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Metallurgy Division

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AUTOCLAVE TESTS OF TUBALLOY SLUGS AND ALLOYS
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1. SUMMARY

1.1 Problem Under Investigation. In previous experiments it had been found that leaks in the coating of a tuballoy slug could be detected by autoclaving at steam pressure of 130 to 150 pounds per square inch gauge (180 to 186°C). If there was a leak in the coating which permitted steam to come in contact with the tuballoy, areas of local swelling, caused by corrosion products, developed.

The objects of the investigation were to obtain data which would lead to a better understanding of the corrosion mechanism which occurs in the steam autoclave. This called for a study of the corrosion behavior of tuballoy in steam and liquid water at the same experimental conditions.

1.2 Important Results.

(a) In liquid distilled water, heated with steam, 130 to 150 pounds per square inch gauge, bare tuballoy slugs approximately 1.4 inches in diameter corroded uniformly at an average linear rate of 139 mg/cm²/hr based on original area.

(b) Bare tuballoy slugs approximately 1.4 inches in diameter, tested in steam at 130 to 150 pounds per square inch corroded for the first 2 hours in essentially the same manner and at the same rate as in liquid water. Beyond this time deep areas of local penetration developed, and the measured average rate rose to as high as 1620 mg/cm²/hr. Cut ends of hot α -rolled slugs were attacked more rapidly than the cylindrical surface, and pitting followed the longitudinal axis of the slug.

(c) Pinholed, aluminum jacketed slugs (2S and A13 alloy) corroded in steam at 130 to 150 pounds per square inch with the formation of TuH₂ causing local swelling, or pimpling, of the jacket of the slug and finally cracked it open. Five slugs were tested, all cracked open. In one, when proper precautions were taken to prevent oxidation of the corrosion product, chemical analysis showed that it contained sufficient hydrogen for 89% of the material to be TuH₂.

(d) Several "as cast" tuballoy alloys were tested in liquid distilled water under steam pressure of 130 to 150 pounds per square inch. The columbium and epsilonized silicon alloys were most resistant to corrosion.

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2. INTRODUCTION

At one time, tuballoy slugs, enclosed in a tightly fitting unbonded aluminum jacket, were proposed as the main component of a water-cooled operating unit. Among the tests devised to detect leaks in faulty jackets was exposure to steam in an autoclave at about 140 pounds per square inch gauge.

Some slugs failed in the steam autoclave in 2 or 3 hours; some failed after days. Those that failed, corroded with the formation of products of considerably lower density than that of tuballoy, resulting in swelling of the jacket and eventually rupture. A better understanding of the mechanism of this corrosion was the prime objective of this investigation.

Tuballoy alloys were also tested to provide data on their possible use as substitutes for pure tuballoy metal in an operating unit.

3. EXPERIMENTAL WORK

3.1 Materials Tested and Equipment Used. In order to provide reference materials for the study of tuballoy corrosion, a bar of metal was obtained and was referred to as "standard" tuballoy. This metal was hot rolled at 600 to 650°C from a billet cast in a graphite mold and designated as Site B heat C255. The rolled bar was cut into two equal lengths, one of which was set aside for the corrosion work. The chemical analysis of the material, which proved to be of uniform chemical content (3 separate samples analyzed), is expressed below in parts per million:

Ag	< 5	Co	< 20	Na	< 5
Al	100	Cr	20	Ni	< 20
B	0.5	Cu	10	Pb	5
Be	< 0.2	Fe	200	Sb	< 5
Bi	1	K	< 20	Si	50
C	623	Li	< 2	Sn	< 5
Ca	< 5	Mg	< 5	Ti	< 100
Cd	< 0.2	Mn	< 5	Zn	< 50
		Mo	< 100		

Plates 4461-4466 and 4478-4482

After the bar had been centerless ground to 1.375 inches in diameter it was cut into full circular sections of varying length for use in tests.

Most of the data reported below were obtained from tests on metal other than standard tuballoy. All of the samples used were from the same general source, however, and were the same diameter as standard metal. These other samples were hot-rolled from Site B castings C245 and C250.

The specimens were regularly prepared for exposure by machining the flat ends on a surface grinder and the cylindrical surface on a lathe. The surfaces were then polished on No. 1 grit emery paper. The specimens were dipped in 25 weight percent nitric acid at room temperature for 5 minutes, rinsed in distilled water, then in clean acetone, dried, weighed, and put on test.

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After the corrosion test, each sample was removed and washed under a faucet, using a stiff brush to remove any loose corrosion product. It was then dipped in 25 weight percent nitric acid for 5 minutes at room temperature, washed successively in tap water, distilled water, then acetone dipped, and dried before weighing.

The two types of jackets on tested slugs were:

(1) An aluminum can, which was fitted tightly to the slug by drawing through a die that reduced the can wall thickness, with a brazed or welded end cap closure.

(2) A die-cast aluminum alloy A-13, "bonded" to the slug (No. 252 and No. 283) by a pre-dip in the molten alloy. (1)

The autoclave used for the experiments at 130 to 150 psi was 14 inches in diameter and approximately the same depth and was provided with a steam trap. A false bottom kept the samples above the condensed water level at all times.

3.2' Corrosion of Bare Tuballoy in Water. Samples were placed in beakers containing sufficient boiled distilled water to completely immerse the tuballoy. The beakers were covered by watch glasses and placed in the autoclave. After the autoclave was closed, the chamber was flushed with steam until all the air was removed. The autoclave was then brought to the desired pressure where it was maintained for the time interval desired. The steam was tapped from the building lines at a pressure which varied between 130 and 150 psi.

One run was made on standard tuballoy in liquid distilled water under steam obtained from a Besler boiler. The steam pressure averaged about 420 psi gauge and the corresponding saturated steam temperature was 233°C. A metal jacketed thermometer mounted in the head of the autoclave read 212°C, equivalent to a saturated steam pressure of 310 psi gauge. The maximum partial pressure of hydrogen which could have accumulated in the autoclave at the end of the run was 15 psi.

Because of the presence of dichromate in the coolant water, it was necessary to observe its corrosive effect on tuballoy. Bare tuballoy was placed in a beaker containing 1 weight percent sodium dichromate dihydrate solution with a watch glass cover. The beaker was then placed in the autoclave and run at 130 to 150 psi. The corrosion rates of the three samples in dichromate were not reproducible. The sample tested for 25 hours corroded with uniformly wide pit-like holes. Analysis of the corrosion product indicated it to be 90% UO_2 and the remainder U_3O_8 . (2)

Table I is a tabulation of water corrosion data including dichromate corrosion. A constant corrosion rate of bare metal is indicated, calculated values varying about an average of 139 $\text{mg}/\text{cm}^2/\text{hr}$, with an average deviation of 17 $\text{mg}/\text{cm}^2/\text{hr}$ (see Figure I). Figure II shows the longitudinal uniform grooving obtained in water corrosion of a bare slug. Judging by appearance, the

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Figure 1

Corrosion of Bare Tuballoy in Liquid Distilled Water Under Steam Pressure
At 130-150 pounds sq. inch (183-186°C)

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cylindrical surfaces were attacked less than the circular end sections transverse to the rolling direction. This is believed to account for the higher average rate of weight loss obtained for the 1.00 cm long than for the 5.01 and 6.90 cm long pieces exposed in water heated by 420 psi steam.

TABLE I

CORROSION OF BARE TUBALLOY IN LIQUID DISTILLED WATER IN STEAM AUTOCLAVE
Steam Pressure 130 to 150 psi gauge
(183-186°C)

Time in Test (hrs)	Length (cm)	Diameter (cm)	Area (cm ²)	Type of Tuballoy	Original Weight (grams)	Weight Loss (gram)	Average Loss (mg/cm ² /hr) (1)
2	9.42	3.45	119	Stock	1691	28	117
5	4.1	3.45	62	Stock	751	38	122
5	9.5	3.45	120	Stock	1665.5	63.5	106
9	1.5	3.45	35	Stock	280.5	49.7	158
9	9.7	3.40	122	Stock	1703	150.5	137
10	9.95	3.35	122.5	Standard	1479	196	160
10	6.68	3.42	90	Stock	1164	141	157
10	7.04	3.42	90	Stock	1224	136	145
16	9.55	3.42	121	Stock	1663.5	313.5	162
23	10.15	3.45	127.5	Stock	1776	335	114
32	10.0	3.35	123	Stock	1765.5	535.5	136
37½	4.1	3.45	62	Stock	451	366	157
1½	5.01	3.47	75.4	Standard	897	76	690(2)
1½	6.90	3.40	91.7	Stock	1195	101	690(2)
1½	1.00	3.42	29.0	Standard	175	34	785(2)
CORROSION IN 1% DICHRONATE SOLUTION							
5	4.4	3.45	65.5	Stock	739	44	134
25	4.4	3.45	65.5	Stock	739	789	3100
37½	2.2	3.45	42	Stock	390	280	267

(1) Based on original areas.

(2) Steam pressure approximately 420 psi gauge (233°C). A metal jacketed thermometer in autoclave cover indicated 218°C (corrected).

3.3 Corrosion of Bare Tuballoy in Steam. The samples to undergo steam corrosion were placed on a watch glass, resting on the plate above the condensed water level of the autoclave. The cover of the autoclave was then closed, the air flushed out, and the autoclave brought to the desired pressure.

Table II lists the data obtained in the corrosion of tuballoy under steam pressure at 130 to 150 psi. The increase in rate for tests beyond 2 hours is clearly shown.

Figures 3 through 11 picture the drastic corrosion that takes place when tuballoy is under steam pressure for more than 2 hours.

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TABLE II

CORROSION OF BARE TUBALLOY IN STEAM

Pressure 130-150 psi gauge (183-186°C)

Time in test (hours)	Length (cm.)	Diameter (cm.)	Area (cm ²)	Type of Sample	Original wt. (grams)	Wt. loss (grams)	Average loss (mg/cm ² /hr)	(1) Remarks based on observations when lid of autoclave was removed
1	10.2	3.45	128	Bare Tu	1778	13.5	106	
2	10.3	3.45	129	" "	1811	27	105(2)	
2	9.75	3.45	122	" "	1734	50	205(3)	
2	9.42	3.45	119	" "	1691	26	109(4)	
3	9.3	3.45	118	" "	1635	577	1630	
3	9.8	3.45	124	" "	1721	53	143	
4	10.2	3.45	128	" "	1788	778	1520	
5	4.25	3.45	64	" "				
5	9.3	3.45	118	" "	1642	232.5	394	Erupted, flamed in air. Top hollowed out.
5	10.1	3.45	127	" "	1762	895	1410	
5	10.5	3.45	131	" "	1841	738	1127	Flamed - abnormal ridge on top.
5	9.5	3.45	120	" "	1661	705.5	1175	Flamed specimen looked normal.
5	9.5	3.45	120	" "	1662	879	1465	Sample glowed.
5				Die cast Slug	3621	453		
5				Die cast Slug	3621	68		
25	3.6	2.65	41	Bare Tu	385	227	219	Top exploded. Sample in wet oxide.
37½	4.25	3.45	64	" "	763.5	453.5	189	

- 1) Based on original area.
- 2) Sample polished only - no acid dip.
- 3) Sample run "as received".
- 4) Sample polished and acid dipped.

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One experiment was carried out to check the rates of corrosion in steam of samples which had been given different surface preparations. One sample was polished on #1 emery paper and nitric acid dipped, another was polished and not acid dipped; the third was run as received with an oxide layer on it.

TABLE III
SAMPLES TESTED WITH DIFFERENT SURFACE PREPARATION

Sample	Surface Prep.	Medium	Time	Loss in mg/cm ² /hr	Remarks
1.	Polished, Acid Dipped	Steam	2 hrs.	109	Corrosion Uniform
2.	Polished, not Acid Dipped	Steam	2 hrs.	105	Corrosion Uniform
3.	Oxide Layer as received	Steam	2 hrs.	205	Corrosion Uniform

The results show a marked increase in the corrosion rate of the sample which was not cleaned, and no great difference between the samples cleaned by an acid dip and polishing, or by polishing alone.

3.4 Jacketed Slug Tests. Two aluminum bonded die-cast 8 inch slugs were pinholed one and two inches from the end in each case, using a #60 drill. Drilling was stopped when contact was made with the tuballoy. After five hours of steam corrosion in the autoclave at 130-150 psi, the slugs were inspected. The end of the aluminum jacket was torn off one slug, but the coating of oxide was not deep. The rest of the die-cast jacket was in good condition. The jacket on the second slug was cracked open with underlying black oxide quite apparent (Figure 12). Swelling started about half-way between one end of the slug and the nearest pinhole, and 1/2 inch beyond the second pinhole (Figure 13). When the jacket was stripped off, oxide covered the entire slug. When the slug was cleaned, pitting was noted from 1/2 to 3/4 inch around the pinholes, but not at the pinholes (Figure 14). The corrosion product analyzed greater than 90% TuO_2 with the remainder Tu_3O_8 , probably the product of the air oxidation of the hot UO_2 when the slug was removed from the autoclave. (2)

To confirm the suspected presence of hydride in the corrosion product of an unbonded slug, before air oxidation occurred, the experiment described above was repeated, using an unbonded, aluminum jacketed slug. After 2 hours no visible swelling had occurred. After 4 hours and 10 minutes, a swelling 4 mm. high was observed and the slug transferred to a helium filled dry box. Several pimples were observed about 1 1/2 inches from the pinhole. Half of the jacket was removed with a hammer and chisel (Figure 15) and samples of corrosion product were taken from the areas of greatest pimpling. Analysis showed a 1.1% H_2 content or 89% TuH_3 .* Success

* Analytical Report #10366 Section C-IV

in this instance was due, no doubt, to the care taken in not exposing the inner corrosion product to an active atmosphere. This residue did not evolve gas in room temperature H₂O, but a series of experiments has proven that tuballoy hydride will not evolve gas from aerated or oxygenated distilled water at room temperature. (1)

Other experiments were undertaken in order to confirm the presence of hydride as a corrosion product. A slug was encased in a rubber jacket with the ends closed but with one hole for steam entrance. During the course of the run, the center of the rubber tube burst open, either due to corrosion product pressure or the decomposition of the rubber. The corrosion product showed no indication of hydride. Similarly a 4 inch length of a slug, with a tightly fitting rubber tube to simulate jacketing was corroded in liquid distilled water for 5.1 hours at 130-150 psi. It lost 129 mg/cm²/hr which is within the average for bare tuballoy in water, and also it showed no hydride present in the corrosion product. It is more probable that the rubber fitted loose enough to permit water to reach the slug than that there is a significant difference due to a rubber instead of an aluminum jacket.

3.5 Water Corrosion of Tuballoy Alloys. Sample preparation procedure were identical with tuballoy, including the 25% HNO₃ dip before and after the test. The data are presented in Table IV. Two alloys nominally containing 3.8 wt. % Si and above 8.9% Cb showed considerable corrosion resistance. Since the 5 minute dip in 25% HNO₃ at room temperature was found not to affect adequate removal of corrosion product from these alloys, the reported rates of attack are lower than actual.

4. DISCUSSION OF EXPERIMENTAL WORK

4.1 Water Corrosion. The cylindrical surfaces of a water corroded slug were marked with grooves which had a definite tendency to follow the longitudinal axis of the slug (Figure 2). The flat surfaces of the slug are not shown, but here the grooves also extend parallel to the axis of the slug and had no tendency to cut across. The residue obtained from liquid water corrosion of tuballoy analyzed 90% TuO₂ and the remainder Tu₃O₈⁽²⁾. The Tu₃O₈ probably came from the air oxidation of hot TuO₂ which was exposed when the lid of the autoclave was opened and the hot water poured off the sample.

The main mechanism by which tuballoy corrodes in liquid water can be represented by the displacement reaction

with the hydrogen bubbling out through the water. Corrosion product does not protect the reacting surface from the water well enough to allow the appearance of tuballoy hydride.

4.2 Steam Corrosion. At first steam corrosion testing in the autoclave seemed to be the answer to the call for a quick, simple method of testing the corrosion rate of tuballoy slugs in aqueous medium at high

TABLE IV

SUMMARY OF DATA ON "AS CAST" TU-ALLOYS

Corroded in Liquid Water under 130-150 psi gauge
Steam Pressure (183-186°C)

Wt.% of Alloying Element	Alloy No.	Area cm ²	Time of test, hours	Average Rate mg/cm ² /hr ⁽³⁾	Remarks
3.77% Si(1)	952C	7.44	10	0.01	When cleaned after test sample still light blue black.
3.77% Si(1)	952C	7.44	2	0.03	
6.31% Mo	576	9.40	2	15.	Sample pitted badly and started to crack.
7.79% Ta	X129A	18.11	2	76.	Cleaned up completely after test.
2.06% Zr	X137	7.32	2	37.	Sample still black after cleaning after test.
2.01% Cb	971-3	7.64	10	42.	No comment.
4.16% Cb	965-3	5.36	10		Sample completely disintegrated.
14.33% Cb	958-3	5.12	2	0.00	Sharp edge not dulled by attack.
14.33% Cb	958-3	5.12	16	0.00	After cleaning-after test-blue black film still present.
100% Cb(2) Sheet	953-b	5.40	16	0.00	No color change during test. No apparent attack.
8.89% Cb	957-3A	5.16	2	0.03 ⁽⁴⁾	Retained a light blue film after test and 5 min. cleaning in 25% HNO ₃ at room temperature.
14.33% Cb	958-3	5.12	2	0.07 ⁽⁴⁾	
3.77% Si	952-C	7.43	2	0.18 ⁽⁴⁾	

- 1) Heat treated to make Epsilon
2) Sheet approximately 1/32 inch thick
3) Based on original areas

- 4) Heated by steam at approximately 420 psi gauge(233°C). Metal jacketed thermometer read 218°C.

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temperatures. However, before long it was realized that regardless of careful standardization of the testing procedure, a definite and reproducible rate of steam corrosion over a long period of time could not be established. The cut ends of tuballoy specimens were found to be attacked more vigorously and the rate of corrosion was always higher for pieces where the area of the ends was a large percentage of the total area. After this was noted, lengths of 2 inches or more were always selected, still without reproducible corrosion rates being obtained. The physical corrosion characteristics of tuballoy in steam were similar to water corrosion after a two hour run, but after that the rate accelerated and soon the metal disintegrated rapidly. Sometimes when the autoclave was opened, the corrosion product glowed and burned and on occasion sharp explosions were seen.

The mechanism by which steam corrosion takes place is believed to follow this course:

One can assume that for the first two hours of steam corrosion of tuballoy the first is progressing. After this however, parts of the reacting surface are protected from the water well enough so that the second reaction becomes important. As the water penetrates the outer corrosion product it comes in contact with TuH_3 and the third reaction proceeds. The rate of the second reaction has elsewhere been shown to be 4.0 gm/cm²/hr at 180°C bath temperature⁽⁵⁾. In Figure 10 a photomicrograph showing a corroded area and unreacted metal, fingers of penetration into otherwise sound metal can be seen. A view at right angles to Figure 10 is shown in Figure 11. This may be a characteristic phenomenon in steam corrosion, involving hydride formation at reactive areas, perhaps at grain boundaries. Figure 9 shows the porosity in metal produced by penetration and incomplete reaction, possibly by the action of hydrogen on the metal.

In Figure 5 is shown the type of laminar attack which was typical of several slugs corroded in steam. Places of deepest penetration are shown by dark areas in Figure 6. On slug ends, corrosion clearly proceeded most rapidly along certain lines, forming concentric circles. This is believed due to the type of working the metal received during the rolling process (and points out extra danger of attack on slugs prepared in this manner). In one slug, for example, a button of metal was undermined by corrosion and came completely loose from the bulk of the metal at one end.

In pinholed jacketed slugs the reaction follows essentially the same course as for bare slugs. Steam at 130-150 psi penetrates through the pinholes to react with the metal to liberate hydrogen. The hydrogen diffuses between the slug and jacket ahead of the water vapor of heavier molecular weight to form hydride. The powdery hydride expanded several fold under the jacket to form pimples. If the corrosion test is allowed to continue, the pimples rupture the jacket and the hydride is converted to oxide by reaction with steam.

4.3 Corrosion of Tuballoy Alloys in Liquid Water. Only very slight corrosion of "good" alloys was noticed. The relative rapid disintegration of the molybdenum alloy is indicated to be due to intergranular attack, as shown by Figure 16. Other alloys which corrode rapidly without severe disintegration may not be as susceptible to this kind of attack as those of molybdenum.

5. CONCLUSIONS

1/ Bare ^{Tu} ~~tuballoy~~ slugs 1.4 inches in diameter corrode in liquid distilled water in a steam autoclave at 130^t150 psi at an average constant rate of 139 mg/cm²/hr based on original area.

Steam corrosion of bare ^{Tu} ~~tuballoy~~ slugs, at 130^t150 psi is roughly the same as in ~~distilled~~ water for about 2 hours, but is much higher and erratic after that time, measured average values being as high as 1630 mg/cm²/hr.

Pinholed, ^{AL} ~~aluminum~~ jacketed slugs corroded in steam at 130^t150 psi, due to the formation of TuH3 which expanded to pimple the jacket of the slug in every case tested.

^{Tu} ~~Tuballoy~~ alloys, ^(including Si, Mo, Ta, Zr, ~~and Nb~~) were tested in liquid distilled water under steam pressure of 130^t150 psi with certain ~~columbium~~ and epsilonized ~~silicon~~ alloys proving resistant to corrosion. *Lead*

Figure 2

Liquid Water Corrosion of Bare
Alloy No. 576 under 130-150 psi.
Steam Pressure for 2 Hours.
Rate-138 mg/cm²/hr.

Figure 3

Steam Corrosion of Bare alloy-
No. 576 at 130-150 psi for 3
hours. Rate 1630 mg/cm²/hr

Figure 4 - Topview of Figure 3

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Figure 5
Steam corrosion of bare alloy #576
at 130-150 psi for 4 hours; showing
lamination - rate $1520 \text{ mg/cm}^2/\text{hr}$.
Section of laminated metal is shown
in Figure 6.

Figure 6
Photomicrograph of laminated areas
showing three laminations of inter-
nally corroded metal and two lamina-
tion boundaries.

Figure 7
Topview of Fig. 5. Loose button of laminated metal
fitted surface indicated by arrows.

Figure 8. Steam corrosion of Alloy #576, 130-150 psi for 5.1 hrs. Rate - 1105 mg/cm²/hr.

Figure 10. Photomicrograph of Alloy #576 Corroded in steam for 5.5 hrs, rate 1068 mg/cm²/hr. Note fingering from corroded area into metal.

Figure 9. Section of slug pictured in Figure 8 showing sound metal at left and corroded metal of ridge at right.

Figure 11. Photomicrograph of same corroded sample as Figure 10 taken at right angles to Figure 10 looking in direction of penetration.

Figure 12
Pinholed. aluminum bonded die cast
slug after 5 hours in steam auto-
clave at 130-150 psi. Jacket
cracked revealing black oxide.

Figure 13
Slug pictured Figure 12 turned 140°,
showing 2 pinholes near top of slug
and swelling above and below each
pinhole.

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Figure 14

Stripped slug showing areas of attack about 1/2" to 3/4" away from pinhole. Steam corroded for 5 hours at 130-150 psi.

Figure 15

Steam corrosion at 130-150 psi for 4 hours of a pinholed aluminum jacketed unbonded slug. Hydride was recovered from section cut away with hammer and chisel.

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N. _____

500X

Figure 16

Photomicrograph of Water Corroded Alloy Number 576 (6.31%)
Intergranular corrosion is plainly evident.

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207. BIBLIOGRAPHY

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