EXPLOSIVE EVENT IN MON-3 OXIDIZER SYSTEM RESULTING FROM PRESSURE TRANSDUCER FAILURE *

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ABSTRACT

In 2003, a Druck® pressure transducer failed catastrophically in a test system circulating nitrogen tetroxide at NASA Johnson Space Center White Sands Test Facility. The cause of the explosion was not immediately obvious since the wetted areas of the pressure transducer were constructed of materials compatible with nitrogen tetroxide. Chemical analysis of the resulting residue and a materials analysis of the diaphragm and its weld zones were used to determine the chain of events that led to the catastrophic failure. Due to excessive dynamic pressure loading in the test system, the diaphragm in the pressure transducer suffered cyclic failure and allowed the silicon oil located behind the isolation diaphragm to mix with the nitrogen tetroxide. The reaction between these two chemicals formed a combination of 2,4-di and 2,4,6-trinitrophenol, which are shock sensitive explosives that caused the failure of the pressure transducer. Further research indicated numerous manufacturers offer similar pressure transducers with silicone oil separated from the test fluid by a thin stainless steel isolation diaphragm. Caution must be exercised when purchasing a pressure transducer for a particular system to avoid costly failures and test system contamination.

INTRODUCTION

In March 2003, the White Sands Test Facility (WSTF) was performing a life cycle test of a fast-acting valve, using nitrogen tetroxide (NTO) as the test fluid. The test system was designed to circulate the NTO from one 55-gal cylinder, through the test article to an identical cylinder and back again. The test system was constructed of ~ 50 ft of ¾-in. and ¼-in. stainless steel tubing. All wetted components in the test system were compatible with the NTO. The inlet supply of NTO was pressurized to 264 psid (~ 300 psig). The test article was opened for 80 ms each cycle and had a flow rate of 1.91 lb/s.

After performing ~ 36,000 cycles, an explosion was heard from the test cell. The video camera in the test cell was quickly obscured by a cloud of NTO, preventing the test team from being able to immediately determine the cause or location of the leak. The test system was quickly powered down and all valves were closed to minimize the spill. Test personnel were then sent into the test cell with totally encapsulating suits to survey the damage and begin the recovery efforts.

During the inspection, it was immediately obvious a pressure transducer on the test article inlet line failed catastrophically, releasing liquid NTO into the test cell. Figure 1 shows the area of the pressure transducer failure and the approximate previous location of the manifold and pressure transducer. The test team recovered the inlet port and body of the pressure transducer and approximately three-quarters of the diaphragm. The cause of the failure was unknown at this point. An investigation team was assembled to review the situation and determine the cause of the failure and how to avoid similar problems in the future.

* Approved for public release; distribution is unlimited.

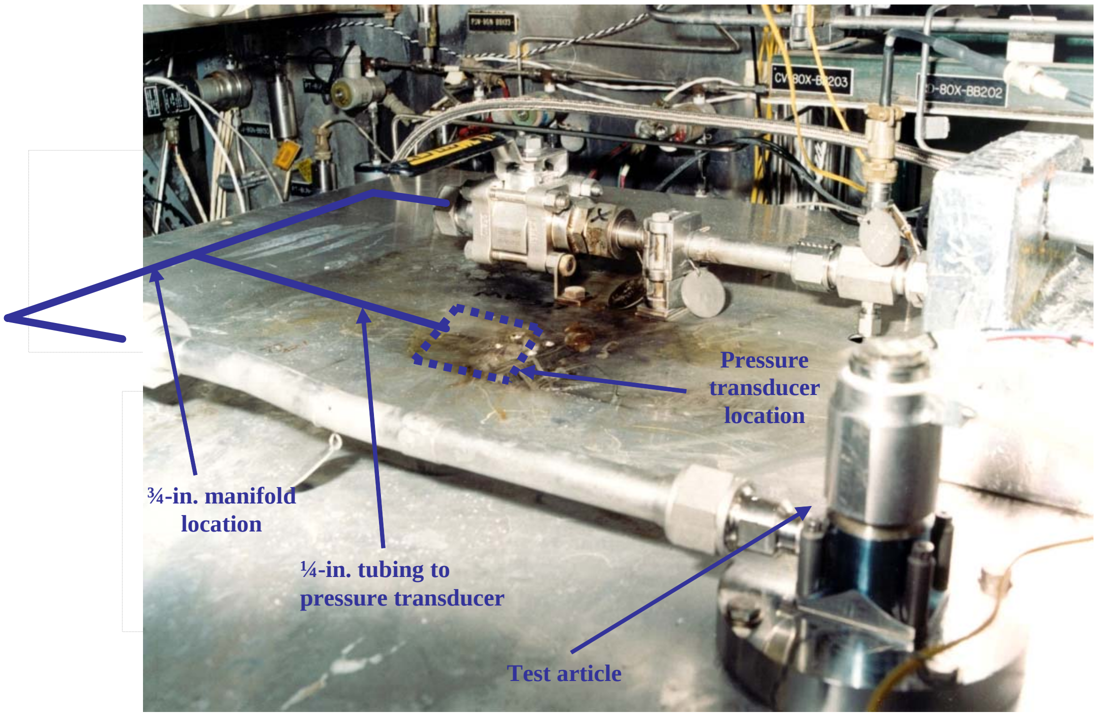

Figure 1: Location of Failed Pressure Transducer

RESULTS AND DISCUSSION

EXAMINATION OF PRESSURE TRANSDUCER AND TEST SYSTEM

The pressure transducer failed at the welded connection between the transducer body and inlet housing (Figure 2). The weld appears to be an electron beam weld (EBW) butt weld configuration. Failure of this weld is from longitudinal stress in the weld fusion zone. The pressure to fail this welded connection was calculated to be 12,700 psi. The ¼ x 0.035-in. wall 300-series stainless steel tubing connecting the pressure transducer to the ¾ x 0.035-in. wall 300-series stainless steel manifold tubing failed close to the ¾-in. tubing. Although the ¼-in. tubing was significantly deformed, the damage primarily appeared to be from thrust generated when the pressure transducer failed. Based on measuring the outside diameter in several locations, the ¾-in. tubing showed no sign of yielding due to pressure. This indicates the system pressure did not exceed the calculated 3,400 psi required to yield this tubing. A small, central area of the failed diaphragm, which matches the inlet port flow passage diameter, was bulged and ruptured (Figure 3). The calculated pressure to burst the diaphragm when supported at the port diameter is ~ 4,000 psi. Based on these observations, the failure appears to be the result of extremely rapid over-pressurization inside the pressure transducer and behind the isolation diaphragm that first exceeded the ~ 4000-psi diaphragm burst pressure. Flow through the 0.157-in. port flow diameter was inadequate to relieve the over-pressure, thus enabling the pressure to reach the ~ 12,700-psi housing burst pressure. After burst, sufficient thrust was achieved to deform and then fail the ¼-in. tubing connected to the pressure transducer. The sensor location of the pressure transducer is shown in Figure 4. Note the oily residue seen in this photo.

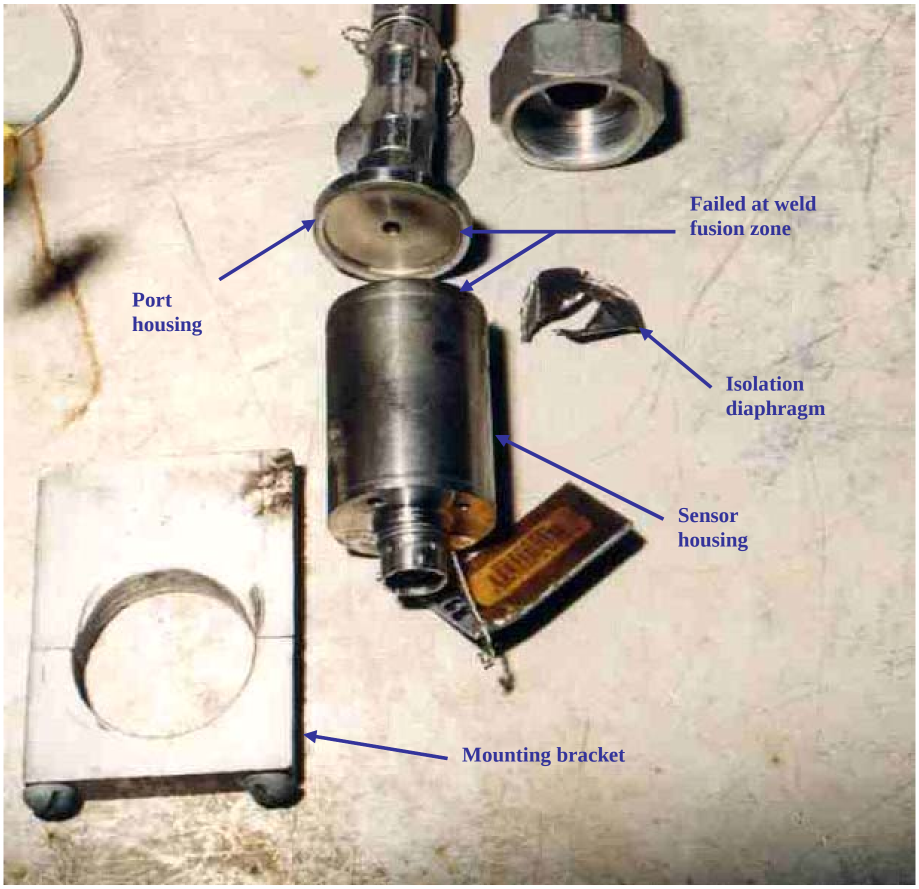

Figure 2: Failed Druck® PDCR 130/W/C Pressure Transducer

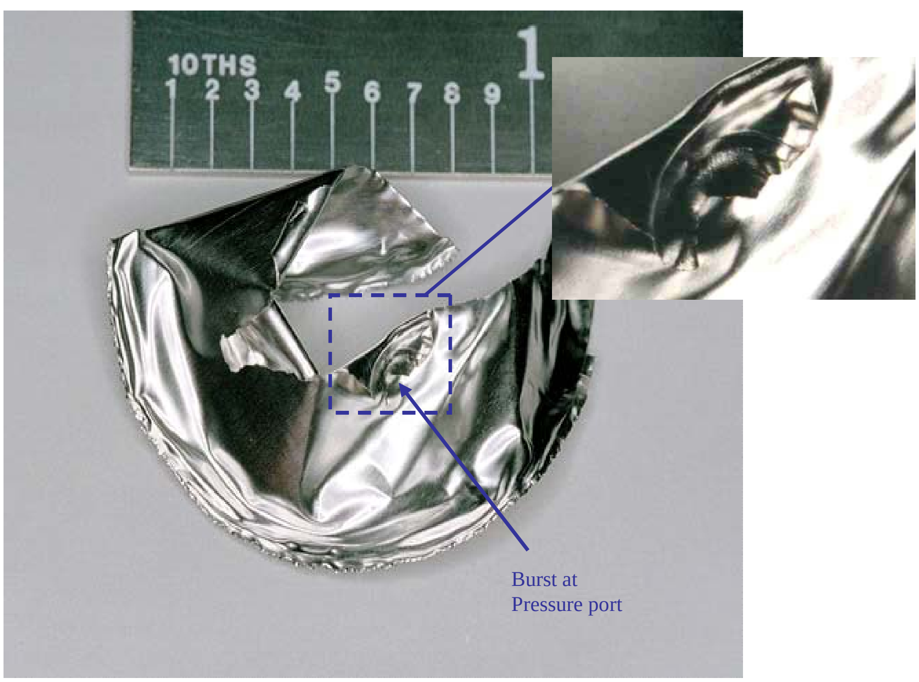

Figure 3: Failed Isolation Diaphragm

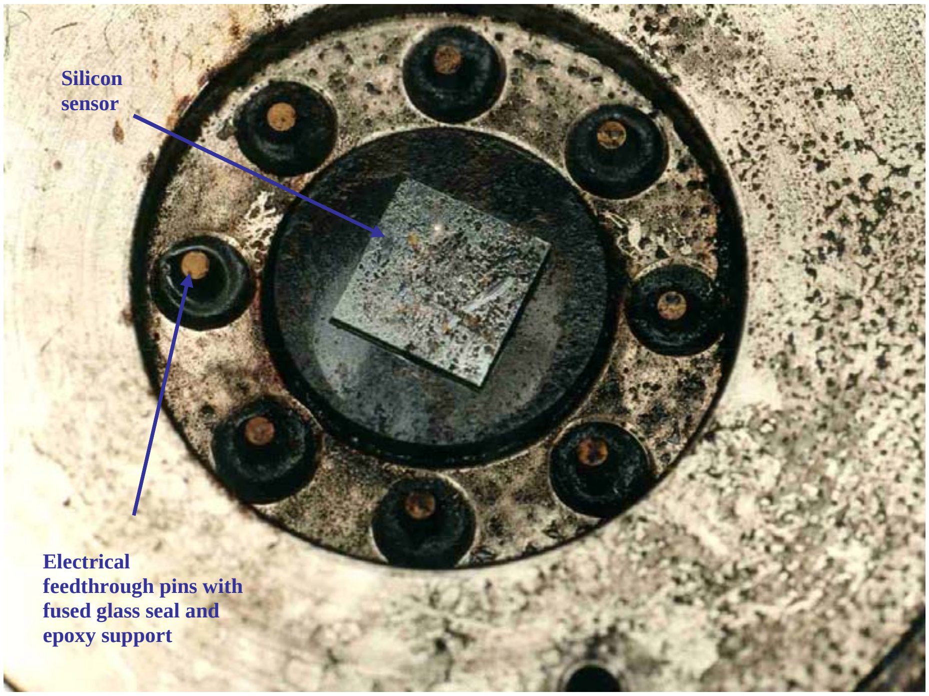

Figure 4: Failed Pressure Transducer Sensor

PRESSURE TRANSDUCER HISTORY

The pressure transducer is a Druck®[†] Model PDCR 130W/C, manufacturer's serial number 460553. The item was purchased in 1994 and first used in a helium system before being used in the NTO test system. The pressure transducers are checked for calibration on a 6-month cycle while in use. One in-place calibration performed in 1996 noted the calibration was out of tolerance per the manufacturer's specs. All other calibrations noted the pressure transducer was within specifications.

DRUCK PRESSURE TRANSDUCER DESIGN AND SPECIFICATION REVIEW

The Druck PDCR 130 pressure transducers utilize a silicon sensor that connects to the electronics through gold-plated feed-throughs that are sealed with fused glass and supported with epoxy. Very thin wires connect the sensor to the feed-throughs. For the basic PDCR 130 pressure transducer, the media pressure acts directly on the silicon sensor. An option, designated by "/W," includes an isolation diaphragm that acts as a barrier membrane and separates the media from the silicon sensor and is required for media that is conductive or not compatible with the sensor. Silicone oil fills the volume between the isolation diaphragm and the sensor and transmits the media pressure to the sensor.

The PDCR 130 Series specification, labeled as USPDCR 130 – 10/93, states that for the 900 to 1000 psi range PDCR/W pressure transducers, the range can be exceeded by 2 times (2,000 psi for the failed unit) with negligible calibration change. The material in contact with the pressure media is stated to be 316 stainless steel. The presence of silicone oil between the isolation diaphragm and the sensor is not mentioned in the specification. Discussions with the company indicated Druck can substitute Halocarbon 4.2 for the silicone oil. This option is not listed in the specification but can be specified during procurement.

It should be noted that other manufacturers supply pressure transducers that have the same basic design as the failed item. Based on a limited review of manufacturers, specifications for pressure transducers generally do not contain reference to the type of oil fill used. Pressure transducers that utilize a

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[†] Druck® is a registered trademark of Druck, Limited, Leicester, England.

silicon sensor quite often include an isolation diaphragm with an oil fill. Specifications for these transducers may state “silicon pressure sensor,” silicon technology,” or list the strain gauge type as “semi conductor.”

WSTF performed a brief study of the most commonly used pressure transducers on-site. The results are listed below.

1. Sensotec® [‡] Transducers – Several models have been researched but not all models have been evaluated.
    a. PPA, H472 models have similar construction to the failed Druck transducer (isolation diaphragm with silicone oil).
    b. STJE, TJE models have bonded foil strain gauge construction and pose no contamination issues.
2. Taber® [§] Transducers – Several models have been researched but not all models have been evaluated.
    a. 2201, 2404, 2415 models have bonded foil strain construction and pose no contamination issues.
3. Kulite® [**] Transducers – The manufacturer has indicated all of their transducers with isolation diaphragms are vacuum backfilled with silicone oil. Based on the type of construction, some may be acceptable for use in NTO systems.
    a. BME-1100 series have similar construction to the failed Druck transducer and are not recommended for use in these systems.
    b. HKM-375 has a flush metal diaphragm construction that may be acceptable for use.

The above list of transducers is not all inclusive and is the result of a brief effort to better understand the types of construction of some of WSTF’s most commonly used transducers.

TEST DATA ANALYSIS

The test data was analyzed to determine the pressure the transducer experienced during the NTO test. Due to the system dynamics, the pressure transducer was exposed to a “water hammer” effect. The failed pressure transducer was the only measurement of the test article inlet pressure. The data from the transducer was stored at a 10 kHz data rate (sampled every 100 μsec). The high-speed data time is recorded without a time stamp and is simply an elapsed time with a window of 150 ms for each test cycle. Each test cycle consists of an 80-ms flow duration followed by a 960-ms delay before the next cycle begins, for a cycle frequency of 1.04 s.

The data from the pressure transducer, prior to the time when the transducer ceased to provide output data, was analyzed to determine the pressure that this portion of the system was subjected to. The data indicated that the dynamic pressure exceeded the 0 to 1,000 psia scaling, resulting in clipped data. Extrapolation of the leading and trailing edges of the trace was used to approximate the peak pressure at ~ 1380 psia. The pressure transducer was also noted to have ceased operation ~ 22 min. prior to the failure.

CHEMICAL ANALYSIS

The failed pressure transducer housing and mounting bracket were coated with a dark residue. The WSTF Chemistry and Metallurgical Lab analyzed the residue on these components and found silicone oil. Druck identified the silicone oil as poly(methylphenylsiloxane) (PMPS) DC550® [††] fluid. An oxidizer beaker test was performed with Dow PMPS DC510®,[††], which is similar to Dow PMPS DC550. The silicone oil reacted with the NTO and the formation of bubbles was noted. A brown residue from this beaker test was

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‡ Sensotec® is a registered trademark of Sensotec, Inc., Columbus, OH.
§ Taber® is a registered trademark of Taber Acquisition Corp., Tonawanda, NY.
** KS Kulite® is a registered trademark of Kulite Semiconductor Products, Inc., Leonia, NJ.
†† Dow Corning 550® and Dow Corning 510® are registered trademarks of Dow Corning Corp., Midland, MI.

analyzed by Fourier transform-infrared (FT-IR) microscopy and found to be 2,4-dinitrophenol, which is a shock-sensitive substance. The dark residue from the pressure transducer housing and support bracket also contained 2,4-dinitrophenol.

The NTO from the catch tank downstream of the test article during the pressure transducer failure was analyzed and the non-volatile residue (NVR) from this analysis was analyzed by FT-IR microscopy. The residue yielded a very close spectral match to picric acid (2,4,6-trinitrophenol), which is also very shock-sensitive. The concentration of this substance in the NTO sample was estimated to be 5 mg/L.

The inlet tube to the test article was flushed, and 0.2 mg of residue was obtained. The residue had a spectrum which was a good match with poly(dimethylsiloxane). This is basically the DC550 silicone oil without the phenyl groups. The oil that remained in the beaker after the oxidizer beaker test also matched with poly(dimethylsiloxane), indicating the loss of phenyl groups from the original oil.

WSTF uses the Druck pressure transducers, with the isolation diaphragm and silicone oil, in several other applications, including hydrazine and monomethylhydrazine (MMH). As a result, beaker tests were performed with the DC510 silicone oil in hydrazine and in MMH. No reaction was observed in either of these tests.

METALLURGICAL ANALYSIS

The pressure port housing and the recovered section of the isolation diaphragm were submitted for analysis of the fracture surfaces. The diaphragm exhibited evidence of fatigue fractures, as shown in Figure 5, propagating from multiple initiation sites. Transgranular, partial penetration, secondary fractures were also observed on the diaphragm surface adjacent to and aligned with the through-thickness circumferential fracture, as shown in Figure 6.

The remainder of the diaphragm fracture surface was described as ductile overload rupture, as shown in Figure 7. No evidence was found that indicated the fatigue fractures were initiated by environmental degradation or a metallurgical discontinuity. X-ray microanalysis indicated the isolation diaphragm alloy was a molybdenum-bearing austenitic stainless steel, consistent with the manufacturer's specification stating 316 stainless steel. The results of microstructural analysis and microhardness testing indicated that the isolation diaphragm material is in the annealed condition.

The transducer housing fracture, which occurred at the weld fusion zone, was found to be a ductile overload failure resulting from a single load application, based on analysis of the pressure port end fracture surface, as shown in Figure 8.

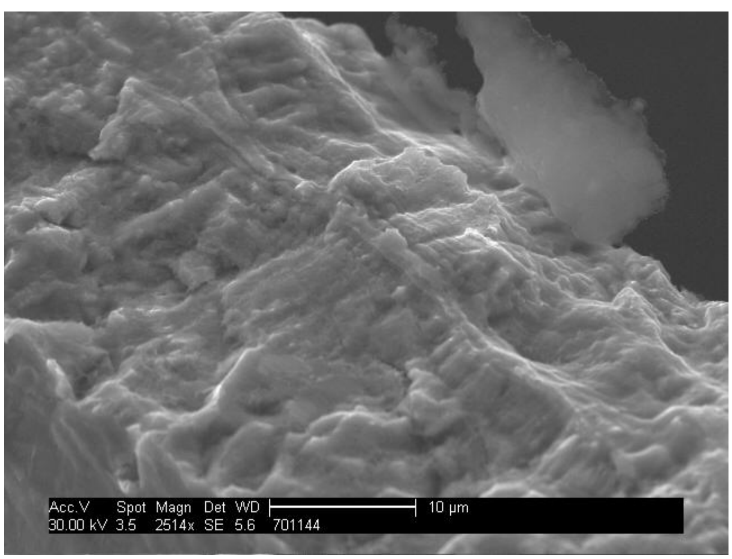

Figure 5. Scanning Electron Photomicrograph of Crack Arrest Marks and Fatigue Striations Observed On Circumferential Diaphragm Fracture

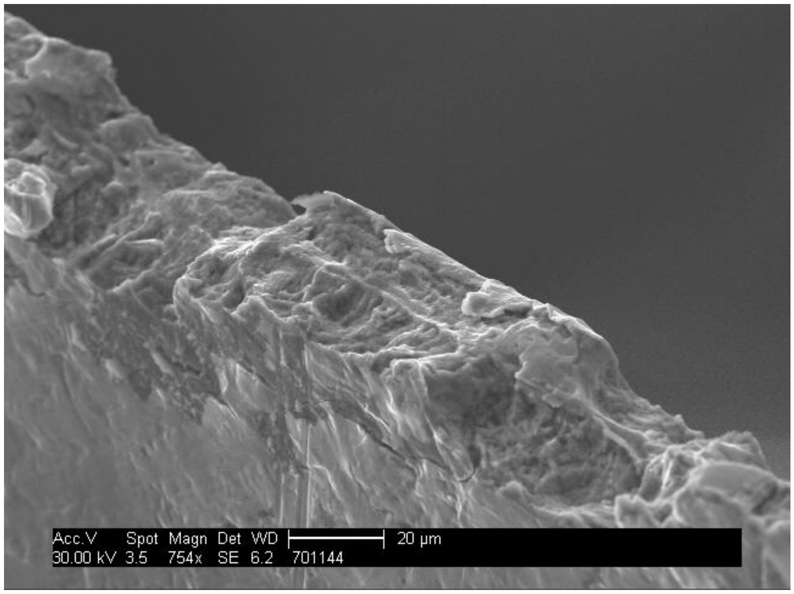

Figure 6. Scanning Electron Photomicrograph of Crack Arrest Marks Observed On Circumferential Diaphragm Fracture

(Note the transgranular, partial penetration, secondary fractures on the diaphragm surface adjacent to and aligned with the through-thickness circumferential fracture.)

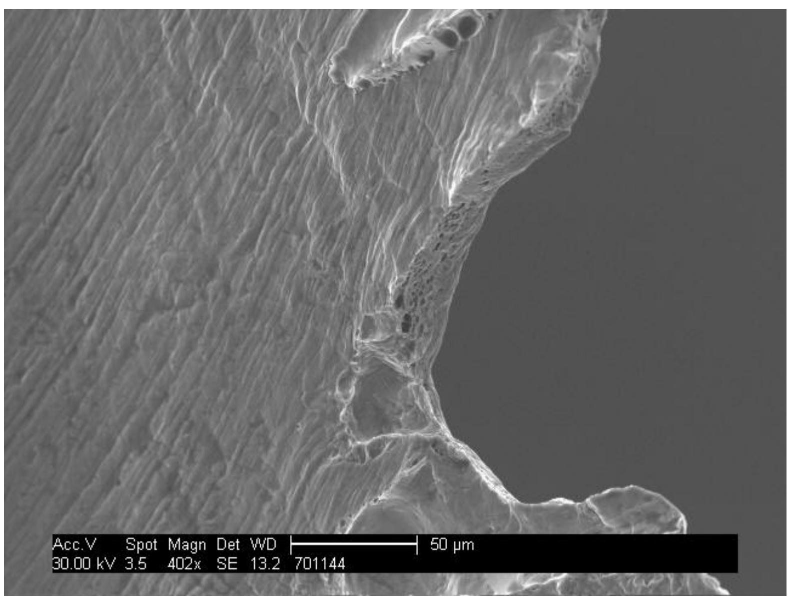

Figure 7: Scanning Electron Photomicrograph of Elongated Dimples Observed on Circumferential Diaphragm Fracture at EBW Root, Indicative of Ductile Behavior

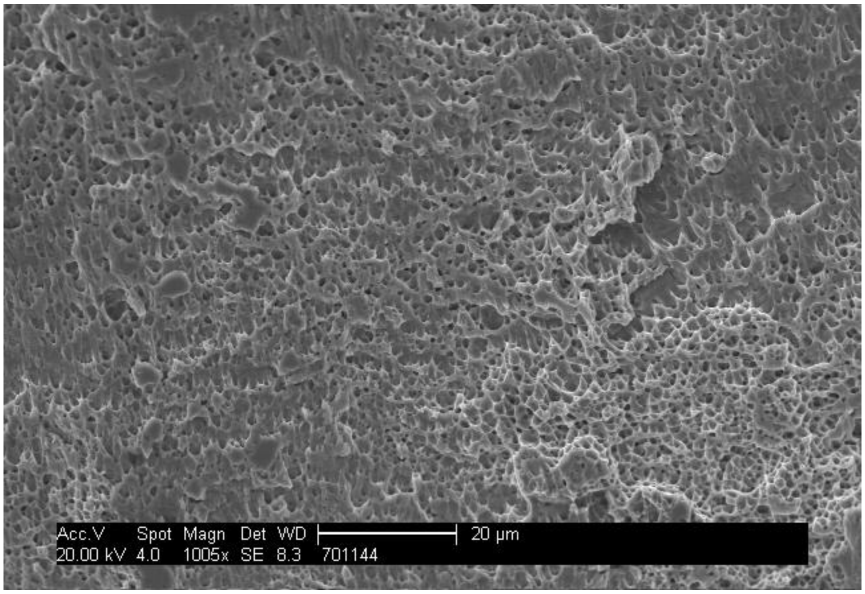

Figure 8: Scanning Electron Photomicrograph Showing Elongated Dimples on Circumferential Pressure Port End Fracture Surface, Indicative of Ductile-Type Overload Rupture

SUMMARY AND CONCLUSIONS

The pressure transducer failure would require an internal pressure of ~ 12,700 psi based on failing the housing weld. If the pressure increased slowly from a chemical reaction in the volume of silicone oil between the isolation diaphragm and the silicon sensor, the isolation diaphragm would first deflect and block the pressure port passage, allowing the pressure to build, but would then burst at ~ 4,000 psi and vent the pressure prior to failing the housing. If the pressure transducer failed due to severe “water hammer” in the test article inlet line, the 3/4-in. tubing would have yielded at ~ 3,400 psi, but no evidence of yielding was detected. The failure appears to be the result of high pressure in the housing with such a rapid pressure increase that venting through the inlet port did not prevent over-pressurization. Based on fatigue fractures in the diaphragm, diaphragm failure did occur prior to the single load application that burst the housing. The formation of shock-sensitive nitrophenol from a reaction between the NTO and silicone oil would be anticipated if the isolation diaphragm fails. The rapid pressure fluctuations resulting in a pressure spike that rises from ~ 300 psi to ~ 1,380 and back to ~ 300 psi in 2.5 ms would appear to be a likely cause of detonating the nitrophenol.

The cause of the pressure transducer output ceasing ~ 22 minutes prior to the failure is not certain, due to the damage that occurred to the hardware. The thin wires between the silicon sensor and the feed-through posts could have been broken or shorted from contact with the isolation diaphragm.

The following failure sequence appears reasonable based on analysis and information obtained during the investigation.

1. The dynamic response of the test article inlet pressure fluctuations to the pressure transducer isolation diaphragm, coupled with the silicone oil in the cavity behind the diaphragm, results in increased diaphragm deflection and stress.

2. One or more fatigue fracture(s) propagate through the 0.002-in. thick 316 stainless steel isolation diaphragm.

3. Silicone oil seeps through the crack into the NTO, and NTO seeps through the crack into the silicone oil cavity.

4. The silicone oil reacts with the NTO, and nitrophenol begins to accumulate as the reaction product. Some of the nitrophenol forms and remains between the isolation diaphragm and the sensor, while the rest on the system side of the isolation diaphragm dissolves in the NTO and circulates into the test system.
5. The diaphragm shorts or breaks the thin wires connecting the silicon sensor to the posts.
6. Impact from the test article inlet pressure cycle detonates the nitrophenol that has formed between the isolation diaphragm and the silicon sensor. (Nitrophenol becomes increasingly impact-sensitive as it dehydrates or becomes a powder. The effect of the various concentrations of nitrophenol in silicone oil and NTO on impact sensitivity was not explored in this investigation. The presence of nitrophenol and the detonation-type failure were viewed as adequate evidence of the cause and failure.)
7. The detonation results in pressure that first ruptures the isolation diaphragm into the pressure port and then bursts the pressure transducer at the housing weld connection.
8. The thrust from the gas release after burst drives the pressure transducer inlet port and electronics housings in opposite directions, with the inlet port housing severely bending the ¼-in. connecting line and then failing the line at the maximum moment location close to the ¾-in. manifold.
9. NTO from the test article inlet side of the test system ejects from the opening left where the ¼-in. line was torn free, releasing the NTO into the test cell.

Pressure transducers that are constructed with isolation diaphragms and a media non-compatible liquid fill are fairly common, produced by several manufacturers, and used in aerospace applications. These transducers may be used in numerous systems at other NASA locations and elsewhere. The lack of consistent, clear, objective information in the manufacturer's specifications regarding the details of construction of this design pressure transducer, including the presence of silicone oil between the isolation diaphragm and the sensor, makes the identification of the potential hazard difficult.

Test systems containing NTO, oxygen, and hydrogen peroxide should be evaluated to determine the type of pressure transducers used. (The silicone oil would not form a shock sensitive substance in oxygen but is considered incompatible in oxygen.) The use of transducers constructed with an isolation diaphragm with silicone oil in these systems should be discontinued. These transducers may include many of the transducers supplied by various manufacturers. Specifications that state "silicon pressure sensor," "silicon technology," or list the strain gauge type as "semi conductor" are probably this design. This design of pressure transducer should be ordered with oil that is compatible with the media. Druck has stated that the oil fill requirement should be stated on the purchase order (PO). Druck also stated the PO could require factory etching of the type of oil used on the transducer case to ease future identification.

Other systems containing isolation diaphragm transducers should be evaluated as well, and the diaphragm should be considered as a single point failure. The analysis should include consideration of media compatibility with silicone oil as well as system or hardware tolerance for silicone oil as a contaminant.

Emphasis on the need to evaluate components for single fault tolerance should be a priority when dealing with hazardous systems. Due to sometimes limited information in component specification, this will often require discussions with the manufacturer or distributor to fully understand the product.

A Government-Industry Data Exchange Program (GIDEP) Alert for pressure transducers that are constructed with isolation diaphragms and a media non-compatible liquid fill has been issued (Appendix A).

GOVERNMENT - INDUSTRY DATA EXCHANGE PROGRAM

SAFE-ALERT

1. TITLE *(Class, Function, Type, etc.)* **Druck PDCR 130/W/C Pressure Transducer (silicone oil filled)**		2. DOCUMENT NUMBER **QB-SA-03-01** 3. DATE *(DD-MMM-YY)* **07-May-03**
4. MANUFACTURER AND ADDRESS **Druck Incorporated** **4 Dunham Drive** **New Fairfield, CT 06812**	5. PART NUMBER **PDCR 130/W/C**	6. NATIONAL STOCK NUMBER
	7. SPECIFICATION	8. TYPE DESIGNATOR
	9. LOT DATE CODE START	10. LOT DATE CODE END
11. MANUFACTURER'S POINT OF CONTACT (POC) **Gary Werner, Vice President – Aerospace**	12. CAGE	13. MANUFACTURER'S FAX **(203) 746-2494**
14. MFR. POC PHONE **(203) 746-0400, X303**	15. MANUFACTURER'S E-MAIL **usa.sales@druck.com**	
16. CROSS REFERENCE VENDOR	17. CROSS REFERENCE CAGE	18. CROSS REFERENCE PART

19. PROBLEM DESCRIPTION / DISCUSSION / EFFECT

The NASA White Sands Test Facility (WSTF) experienced a failure of the referenced pressure transducer during a test operation. The 0-1,000 psia pressure transducer was used to measure a nominal pressure of 276 psia, but the location was upstream of a pressure-operated valve that resulted in a dynamic pressure spike that reached an estimated 1,380 psia for a time duration above 1,000 psia of ~1 ms. The pressure transducer is rated by Druck for 2 times the transducer range with negligible calibration change. The pressure transducer was being used above the span but the failure demonstrates an inherent concern that is the topic of this Alert.

The pressure transducer failed catastrophically during service in nitrogen tetroxide (NTO) resulting in release of NTO and damage to a remote test system. The pressure transducer design utilizes a silicon strain gauge diaphragm with the sensor isolated from the media by a 0.05 mm (.002 inch) thick 316 stainless steel isolation diaphragm. The media pressure acts on the diaphragm and is transferred through the oil to the silicon sensor. The ~0.8 mL volume between the isolation diaphragm and the silicon sensor is filled with silicone oil. Metallurgical examination revealed that the isolation diaphragm had failed due to cyclic fatigue close to the electron beam weld fusion zone around the circumference. The crack allowed NTO to migrate into the volume with silicone oil and silicone oil to migrate into the NTO media. Silicone oil and NTO react forming 2,4-dinitrophenol and 2,4,6-trinitrophenol residue, both of which are shock sensitive. Metallurgical analysis revealed that the failure in the housing weld was a ductile overload failure resulting from a single load application. The shock sensitive residue apparently detonated resulting in pressure failure of the pressure transducer at the weld fusion zone. The burst pressure, based on longitudinal stress at the weld, was calculated to be 12,700 psi. In addition to the catastrophic failure of the pressure transducer, the ~48 gallons of NTO remaining in the test system were contaminated with trinitrophenol.

The presence of silicone oil in the volume behind the isolation diaphragm is not mentioned in Druck's PDCR 130 Series specification (USPCDR130 – 10/93) downloaded from the Druck web site on April 8, 2003. The specification also does not mention that Druck will substitute

Halocarbon 4.2 oil for the silicone oil during manufacturing, if specified during procurement. Other brands of pressure transducers utilize a similar design with an isolation diaphragm and silicone oil. A brief review of various manufacturer's specifications indicates that some of the manufacturers clearly identify the presence of silicone oil behind the isolation diaphragm and alternative oils that can be used and some do not mention the presence of silicone oil.			
20. ACTION TAKEN/PLANNED **1. Discontinue use of pressure transducers that utilize an isolation diaphragm with silicone oil in WSTF NTO systems and systems which interface NTO systems. This includes many of the transducers supplied by Druck as well as other manufacturers. Specifications that state "silicon pressure sensor", "silicon technology", or list the strain gage type as "semi conductor" are probably this design. This design of pressure transducer should be ordered with oil that is compatible with the media. Druck has stated that the oil fill requirement should be stated on the Purchase Order (PO). Druck also stated that the PO could require factory etching of the type of oil used on the transducer case to ease future identification.** **2. Evaluate use of pressure transducers that utilize an isolation diaphragm with silicone oil in WSTF systems, such as oxygen, hydrazine, MMH, etc, based on a hazards analysis. The isolation diaphragm should be considered as a single point failure and the analysis should include consideration of media compatibility with silicone oil as well as system or hardware tolerance for silicone oil as a contaminant. (Initial review indicates that silicone oil filled pressure transducers are not suitable for oxygen systems.)** **3. Emphasize the need to evaluate components for single fault tolerance. Due to sometimes limited information in component specification, this will often require discussions with the manufacturer or distributor to fully understand the product.**			
21. DATE MFR. NOTIFIED **May 12, 2003**	22. MANUFACTURER'S RESPONSE [X] REPLY ATTACHED [ ] NO REPLY	23. ORIGINATOR ADDRESS/POINT OF CONTACT **John Anderson, 201LD Phone: (505) 524-5693** **NASA-JSC-WSTF** **12600 NASA Road** **Las Cruces, NM 88012**	
24. GIDEP REPRESENTATIVE **Donald Hall**		25. SIGNATURE	26. DATE **May 12, 2003**