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FAILURE ANALYSIS REPORT - S/N 2H NOZZLE

MAY 1965

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Space Nuclear Propulsion Office
Cleveland Extension
National Aeronautics and Space Administration
21000 Brookpark Road
Cleveland, Ohio 44135

Attention: L. C. Corrington
Assistant Chief for Technical Operations

Subject: AGC Report RN-TM-0207, Failure Analysis Report
S/N 2H Nozzle, May 1965

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- (1) RN-TM-0207, Failure Analysis
Report - S/N 2H Nozzle, May 1965
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MAY 1965

REON TECHNICAL MEMORANDUM

FAILURE ANALYSIS REPORT - S/N 2H NOZZLE

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AUTHOR:

S. Goodman

S. GOODMAN
METALLURGICAL ENGINEER
REON MATERIALS & PROCESSES

APPROVED:

C. W. Funk

C. W. FUNK
MANAGER
REON MATERIALS & PROCESSES

REON TECHNICAL STAFF:

J. J. Peterson

J. J. PETERSON
REON TECHNICAL STAFF

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FAILURE ANALYSIS REPORT - S/N 2H NOZZLE

I. SUMMARY

A. Results

Flame and pressure instabilities were reported to have caused the shut-down of the chemical firing test, after which damage to the nozzle was observed. Metallurgical examination of specimens taken from the tube crowns in five locations where damage was observed, showed that the material was sound initially. Microstructures indicated temperatures above 1900°F. Micrograph indications included thermal fatigue, grain growth, columbium-carbide solution and columbium carbo-nitride liquation. The sample examined for metal erosion showed a loss of only 0.0009 in. from the nominal 0.014 in. The two damaged areas were in the throat and 180° apart.

B. Conclusions

The material was sound and of the desired thickness initially. The engine was observed to malfunction sufficiently to cause the test to be stopped. The metal specimens give evidence of temperatures reaching the range of 2100°F-2600°F. Damage was observed in two areas in the throat which were 180° apart. It is concluded from this that failure of the S/N 2H nozzle was due to severe local overheating caused by flame instability.

C. Recommendations

It is recommended that vacuum treated stainless steel Type 347 be used for the U-tube material. This will tend to prevent the formation of columbium carbo-nitrides and improve stress-rupture strength.

I. INTRODUCTION

On July 6th, 1964, the serial number 2H nozzle (P/N 296500-9) was found to have suffered damage during chemical firing. The principal damage consisted of a number of cracked tubes in the throat area. Upon hydro-testing on July 14th, a leak rate of 87 lbs/min. was recorded.

Abnormalities experienced during the test run in which failure occurred included: excessive pressure variations (greater than 100 psi) and cycling of pressure variations (greater than 1000 cycles/sec). The combustion stability monitor ended the test as a result of these conditions. It was later determined that the fuel had entered the chamber as a liquid, rather than a gas.

The test in which failure was experienced was the tenth for this nozzle. In Table I, the dates and durations of tests are listed, as are pertinent comments. Cracking was not experienced until the tenth and final run. To date of May 6th, this nozzle has not been re-tested.

II. PROCEDURE

REON technical personnel visually examined the interior of nozzle S/N 2H. The observations recorded herein are graphically presented in Figure 1. Based upon these observations, an estimate of event sequence was developed in order to permit analysis of the data and an interpretation of the factors contributing to this failure. This estimate is presented in Figure 2. A second graphical presentation of data was developed to clarify the possible interrelationship of tube dents and cracks. This is presented in Figure 3.

The service history of the S/N 2H nozzle is given in Table I.

The metal specimens taken (see Figure 4) were visually examined and macrophotographed. They were then sectioned, mounted and prepared for metallography. (See Table II.) The significant specimens were microphotographed. Micro-hardness

tests were made. (See Table III.) In addition, two specimens were submitted for electron fractography. One specimen was used for spectroscopic analysis for composition.

III. DISCUSSION

Three potential causes of tube crown leaks (see Ref. 1) can be enumerated: overstress, defective material, and excessive temperature.

An overstress could be generated as a result of an over-pressurization of the system. Since it was necessary for the combustion stability monitor to end the test, over-pressurization might have resulted.

Wall thinning through fabrication malpractice, creep, oxidation or erosion could also result in an overstress of the remaining material, causing failure of the system.

Defective material used for the tube could lead to failure. The material could be defective because of improper chemical content, local defects, corrosion attack, or an unsatisfactory microstructure.

The only evidence of overpressurization that we have is the information that a shut-down was required. This was done in order to prevent damage by overpressurization. Presumably the shut-down was effective in accomplishing this. We may therefore discount this factor as a major cause of failure, although not as a contributing cause.

Figure 5 provides some of the data for the evaluation of wall thinning as a factor in the failure of the tubes. The upper surface shows roughening caused by material loss. The original nominal thickness of the tube wall is .014 in. The wall thickness measurement of the specimen in Figure 5 is .0131 in. (average of three readings). Since the samples that could be taken from the 2H nozzle were small and few, additional testing was performed to evaluate the wall thickness

loss of AISI 347 under simulated firing conditions. The results of these tests were published in References (2) and (3). In addition, Reference (3) includes an analysis of the relative effects of wall thinning and stress-rupture behavior on the failure of tube walls. The wall thinning observed in the supplementary tests is consistent with the measurements reported for Sample 2 (Figure 5). Further, the study in Reference (3) shows that, for temperatures above 1900°F and for very short times, the stress-rupture strength is more critical than the wall thickness loss.

We may then conclude that wall thickness loss did not contribute significantly to the failure of the tubes of Nozzle S/N 2H.

The possibility that defective material was used needs to be considered. The only direct evidence we have is that presented by the micro-hardnesses and the microphotographs. As Table III shows, the hardnesses found are characteristic of annealed AISI Type 347. The microphotographs do not show a high incidence of non-metallic inclusions. The chemical analysis attempted by spectroscopy was not successful, due to the excessively small sample available for this test. Figure 5 shows a normal grain size at the fuel side indicating that this material was initially satisfactory in this respect. Here we have a potential cause of failure which cannot be completely evaluated.

The possibility of excessive temperature rise must now be considered. Viewing Figure 1 we see that there is some evidence of temperature effects inasmuch as oxide deposits are listed. However, Figure 2 shows that not only overheating but back flow on shut-down may have contributed to this. Figure 5 shows us a section through the thickness of sample 2. Note the grain growth from ASTM micrograin Size No. 9 to No. 6 and the partial solution of the finely dispersed columbium carbide grains. Here the conclusion is drawn that a temperature range of 2000° - 2200°F was experienced. Figures 6 and 7 show the cracking which led to sampling of these areas. While the main crack is longitudinal, there are a number of transverse cracks branching from it. The irregular path of the

crack, seen most clearly in Figure 7, suggests an intergranular mode of rupture. This same intergranular, branching mode of cracking is also seen in Figures 8 and 9. Figure 10 shows the locations on sample 3 (Figures 8 and 9) from which the microspecimens for the following figures were taken:

Figure 11 is a view looking down on the crack in Figure 8. The magnification is 75X. The intergranular nature of this crack is confirmed at this magnification. Visible evidences of high temperature are the enlargement of grains and the massive amounts of coarse precipitates.

Figure 12 shows the face of the fractured surface. The massive precipitation of columbium carbides and columbium compound could only have been caused by extremely high temperature since the total heating time (total time for all runs) given in Table I is only 69 seconds. The temperature reached must have been on the order of 2350°F.

Additional evidence of high temperature is given by Figure 13. The zones which present a cross-hatched appearance are columbium carbo-nitrides which melt at about 2400°F (see Ref. 4). Had the rate of heating been slow, the columbium carbo-nitrides would have dissolved in the iron-chromium-nickel matrix, rather than retain their identity and melt.

Another view of the microstructure of sample 3 is presented by Figure 14. At the upper right-hand corner, evidence of melting may be seen. Here, the tree-like, or dendritic, pattern common to cast metal proves that the temperature reached 2550° - 2600°F.

Examples of transverse cracking are shown in Figure 15.

Figure 16 shows the manner in which sample 5 was cut and the resulting segments mounted for micro-hardness measurements and metallography.

Figure 17 shows a surface perpendicular to the fracture surface. The edge at the right is the edge of the fracture surface. The presence of ferrite stringers and enlarged grains point to a temperature on the order of 2350°F. The specimen exhibits two intergranular cracks.

Figures 18, 19, and 20 are electron fractographs at 10,000X. Figure 18 is representative of almost all of the area of the fracture surface of sample No. 1. It is interpreted as an indication of ductile tensile failure. However, Figure 19, which was also made from sample 1, gives evidence of thermal fatigue in the center of the field of view. Figure 20 was obtained from sample 4 and indicates a ductile tensile failure.

The indication of thermal fatigue in Figure 19 is not the only one. References (5) and (6) deal with thermal fatigue and contain illustrations which show the same characteristics as the nozzle 2H failure specimen. The cracking shown in both the references, and that observed in the failed material, shows an intergranular initial failure with a rounding of the fracture surface projections developing afterward. These features may be readily observed in Figures 11, 12, and 17.

We may see from the weight of evidence above that the most important factor acting to cause failure of the tubes was the development of a metal temperature range of 2100 - 2600°F at various locations. Data available in Ref. (7) shows the tensile strength of AISI 347 to be 4000 psi at 2400°F. Coupled with the internal fuel pressure and the compressive stresses caused by the thermal expansion of the tube material, the lowered strength and the thermal fatigue effects caused the metal to bulge (see Figures 6, 7, and 15) and fail.

The cause of the metal temperature rise has not yet been identified. Returning to Figure 2, see that both general and local overheating are listed. However, the pattern of tube cracking as shown in Figure 3 is highly localized, with 15 out of 16 cracked tubes being concentrated in two places.

The localized overheating could have been caused by coolant passage blockage. Had this condition existed from the start, it would not have been possible to conduct so many runs with temperatures rising to the melting point. Later obstruction could have been generated by the igniter failure during the ninth run. Fragments of the device did cause denting. However, a look at Figure 3 shows no correlation between the distribution of dented tubes and those which cracked.

With these potential causes eliminated, the remaining evidence consists of the experience obtained during the tenth run when it was necessary to stop the test due to excessive pressure variation and cycling.

From this we conclude that local overheating resulting from flame instability caused the failure of the coolant tubes by reducing the strength level and causing high-temperature, low-cycle, thermal fatigue.

TABLE NO. I

SERVICE HISTORY - NOZZLE, S/N 2H

<u>Date</u>	<u>Duration of Run (sec.)</u>	<u>Remarks</u>
3/19	2.543	No Damage
3/28	1.050	No Damage
3/30	6.550	No Damage
4/1	10.100	No Damage
4/3	3.630	No Damage
4/6	15.380	No Damage
4/30	10.800	No Damage
5/6	15.900	No Damage
7/1	1.400	No Damage - But Igniter Failed.
7/6	2.173	Cracks in tubes at throat
Total firing time	69.526 sec.	

TABLE NO. II

MICRO-SPECIMEN ETCHING TECHNIQUE

ETCHANT:	Freshly prepared
	25 gms Ammonium Bifluoride (NH_4FHF)
	50 ml. Concentrated Nitric Acid (HNO_3)
	100 ml. Distilled water
ETCHING TEMPERATURE:	Room Temperature
ETCHING TIME:	10 seconds
ETCHING METHOD:	Swab Application

TABLE NO. III

MICROHARDNESS VALUES*

<u>Sample No.</u>	<u>Mount No.</u>	<u>Average Microhardness</u>
2	8844	R _B 87.1
3A	8845	R _C 20
3B	8846	R _B 87.7
5A	8848	R _B 84.8
5B	8849	R _B 94.3
5C	8850	R _B 84.2
5D	8851	R _B 92.0

*Typical Hardness-Annealed Sheet = R_B 85 (Ref. 8)

Figure 1
Available Evidence - 2H Nozzle

Figure 2
Estimate of Event Sequence - 2H Nozzle

Figure 3
Nozzle Cross Section
Crack and Dent Distribution (Looking Aft)

* 12 O'CLOCK POSITION IS IN LINE WITH
FUEL INLET LOCATION, WHILE 6 O'CLOCK IS IN
LINE WITH THE OXIDIZER INLET

Figure 4
Nozzle Interior
Metallographic Specimen Locations

Figure No. 5 Sample No. 2

250X

This specimen was taken from the throat section at the two o'clock position. It was selected because it was in an area of the tube which had been flattened. A check of the thickness was desired to determine if there had been significant metal loss through erosion.

The photomicrograph shows a cross-section through the thickness of the tube sample. The measured thickness is 0.0131 in. (average of three readings) as compared with an original nominal thickness of 0.014 in. This measurement was reported in Ref. 9. Not only this data, but the results of subsequent testing, as reported in Ref. 2 and Ref. 3, show that wall erosion is not the primary factor in determining U-Tube life.

In the view shown, the upper edge is at the face exposed to the ^{fuel} flame, while the lower edge is at the face in contact with the ^{flame} ~~fuel~~.

In traversing the metal thickness from the fuel side to the flame side, it will be noted that the grain size increases. At the fuel side, the grain size is ASTM Micro-grain size 9 (per Ref. 10). This is the same grain size exhibited by as-formed tubing (see Ref. 11). At the flame side, the grain increases to ASTM Micro-grain Size No. 6. Since there has been no grain growth at the fuel side, the temperature reached here was below 1900°F, just how much below is not indicated by the microstructure. At the flame side, the grain size is indicative of a temperature range of 2000° - 2200°F. This temperature estimate may be confirmed by analyzing the columbium carbide distribution in the sample. The small, black spots are the columbium carbides. It can be seen that they are less numerous at the flame side. Since they have not completely disappeared, we can see that the carbide solution temperature of 2200°F has not been exceeded. (See Ref. 12.)

It should be noted that there is no evidence here of grain boundary precipitation nor of any cracking.

Figure No. 6 Sample No. 1

7X

This specimen was removed from the 5 o'clock position in order to permit study of the defect. The flame surface is shown and is discolored by an oxide deposit. The crack is primarily longitudinal with relation to the tube axis and branches transversely at several points.

Figure No. 7 Sample No. 1

7X

The fuel surface of this specimen is shown. The extent and branching nature of the crack may be more easily seen than in the photograph of the flame surface. The black ink marking indicates the forward end of the specimen.

Figure No. 8 Sample No. 3

5X

This sample was removed from the throat area at 3 o'clock. The forward end (at left) is identified by the black ink mark. The surface is coated with an oxide scale. The crack is longitudinal with branching, transverse cracks.

Figure No. 9 Sample No. 3

5X

The fuel side of the same specimen as shown in Figure 8 is represented here. The surface is bright and free of oxide. The crack pattern may be more readily seen here. The black ink mark indicates the forward direction.

Figure 10
Schematic of Microspecimen Selection
From Sample No. 3

Figure No. 11 Sample 3C-1

75X

This photomicrograph presents a view of a section of one of the transverse cracks found in Sample 3. The junction of the transverse crack with the longitudinal crack is shown at the left. The form of thermal fatigue cracking is intergranular and this may be seen most clearly at the various points at which small cracks lead away from the main fracture, such as at the extreme right. (see Refs. 5 and 6).

Figure No. 12 Sample 3B

250X

The sample was taken from the left hand end of the crack shown in Figures 8 and 9. The face of the transverse crack is shown.

The salient feature of the microstructure is the massive precipitation in the grain boundaries. The precipitate actually forms a very nearly continuous network. It will be noted that in these areas the small, black particles of columbium carbide have disappeared. The precipitate here is made up of agglomerated columbium carbides and columbium compound, (see Ref. 12). Such precipitation occurs at 2350°F and higher, as does the agglomeration effect.

The networks formed tend to embrittle the metal and this is partly responsible for the loss of material in specimen preparation. Some evidence of thermal fatigue cracking is to be found at the edges of the larger voids which extend inward from the surface. This too, would lead to loss of metal.

It is highly probable that the formation of the precipitate in the grain boundaries inhibited the grain growth which might have been expected upon heating this metal to temperatures of 2350°F and above.

Figure No. 13 Sample 3C - 3

250X

The field of view in this photomicrograph is the intersection of a plane tangent to the tube crown of sample 3, and the crown. This field is in the vicinity of a crack (see Figures 8, 9, and 11).

Those areas in the view with a cross-hatched appearance (dendrites) are zones of liquation of columbium carbo-nitrides. The theory which explains this does so by indicating that the carbo-nitrides do not have time to decompose when heating is very rapid. Upon slow heating, these compounds decompose and dissolve in the matrix, thereby avoiding liquation. (see Ref. 4). The temperature at which liquation, or melting, occurs is 2400°F , below the melting point of the metal.

The black areas at the center of the photograph are caused by the removal of material during specimen preparation. The black area at the lower right is part of the crack.

Figure No. 14 Sample 3C - 2

250X

This sample was taken from the throat section at the 2:50 o'clock position. A cast, dendritic structure may be seen, being most prominent at the upper right. In order for such a structure to appear, the metal must be heated to its melting range of 2550 - 2600°F.

Figure No. 15 Samples 4 (L) and 5 (R)

5X

Samples Nos. 4 and 5 were taken from the same tube as sample no. 3. The flame side of sample 4 is shown at the left and the fuel side of sample 5, is shown at the right. Both samples have transverse cracks and are marked with black ink to indicate the forward end. At the right-hand edge of sample 5 a portion of a weld repair may be seen.

Figure 16
Schematic of Microspecimen Selection
From Sample No. 5

Figure No. 17 Sample 5A

250X

This photomicrograph shows a plane through the thickness of the sample and perpendicular to the surface of the transverse crack. The edge at the right is the edge of the fracture surface.

The grain size here has become enlarged as a result of exposure to high temperature. The ferrite phase has formed elongated stringers. This is indicative of temperatures on the order of 2350°F. (See Ref. 12.) Also, visible are two intergranular cracks, one extending inward from the center of the fracture surface and one originating at the lower right-hand corner of the sample.

Figure No. 18 Sample No. 1
Electron Micrograph #441

10,000X

The view above is of the fracture surface of the specimen. The areas which appear to be domed are, in fact, representative of depressions in the fracture surface against which the replica is cast. These domes are interpreted as areas of ductile failure, while the flat areas are interpreted as zones of brittle failure. The overall surface is described as representative of a ductile tensile failure. (See Ref. 13.)

Figure No. 19 Sample No. 1
Electron Micrograph No. 442

10,000X

The view above is of an area of the fracture surface which gives evidence of thermal fatigue in the center. (See Ref. 13.)

Figure No. 20 Sample No. 4
Electron Micrograph No. 443

10,000X

The view above is of a portion of the fracture surface of sample no. 4. The areas which appear to be domed are interpreted as areas of ductile fracture while the flat areas are considered to be areas of brittle fracture. The overall view is taken to be indicative of a ductile tensile failure. The entire surface examined is represented by the view above. No evidence of thermal fatigue was found here. (See Ref. 13.)

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