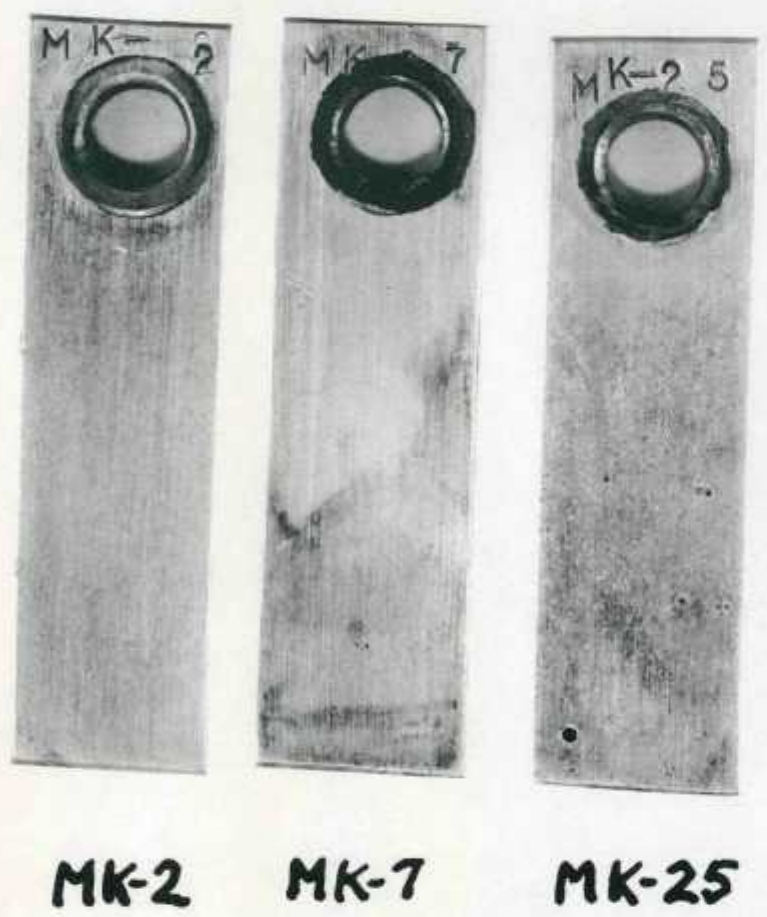
MK-2

MK-7

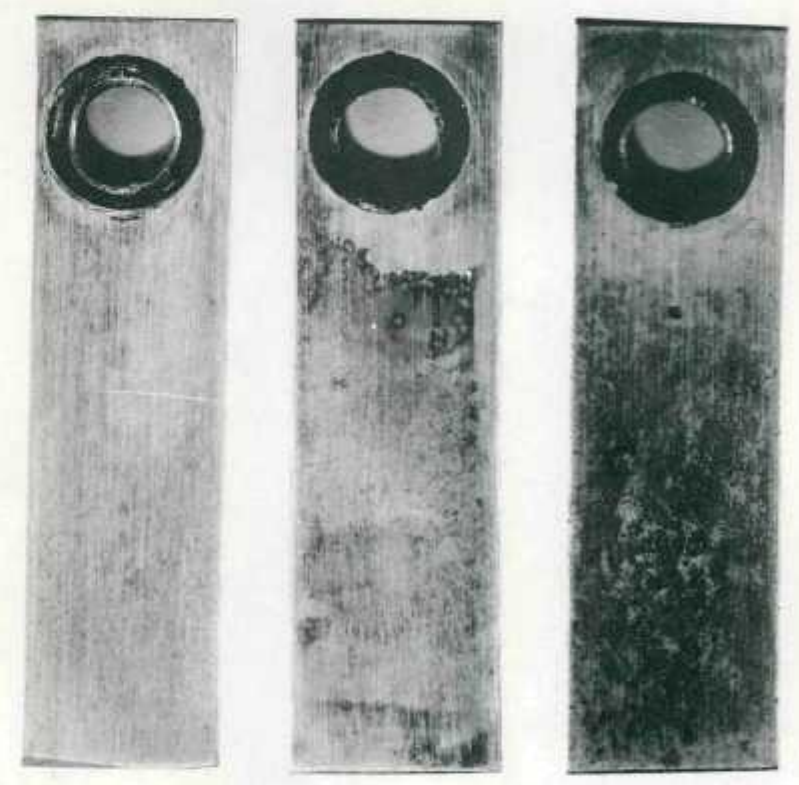
MK-25

Fig. 32 Coupons as Removed. (2.3X)

Fig. 33 Coupons as Removed. (2.3X)



MK-2 MK-7 MK-25



MK-2 MK-7 MK-25

Fig. 34 Coupons Defilmed. (2.3X)

Fig. 35 Coupons Defilmed. (2.3X)

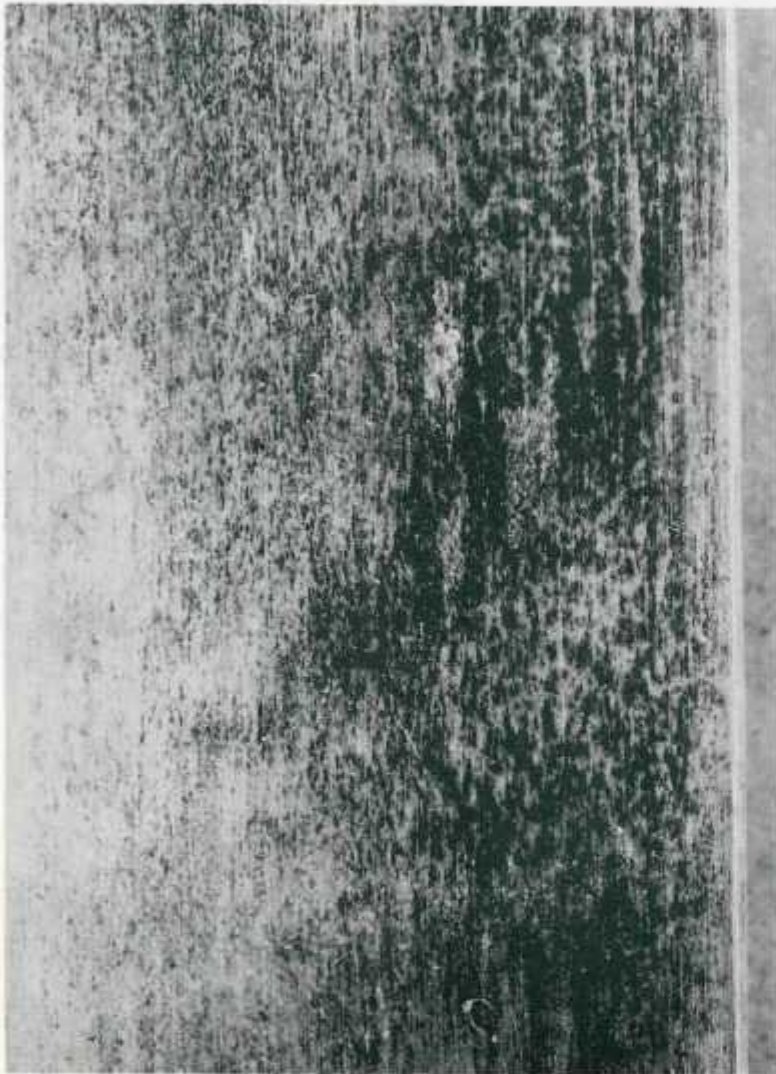


Fig. 36 Coupon MK-20 Defilmed. (10X)



Fig. 37 - Coupon MK-20 Defilmed. (10X)

Plate-1670-1
Plate-1670-2

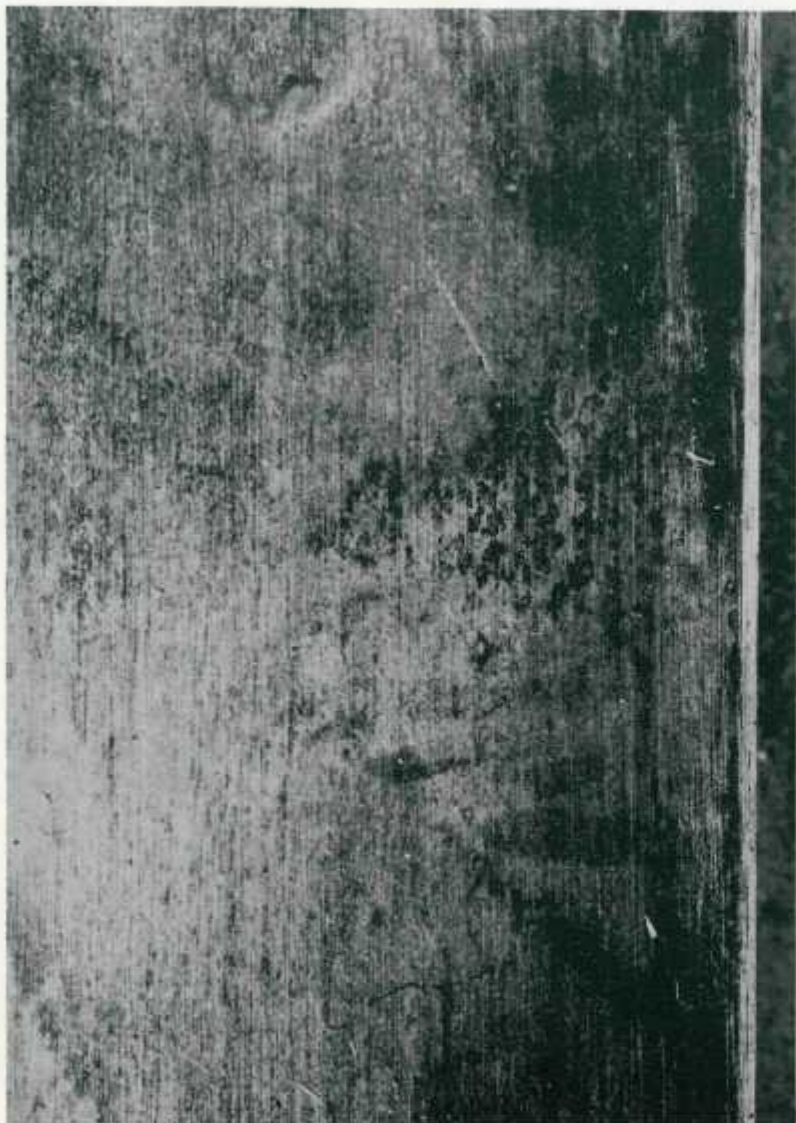


Fig. 38 - Coupon MK-23 Defilmed. (10X)



Fig. 39 - Coupon MK-23 Defilmed. (10X)



Fig. 40 - Coupon MK-25 Defilmed. (10X)

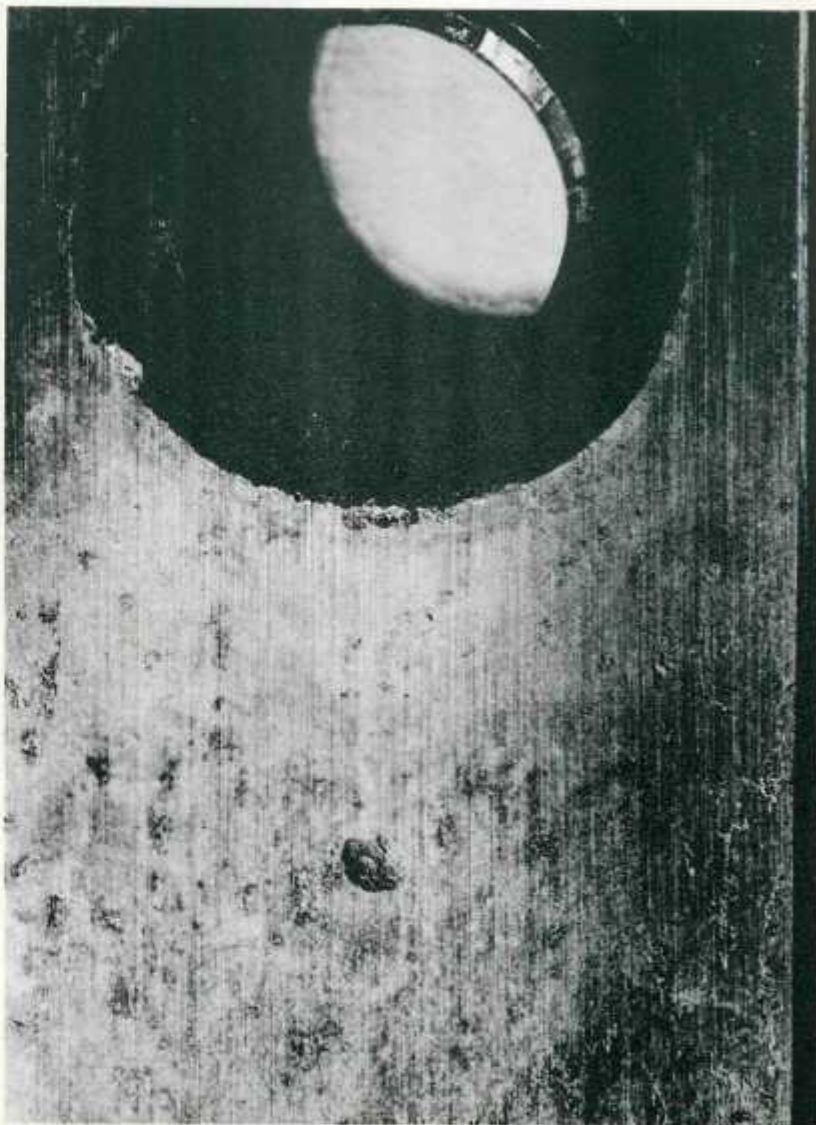


Fig. 41 - Coupon MK-25 Defilmed. (10X)

Plate-1672-1
Plate-1672-2

1-32