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SUBJECT: Metallographic Exam. of Type 347 SS And Titanium 75A Corrosion Specs.
Exposed to Vapor Above Oxygenated 0.14 m UO_2SO_4 Containing Chlorine
or Iodine. Metallographic Report No. 21 (Y-12)

TO: G. E. Moore

FROM: T. M. Kegley, Jr.

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Introduction And Procedure:

Type 347 stainless steel and titanium 75A specimens were received for metallographic study. These specimens were selected from a group of specimens which had been subjected to vapor phase exposure above oxygenated $0.14 \text{ M } \text{UO}_2\text{SO}_4$ solutions containing varying amounts of chlorine or iodine. The test conditions, described briefly in the accompanying table, are described more fully in ORNL-CF-55-12-70.

The specimens were first examined with a low power microscope and appropriate photomicrographs made of the specimen surfaces. Sections of each of the specimens were then taken for metallographic examination.

Results:

The metallographic results are presented mainly in the photographs and table accompanying this report.

Features, which were particularly striking, were the transgranular cracking and undercutting experienced by the 347 SS specimens which were not subjected to applied stresses. An example of the transgranular cracking found in a specimen (C3-25) exposed to vapor above a solution containing 10 ppm Cl^- is given in Figs. 30 to 35. Undercutting, as used in this report, is the internal deterioration of metal which occurred principally in thin 19 mil type 347 SS sheet specimens. Such undercutting is illustrated in Figs. 4 and 5, specimen C1-1, and Figs. 25 and 26, specimen C3-25. Presumably such undercutting occurred from the enlargement by corrosion of deep surface pits.

The attack of type 347 SS by chloride-containing solutions in some instances appeared to be of a localized nature. This was particularly true in a 1/4 inch wafer specimen (C3-S2) and a 19 mil sheet specimen (C4-42). The localized attack resulted in pits as shown in Figs. 12, 13, 47, 48, 49, 50, 51 and 52. The pits in the 1/4 inch wafer specimen may be related in some way to the rolling direction. No explanation is evident for the pits in the 19 mil sheet specimen (C4-42). The intergranular nature of the attack in one of the pits in specimen C4-42 is shown in Fig. 52.

The use of sheared corrosion specimens, such as the 19 mil sheet specimens, probably intensified any stress corrosion effects. This was particularly noticeable in the cracking found in the sheared region of specimen C3-25 as illustrated in Fig. 29.

Two type 347 SS 19 mil control specimens exposed above oxygenated 0.14 M UO_2SO_4 solutions to which no chlorine (Cl^- or Cl_2) was added (residual chlorine content about 2 ppm) showed no cracking or undercutting as was experienced by the specimens exposed above solutions which contained chlorine additions. One specimen, I1-81, did show slight surface roughening following exposure, see Fig. 71.

Two transgranular cracks were also found in one type 347 SS 19 mil specimen exposed above an oxygenated 0.14 M UO_2SO_4 solution containing 7 ppm iodine ($\text{I}_2\text{-H}_2\text{O}$). See Fig. 75. Three other 347 sheet specimens exposed above oxygenated 0.14 M UO_2SO_4 solutions containing 7 or 9 ppm iodine ($\text{I}_2\text{-H}_2\text{O}$ or KI) exhibited no cracking. Pitting also occurred in the iodine specimens but was not as serious as the undercutting which occurred in the specimens exposed to vapor above solutions containing chlorine.

The two titanium specimens, T2 and T10 appeared to have no appreciable attack after vapor exposure above oxygenated 0.14 M UO_2SO_4 solutions containing 80 and 90 ppm Cl ($\text{Cl}_2\text{-H}_2\text{O}$).

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Transgranular cracking was found in stress specimen C8-42 which was stressed to a maximum outer fiber stress of 60,000 psi in a three-point loaded beam type stress assembly and exposed to vapor above oxygenated 0.14 M UO_2SO_4 solution containing ≤ 200 ppm chlorine ($\text{Cl}_2\text{-H}_2\text{O}$). The transgranular cracking in stress specimen C8-42 is shown in Figs. 56 and 60. A deep pit (90 mils depth) at the edge of stress-base specimen C8-53, which was exposed to vapor above oxygenated 0.14 M UO_2SO_4 solution containing 90 ppm chlorine ($\text{Cl}_2\text{-H}_2\text{O}$), is shown in Figs. 62 and 63. A pit (60 mils depth) in metal adjacent to weld in stress-base specimen C8-53 is illustrated in Figs. 64 and 65.

Summary:

Although the use of sheared specimens may account for some of the extreme corrosion effects noted, transgranular (stress corrosion) cracking and undercutting have been found in the vapor phase corrosion of 347 SS specimens (free of applied stresses) exposed above oxygenated 0.14 M UO_2SO_4 solutions containing as little as 10 ppm chlorine ($\text{Cl}_2\text{-H}_2\text{O}$). Transgranular cracking was found in one 347 SS specimen exposed above 0.14 M UO_2SO_4 solution containing 7 ppm iodine ($\text{I}_2\text{-H}_2\text{O}$). However, three other 347 sheet specimens exposed above 0.14 M UO_2SO_4 solutions containing iodine exhibited no cracking. Two specimens of titanium 75A showed good corrosion resistance when exposed above 0.14 M UO_2SO_4 solutions containing 80 and 90 ppm chlorine ($\text{Cl}_2\text{-H}_2\text{O}$).

Reference:

G. E. Moore, "Summary of Laboratory Corrosion Tests. The Solution and Vapor Phase Corrosion of Type 347 SS, Ti 75A, and Zircaloy-2 Exposed to 0.14 M Uranyl Sulfate in the Absence and Presence of Chlorine or Iodine." Dec. 12, 1955, ORNL-CP-55-12-70.

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TEST CONDITIONS AND METALLOGRAPHIC OBSERVATIONS FOR TYPE 347 SS AND
T1-75A SPECIMENS EXPOSED TO VAPOR ABOVE OXYGENATED 0.14 M URANYL SULFATE

Specimen	Material	Temp (°C)	Additions	ppm Cl or I	Time Hrs.	
C1-1	347 SS 19 mil sheet	100 ¹ (250)	NiCl ₂ + 360 psi O ₂ at 100°C	2 10 20 50	840 504 504 2160	Considerable undercutting. Transgranular cracks near edge.
C3-52	347 SS wafer	100 ¹ (250)	Cl ₂ -H ₂ O + 360 psi O ₂ at 100°C	10 80	1848 1680	Surface blister. Transgranular cracks beneath blister. Pits probably related to rolling direction of rod. One pit measured 6 mils depth.
C3-25	347 SS 19 mil sheet	100 ¹ (250)	Cl ₂ -H ₂ O + 360 psi O ₂ at 100°C	10	1008	Pit-depth 6-7 mils. Almost complete deterioration by undercutting in some areas. Transgranular cracking in one area. "Crack," near edge, 7 mils depth.
C4-34	347 SS 19 mil sheet	250	NiCl ₂ + 450 psi O ₂ at 250°C	13	2664	Enlarged pits--4 mils max. depth. Transgranular cracking, max. depth 13 mils.
C4-42	347 SS 19 mil sheet	250	Cl ₂ -H ₂ O + 450 psi O ₂ at 250°C	9	2664	Enlarged pit, 8 mils depth. Intergranular crack connect- ed with pit. Intergranular attack exhibited in pit.
C8-42	347 SS ² stress specimen	100 ¹ (250)	Cl ₂ -H ₂ O + 290 psi O ₂ at 100°C	7 ≪ 200	1680 1008	Transgranular cracking. Surface pit--7 mils depth.
C8-53	347 SS ² base plate	100 ¹ (250)	Cl ₂ -H ₂ O + .008 M CuSO ₄ + 290 psi O ₂ at 100°C	7 90	1680 2136	Pit in end probably related to rolling direction--90 mils depth, general attack. Pit in base metal-heat affected zone, 60 mils depth, general attack. Crack in weld-60 mils depth.
I1-70	347 SS 19 mil sheet	100 ¹ (250)	360 psi O ₂ at 100°C		2520	Control specimen. No appreciable attack.
I1-81	347 SS 19 mil sheet	250	450 psi O ₂ at 250°C		2520	Control specimen. Surface roughened. Pit, depth 1.5 mils.

Specimen	Material	Temp (°C)	Additions	ppm Cl or I	Time Hrs.	
Il-74	347 SS 19 mil sheet	100 ¹ (250)	I ₂ -H ₂ O + 450 psi O ₂ at 250°C	7	2520	Enlarged pits, max. depth 6 mils. Two transgranular cracks, 3.5 and 1 mil in depth.
Il-86	347 SS 19 mil sheet	250	I ₂ -H ₂ O + 450 psi O ₂ at 250°C	7	2520	Surface roughening. About 1 mil variation.
Il-78	347 SS 19 mil sheet	100 ¹ (250)	KI + 360 psi O ₂ at 100°C	7	2520	Pit near edge, 1.6 mils depth. Pit near punched hole, 6 mils depth.
Il-90	347 SS 19 mil sheet	250	KI + 450 psi O ₂ at 250°C	9	2520	Pit and crack system near sheared edge, 11 mils depth. Crack appears to be mostly intergranular.
T2	Ti 75A 10 mil sheet	250	Cl ₂ -H ₂ O + 450 psi O ₂ at 250°C	8	1008	No appreciable attack
				17	840	
				90	2688	
T10	Ti 75A 10 mil sheet	100 ¹ (250)	Cl ₂ -H ₂ O + 360 psi O ₂ at 100°C	5	1680	No appreciable attack
				80	2136	

- Initially heated 2 hrs. at 250°C.
- Three-point loaded, beam-type stress assembly, welded base plate. Maximum outer fiber stress of 60,000 psi.

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Fig. 1

T8826

Spec Cl-1. Type 347SS 19 Mil Sheet After Exposure To Vapor
Above Oxygenated 0.14 m UO_2SO_4 Containing 50 ppm Cl.

Fig. 2

T8827

4.1X

Region of Spec Cl-1 Through Which Sec. A Was Taken.

Fig. 3
Enlargement of Sec. A Region Shown in Fig. 3.

T8903

50X

Fig. 4
Section A of Spec Cl-1 Taken as Indicated in Fig. 1.

T8935

50X

Fig. 5 T8938 250X
Undercutting Exhibited in Sec. A of Spec. Cl-1, See Figs. 1 to 4.

Fig. 6 T8934 250X
Enlarged Crack in Sec. B of Spec. Cl-1, See Fig. 1.

Fig. 7 T8933 250X
Edge of Crack Found in Sec. B of Spec. C1-1, See Fig. 1.

Fig. 8

T8944

150X

Sec. C. of Spec C1-1, See Fig. 1.

Fig. 9

T8946

750X

Transgranular Cracking Found in Sec. C of Spec. C1-1, See Fig. 1.

Fig. 10
Spec. C3-S2. Type 347SS Wafer After Exposure To Vapor
Above Oxygenated 0.14 m UO_2SO_4 Containing 80 ppm Cl.

T8828

4.1X

Fig. 11
Surface of Spec. C3-S2 Showing Blister And Pits.

T8906

10X

Fig. 12

T8907
Pits Found in Wafer Spec. C3-S2.

50X

Fig. 13

T8997
Section Taken Through Pit in Wafer Spec. C3-S2. Sec. A, See Fig. 10.

250X

Fig. 14 T8908
Blister Found On Surface of Wafer Spec. C3-S2.

50X

Fig. 15 T8992 150X
Section Through Blister in Wafer Spec. C3-S2. Sec. B, See Fig. 10.

Fig. 16 T8993 250X
Section Through Blister in Wafer Spec. C3-S2. Part of Region
Shown in Fig. 15.

Fig. 17 T8994 750X
Section Through Blister in Wafer Spec. C3-S2. Part of Region
Shown in Fig. 16. Note Transgranular Cracking.

Fig. 18 T8829 3.5X
Spec. C3-25. Type 347SS 19 mil Sheet After Exposure to Vapor
Above Oxygenated 0.14 m UO_2SO_4 Containing 10 ppm Cl.

Fig. 19 T8915 10X
Pit in Spec. C3-25, Section A-1 Taken Through This Region.

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Fig. 20

T8916

50X

Pit in Spec. C3-25, See Fig. 19.

Fig. 21

T9103

150X

Section A-1 Through Pit Shown in Figs. 19 and 20.

Fig. 22

T9104
Surface of Pit Shown in Figs. 19 to 21.

750X

Fig. 23

T8917
Region of Spec. C3-25 Through Which Section B Was Taken.

10X

Fig. 24 T8918 50X
Region of Undercutting Through Which Section B of Spec C3-25
Was Taken.

Fig. 25 T9107 75X
Undercutting Found in Section B of Spec. C3-25.

Fig. 26

T9108

250X

Same Region as Fig. 25 Showing Undercutting in Spec. C3-25.

Fig. 27

T8913

10X

Region of Spec. C3-25 Through Which Section C Was Taken, See Fig. 18.

Fig. 28 T8914 50X
Region of Spec. C3-25 Through Which Section C was Taken, Same
Region as Fig. 27.

Fig. 29 T9110 200X
Crack at Sheared Hole in Section C of Spec. C3-25.

Fig. 30

T9111
Section C of Spec. C3-25.

50X

Fig. 31

T9112
Section C of Spec. C3-25. Same Region as Fig. 30.

150X

Fig. 32 T9113 750X
Sec. C of Spec C3-25. Part of Same Region Shown in Fig. 31.

Fig. 33

T9114
Section C of Spec. C3-25.

50X

Fig. 34 T9115 250X

Transgranular Crack in Sec. C
of Spec. C3-25. Same Region
as Fig. 33.

Fig. 35 T9116 750X

Same Transgranular Crack As
That Shown in Fig. 34

Fig. 36

T8830
Other Side of Spec. C3-25, See Fig. 18.

Fig. 37

T8912
Region of Spec. C3-25 Through Which Section A-2 Was Taken, See
Fig. 36. Note Crack at Edge.

25X

Fig. 38

T9105
Section A-2 Through Crack Shown in Fig. 37. 150X

Fig. 39

T8832 3.5X
Spec. C4-34. Type 347SS 19 mil Sheet After Exposure to
Vapor Above Oxygenated 0.14 m UO_2SO_4 Containing 13 ppm Cl.

Fig. 40 T8920 50X
Region of Spec. C4-34 Through Which Section A Was Taken,
See Fig. 39.

Fig. 41 T9642 100X
Section A of Spec. C4-34, See Figs. 39 And 40.

Fig. 42

T9643

250X

Cracks in Sec. A of Spec. C4-34, See Figs. 39 to 41.

Fig. 43

T9644

750X

Enlarged View of Crack Shown in Fig. 42.

Fig. 44

T8831

Other Side of Spec. C4-34, See Fig. 39.

Fig. 45

T8921

10X

Region of Spec. C4-34 Through Which Section B Was Taken,
See Fig. 44.

Fig. 46

T9645
Pits in Section B of Spec. C4-34. See Figs. 44 and 45.

100X

Fig. 47

T8833
Spec. C4-42. Type 347SS 19 mil Sheet After Exposure to Vapor
Above Oxygenated 0.14 m UO_2SO_4 Containing 9 ppm Cl.

Fig. 48

T8922

10X

Surface of Spec. C4-42, See Fig. 47.

Fig. 49

T8923

25X

Pits in Spec. C4-42. Same Region as Fig. 48.

Fig. 50

Pits in Spec. C4-42. Same Region as Fig. 48.

T8924

50X

Fig. 51

Section Through One of the Pits of Spec. C4-42.

T9640

100X

Fig. 52 T9641 250X
Same Pit as Shown in Fig. 50. Note Intergranular Attack. Spec. C4-42.

Fig. 53 T8835
Spec. C8-42. Type 347SS Stress Specimen After Exposure to Vapor Above Oxygenated 0.14 m UO_2SO_4 Containing $\ll 200$ ppm Cl.

Fig. 54 T8927 10X
Region of Spec. C8-42 Through Which Section A Was Taken,
See Fig. 53.

Fig. 55 T8928 50X
Region of Spec. C8-42 Through Which Section A was Taken.
Same Region As Fig 54.

Fig. 56

T9768

100X

Crack And Pit in Section A of Spec. C8-42. See Figs. 53 to 55.

Fig. 57

T8836

Other Side of Spec. C8-42, See Fig. 53.

Fig. 58 T8925 10X
Region of Spec. C8-42 Through Which Section B Was Taken, See
Fig. 57.

Fig. 59 T8926 50X
Region of Spec. C8-42 Through Which Section B Was Taken. Same
Region As Fig. 58.

Fig. 60

T9769

100X

Section B of Spec. C8-42, See Figs. 57-59.

C8-53

Fig. 61

T8837

Spec. C8-53. Type 347 SS Stress Base Plate After Exposure To Vapor Over Oxygenated 0.14 m UO_2SO_4 Containing 90 ppm Cl.

Fig. 62 T8929 10X
Side of Stress-Base Plate Spec. C8-53 Containing Deep Pit
Through Which Section A Was Taken.

Fig. 63 T9766 50X
Deep Pit in Section A of Spec. C8-53, See Figs. 61 and 62.

Fig. 64 T8931 10X
Deep Pit in Region of Section B of Spec. C8-53, See Fig. 61.

Fig. 65 T9764 50X
Pit in Base Metal Adjacent to Weld in Section B of Spec. C8-53.
Pit is Same as That Shown in Fig. 64.

Fig. 66

T9765

50X

Crack in Weld of Section B in Spec. C8-53.

Fig. 67

T9767

250X

End of Crack Shown in Fig. 66.

Fig. 68

T9188

Control Spec. Il-70 Type 347SS 19 mil Sheet After Exposure to Vapor
Above Oxygenated 0.14 m UO_2SO_4 Free of Cl. 100°C.

Fig. 69

T9189

250X

Surface of Control Spec. Il-70, See Fig. 68.

Fig. 70 T9183
Control Spec. II-81 Type 347SS 19 mil Sheet After Exposure to
Vapor Above Oxygenated 0.14 m UO_2SO_4 . 250°C.

Fig. 71 T9185 250X
Roughened Surface of Control Spec. II-81, See Fig. 70.

Fig. 72

T9186

250X

Pit Found in Control Spec. 11-81, See Fig. 70.

A

B

1 INCH

Fig. 73

T9513

Spec. 11-74, Type 347SS 19 mil Sheet After Exposure to Vapor
Above Oxygenated 0.14 m UO_2SO_4 Containing 7 ppm $\text{I}(\text{I}_2\text{-H}_2\text{O})$. 100°C.

Fig. 74 T9515 25X
Region of Spec. Il-74 in Which Section A Was Taken, See Fig. 73.

Fig. 75 T9760 250X
Pit And Cracks Found in Section A of Spec. Il-74, See Figs. 73 and 74. Note Transgranular Nature of Cracks.

Fig. 76 T9514 25X
Region of Spec. 11-74 in Which Section B Was Taken, See Fig. 73.

Fig. 77 T9762 250X
Pit Found in Section B of Spec. 11-74, See Figs. 73 and 76.

Fig. 78 T9517 3.5X
Spec. Il-86, Type 347SS 19 mil Sheet, After Exposure to Vapor
Above Oxygenated 0.14 m UO_2SO_4 Containing 7 ppm $\text{I}(\text{I}_2\text{-H}_2\text{O})$. 250°C.

Fig. 79 T9518 25X
Region of Spec. Il-86 in Which Section A Was Taken, See Fig. 78.

Fig. 80

T9772

250X

Surface in Section B of Spec. 11-86, See Fig. 78.

Fig. 81

T9516

3.5X

Other Side of Spec. 11-86, See Fig. 78.

Fig. 82 T9521 25X
Region of Spec. 11-86 in Which Section B Was Taken, See Fig. 81.

Fig. 83 T9773 250X
Surface in Section B of Spec. 11-86, See Figs. 81 and 82.

Fig. 84

T9523

Spec. 11-78, Type 347SS 19 mil Sheet, After Exposure to Vapor
Above Oxygenated 14 m UO_2SO_4 Containing 7 ppm I(KI). 100°C.

Fig. 85

T9771

250X

Surface at Sheared Edge of Spec. 11-78. Location is at A
in Fig. 84.

Fig. 86 T9522
Other Side of Spec. Il-78, See Fig. 84.

Fig. 87 T9770 250X
Pit Near Punched Hole in Spec. Il-78. Location is at B in
Fig. 86.

Fig. 88 T9527
Spec. IL-90, Type 347SS 19 mil Sheet, After Exposure to Vapor
Above Oxygenated 0.14 in UO_2SO_4 Containing 9 ppm I(KI). 250°C.

Fig. 89 T9529
Pit at A in Fig. 88, Spec. IL-90.

50X

Fig. 90 T10189 250X
Pit And Crack System Found in Spec. T1-90 at A, See Figs. 88 and 89.

Fig. 91 T9309 4X
Spec. T-10, Titanium 75A 10 mil Sheet, After Exposure to Vapor
Above Oxygenated 0.14 m UO_2SO_4 Containing 90 ppm Cl ($\text{Cl}_2\text{-H}_2\text{O}$).

Fig. 92 T9311 250X
Section Taken Through Titanium Spec. T-10, See Fig. 92.

Fig. 93 T9312 750X
Section Taken Through Titanium Spec. T-10, See Fig. 92.

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