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DATE: August 15, 1956

COPY NO. 9

SUBJECT: Failures of Titanium Alloy
Trim in HRP Dump Valve Loop

TO: Distribution

FROM: Joseph P. Hammond, Th. M. Kegley, Jr., and G. M. Adamson

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SUMMARY

Incident to the HRP valve trim testing program, failures in titanium alloys have occurred on two separate occasions. Both failures resulted largely from a rapid burning of the trim material, which occurred, probably on opening the valve, during a single cycle of operation.

The first failure involved a 6 Al-4 V alloy and occurred after 75 cycles in the uranyl sulphate solution. The second failure involved a 4 Al-4 Mn alloy and occurred after 112 cycles in the uranyl sulphate solution followed by 292 cycles with oxygenated wash water.

Both failures resulted in the consumption of about 4 grams of metal, which came largely from the poppets. Characteristic craters were found in the poppets in addition to a more widespread cavitation which was similar in appearance to cavitation corrosion.

Evidence of an explosive reaction was found on the valve bodies in the case of each failure. An almost negligible heat-affected zone in the poppet microstructures, was indicative that the failures had occurred in a short span of time.

The evidence suggested that the craters may have resulted from initial explosions involving pyrophoric material produced under the conditions of testing, and that the more prevalent erosive-type cavitation resulted from a subsequent steady-state burning made possible by the dynamic flow of a vapor-liquid medium past the trim parts.

The scales removed from the cavities on the valve trim parts were largely $TiO_{1.25}$, whereas spatter that had been transferred from the poppet to the seat was TiO_2 and other oxygen-rich oxides. Ingredients of the stainless steel valve body were found in the residues removed for the valve trim in the case of one of the failures.

INTRODUCTION

Since November of 1955 a program has been underway to evaluate various alloys for the trim of the HRP dump valve. Recently there have been two failures of valve trim; first, a MST-6Al-4V (6% Al-4% V) alloy and then a C-130 AM alloy, following just five weeks apart. Trim of two titanium alloys, the C-130 AM and an A-110 AT, had been tested earlier without incident and with very good results when compared to the other alloys tested. While zirconium alloys had been scheduled for test, no trim in this material has been run to date.

As will be shown later, these failures, if not catastrophic in nature, resulted from rapid burning and, with the failure of the titanium impeller hub of last year¹, presents a potentially serious aspect in

1. J. C. Griess, et al, "Report on Corrosion of Titanium Impeller No. One," ORNL-CF 55-9-65, Sept. 15, 1955.

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the application of titanium alloys in HRP.

The dump valve test loop is designed to discharge twenty gallons of oxygenated uranyl sulphate solutions at 2000 psi and 300°C through a 1/2-in. diameter shut-off type test valve to a low pressure system and automatically repeat this cycle. The discharge into the dump tank requires approximately one minute, while fourteen minutes are required to pump the solution to the core tank, preliminary to the next cycle. The pumping is continuous and in a closed system.

Oxygen is introduced into the line upstream of the core tank under pressure controlled to give a solution assay of 500 ppm.

The design of the trim seat and poppet used at the time of the failures is shown in Fig. 1. It will be noted that the mating is essentially by line contact, since the included angle between the slight chamber on the seat and the diagonal on the poppet is one degree. Seating is effected by a downward motion of the poppet onto the valve seat. No twisting of the poppet accompanies the mating. The valve housing in which the trim is mounted (shown later, Figs. 1 and 2) is of 347SS.

The first valve trim failure, the 6% Al-4% V alloy, occurred on June 5 after 75 cycles with the uranyl sulphate solution. The temperature of the solution (measured as introduced into the core tank) was 220°C, while the discharge pressure (measured upstream of valve) was 1,150 psi. Examination of the pressure chart upstream of the valve made it clearly evident that the failure had occurred completely within the final cycle of testing. However, it was not discernible from the upstream, downstream (with respect to valve), and dump tank pressure charts together whether the failure initiated with opening or closure of the valve.

The second failure, the 4 Al-4 Mn alloy, occurred on July 13 after 112 cycles with the uranyl sulphate solution followed by 292 cycles with water charged with 6 lbs. of trisodium phosphate. The trisodium phosphate had been introduced to facilitate cleansing as the purpose of the final operation had been strictly to clean the system. The mechanism for introducing oxygen into the system had been running during the cleansing operation.

The discharge pressure upstream of the valve was 1300 psi and the temperature as measured upstream of the core tank was 200°C. Again the failure ostensibly occurred completely within the final cycle, and it could not be determined from pressure charts whether it was initiated with opening or closing of the valve.

The valve trim and body parts in the case of each failure were submitted to macroscopic examination, while the titanium trim parts were subjected to careful metallographic and X-ray diffraction examination as well. In addition, scrapings were taken from the spatter and/or scale on the valve trim and body parts for analysis by wet chemical, spectrographic, and X-ray

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diffraction means. Chemical analyses were made of the system solutions after each failure. The results of these examinations are reported in the following sections.

MACROSCOPIC EXAMINATION

The dump valve body and flange with the titanium alloy trim in place are shown respectively in Figs. 2 and 3. With discharge, the solution enters from a line beneath the valve body in Fig. 2 and leaves through the line to the right.

Valve trim parts at higher magnification are shown in Fig. 4. Figure 4a illustrates how the poppet mated with the seat at time of failure. The extremities of the "burnout" on the poppet, "A" and "B", coincide with flash markings "A" and "B" on the seat. The crater "C" on the poppet is the focal point for the burned area "D" on the seat. The "burn out" area of the poppet was just adjacent to the spatter shown on the valve body in Fig. 2.

The valve flange directly above the "burnout" of the poppet (see position "A" in Fig. 3) was eroded or burned to a depth easily discernible upon raking a finger nail within the area.

Views of this portion of the flange at higher magnification are shown in Fig. 5. The recession illustrated at high magnification in Fig. 5b corresponds to position "A" in Fig. 5a; however, Fig. 5b requires a clockwise rotation of 90° to be of like orientation. The various scratches and markings in these particular illustrations resulted when scrapings were being taken for chemical and X-ray diffraction examinations.

The poppet "burnout", shown in Fig. 4b at higher magnification, is similar in appearance to cavitation corrosion, except for the crater in the center. Note that the "burnout" is to a large depth (about 1/4 in.) and encompasses areas both above and below the ring of contact. It may be noted that the surface of the seat along the ring of contact, Fig. 4a, is not burned completely from flash marking "A" to marking "B", but rather there are areas directly beneath the poppet cavity that were unaffected.

In the rush to prepare metallographic specimens, the valve trim parts were sectioned before after-test weighings could be made. However, it is estimated that the weight loss to the poppet alone was about four grams in the case of each failure.

With minor exceptions, the manifestations resulting from the second valve failure were quite similar to those of the first. A bottom view of the poppet is shown in Fig. 6, whereas the valve seat is shown in Fig. 7a.

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In addition to the large "burnout" on the poppet, extending from the one o'clock position to the five o'clock position in Fig. 6, a second very small crater is evidenced at point "A". Figure 7a, position "A", scarcely shows the corresponding point for this crater along the contact ring of the seat. Figure 7b, as viewed normal to the shoulder of the seat, reveals this position at higher magnification as a small crater.

Views of this crater on the poppet at higher magnifications are shown in Figs. 8a and 8b. It will be noted that the crater appears to emanate from the upper edge of the ring contact. The upper side of Fig. 8a represents the larger-diameter portion of the poppet, which is downstream from the ring of contact.

The crater and part of the streamer shown in Fig. 8b are in recession, while the remainder of the streamer shown in Fig. 8a is in relief. The "tear drops" shown at "A" and "B" in Fig. 8a reveal that surface tension was at work and that early stages of the "burnout" involved melting of the titanium trim.

While the point may not be pertinent, the poppet and the valve seat revealed a very striking iridescent straw to brown color on the surfaces upstream of the ring of contact. This discoloration was not particularly noticeable in the case of the first failure. It has been suggested that the caustic condition resulting from the addition of the trisodium phosphate to the wash water may have caused the discoloration.

Close examination of the ring of contact on a number of poppets and valve seats under the stereoscopic microscope revealed a highly worked condition with decided evidence of galling (see Figs. 8a and 8b for this condition in case of second failure). It is understood that during the first several cycles of the testing operation these markings become about as prominent as they are after large numbers of cycles. While work hardening may approach saturation during the early stages of testing, galling no doubt proceeds throughout.

Metallographic Examination

A photomicrograph of an etched cross-section through the poppet for the first failure is shown in Fig. 9a, while Fig. 9b shows the corresponding structure on the second failure. Figure 9a shows a shallow white-appearing band along the contour of the failure cavity which is the heat-affected zone of the microstructure. Actually, these particular macrographs accentuate this effect, and, as will be seen in Figs. 10 and 11, the internal microstructure was only slightly affected by the heat.

For the first failure, the heat-affected zone (the white-appearing rim in Fig. 9a) in the area of the cavity was only four mils in depth and ranged up to about 13 mils in the area above the cavity, position "B" of Fig. 9a. The latter area was affected to a greater depth as the reaction products were swept in this direction.

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The scale, which proved by X-ray diffraction to be chiefly $TiO_{1.25}$, was only about 1 mil thick in the area of the cavity (note the scale at the upper right, Fig. 10a) and about 3 to 5 mils thick in the area immediately above the cavity (Fig. 10b).

Except for an absence of discernible patches of alpha phase, the basket-weave, alpha-beta structure shown in Fig. 10a is that of an annealed structure. A slightly blurred structure is noted just adjacent to the scale, which corresponds to the heat-affected zone.

The microstructure in the case of the second failure was affected even less by the heat of reaction. The microstructure in the vicinity of the cavity, Fig. 11a, is not greatly unlike that far removed from the cavity, Fig. 11b. The microstructure here shows alpha plus transformed beta (basket weave), which corresponds to the annealed state for this alloy.

The fact that the heat-affected zone was very shallow in the case of these failures indicates that the periods of reaction were very short and that the heat was rapidly removed from the burning surfaces, away from the metal, as it was evolved.

Chemical, Spectrographic, and X-Ray Diffraction Analyses

Chemical analyses of the respective alloys revealed their identification to be correct:

Alloy	Weight %				
	Al	V	Mn	C	N
MST-6Al-4V (1st Failure)	6.05	3.7			
	6.10				
C-130AM (2nd Failure)	4.2		4.1	0.10	0.04

The results of the X-ray diffraction analyses on the residues removed from the valve trim parts are presented in Table I. It will be noted that the reaction products found on the titanium trim parts are essentially oxides of titanium, except for the analyses made on the valve seat in the case of the second failure. Here the austenitic stainless and alpha iron structures are evidenced as well as the titanium oxides. The fact that the stainless material from the valve body very apparently was washed on to the trim seat would indicate that at one stage the reaction was quite violent, particularly if it may be assumed that the system was in process of dumping at the time of failure.* In addition to the stainless

*Arguments in support of the failures occurring with opening of the valves are offered later.

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material appearing as separate phases, elemental iron is indicated as a contaminant in the titanium oxide for both the valve seat and the poppet in connection with the second failure.

It will be observed that the scale formed on the poppet in the case of each failure is comprised essentially of the lower oxides, and primarily of $TiO_{1.25}$. It appeared that when the scale was very shallow and tightly adherent, such as in the poppet cavities and the deeply burned areas on the seats directly below the poppet cavities, it consisted largely of $TiO_{1.25}$ or lower oxides. On the other hand, the loosely-adhering residue, which appeared to represent material which had been transferred from one part to another during the course of the failure, was TiO_2 and other oxygen-rich structures. For example, in Fig. 4a the scale in the cavity of the poppet was $TiO_{1.25}$ as well as that in the deeply-burned area of the seat at "D". However, the loose residue on the seat at "E" (the surface below "E", after removal of the scale, was intact and unaffected) was largely TiO_2 (see Table I for analyses).

The results of the X-ray analyses taken on the residues removed from the 347SS valve body parts are presented in Table II. In addition to these data, both chemical and spectrographic analyses were taken of the residue removed from the valve pot for the first failure only, giving the following results:

Chemical Analysis						
Cr	Fe	Ni	Ti	Al		
mg/g						
16.3	215	2.81	191	11.3		
Spectrographic Analysis						
Ti	Al	V	Cb	Cr	Fe	Ni
>5	1	1	1	>5	>10	>2

The chemical and spectrographic analyses agree with one another reasonably well, showing the presence of the ingredients of both the stainless and the titanium alloy parts to about equal extents.

The results of the X-ray diffraction analyses for the valve body parts revealed oxides of iron as well as oxides of titanium and in certain instances double oxides of these metals. It will be noted that two separate groups analyzed the residues from the second-failure pot and the second-failure flange and the results are not in agreement. The Metallurgy Division group found very substantial amounts of austenitic stainless and alpha iron structure in their patterns, whereas the HRP group got predominantly oxides based on iron and titanium.

Because of this discrepancy, the X-ray pattern readings obtained by the HRP group were given to the Metallurgy Division group for interpretation as a means of a check. Their interpretations were very close to those of the other group. Consequently, it may be assumed that the samples given

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the individual analyzers for these parts were different in constitution and the results reported in Table II are correct. It is believed that the sampling technique was in fault and that both oxide and metal structures were represented in the residue at large. Possibly the more loosely-adhered spatter was predominantly oxide, whereas the dense scale was largely solidified metal.

The chemical analyses taken on the system solutions subsequent to the failures were as follows:

<u>First Failure, Uranyl Sulphate Solution</u>						
<u>U</u>	<u>SO₄</u>	<u>Cu</u> mg/ml	<u>Ti</u>	<u>Ni</u>	pH	N
6.1	4.1	0.11	0.002	0.229	1.6	0.031

<u>Second Failure, Phosphate-Laden Water</u>						
<u>Fe</u>	<u>Cr</u>	<u>Ni</u>	<u>Cl</u>	<u>Ti</u>	<u>Cu</u>	<u>Total Solids</u>
micrograms/ml						
25.0	<1.0	<1.0	4.0	<1.0	<1.0	46.0 mg in 40 cc

In comparison to the original uranyl sulphate solution, the analyses for the uranium and copper are low. Other items reported for the first failure appear to be normal. The 6.1 mg/ml value for U and 0.11 mg/ml for Cu as compared to 10.0 and 0.33 for a normal solution, suggest that the failure may have caused partial precipitation of these ingredients, probably in the form of oxides.

The only unusual feature associated with the analysis on the wash water is the high content of insoluble solids. Failure of the valve, no doubt, is responsible for this high value.

DISCUSSION AND CONCLUSIONS

The small crater found on the poppet in the case of the second failure demonstrates that such processes may, in certain instances, proceed little beyond an initial small-crater producing reaction, whereas in other instances the process may proceed on from such an initial reaction to consume large quantities of material. When this occurs a large cavity is produced which is quite similar in appearance to cavitation corrosion.

It is suggested that the crater-type cavities resulted from reactions of explosive character whereas the portions of the cavities which have the definite erosive characteristics, resulted from a non-explosive but

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nonetheless self-sustaining reaction. Such a point of view immediately poses the questions: (1) How are such processes initiated? (2) Once initiated, how are they sustained? (3) How are these processes ultimately terminated?

In regard to the first point, a parallel may appear to be at hand in the case of the titanium-fuming nitric acid explosions wherein a pyrophoric material is set off by impact². Unfortunately, however, the pyrophoric condition in this case resulted from fine particles which were produced by corrosive attack and no such corrosive attack or particles were found on the trim parts. On the contrary, the all-around resistance of titanium to the corrosive medium involved is considered to be extraordinarily good.

Rather than a corrosion-induced condition for setting the stage for failure, one produced by mechanical effects appears more likely. A study of possible pyrophoric conditions in titanium as produced by cycles of mechanical working, galling, and film formation may be worthy of consideration. Or possibly an area of copious debris is not required and that the reaction can be started by a single fragment extending from the surface. Such a fragment, created as a result of opening the valve, would have an active (as opposed to passive) surface and high surface-to-volume ratio, and under the environmental conditions would be ignited spontaneously. This, however, does not appear as likely as the former suggestion, since it is difficult to envision transfer of sufficient heat from the peninsula to the surface to carry the reaction on over to an autocatalytic state.

On the questions of sustaining the process, once initiated, and its termination, these must obviously relate to the reaction heat balance, i.e., to sustain the reaction, the rate of heat generation must exceed the rate of its dissipation. Otherwise, the temperature would decrease and the reaction subside.

To account for the reaction sustaining itself in the case of the valve parts, a dynamic condition may be required wherein the oxidation products are blown free of the reacting surface so as not to retard the rate of reaction as a result of obstructing the diffusants responsible for the reaction. If the diffusants are retarded too greatly, the heat-balance is upset and the reaction extinguished. On the other hand, the onrushing solution must not extract heat at too great a rate lest the heat balance be upset in this way. While the low thermal conductivity of the metal would favor the maintenance of surplus heat, a refrigerating effect stemming from pressure drop and vapor formation, would work in the opposite direction.

2. V. B. Rittenhouse, et al, "Corrosion and Ignition of Titanium Alloys in Fuming Nitric Acid," CIT Progress Reports 26-1, 26-2, 26-3, 26-4, AF33 (616)-3066, Sept. 1955-March 1956.

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With the pressure drop and high turbulence inherent in the valve design, it is quite likely that the system solution would contain a high percentage of oxygen-rich vapor by the time it passes the area of reaction on the poppet and seat. Such a mixture of liquid and vapor would have a high degree of erosiveness in the removal of scale and molten metal and at the same time would not remove heat as rapidly as would liquid alone.

Although the various pressure charts did not indicate whether the failures started with opening or closing of the valve, other considerations lead to the conclusion that it occurred with opening. First of all, if the reaction had started with closing it would have had to sustain itself largely under static conditions, which is not likely. In the second place, the reaction markings on the trim seat in the case of both failures point to the reaction having started with opening. Along the ring of contact of the valve seat within the confines of the poppet cavity above, there are places where the reaction failed to burn or mar the seat. This could not be if the reaction had proceeded with the valve in the closed position.

The mode of the failure in the case of the impeller hub was somewhat different to the present failures and in no way do the facts connected with that failure refute the arguments put forth in the present case.

While it is not inconceivable that the pump failure could have stemmed from a pyrophoric condition also, it is more simply explained in terms of frictional heat produced by galling and wear. Once the reaction was started, the metal expanded thermally which produced greater interference and additional reaction. Confined to the small space immediate to the hub, the reaction products probably contributed to further wear and abrasion.

The pump was run at 3600 RPM. While the velocity of the solution with respect to the metal surface was very low compared to the case of valve failures, some degree of turbulence probably abetted the reaction as did the surrounding heat, slow in its removal from the area. The heat-affected zone on the hub was about 1/10-in. at the point of greatest depth, whereas in the case of the valve trim parts the heat-affected zone was almost negligible.

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TABLE I. X-RAY DIFFRACTION ANALYSES OF RESIDUES ON TITANIUM ALLOY TRIM PARTS

Part	Description	Results
1st failure poppet	Tightly adhering metallic scale, sl. magnetic, Fig. 4b, "A"	Med. $TiO_{1.05}$ Med. 2 lines (un- identified)
1st failure valve seat	Tightly adhering black scale, sl. magnetic, Fig. 4a, "D", for position	Med. $TiO_{1.25}$ Med. 5 lines (unidentified)
1st failure valve seat	Loosely adhering black spatter, str. magnetic, Fig. 4d, "E", for position	Str. TiO_2 , brookite Str. αTi_2O_3 Wk. TiO_2 , anatase Wk. 14 line (unidentified)
2nd failure poppet	Tightly adhering black scale, ranging from str. magnetic to no magnetism, Fig. 6, "B"	Med. $TiO_{1.25}$ Med. Fe_2TiO_2 Wk. αFe (one line) Wk. αTi_2O_3 αFe_2O_3 Wk. 2 line (unidentified)
2nd failure valve seat	Black residue, major amt. very magnetic remainder sl. magnetic, Fig. 7a, "B"	Str. 347 SS(FCC) Med. αFe (BCC) Med. TiO_2 anatase Wk. αTi_2O_3 αFe_2O_3 Wk. 3 lines (unidentified)
2nd failure valve seat	Brown residue, str. magnetic, Fig. 7a, "C"	Str. 347SS (FCC) Str. αFe (BCC) Med. 6 lines (unidentified)

NOTE: All analyses were conducted by X-Ray Diffraction Department, Metallurgy Division. Except for the first entry, all analyses were made by the powder technique, using a Debye-Scherrer type camera. The first determination was made by a black reflection shot directly on the cavity of the poppet.

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TABLE II. X-RAY DIFFRACTION ANALYSES OF RESIDUES ON 347SS VALVE BODY PARTS

Part	Description	Results
1st failure valve pot	Loosely adhering black spatter and scale, V. str. Fe_2O_3 * part str. magnetic, part sl. magnetic, sample taken at large Fig. 2	(alloyed) Wk. TiO_2 , anatase Wk. NiO Wk. other lines
2nd failure valve pot	Loosely adhering black spatter and scale, Str. Fe_2O_3 ** v. str. magnetic	Med. 347SS (FCC) Med. alpha Fe (BCC)
2nd failure valve pot	Loosely adhering black spatter and scale, Str. Fe_2TiO_5 *** str. magnetic	Med. FeTiO_3 Wk. 5 lines (unidentified)
2nd failure valve flange	Black scale and spatter, v. str. magnetic	Str. 347SS (FCC) ** Med. alpha Fe (BCC) Wk. Fe_2O_3 Wk. 1 line (unidentified)
2nd failure valve flange	Black scale and spatter, v. str. magnetic	Str. Fe_2TiO_5 *** Med. FeTiO_3 Med. alpha Fe (BCC) Med. Fe_2O_3 Wk. TiO_2

* Conducted by Spectroscopy Research Dept., Stable Isotopes Research and Development Division.

** Conducted by X-Ray Diffraction Dept., Metallurgy Division.

*** Conducted by Metallurgy Group, HRP:

NOTE: All analyses were made by the powder diffraction method. Except for entry 3, where a diffractometer was employed, a Debye-Scherrer type camera was used. Samples were taken at large from the individual parts.

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Fig 1. VALVE POPPET AND SEAT

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Fig. 2. First Failure, Valve Body with Titanium Alloy Seat in Place.

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Fig. 3. First Failure, Valve Flange with Titanium Alloy Poppet in Place.

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Fig. 4. First Failure, Valve Trim. (a) Poppet and seat as removed from mating position, 1X. (b) Poppet, 3.5X. Reduced 11.5%.

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Fig. 5. First Failure, Eroded Area on Flange Resulting from "Burnout". (a) Flange. (b) Recession at point "A" in illustration (a), 10X. Reduced 11.5%.

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Fig. 6. Second Failure, Poppet Viewed from Inlet Side.

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Fig. 7. Second Failure, Valve Seat Viewed from Outlet Side. (a) Note small crater at position "A" along contact ring. (b) The same crater at 50X magnification.

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Fig. 8. Second Failure, Crater on Poppet in Position Opposite Small Crater Shown in Fig. 7(b). Downstream of ring of contact is up. (a) 15X, (b) Same, 50X.

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Fig. 9. Cross-section of Poppets Showing Extent of Heat-Affected Zones. (a) First failure, section "a,b" of Fig. 4b, 7.5X. (b) Second failure, section "a,b" of Fig. 6, 5X. Reduced 11.5%.

Figure 10. First Failure, Microstructure at Cavity of Poppet. (a) Junction of cavity with unaffected surface, "A" of Fig. 9a, 250X. (b) Scale formation at "B" of Fig. 9a, unetched, 250X.

Fig. 11. Second Failure, Microstructure at Cavity of Poppet. (a) At cavity surface, 250X. (b) Distant to cavity surface, 250X.