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Title: Gas Generation of Heated PBX 9502

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Gas Generation of Heated PBX 9502

Progress Report

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Abstract

Uniaxially pressed samples of PBX 9502 were heated until self-ignition (cookoff) in order to collect pressure and temperature data relevant for model development. Samples were sealed inside a small gas-tight vessel, but were mechanically unconfined. Long-duration static pressure rise, as well as dynamic pressure rise during the cookoff event, were recorded. Time-lapse photography of the sample was used to measure the thermal expansion of the sample as a function of time and temperature. High-speed videography qualitatively characterized the mechanical behavior and failure mechanisms at the time of cookoff. These results provide valuable input to modeling efforts, in order to improve the ability to predict pressure output during cookoff as well as the effect of pressure on time-to-ignition.

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1. Introduction

PBX 9502, subjected to sufficiently high temperatures, can undergo thermal runaway and self-ignition; this is known as "cookoff". The detailed cookoff behavior of PBX 9502 is of particular relevance to weapon safety scenarios. Prior work has explored in detail the temperatures, location, and time-to-cookoff, resulting in mature modeling capabilities. Among other findings, this existing body of research has shown that the cookoff behavior exhibits a strong pressure dependence. PBX 9502 contained in a gas-tight vessel with no free volume cooks off at an earlier time than a sealed vessel with moderate free volume, which cooks off at an earlier time than a vented vessel. The quantity of gas generated will determine the pressure of the enclosing vessel, and subsequently affect the cookoff time.

To date, we have limited ability to predict this gas generation. The primary purpose of the experiments described herein is to provide data to inform model development, thereby advancing the predictive capability to account for pressure rise resulting from gas generation.

Additionally, the pressure rise is of vital importance for the consideration of vessel design: at what point along the path to cookoff will a gas-tight containment vessel enclosing heated PBX 9502 fail? We wish to know the pressure rise due to the PBX 9502 in order to predict the time at which the vessel will fail.

2. Motivation

The Intermediate Scale Bucket experiments performed by M-6 in 2012 demonstrated that the time-to-cookoff is dependent on pressure[1]. Experiments were performed with three different pressure configurations: vented, sealed, and with an ullage volume. Time-to-cookoff was shortest for the sealed configuration, longest for the vented configuration.

Permeability testing of PBX 9502 was performed by M-6 personnel in 2014[2]. The permeability of pristine samples was compared to that of thermally cycled samples (thermally cycled samples were soaked at 250˚C for 2.5 hours then returned to room temperature). The results showed that thermally cycled samples had significantly greater permeability than pristine samples; however, the permeability of both pristine and damaged remained extremely small—orders of magnitude lower than believed necessary to contribute strongly to cookoff behavior[3,4]. Note that the permeability of PBX 9502 has not been measured while it is held at an elevated temperature near to that where cookoff occurs. The practical obstacle to such a measurement is that there does not exist a suitable polymer sealing material that remains both mechanically viable and sufficiently impermeable (relative to the explosive that is) at the temperature of interest (which is in the vicinity of 300˚C). Furthermore, it is unclear whether such a measurement would have any meaning: the sample is mechanically confined in order to perform the measurement, and the mechanical confinement itself will significantly affect the outcome at elevated temperatures (owing to thermal expansion, permitting the opening of porosity).

The impermeable nature of PBX 9502 is difficult to reconcile with the pressure-dependence demonstrated in the Bucket Tests. The location of ignition in the Bucket Test experiments was at the center of the explosive sample. In order for the pressure rise in the gas space surrounding the explosive to have an effect on the time-to-ignition, one would expect that the pressure must be communicated to the ignition location at the center of the sample. However, if the explosive is impermeable, how is the pressure of the gas space surrounding the sample communicated to the center of the sample? Gas transport can only occur with sufficient permeability.

Additionally, gas must be generated by the explosive in order for the pressure to rise in the surrounding volume. However, if the explosive is impermeable to gas transport, from whence is the gas generated? Potentially, the entirety of the generated gas arises from a surface "skin" of exposed explosive. Alternatively, the sample—though initially impermeable—may develop permeability during the thermal trajectory; this would result from mechanical and/or morphological changes (either sparse fractures or intricately connected porosity) occurring from the thermal damage. If the sample becomes sufficiently permeable, then gas generated throughout the volume of the sample can be transported to the exterior volume, thus contributing to the pressure rise.

It can be helpful to identify two independent pressures of interest: the "ballast pressure" that we measure in the volume external to the explosive, and an internal "pore pressure" resulting from a pocket of solid reacting into gas within the interior of the explosive sample. If the material is sufficiently permeable, then the gas generated interior to the sample is transported quickly enough into the ballast volume for the ballast pressure and the internal pore pressure to equalize. If the explosive is impermeable, then the ballast pressure and the pore pressure remain separate. If the pore pressure grows large enough relative to the external pressure, the strength of the material will be exceeded and the sample will fracture into pieces.

The pressure that is measured during an experiment is the ballast pressure. As mentioned, this pressure may result from two sources: if the material is impermeable, the gas generation may result solely from decomposition of the surface "skin" of the explosive. If the material is sufficiently permeable, the gas may arise from a percentage of the entire bulk of IHE decomposing and being transported to the free volume.

The experiments reported here were designed to address these related issues of permeability and pressure-dependence, with the specific goal of providing additional data required to improve efforts to model the location and time-to-ignition of cookoff.

3. Experiment Design

3.1. Apparatus

The apparatus is a "cookoff oven"—a gas-tight copper vessel with visual access through opposing sapphire windows. The oven is designed to create the most uniform possible heating of the sample, through radiant transfer from the surrounding copper walls and sapphire windows. The sample is placed atop a pedestal of either PEEK (ultra high-temperature plastic) or ceramic which contacts the sample in only four spots to minimize surface area, thereby minimizing conductive heat pathways.

Visual access to the sample allows us to qualitatively characterize the mechanical failure of the sample due to thermal damage. If the sample cracks into pieces as a result of internal pore pressure exceeding the material strength, we can witness at what temperature that occurs, and what sort of fragmentation occurs.

The design permits the use of an optional pressure relief device, in order to mitigate the potential damage to the oven and pressure transducer. The dynamic pressure rise that occurs with cookoff is likely to exceed the pressure capability of a sensitive pressure transducer (which is intended to measure the slow, quasi-static pressure build-up). The transducer must be protected by a pressure relief device in order to perform the quantity of experiments required for a thorough analysis.

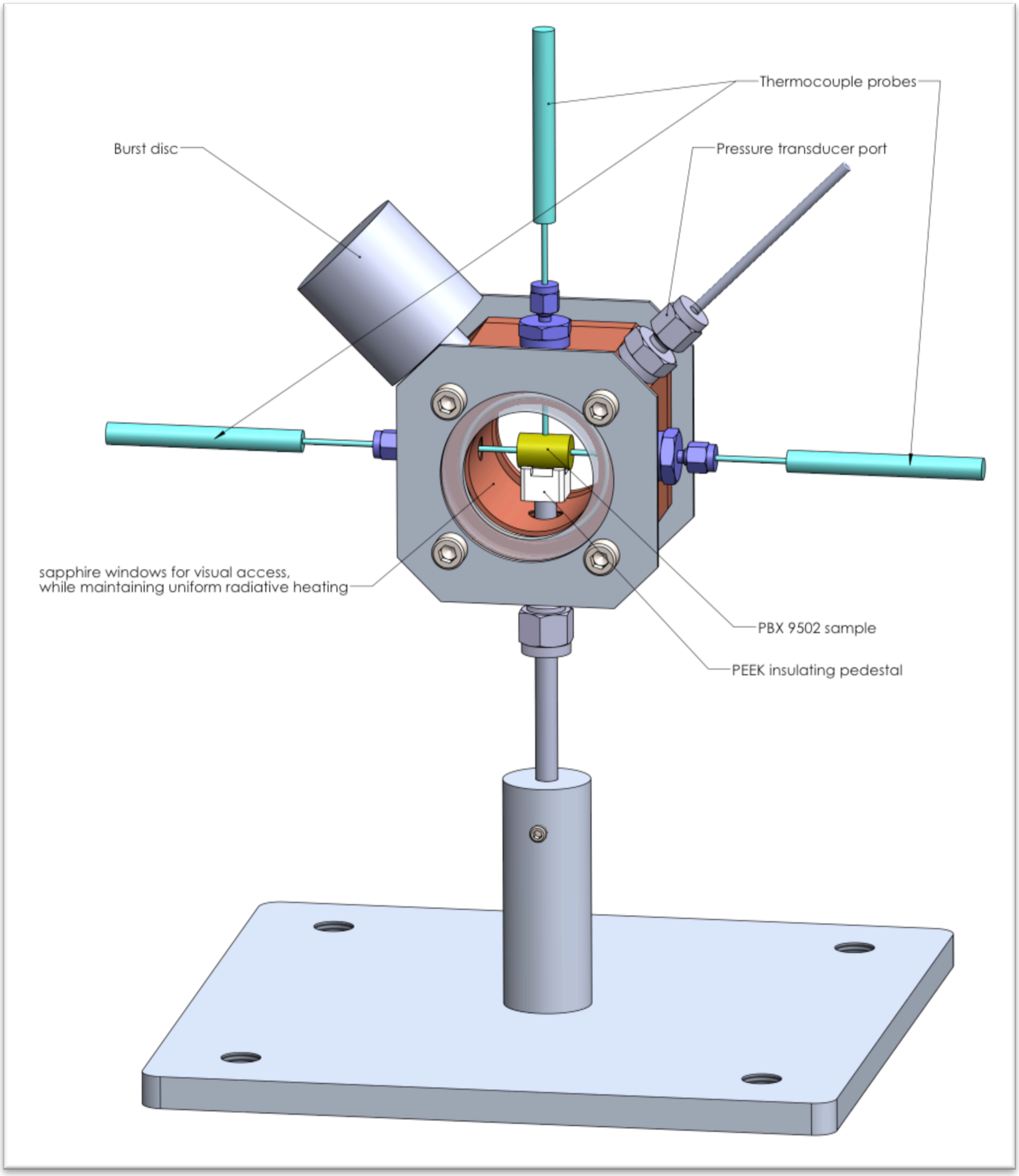


Figure 1. Gas generation experiment diagram.

3.2. Heating

Heating was accomplished with a fiberglass "rope" heater around the exterior of the copper oven. The heater was 50 W/ft, 96 inches long, for a total of 400W, powered by 120VAC, 3.3A, McMaster-Carr part number 3641K26. Numerous layers of fiberglass fabric strip were then wrapped over the heater for insulation.

Heater power is controlled via a custom-designed box which incorporates a Omega brand temperature controller, model CN2301-DC1-DC2-DC3, which utilizes a PID algorithm. The controller was tuned using a mock assembly; Table 1 contains the final PID values used for all experiments.

Table 1. PID tuning values for temperature controller

Proportional band	3.3% (of 0-300°C)
Integral Time	58 sec
Derivative Time	14 sec

3.3. Diagnostics

3.3.1. Pressure

The oven is gas-tight and is fitted with a single pressure transducer capable of simultaneous static and dynamic pressure measurements. The transducer was Omega model PX1009L0-100AV with a calibrated range of 0-100 psia (Figure 30). The Omega transducer was serial number 608837 with a scaling of .3026 mV/psi (Figure 31). The transducer is a full-bridge strain-gauge design, capable of exposure to temperatures up to 400°C.

Static, long duration pressure is measured at a rate of 1Hz using a NI 9237 module mounted in a cDAQ-9188 chassis. A custom-designed National Instruments VI running on a laptop served to record long duration temperature and pressure data simultaneously.

Dynamic pressure rise during the explosive event was recorded using a Tektronix DPO-4104B-L oscilloscope, at a capture rate of 500kS/s, 5M samples, for a duration of 10 seconds. The NI 9237 module mentioned above provides an excitation voltage of 5V to the pressure transducer bridge circuit; the scope monitors the same voltage across the bridge as the NI DAQ. A filtering capacitor of 0.01 μF and a resistor of 15 kΩ in parallel, and a resistor of 15 kΩ in series, were connected at the oscilloscope input in order to reduce noise coupling. Additionally, the oscilloscope was powered through an isolation transformer in order to isolate the signal from noise carried on the ground conductor. Triggering for the dynamic pressure event was accomplished using a microphone connected to a StopShot™ brand microphone trigger box.

3.3.2. Temperature

The exterior surface temperature of the explosive sample is measured in three places with probe-style thermocouples. The thermocouple used is Omega model TJFT72-K-SS-116G-6-SMPW-M; this style probe uses a smaller wire diameter at the bead, in order to best reflect the temperature of the sample without undue effect from heat conducted down the probe from the vessel wall. When viewing the oven as in Figure 2, TC2 is on the left, TC3 on the top, TC4 on the right. Three external thermocouples are taped to the outside wall of the copper vessel. The heater control TC and TC1 are taped to the top copper surface of the oven, as close together as possible, in a location that is not directly underneath the heating tape (Figure 5). TC5 is also taped to the top surface of the oven, but in a location that falls underneath the heating tape. A final thermocouple (TC6) is taped to the tubing next to the pressure transducer in order to monitor the gas temperature in the plumbing.

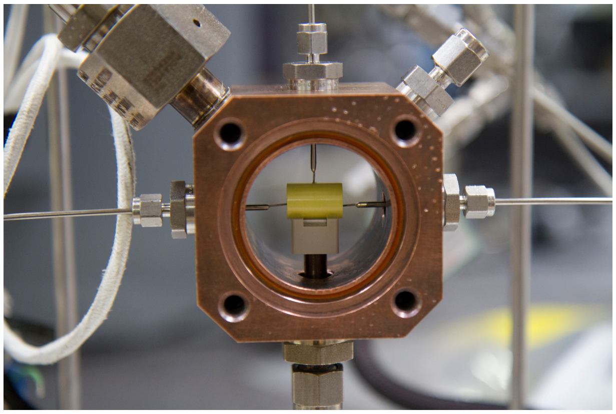


Figure 2. View of assembled design with no windows/heaters/insulation.

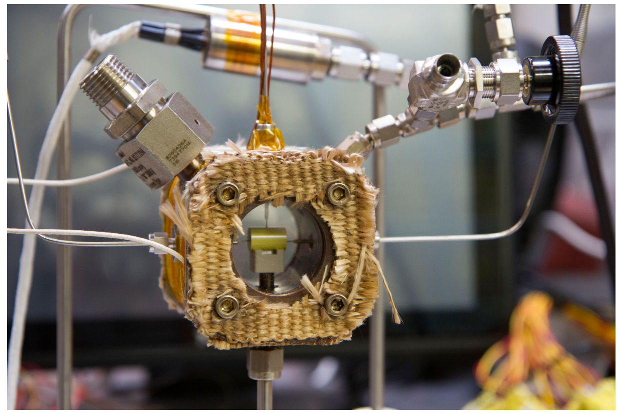


Figure 3. View of assembled design showing insulation around window.

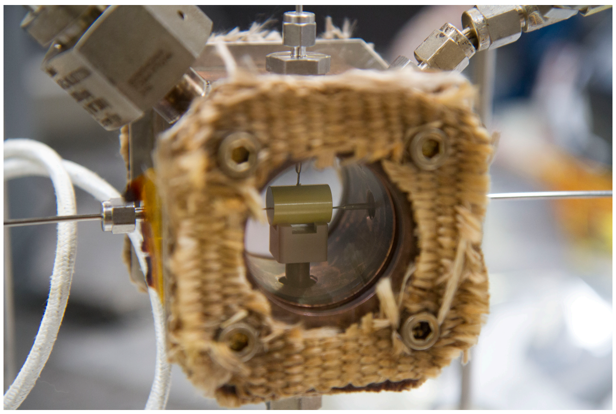


Figure 4. View of assembled design showing sample resting on pedestal.

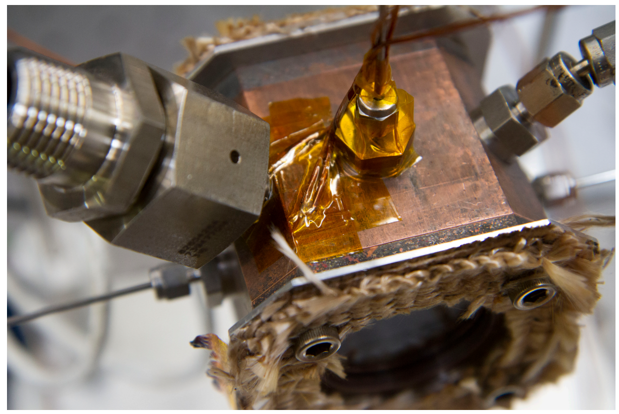


Figure 5. View of assembled design showing location of thermocouples taped to top of oven surface.

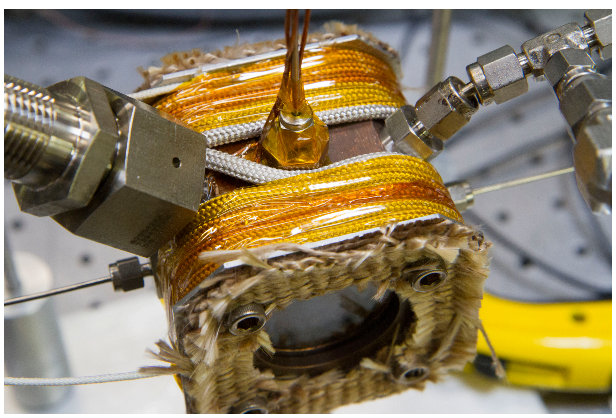


Figure 6. View of oven design showing wrapped heater tape.

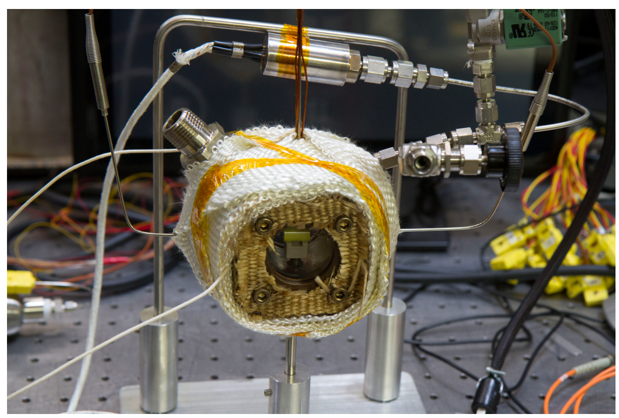


Figure 7. View of assembled design showing insulated apparatus ready for testing.

Temperature was recorded at a rate of 1Hz using an NI 9214 module mounted in a cDAQ-9188 chassis. A custom-designed National Instruments VI running on a laptop served to record long duration temperature and pressure data simultaneously.

3.3.3. Time-lapse Photography

A DSLR camera images one side of the sample. It is rigged with an intervalometer in order to record a single still photograph every 5-30 seconds (varied test-to-test). The resulting time-lapse video can be analyzed in order to measure the thermal expansion of the sample, as a function of time and temperature. A Canon™ 7D DSLR was paired with a Canon™ 200-400 f4 lens at an aperture of f22, zoomed fully to 400mm, ISO 100, shutter speed of 1/3 s. Both EF12 and EF25 extension tubes were used (stacked) to sufficiently reduce the focusing distance.

3.3.4. High-speed Videography

High-speed video was captured using a Phantom™ brand camera, model "Miro", through the sapphire window opposite the DSLR. A capture rate of 1000fps was achieved with an exposure of 990μs at a resolution of 768 x576 using a Canon™ 180mm fixed focal length macro lens, with a 2x teleconverter, at an aperture of f7, for a total capture duration of 9.3s. The high-speed videography revealed aspects of macro-cracking, smoke generation, and burning when cook-off occurred.

4. Experiment

In order to eliminate humidity from the air, the apparatus was purged with dessicated compressed air prior to testing. Testing was performed with the internal gas pressure of the oven at the ambient pressure. The internal volume of the oven was measured by filling with ethanol and weighing the unit before and after filling.

4.1. Explosive

All explosive samples were the Insensitive High Explosive (IHE) PBX 9502, a two-component plastic bonded explosive consisting of 95% TATB and 5% Kel-F 800. All PBX 9502 was pressed from virgin Lot 008 prills.

Two different samples were studied, which differed in pressing orientation (see Figure 8). A billet 4in diameter x 0.7874in thick was uniaxially pressed, and samples 10mm diameter x 15mm length were machined out of this billet in two orientations, "axial" and "orthogonal". Density was 1.89g/cc ±0.008g/cc. TATB crystals are platelet shaped, and when uniaxially pressed will preferentially orient parallel to the pressing anvil. The resulting PBX is morphologically anisotropic. The "axial" orientation is machined such that the cylindrical axis of the sample was parallel to the uniaxial pressing direction. The "orthogonal" orientation is machined with the cylindrical axis normal to the pressing direction.

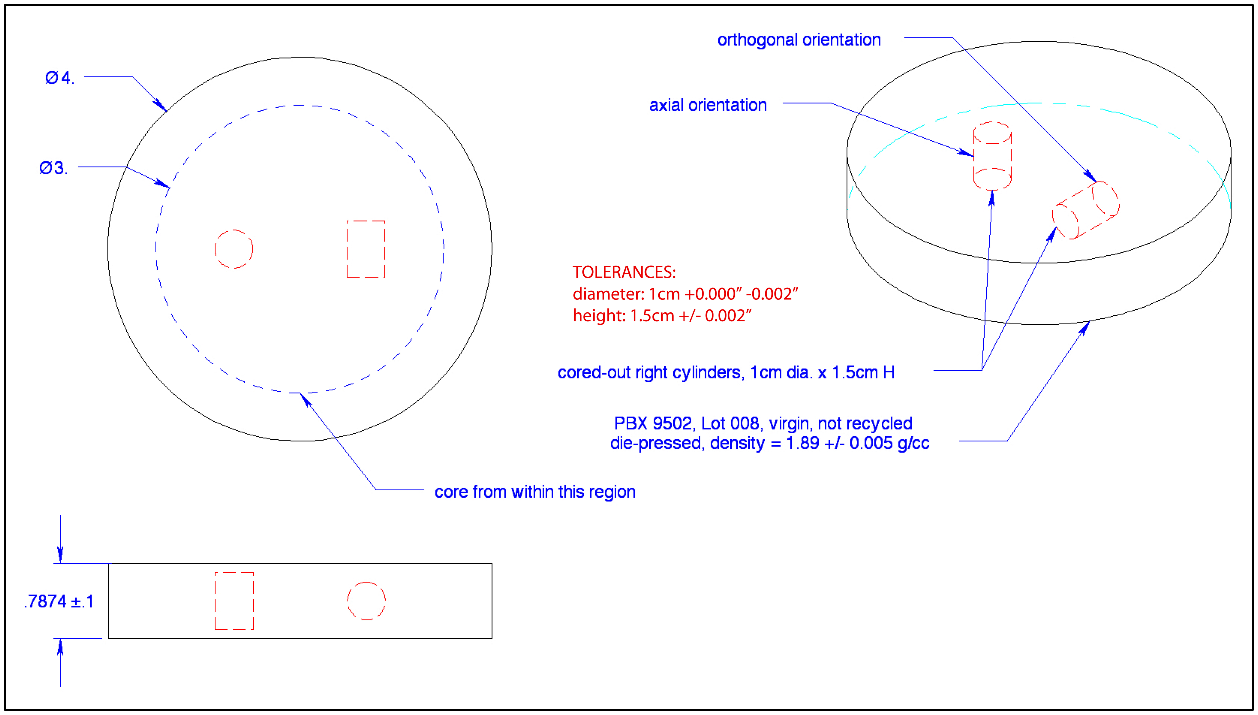


Figure 8. Samples were machined out of a uniaxially pressed billet in the two orientations shown here.

5. Results

5.1. Test Record

Test #	Date	Sample Diameter (mm)	Sample Length (mm)	Sample Mass (g)	Sample Volume (cc)	Sample Density (g/cc)	Pressing Orientation	Burst Disc Pressure Relief (psi)	Setpoint Temp #1	Control TC (under heater)	Heater surface temp	Steady sample temp resulting from setpoint #1	Thermal Profile description	Time to cookoff (min from start)
1	1/13/16	9.98	15	2.2	1.17339	1.875	Axial	50	290	290	269	260	multiple steps, from initial setpoint 290 to final setpoint 380	257
2	4/18/16	9.98	15	2.21	1.17339	1.883	Axial	150	350	359	335	323	one soak	38
3	5/12/16	9.99	15.02	2.22	1.17731	1.886	Axial	150	330	338	320	308	one soak	74.7
4	7/18/16	9.99	15	2.21	1.17574	1.880	Orthogonal	150	330	338	316	307	one soak	72
5	7/21/16	9.96	15.01	2.2	1.16947	1.881	Orthogonal	1500	330	331	305	301	one soak	106
baseline	7/19/16	N/A	N/A	N/A	N/A	N/A	N/A	150	330				one soak	

Figure 9. Test record, including temperatures and cookoff times.

5.2. Thermal Profiles & Static Pressure

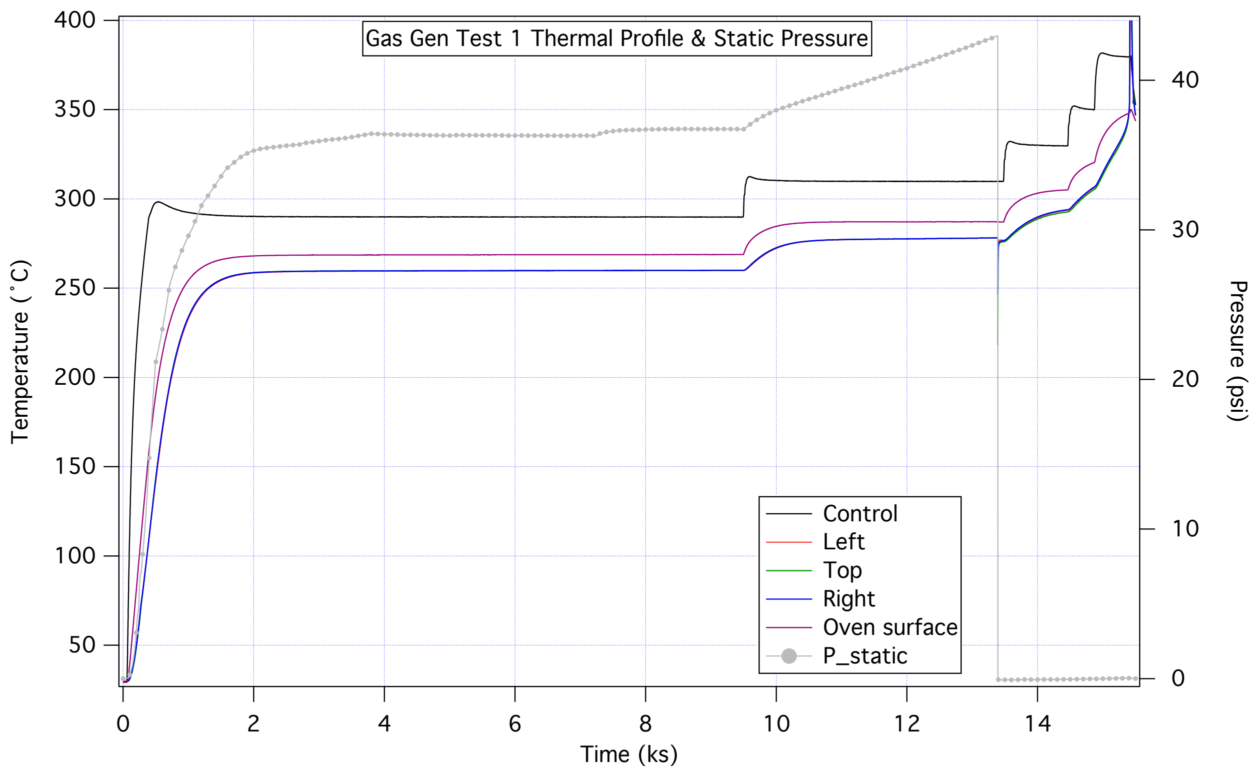


Figure 10. Test 1, graph of thermal profile & static pressure.

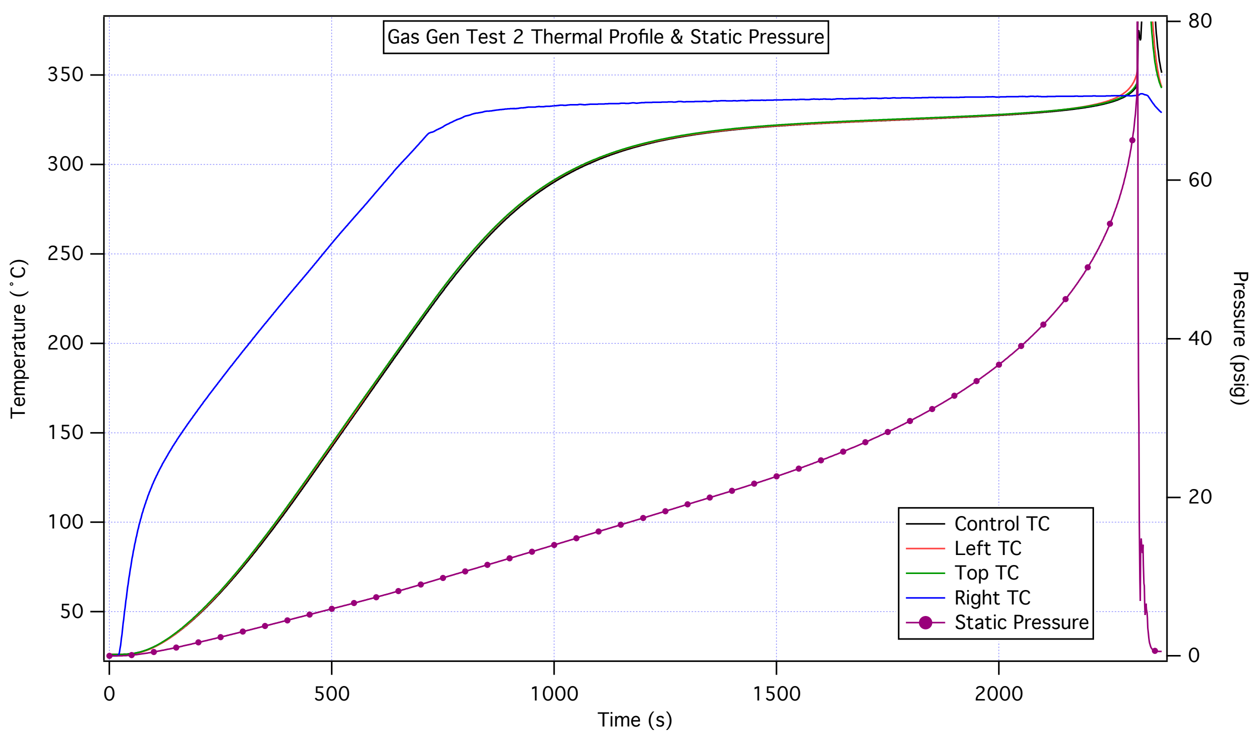


Figure 11. Test 2, graph of thermal profile and static pressure.

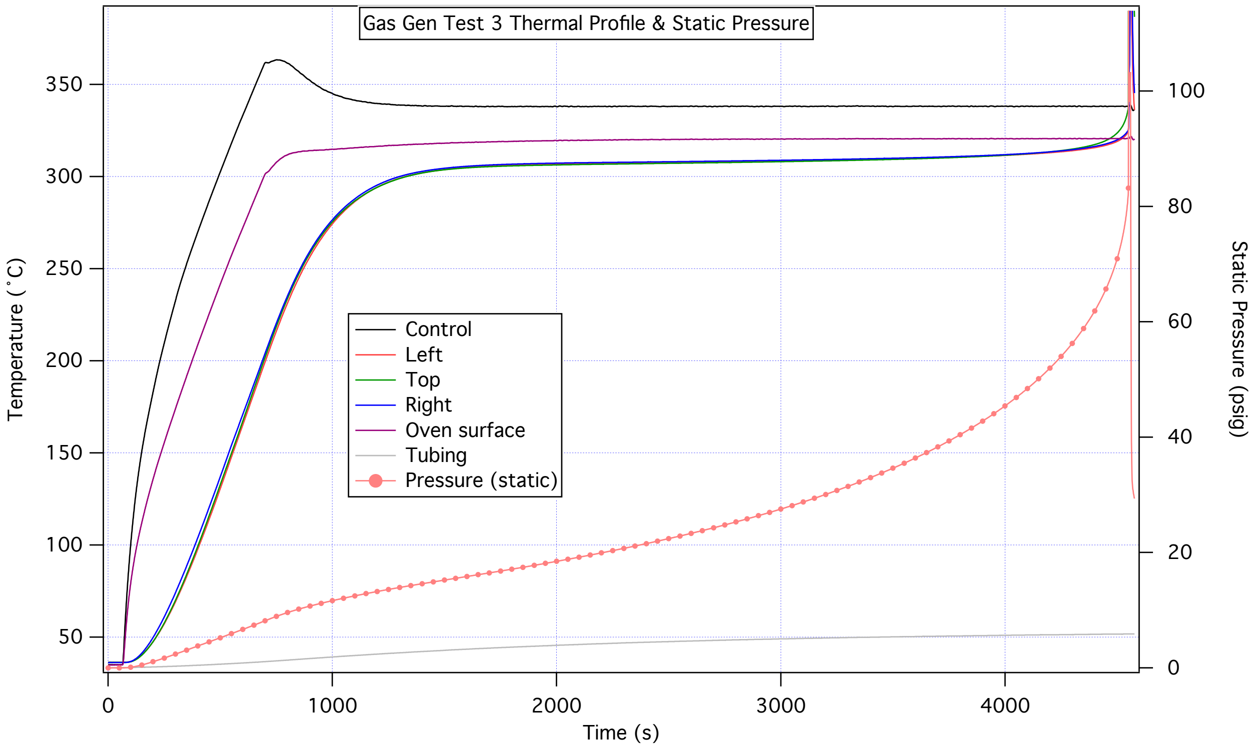


Figure 12. Test 3, graph of thermal profile and static pressure.

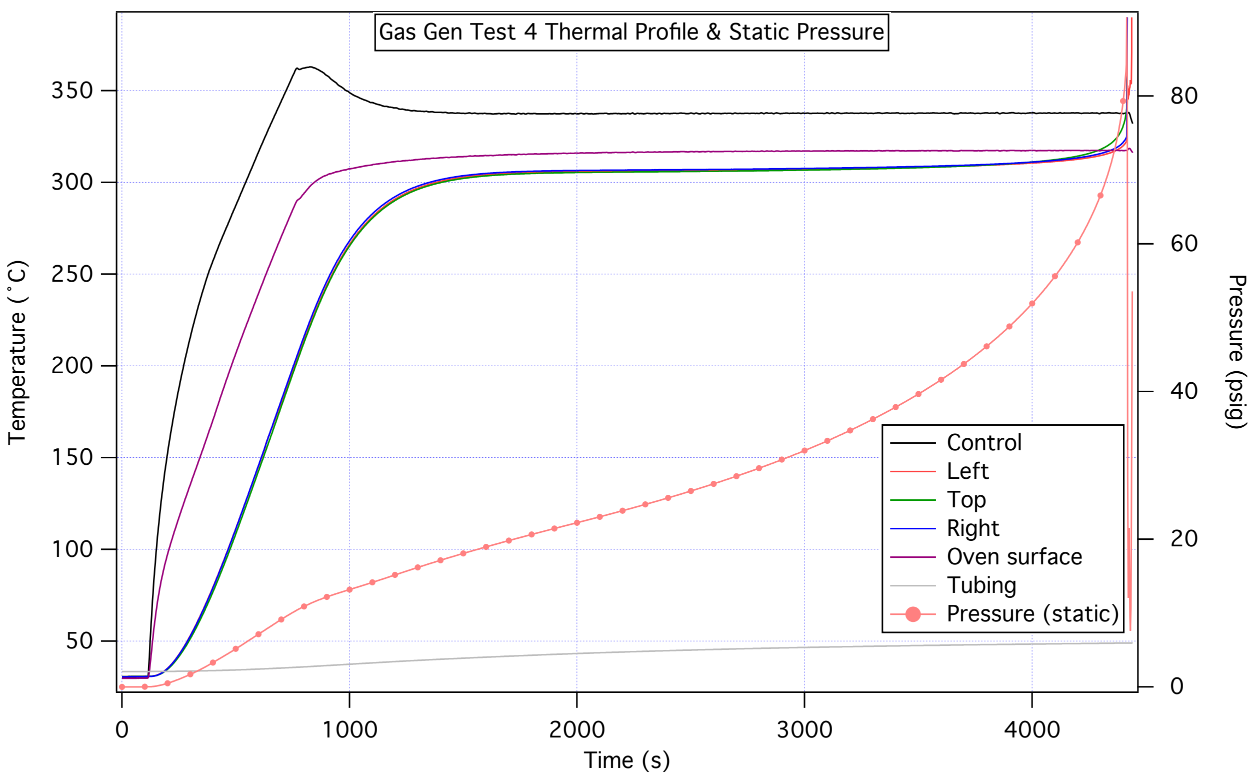


Figure 13. Test 4, graph of thermal profile and static pressure.

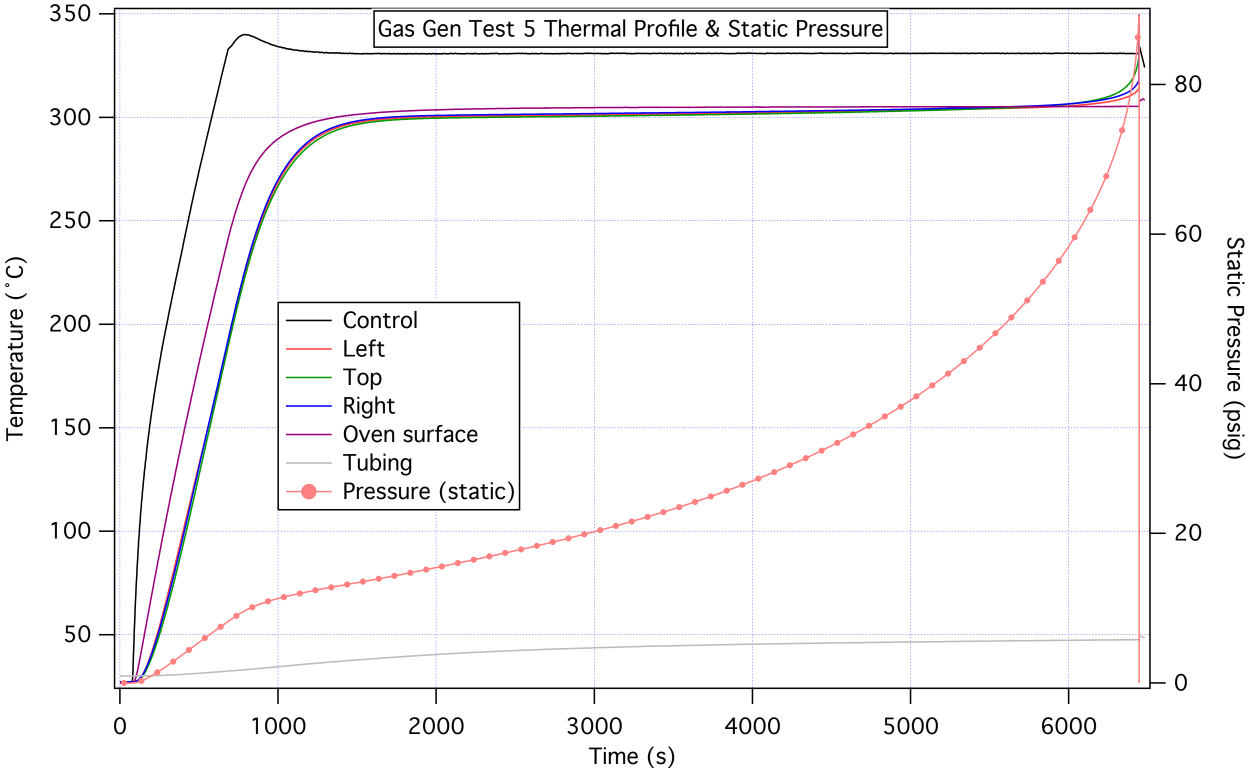


Figure 14. Test 5, graph of thermal profile and static pressure.

5.3. Dynamic Pressure

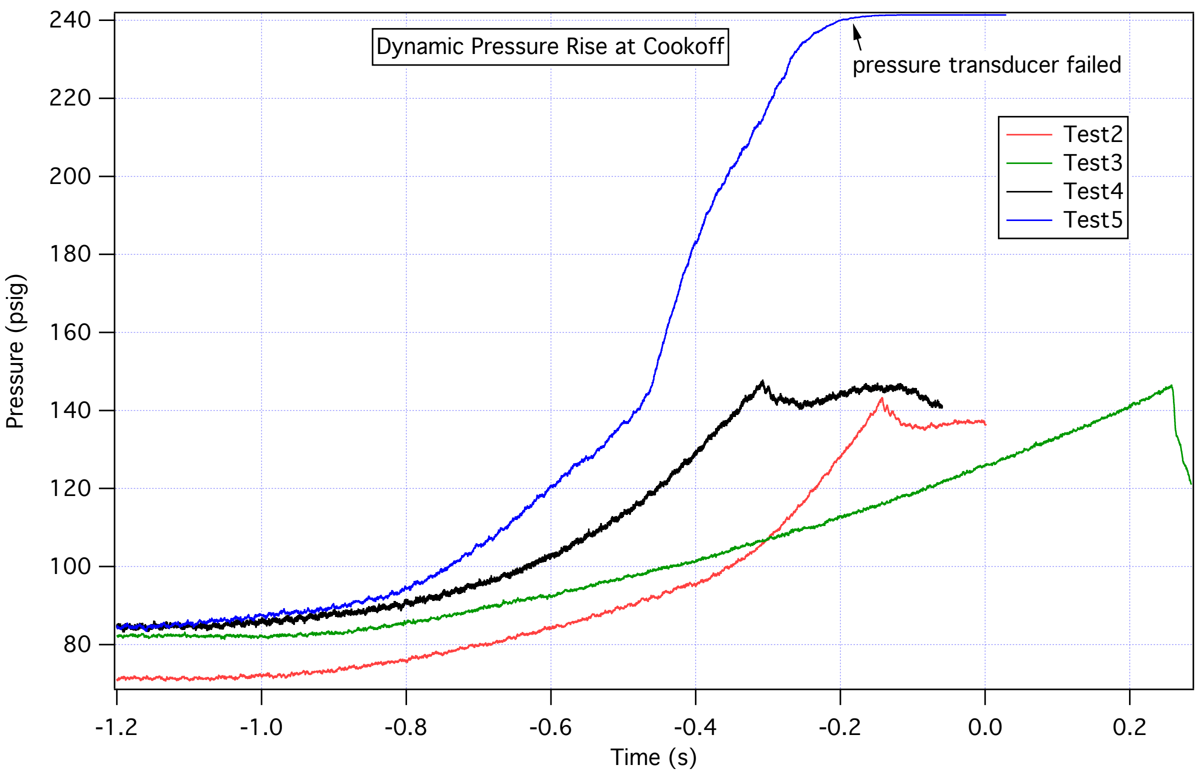


Figure 15. Dynamic pressure rise in Tests 2-5 where data was recorded.

5.4. Thermal Expansion

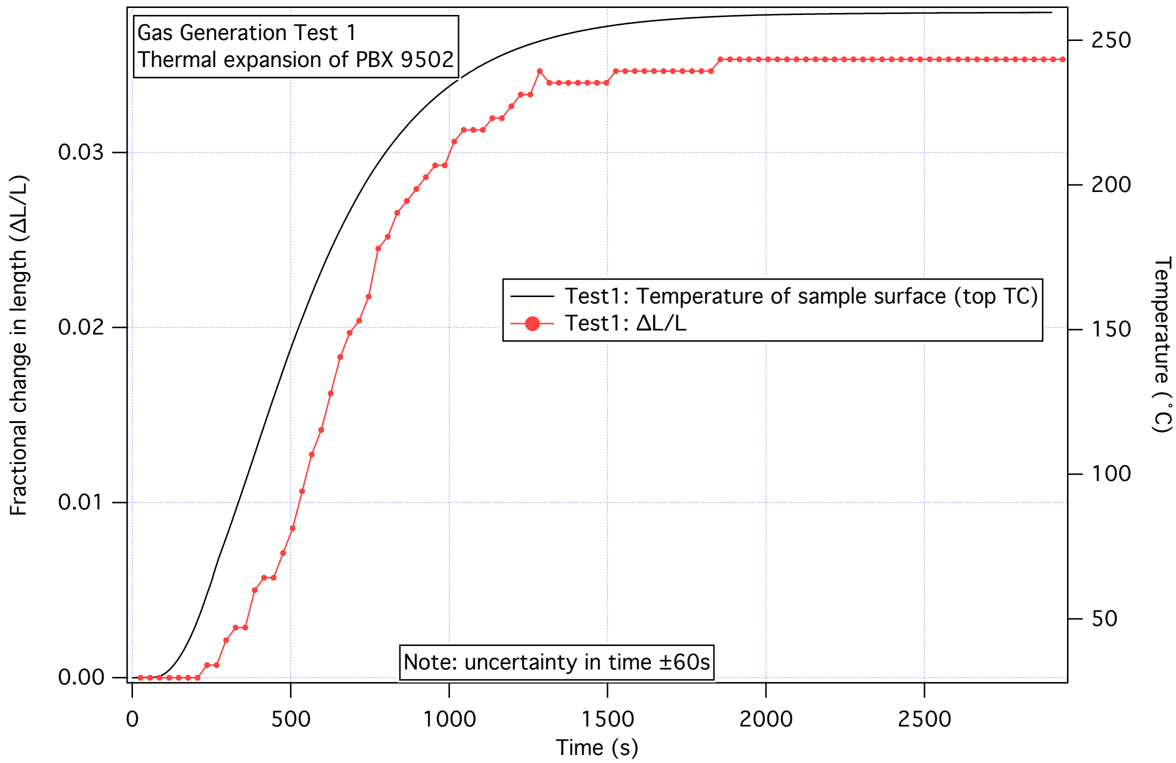


Figure 16. Test 1 thermal expansion.

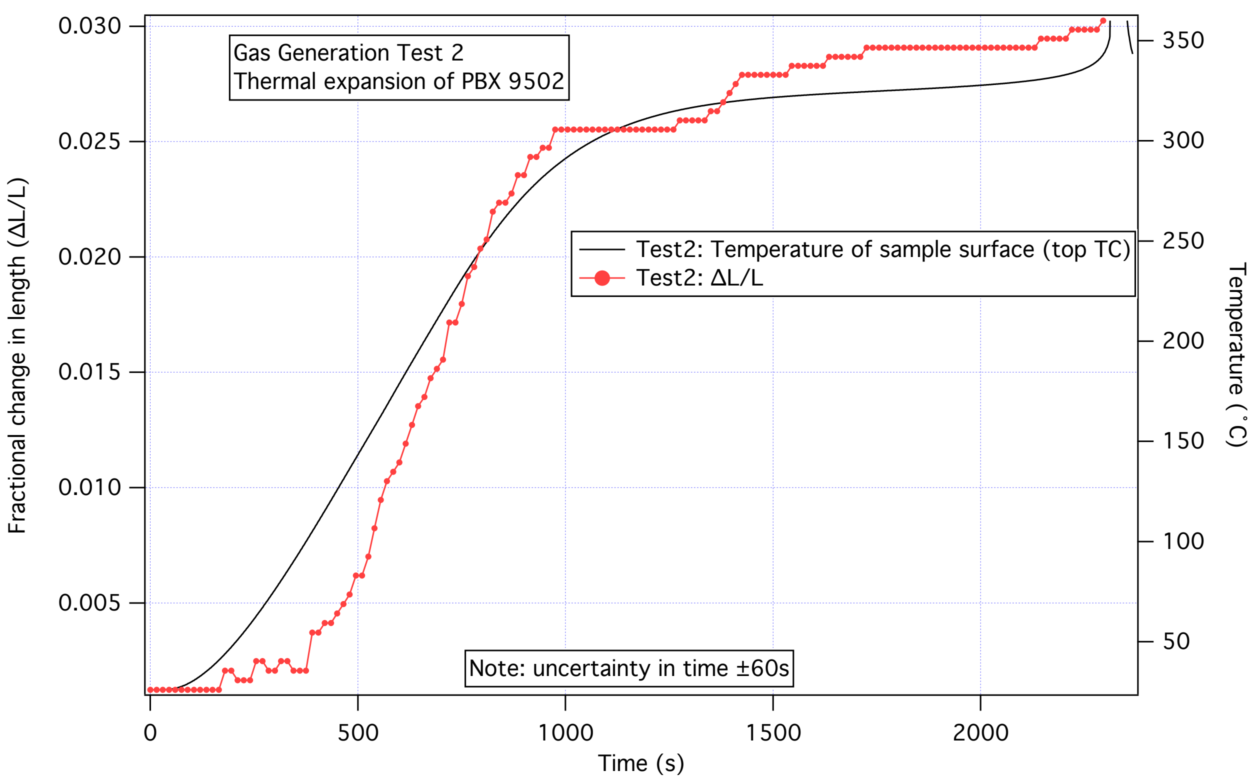


Figure 17. Test 2 thermal expansion.

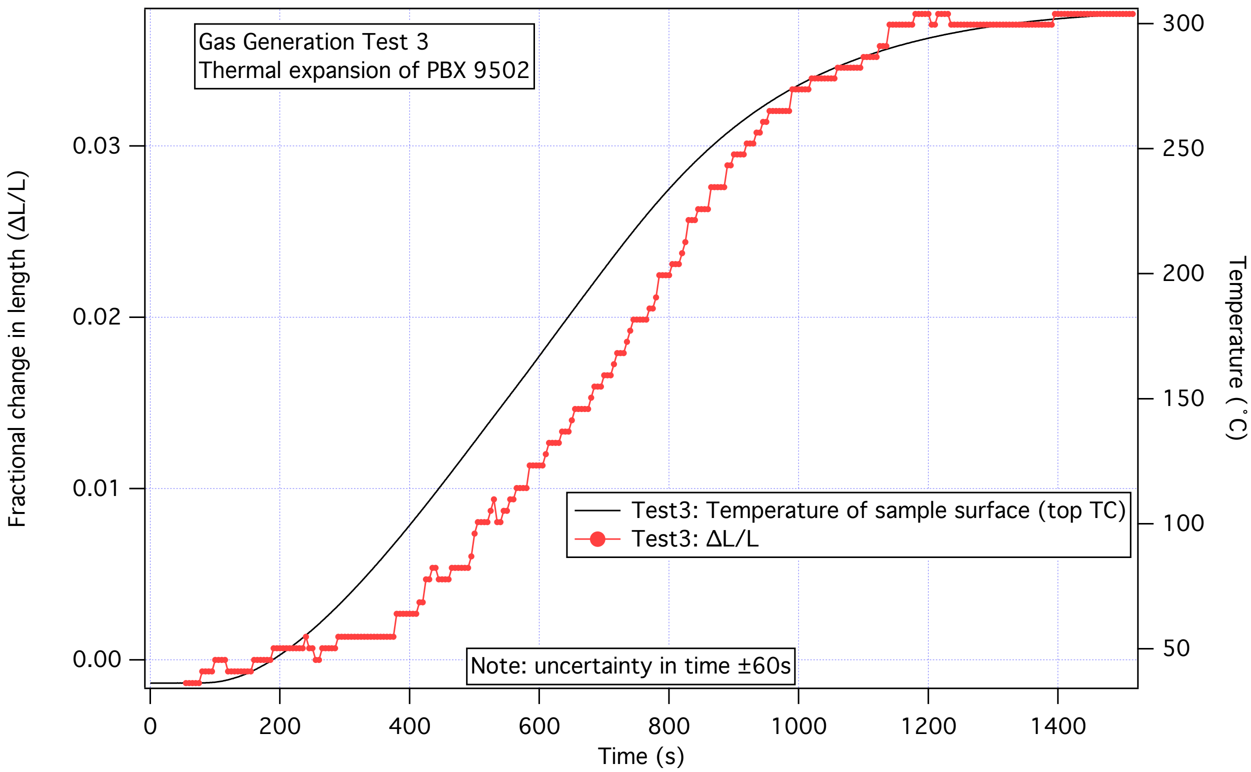


Figure 18. Test 3 thermal expansion.

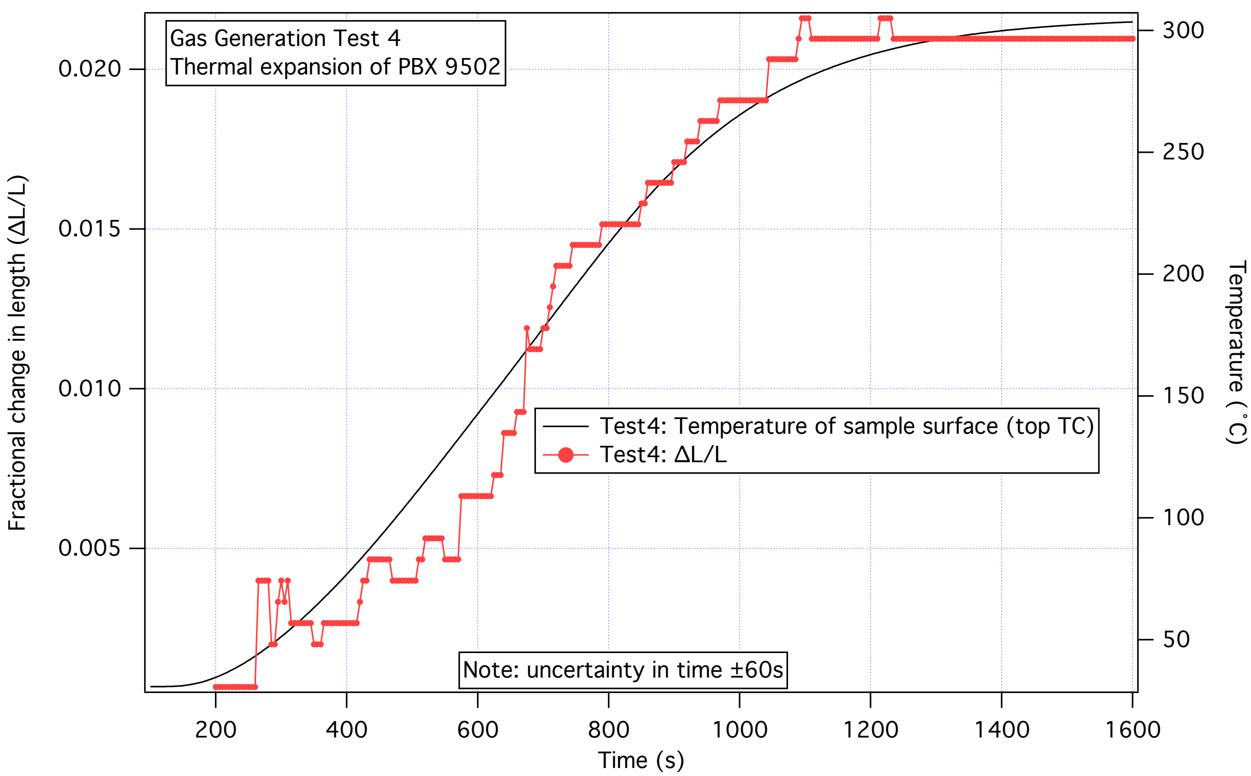


Figure 19. Test 4 thermal expansion.

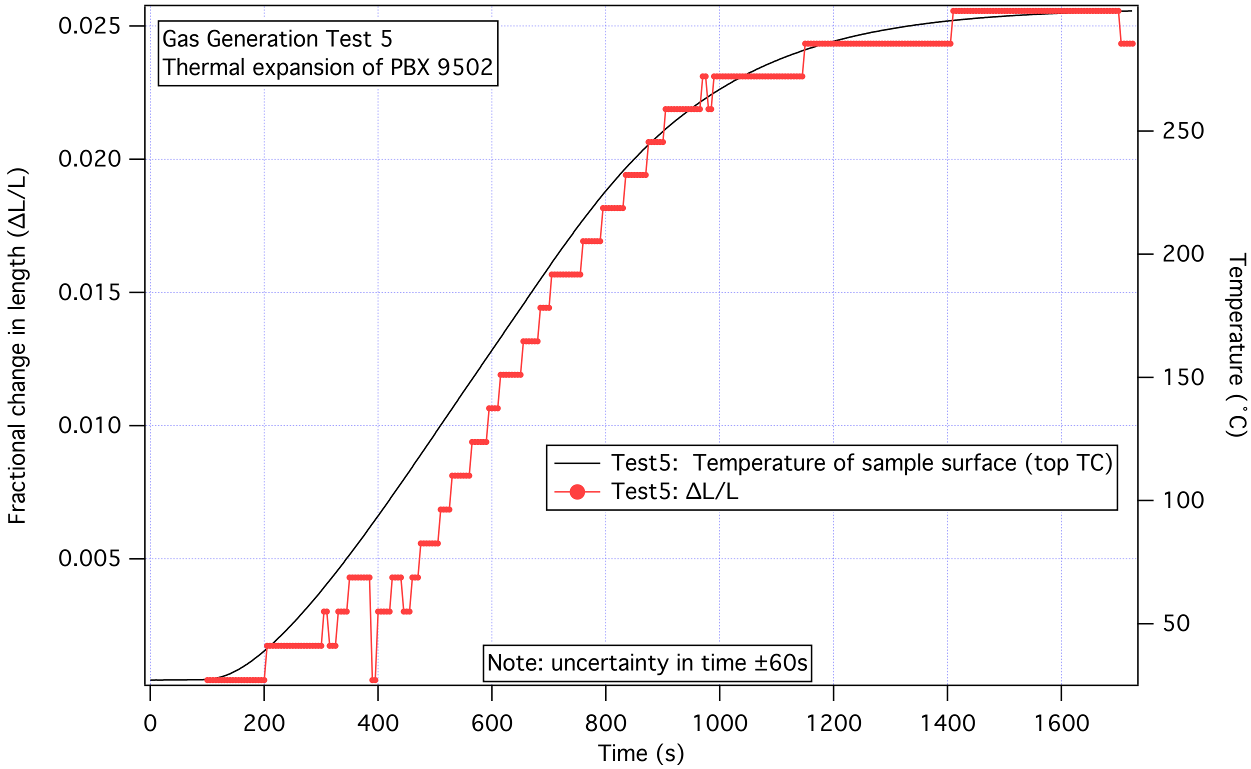


Figure 20. Test 5 thermal expansion.

5.5. Post-mortem Images

Figure 21. View of charred sample remnant from Test 2.

Figure 22. View of charred sample remnant from Test 3.

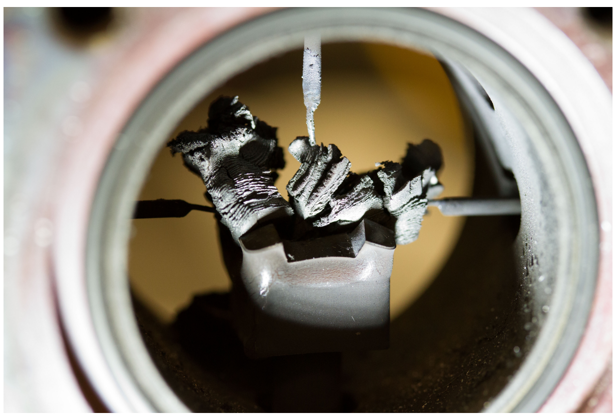


Figure 23. View of charred sample remnant from Test 4.

Figure 24. Detail view of sample remnant from Test 4.

Figure 25. Another view of sample remnant from Test 4.

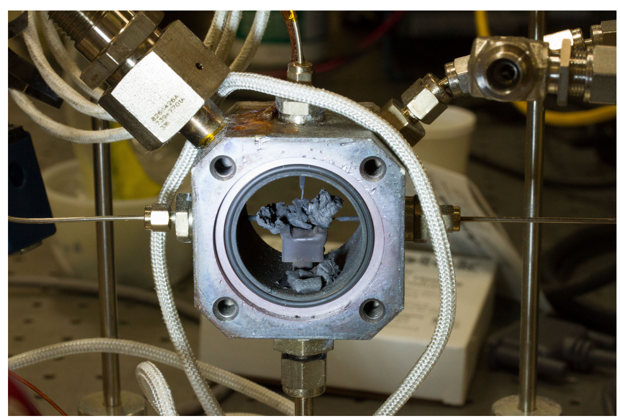


Figure 26. View of sample oven post-mortem Test 4.

5.6. Discussion

Test 1 was used as a scoping test to determine appropriate setpoints for cooking off. Note the multiple steps in the thermal profile: at each step, as the behavior levelled off with no signs of selfheating, the setpoint was raised. Note that cookoff did not occur until after the oven temperature had been raised to 380˚C and the sample surface temperature exceeded 340˚C (though admittedly the temperatures were not quite in equilibrium during the last few setpoint bumps). In contrast, heater setpoint temperatures in the Intermediate Scale Bucket test ranged from 250-280˚C[5], in the High Fidelity an oven temperature of 255˚C was used, and 175-253˚C in the Hockey Puck experiments[6]. The hotter oven temperature required for cookoff to occur in this test is a reminder that details including boundary temperature, sample size, and the thermal pathways permitting heat transport can have a large effect on cookoff behavior. Figure 9 tabulates the key temperatures and times for these cookoff tests.

All five tests behaved similarly on the photo record—including both the time lapse and the high-speed video captured at cookoff. The samples discolored slowly over the entire duration of the test, moving from the pristine bright yellow color into a putrid green and then charcoal black. Figure 27 shows selected still photos from the time-lapse sequence of Test 2, which is representative of all of the tests. No macro cracking occurred during the long-duration thermal damage (unlike, in contrast, a similar thermal damage treatment of PBX 9501).

Figure 27. Selected stills from the time-lapse sequence of Test 2, showing evolution of discoloration of the sample. Total duration of this sequence is 38 minutes.

The cookoff event itself begins with a single large crack opening, from whence a grayish white smoke is expelled. The smoke fills the interior of the oven, obscuring view of the sample. In Tests 1-4, no luminous flame accompanied the cookoff. Figure 28 shows stills from Test 3, which was typical of Tests 1-4. That is to say that the sample combusted, with rapid smoke production, without sufficiently exothermic reaction in the product gases to cause luminous flame. The combustion process is relatively long-lived—on the order of ~1s in duration.

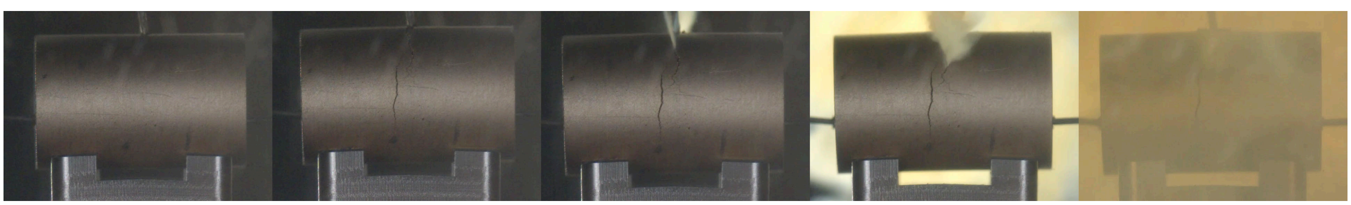
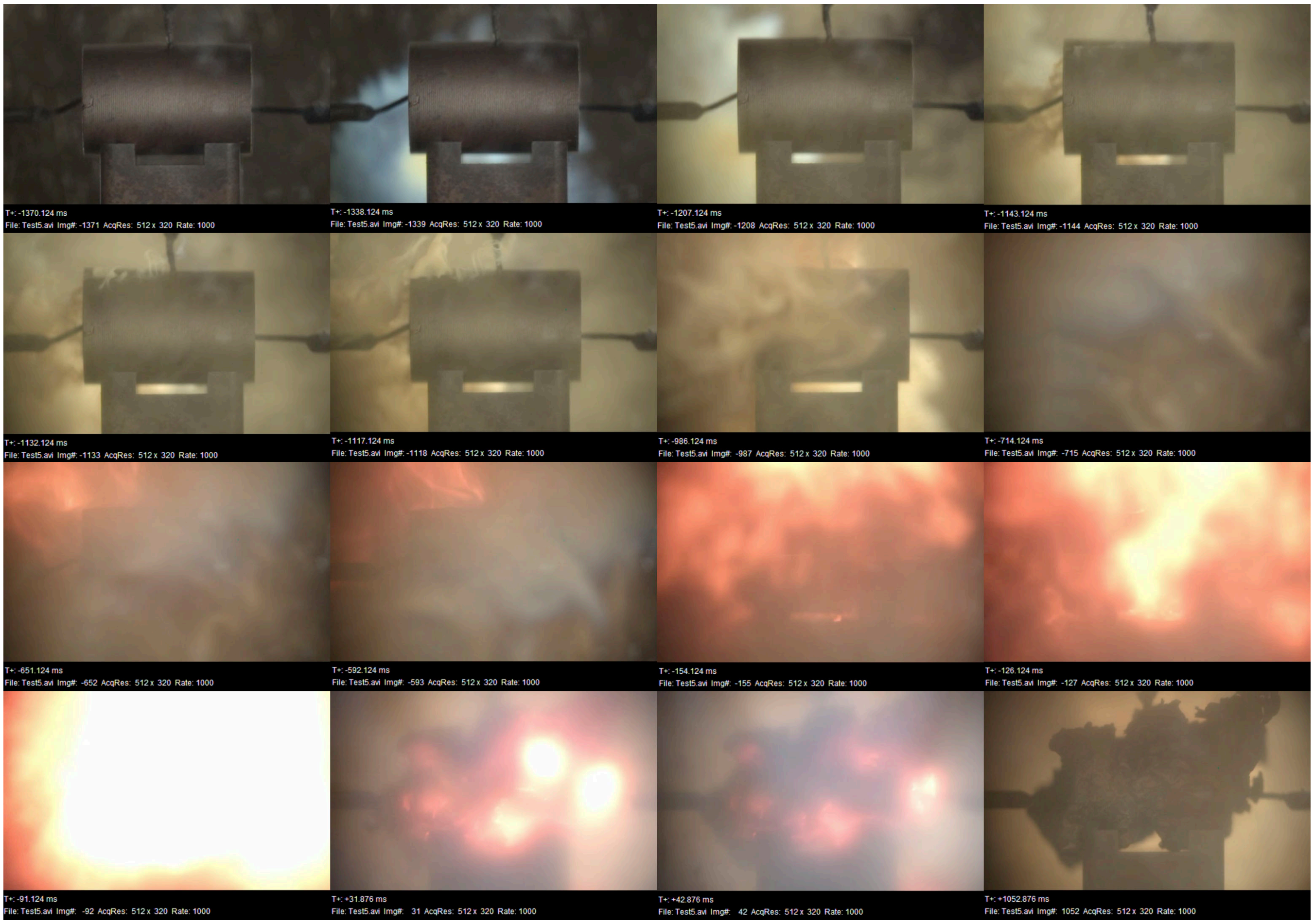


Figure 28. Sequence of selected stills from the high-speed video record, showing the formation of a crack in the second frame, which begines spewing smoke in the third frame. Total duration of this sequence is ~1 second.

Figure 15 shows the dynamic pressure rise for all five tests, and gives a quantitative indication of the reaction rate. In Tests 1-4, the pressure attainable in the oven was capped at 150psig with a burst disc. However, for Test 5 the burst disc rating was changed to 1500psig, permitting the oven to reach higher pressure before venting. In Test 5 we observed: 1) the same initial expulsion of white smoke from a crack 2) oven filling with smoke 3) ignition of the gases with a bright flame 4) a flaring of combustion that saturated the camera sensor 5) the re-appearance of the sample, glowing with flame in areas 6) the extinguishing of the sample and the final appearance of the charred remnant. The higher-rated burst disc provided pressure confinement that permitted the reaction to access a qualitatively different mode of combustion.



In all tests, the combustion reaction generated sufficient pressure to "pop" the burst disc and relieve pressure. In all tests, the sample was not quite fully consumed, and a charred remnant was left on the pedestal. The remaining material was extremely low density and brittle—a carbon charcoal. Figure 21 through Figure 26 are selected post-mortem images of this remnant, from various tests, which exhibits a layered morphology. It is unclear whether the layered nature of the remnant is linked to the laminar anisotropy characteristic of uniaxially pressed PBX 9502.

The mechanical failure demonstrated in these tests—namely the formation of macro-cracks within seconds of cookoff—is consistent with the permeability testing performed in 2013[1]. Those permeability results found that PBX 9502, even when thermally damaged, remains quite impermeable. As discussed in the introduction, this result is difficult to reconcile with cookoff behavior, which seems to suggest a mechanism for gas pressure communication from the exterior to the interior of the sample. The timelapse and video record show that the sample remains intact—at least on the large scale—up until the cookoff event occurs. It seems plausible that the internal pressure liberated by reaction is contained within interior pores of the material, unable to escape, until the pressure builds and exceeds the mechanical strength of the material itself. At which point the internal gas pressure cracks the sample, and interior product gases are liberated.

The thermal expansion of the sample was measured along the sample axis via image analysis (Figure 16-Figure 20). The linear coefficient of thermal expansion, also abbreviated "CTE", or mathematically represented as α_L is defined as the fractional change in length per unit change in temperature, and is given in units of per Kelvin:

$$\text{CTE} = \alpha_L = \frac{\Delta L / L}{\Delta T} \quad \text{where} \quad \begin{aligned} &\alpha_L = \text{linear expansion coefficient} \\ &\Delta L = \text{change in specimen length} \\ &L = \text{specimen length} \\ &\Delta T = \text{temperature change that produced } \Delta L \end{aligned} \tag{1}$$

The image analysis is referenced to the initial length, L_0. The adjusted form for the change in length based on the length at temperature is:

$$\frac{\Delta L}{L} = \frac{\Delta L / L_0}{1 + (\Delta L / L_0)} \tag{2}$$

The CTE was calculated for each test, at steady state after the temperature reached a constant. These values are tabulated in Figure 29. The average CTE[1] for the three axial orientation samples is $1.3 \times 10^{-4} \pm .3 \times 10^{-4}$ °K^{-1}. The average CTE for the two orthogonal orientation samples is $.83 \times 10^{-4} \pm .08 \times 10^{-4}$ °K^{-1}. Note that the axial orientation samples demonstrated a higher thermal expansion than the orthogonal orientation. These results are consistent with the review by Skidmore[7] and data reported by Maienschein and Garcia[8] for LX-17 (92.5 wt.% TATB, 7.5 wt.% Kel-F 800). As in our results, Maienschein and Garcia report a slightly higher CTE for uniaxially-pressed samples expanding along the axial orientation than along the radial orientation.

Test	Steady-state temperature	$\Delta L/L_0 \pm .002$	CTE calculated at temperature	Pressing Orientation
1	259°C	.035	1.54×10^{-4}	Axial
2	325°C	.029	$.973 \times 10^{-4}$	Axial
3	304°C	.038	1.41×10^{-4}	Axial
4	302°C	.021	$.772 \times 10^{-4}$	Orthogonal
5	299°C	.024	$.893 \times 10^{-4}$	Orthogonal

Figure 29. Coefficient of thermal expansion calculated via image analysis.

6. Conclusions

In this test series we designed a small cookoff oven with the purpose of recording the long, quasi-static pressure generation of an unconfined sample of PBX 9502 as it is heated through to self-ignition. The apparatus contained visual access on two axes in order to capture both high-speed video and long-duration time-lapse photography.

Five tests were performed; the thermal profile, and the static and dynamic pressure data were recorded. High-speed video shows large, macroscopic crack formation in the final seconds prior to self-ignition, followed by the production of smoke which is expelled from the cracks. These data suggest that cracks are the dominant gas transport features.

In Tests 1-5, deflagration occurred without visual flame. In Test 5, which incorporated a higher-pressure burst disc and was thus able to access higher pressure gas pressure confinement during cookoff, product gases deflagrated with luminous flame.

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[1] One source of error in these measurements is that the temperature used for the calculations is measured with a finite-sized thermocouple bead on the surface of the sample, which may differ from the average internal temperature of the sample. Another source of error is the pixel resolution of the image analysis.

Coefficients of thermal expansion were calculated from image analysis. Dynamic pressure rise was obtained up to a cap resulting from the limitation of the diagnostics used. This experimental series demonstrated the potential for this apparatus, with only slight design modifications, to capture the entire pressure pulse of a small sample of PBX 9502 heated to self-ignition.

7. Future Work

7.1. Full pressure record during cookoff

The tests reported here concentrated on resolving the long-duration behavior of the sample, including the much lower static pressure rise that provides evidence of the permeability evolution of the PBX 9502. It is desirable to construct a stronger oven capable of containing the maximum pressure reached during the cookoff event, and to place a high-temperature dynamic transducer directly adjacent to the oven, in order to capture the full pressure record during cookoff behavior.

7.2. Effect of confinement on gas generation and cookoff time

Evidence exists to suggest that the "ullage"—the amount of free volume available, into which the explosive charge is able to thermally expand—has an effect on the cookoff behavior of PBX 9502. The Gas Generation apparatus can be used to explore the effect of ullage, by utilizing confinement sleeves (for radial confinement) or caps (for axial confinement) inside the oven. If a clear material is used as a radial sleeve, the expansion and behavior can still be visually imaged. Obtaining the dependence of gas generation on mechanical confinement would provide valuable data on cookoff behavior.

7.3. Effect of surface area to volume ratio on the gas production

An unanswered question is whether the observed pressure rise is associated with the production of gas from the surface of the explosive or from the interior volume. By varying the surface area to volume ratio of samples, it may be possible to determine which is the source of the gas production.

Appendix A: Experimental Procedure

SETUP NOTES:

- Using a strain-gauge pressure transducer so that both long-term static and dynamic can be recorded from same device. Long-term static measured using NI chassis with strain gauge module, and custom VI. Dynamic measured with scope. Custom built distribution box provides the connections to split the signal.
- Using a microphone trigger, and a panic button trigger, set up with a logic trigger on a scope. Scope output triggers a)phantom b) pressure transducer scope
- Using 4 rectangular LED lights in the corners of the boombox

PRE-TEST

1. Measure and mass sample
2. Assemble oven.
    a. Install burst disc into holder & mount holder onto oven. Record bust disc details in notebook.
    b. Place sample in oven on pedestal.
    c. Clean sapphire windows, bolt to oven (use Kalrez o-rings).
    d. Carefully mount thermocouple probes into oven, replacing o-rings on fittings as necessary.
    e. Tape TCs to oven surface for control, diagnostic
    f. Wrap heating rope around oven, keeping flat on oven (any bits not touching copper will burn out)
    g. Wrap oven with fiberglass insulation
    h. Tape TC to gas tubing next to pressure transducer
    i. Check resistance of heater to make sure it's not broken, record in notebook.
3. Run cable for remote valve
    a. Check operation of valve
4. Run two long e-net cables, for
    a. Phantom
    b. NI DAQ VI
5. Setup pre-test configuration
    a. Connect apparatus to manifold with vacuum pump, pressure transducer, vacuum gauge.
    b. Connect and start DAQ to record pressure.
    c. Prepare scope for checking scope pressure measurement.
6. Purge volume
    a. Evacuate volume.
    b. Fill to 30psig with nitrogen.
    c. Repeat steps a and b two more times.
7. Check pressure readings on both DAQ devices, at multiple pressures. Note that scope will report a voltage offset at ambient pressure.
8. Record scope offset at ambient pressure.
9. Let DAQ run overnight to check leak rate.
10. Open vent valve briefly to allow volume to equalize to ambient, or just above.
11. Place assembly into boom box (elevate using two layers of 1/2" thick steel plates).
12. Connect
    a. Remote vent valve wire
    b. Heater power
    c. Thermocouples
    d. Pressure transducer cable
13. Arrange 4 LED lights around assembly.
14. Setup cameras
    a. Aim square to sample
    b. AC adapter
    c. Use 125GB
    d. Attach intervalometer
    e. Block flare light
    f. Focus.
    g. Check settings.
15. Set up microphone trigger.
16. Set up transducer scope
    a. Use isolation transformer.
    b. Use filtering cap & resistors at scope.
    c. Connect to the LAN in order to be able to remotely reset trigger.

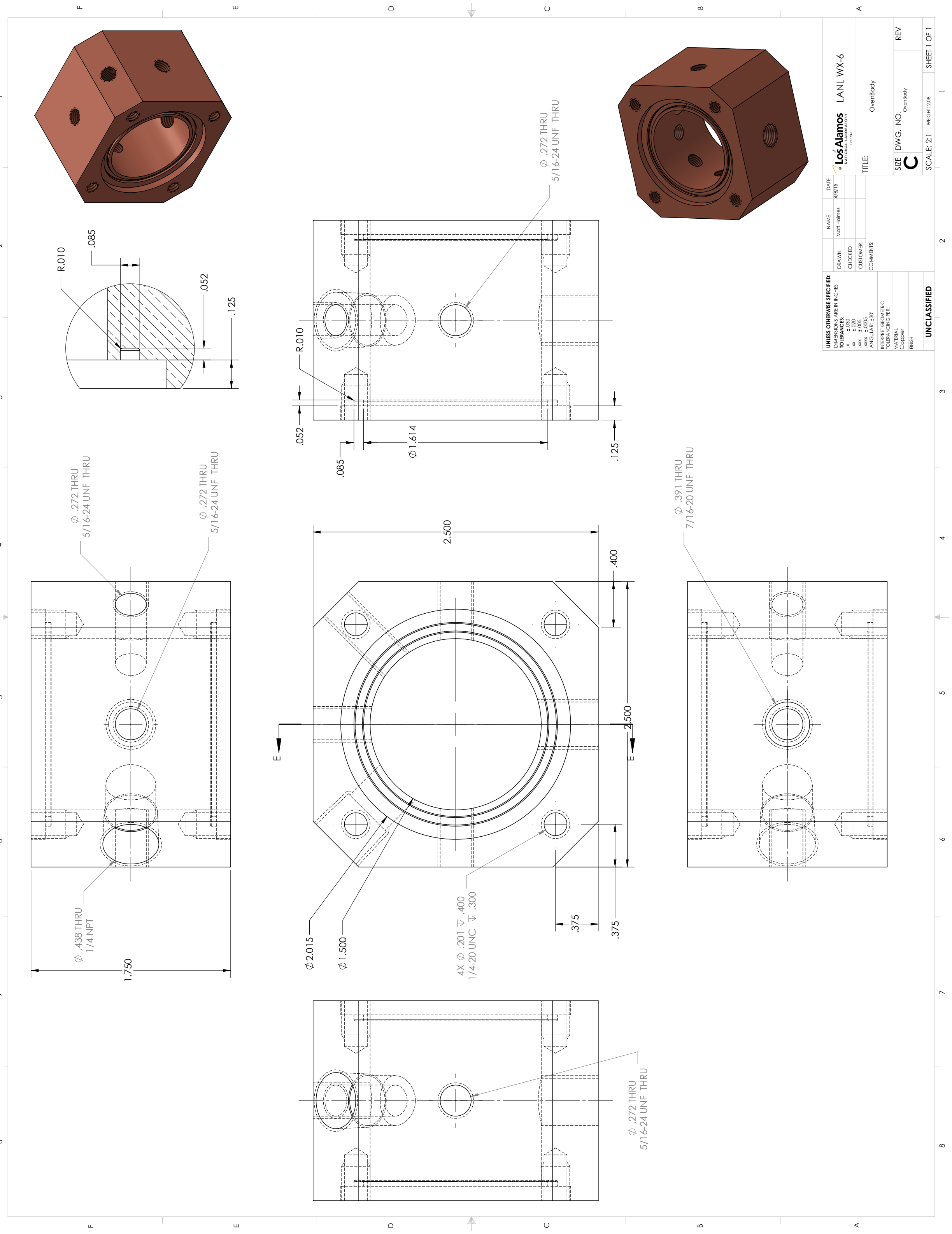
TEST TIME

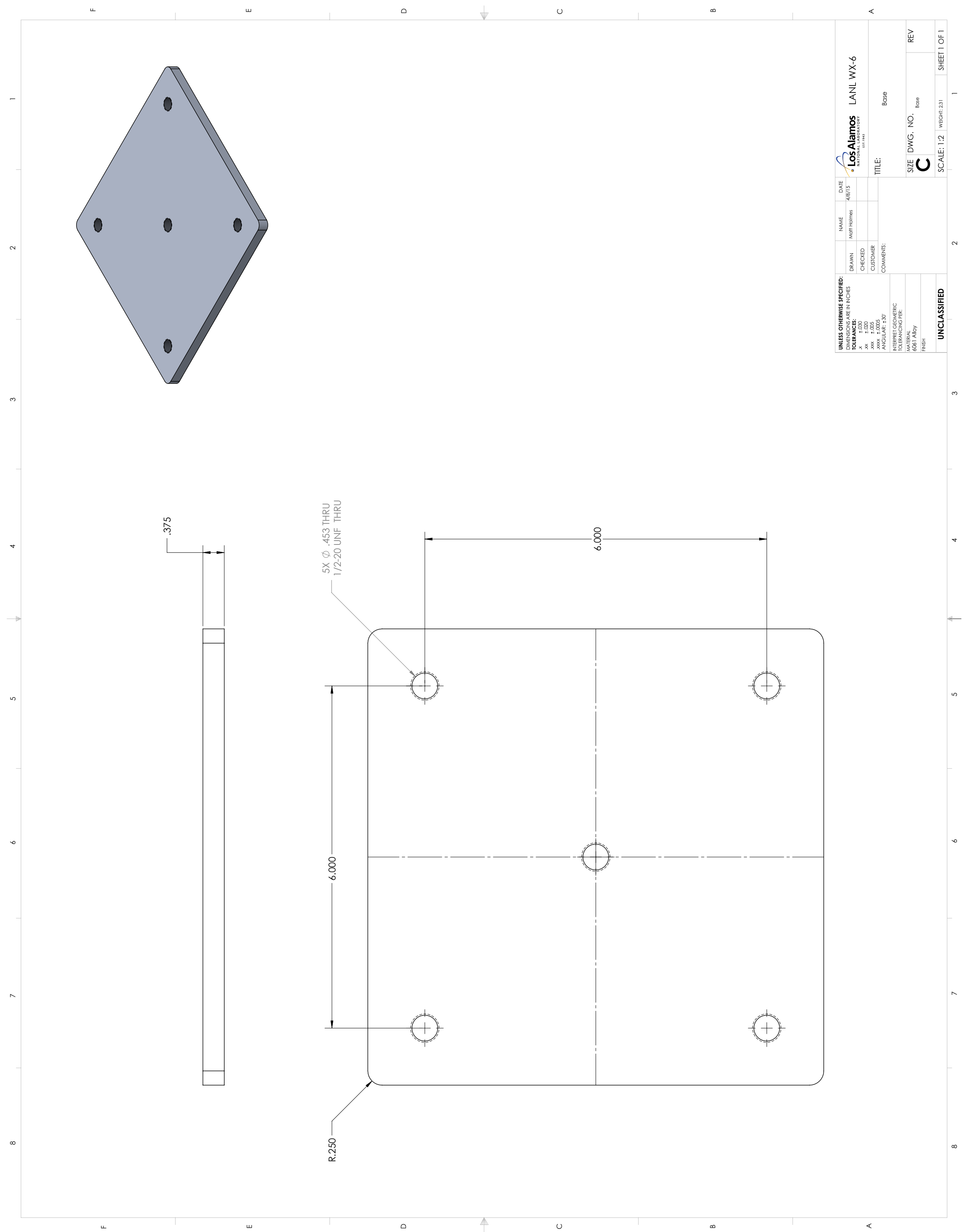
1. Check both triggers (microphone to scope and phantom).
2. Measure heater resistance at plug end and record.
3. Force trigger scope and measure average voltage to obtain the value for ambient pressure.
4. Use Heise gauge, record absolute ambient pressure.
5. Lock down boombox
6. Place Nederman exhaust over vent.
7. Check heater controller settings, adjust setpoint.
8. Lock out room.
9. Start VI.
10. Start canon camera intervalometer.
11. Plug in heater.
12. Start heater; record time.

DURING TEST
In event of spurious trigger, rapidly decide whether there's any value in saving data, then reset the triggers

POST-TEST
1. Turn off and unplug heater
2. Stop DAQ
3. Save pressure scope data
4. Save phantom video as raw (.cine)
5. Stop canon intervalometer
6. Transfer all data to office machine

Appendix B: Drawings

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REV
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DRAWN
Matt Holmes
CHECKED
CUSTOMER
COMMENTS:
UNLESS OTHERWISE SPECIFIED:
UNCLASSIFIED
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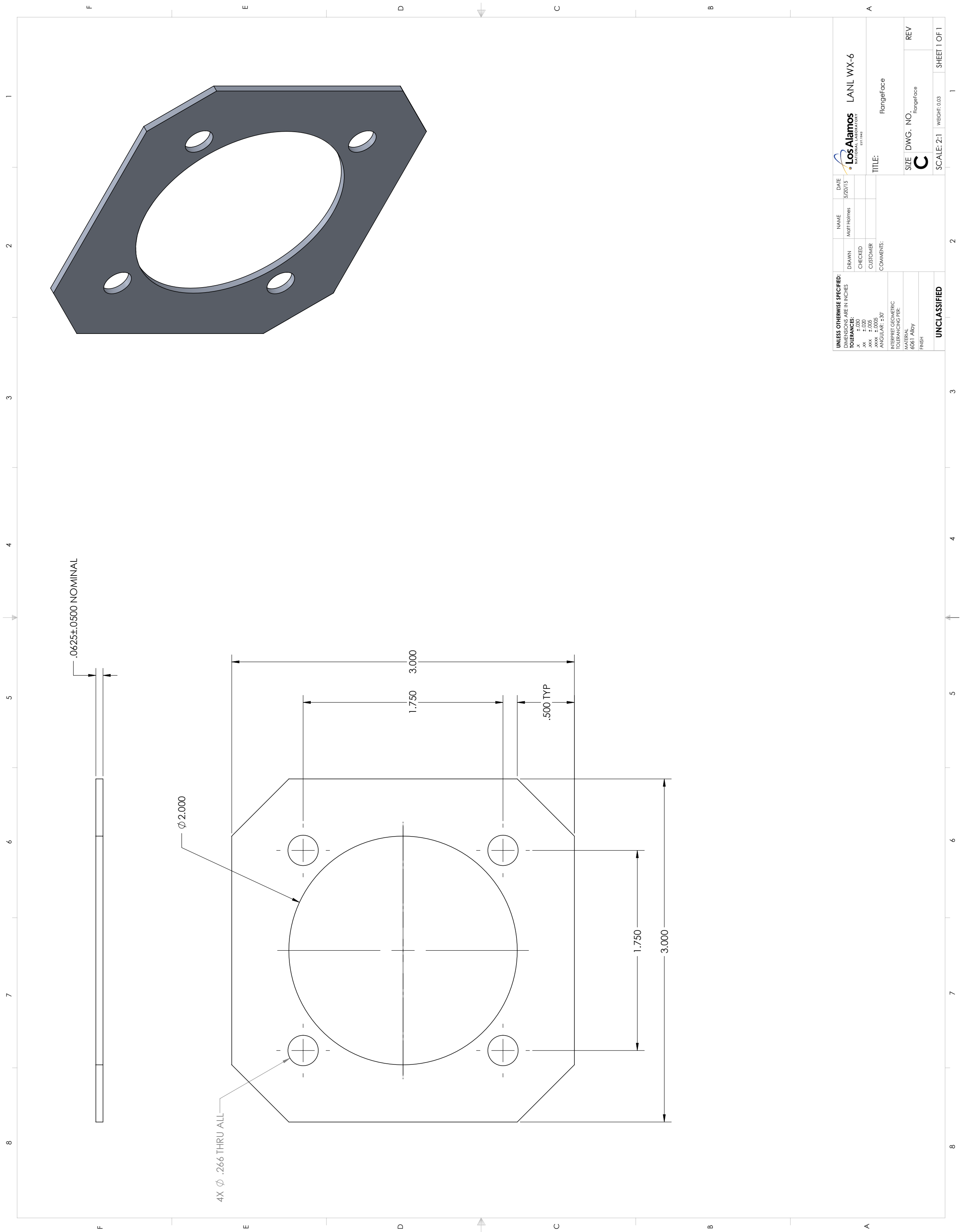
6.000
6.000
5X ∅ .453 THRU
1/2-20 UNF THRU
R.250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Los Alamos
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FlangeFace
SIZE
C
DWG. NO.
FlangeFace
REV
SCALE: 2:1
SHEET 1 OF 1
UNCLASSIFIED
DRAWN
Matt Holmes
5/20/15
CHECKED
CUSTOMER
COMMENTS:
UNLESS OTHERWISE SPECIFIED:
DIMENSIONS ARE IN INCHES
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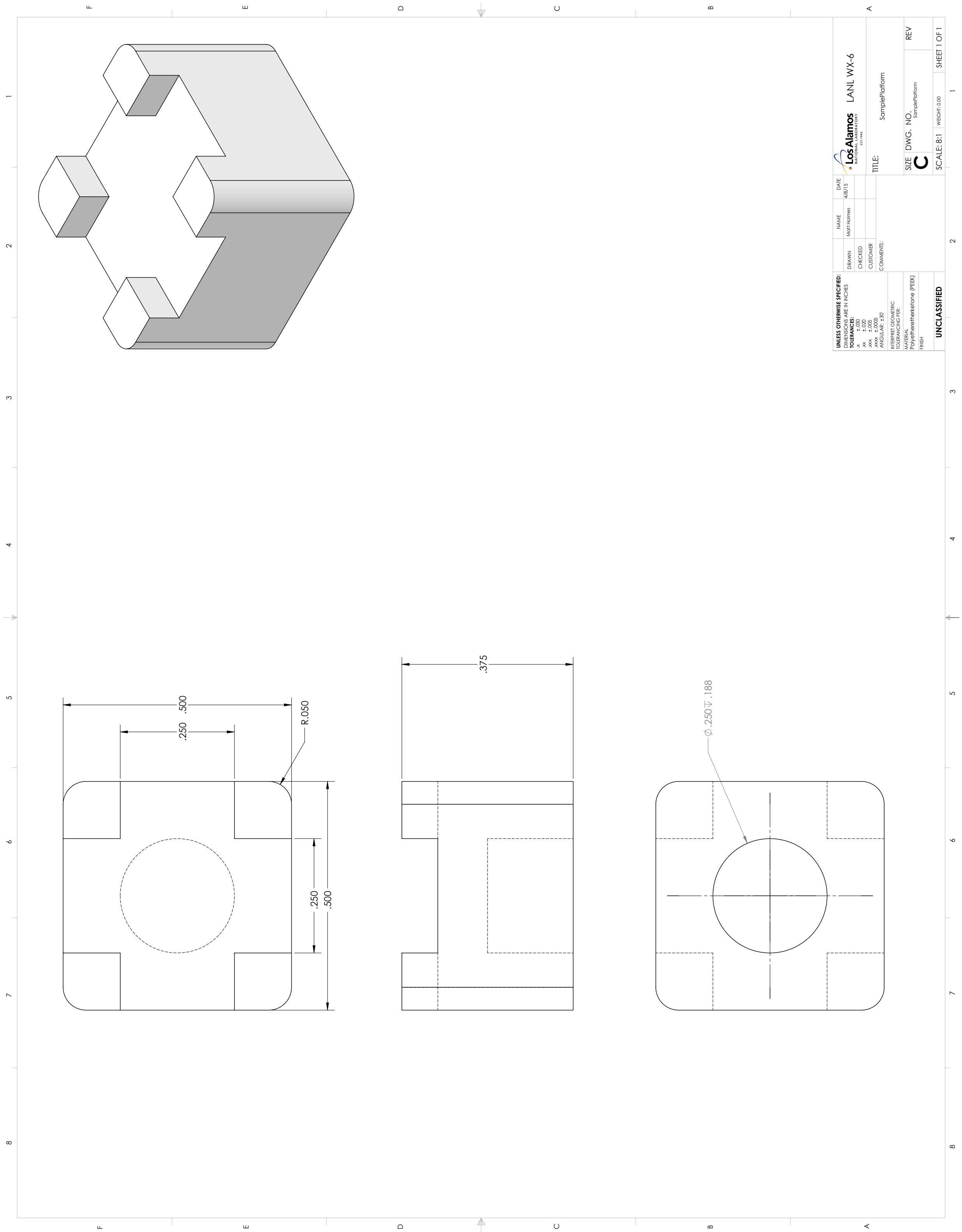
3.000
1.750
.500 TYP
∅2.000
1.750
3.000
4X ∅ .266 THRU ALL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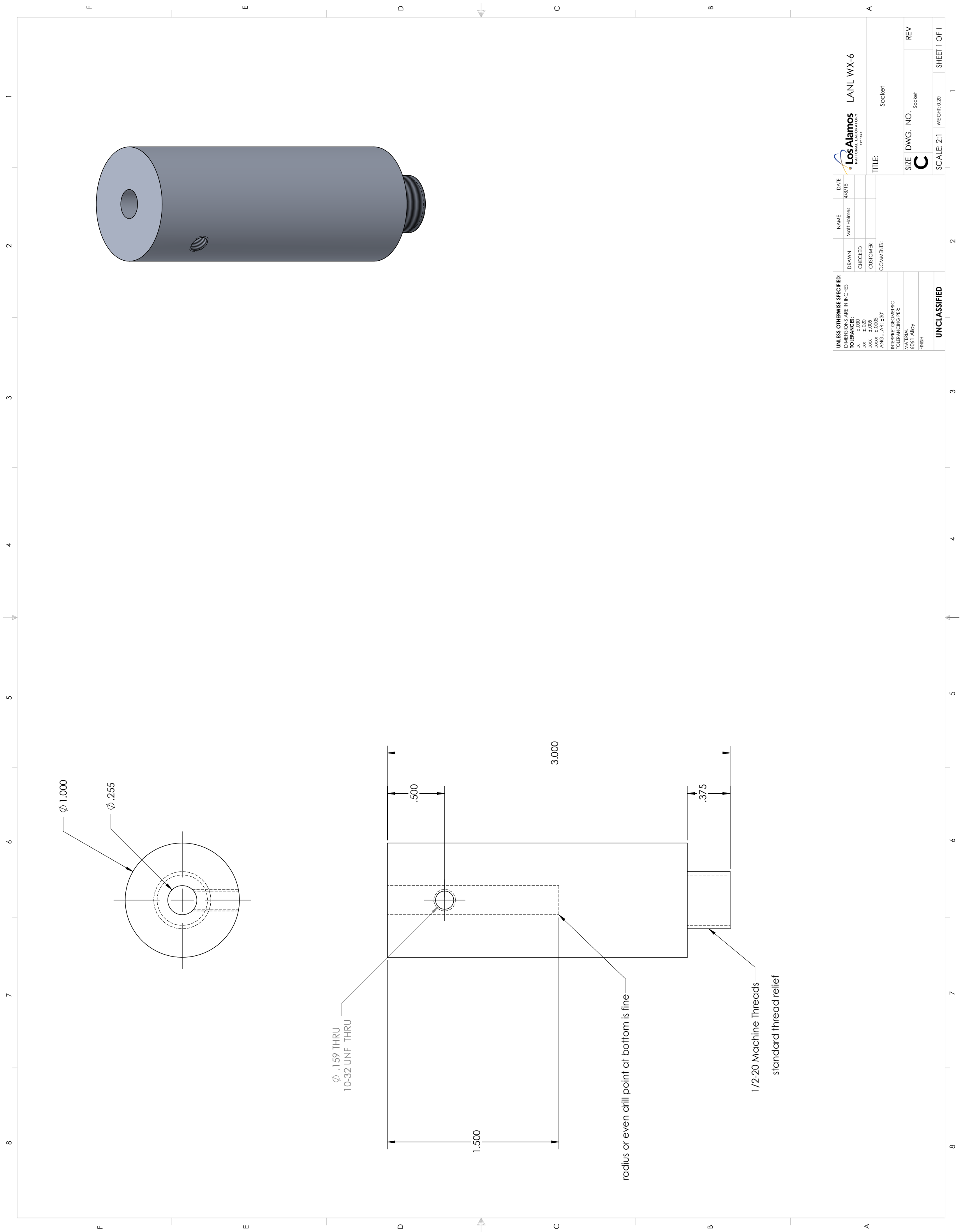
.250
R.050
.375
.250
.500
LANL WX-6
SamplePlatform
UNCLASSIFIED
SCALE: 8:1
SHEET 1 OF 1


LANL WX-6
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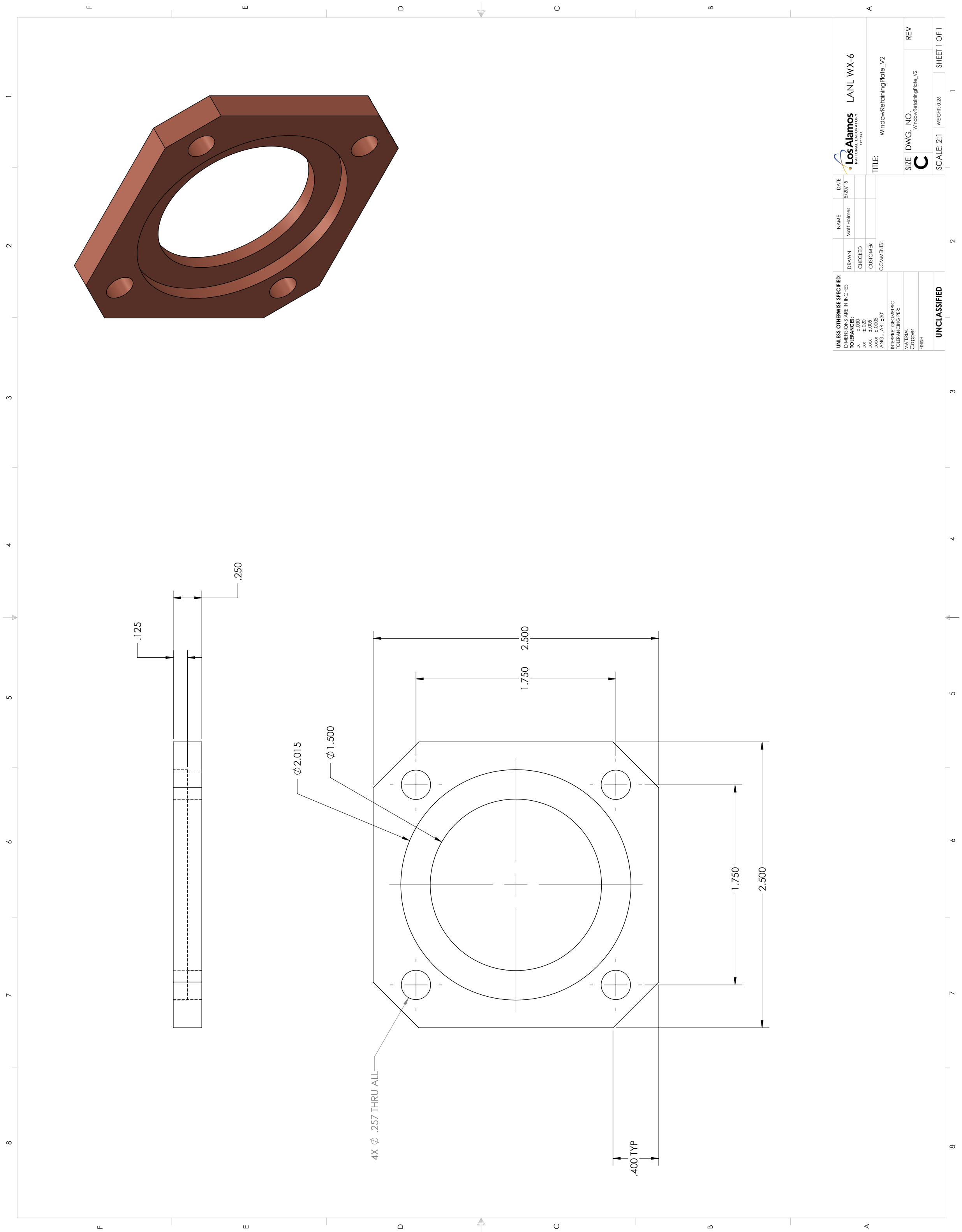
Socket
SIZE
C
DWG. NO.
REV
SCALE: 2:1
SHEET 1 OF 1
UNCLASSIFIED
3.000
.500
.375
Ø1.000
Ø.255
Ø .159 THRU
10-32 UNF THRU
radius or even drill point at bottom is fine
1/2-20 Machine Threads
standard thread relief
1.500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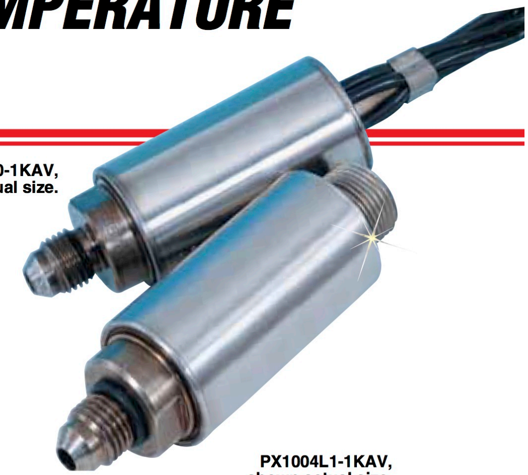
SIZE
C
SCALE: 2:1
SHEET 1 OF 1
REV
DRAWN
Matt Holmes
5/20/15
CHECKED
CUSTOMER
COMMENTS:
UNLESS OTHERWISE SPECIFIED:
DIMENSIONS ARE IN INCHES
MATERIAL
Copper
UNCLASSIFIED
.250
.125
2.500
1.750
Ø2.015
Ø1.500
4X Ø .257 THRU ALL
.400 TYP

Appendix C: Datasheets

HIGH- AND VERY-HIGH-TEMPERATURE PRESSURE TRANSDUCERS

mV/V Output
0-15 to 0-10,000 psi
0-1 to 0-700 bar

1 bar = 14.5 psi
1 kg/cm² = 14.22 psi
1 atmosphere = 14.7 psi = 29.93 inHg = 760.2 mmHg = 1.014 bar



PX1004/PX1009 Series

NIST CE
Standard

- **Two Versions That Operate from -54 to 232°C (-65 to 450°F) (PX1004) or -54 to 343°C (-65 to 650°F) (PX1009)**
- **High Performance**
- **Proven High Reliability**
- **Exceptional Calibration Stability**

Applications

- **Engine and Test Stands**
- **High-Temperature Testing**
- **Process Monitoring**
- **Ground Support Equipment**
- **High-Temperature Geothermal Generators**

OMEGA's PX1004 and PX1009 sputtered thin-film pressure transducers are designed for high-temperature service. These instruments can operate in temperatures from -54 to 232°C or 343°C (-65 to 450°F or 650°F).

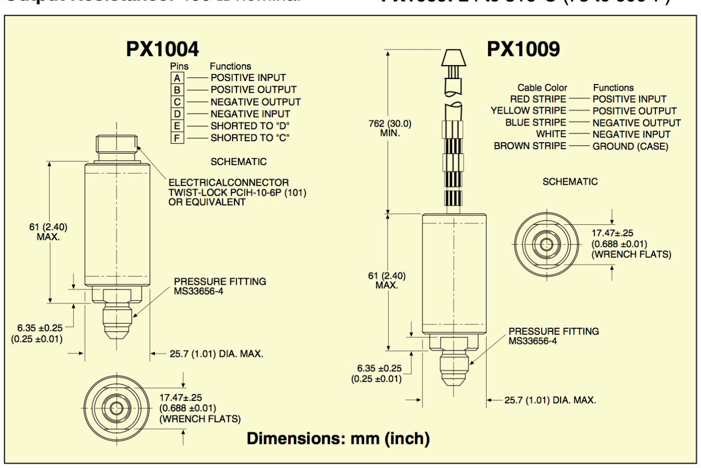
Yet even in these extreme temperatures, they provide outstanding accuracy, long-term calibration stability, and reliability. Static accuracy is ±0.25%, and thermal zero and sensitivity shifts over their compensated ranges are less than ±0.01%/°F.

OMEGA's thin-film technology makes this premium performance possible. The strain gages are sputter-deposited, forming a molecular bond with the substrate. There is virtually no shift, drift, or creep to cause the transducer's calibration to change. The all-welded stainless steel pressure cavity and double-isolated case ensure pressure integrity and reliability in adverse environments.

These high-temperature transducers are available in many standard ranges from 15 psi to 10,000 psi. A NIST-traceable calibration record is available for these units.

SPECIFICATIONS

Excitation: 10 Vdc
Full Range Output: 30 mV nominal, 26 mV minimum
Residual Unbalance: 0 mV ±5% FSO at zero pressure
Input Resistance: 400 Ω nominal
Output Resistance: 400 Ω nominal
Insulation Resistance:
PX1004: 100 MΩ or greater @ 45 Vdc
PX1009: 50 MΩ or greater @ 45 Vdc
Electrical Connections:
PX1004: PCIH-10-6P or equivalent
PX1009: Color-coded pigtail leads, 0.8 m (30") long, minimum
Sensing Element: 4-active-arm bridge using sputter-deposited thin-film elements.
Accuracy: Combined linearity, hysteresis and repeatability: ±0.25% FSO, BSL (over the compensated temperature range)
Note: *Performance is determined at 25°C ±1°C (77°F ±2°F), open circuit, rated excitation, unless otherwise specified*
Operating Temp Range:
PX1004: -54 to 232°C (-65 to 450°F)
PX1009: -54 to 343°C (-65 to 650°F)
Compensated Temp Range:
PX1004: 24 to 204°C (75 to 400°F)
PX1009: 24 to 316°C (75 to 600°F)



B-75

Figure 30. Omega model PX1009 datasheet

HIGH AND VERY HIGH TEMPERATURE PRESSURE TRANSDUCERS

MILLIVOLT OUTPUT PRESSURE TRANSDUCERS

B

Thermal Effects:
Span: ±0.01% FSO/°F
Zero: ±0.01% FSO/°F

Natural Frequency:
50 kHz at 5000 psi decreasing logarithmically to 5 kHz at 15 psi

Vibration Sensitivity: At 35 g peak from 10 Hz to 2000 Hz (limited to ½" D.A.), the output shall not exceed:

	PX1004	PX1009
15 psi	0.04% FSO/g	0.15% FSO/g
50 psi	0.02% FSO/g	0.07% FSO/g
100 psi	0.015% FSO/g	0.05% FSO/g
500 psi	0.006% FSO/g	0.02% FSO/g
1000 psi	0.003% FSO/g	0.02% FSO/g

Shock: 100 g, 11 ms half sine wave without calibration shift or damage

Humidity:
Per MIL-STD-810, Method 507.1
Standard ranges: 0 to 15, 25, 50, 100, 250, 500, 1000, 1500, 2000, 2500, 5000 and 10,000 psia. Sealed gage available in ranges of 100 psi and above.

Proof Pressure:
PX1004: 200% of rated pressure
PX1009: 150% of rated pressure

Burst Pressure:
PX1004: 300% of rated pressure
PX1009: 200% of rated pressure

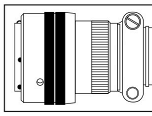
Wetted Parts: 17-4 PH vacuum melt or 15-5 stainless steel

Pressure Fitting:
7⁄16-20 male per MS33656-4

Mounting Isolation: Case isolation ensures that the sensing element will not be affected by external stresses

Mating Connector (PX1004):
PCS06E-10-6S(SR) (sold separately)

Weight: 145 g (5 oz) maximum



PX1004 Mating Connector
PCS06E-10-6S(SR)

To Order

RANGE		MODEL NO. PX1004	MODEL NO. PX1009	COMPATIBLE METERS*
ABSOLUTE PRESSURE ***(All Ranges Available in Sealed Gage Pressure)***				
0 to 15 psi	0 to 1.0 bar	**PX1004L1-015AV**	**PX1009L0-015AV**	DP41-S, DP25B-S
0 to 25 psi	0 to 1.7 bar	**PX1004L1-025AV**	**PX1009L0-025AV**	DP41-S, DP25B-S
0 to 50 psi	0 to 3.4 bar	**PX1004L1-050AV**	**PX1009L0-050AV**	DP41-S, DP25B-S
0 to 100 psi	0 to 6.9 bar	**PX1004L1-100AV**	**PX1009L0-100AV**	DP41-S, DP25B-S
0 to 250 psi	0 to 17.2 bar	**PX1004L1-250AV**	**PX1009L0-250AV**	DP41-S, DP25B-S
0 to 500 psi	0 to 34.5 bar	**PX1004L1-500AV**	**PX1009L0-500AV**	DP41-S, DP25B-S
0 to 1000 psi	0 to 68.9 bar	**PX1004L1-1KAV**	**PX1009L0-1KAV**	DP41-S, DP25B-S
0 to 1500 psi	0 to 103 bar	**PX1004L1-1.5KAV**	**PX1009L0-1.5KAV**	DP41-S, DP25B-S
0 to 2000 psi	0 to 138 bar	**PX1004L1-2KAV**	**PX1009L0-2KAV**	DP41-S, DP25B-S
0 to 2500 psi	0 to 172 bar	**PX1004L1-2.5KAV**	**PX1009L0-2.5KAV**	DP41-S, DP25B-S
0 to 5000 psi	0 to 345 bar	**PX1004L1-5KAV**	**PX1009L0-5KAV**	DP41-S, DP25B-S
0 to 10,000 psi	0 to 689 bar	**PX1004L1-10KAV**	**PX1009L0-10KAV**	DP41-S, DP25B-S

Comes complete with 5-point calibration. ***** *See* ***omega.com*** *for compatible meters.* *Metric ranges available – Consult Engineering.*
To order sealed gage pressure, replace ***"A"*** *in model number with* ***"S"*** *(no extra charge).*
Ordering Examples: PX1004L1-100AV, *100 psi absolute pressure transducer with male pressure connection per MS33656-4 and PCHI0-10-6P electrical connector.* ***PCS06E-10-6S(SR),*** *mating connector (sold separately).* ***PX1009L0-1KSV,*** *1000 psi sealed gage pressure transducer with MS33656-4 male pressure connection and 0.8 m (30") high-temperature cable.*

ACCESSORY

MODEL NO.	DESCRIPTION
PCS06E-10-6S(SR)	Mating connector for PX1004 transducers

CUSTOM CONFIGURATIONS

PX1004/1009 SERIES	PRESSURE PORT [1]	ELECTRICAL CONNECTION [2]	RANGE [3]	UNITS [4]	OUTPUT [5]	OPTIONS [6]
	L = MS33656-4	**PX1004 only 1** = **PCIH0-10-6P** Connector (or equal). Mating connector (sold separately), order **PCS06E-10-6S(SR)**	015 psi	A	**V** = mV/V Output	**CAL11 =** 11-point NIST-traceable calibration
		PX1009 only 0 = color-coded cable, 0.8 m (30") min.	025	A	Base Price PX1004:	**extra cost**
			050	A	Base Price PX1009:	
			100	A, S		
			250	A, S		
			500	A, S		
			1K	A, S		
			1.5K	A, S		
			2K	A, S		
			2.5K	A, S		
			5K	A, S		
			10K	A, S		

Metric ranges available – Consult Engineering.

To order a custom configuration:
1. Select a pressure port
2. Select electrical connection
3. Select a pressure range
4. Select pressure units
5. Select output
6. Select options and agency approvals

Ordering Examples: PX1004L1-100AV, *MS33656-4 male pressure port, twist-lock connector, 100 psi absolute pressure range and mV/V output.* ***PCS06E-10-6S(SR),*** *mating connector (sold separately).* ***PX1004L1-015SV,*** *MS33656-4 male pressure port, twist-lock connector, 15 psi sealed gage pressure range and mV/V output.* ***PCS06E-10-6S(SR),*** *mating connector (sold separately).*
PX1009L1-1KSV-CAL11, *MS33656-4 male pressure port, 0.8 m (30") high-temperature cable, 1000 psi sealed gage pressure range, mV/V output and optional 11-point NIST-traceable calibration.*

O M E G A E N G I N E E R I N G I N C.

PRESSURE TRANSDUCER
FINAL CALIBRATION

0.00 - 100.00 PSIA
Excitation 10.000 Vdc

Job:
Model: PX1009L0-100AV
Date: 6/26/2015
Calibrated: 0.00 - 100.00 PSIA

Serial: 608837
Tested By: GP
Temperature Range: +75 to +600 F
Specfile: PX1009

Pressure PSIA	Unit Data mVdc
0.00	0.758
50.00	15.835
100.00	31.018
50.00	15.846
0.00	0.770

Balance	0.758	mVdc
Sensitivity	30.260	mVdc
In Resist	400.70	Ohms
Out Resist	374.30	Ohms

Calibration Factors:
Sensitivity = 3.026 mV/V

ELECTRICAL LEAKAGE: PASS
PRESSURE CONNECTION/FITTING: MS33656-4 7/16-20 MALE
ELECTRICAL WIRING/CONNECTOR: RED STRIPE = +INPUT
YELLOW STRIPE = +OUTPUT
BLUE STRIPE = -OUTPUT
WHITE = -INPUT
Brown STRIPE = Ground (Case)

This Calibration was performed using Instruments and Standards that are traceable to the United States National Institute of Standards Technology.

S/N	Description	Range	Reference	Cal Cert
1742/94-8	AUTO 150 PSI DRUCK	0 - 100.00 PSIA	C-2509	C-2509
US37015149	AT34970A DMM UUT	Unit Under Test	C-3004	C-3004

Q.A. Representative : Gary Perren Date: 6/26/2015

This transducer is tested to & meets published specifications. After final calibration our products are stored in a controlled stock room & considered in bonded storage. Depending on environment & severity of use factory calibration is recommended every one to three years after initial service installation date.

Omega Engineering Inc., One Omega Drive, Stamford, CT 06907
http://www.omega.com email: info@omega.com phone (800) 826-6342

Figure 31. Transducer calibration sheet.

Appendix D: References

1. Holmes, M. D., Parker, G. R., Dickson, P., Meyer, B. A. and Schmidt, C. C., "Pressure Dependence of Slow Cookoff Behavior in PBX 9502 Bucket Tests" *Proceedings of the 15th Internation Detonation Symposium*, pp. 1506-1517, San Francisco, CA, 2014.

2. Meyer, B. A., Schmidt, C. C., Holmes, M. D. and Parker, G. R., "Measurement of PBX 9502 Flow Parameters" *LANL Report: W-7-TR-0023*, 2014.

3. Erikson, W. W., Martinez, M. J., Hobbs, M. L., Kaneshige, M. J. and Dodd, A. B., "Modeling Explosive Decomposition and Cookoff with Porous Flow" *Proceedings of the 41st JANNAF Conference*, San Diego, CA, Dec 2006.

4. Zerkle, D. K. and Luck, L. B., "Modeling Cook-off of PBX 9501 with Porous Flow and Contact Resistance" *LANL Report: LA-UR-03-8077*.

5. Parker, G. R., Holmes, M. D. and Dickson, P., "The Effect of Pressure and Venting on the Slow Cookoff of PBX 9502 in the Intermediate-scale Bucket Test" *Los Alamos National Laboratory Report: LA-UR-13-25716*, 2013.

6. Holmes, M. D., Parker, G. R., Broilo, R. M., Heatwole, E. M., Vaughan, L. D., et al., "Hockey Puck Cookoff" *Los Alamos National Laboratory Report: LA-UR-16-22340*, 2016.

7. Skidmore, C. B. and Butler, T. A., "The elusive coefficients of thermal expansion in PBX 9502" *LANL Report: LA-14003*, 2003.

8. Maienschein, J. L. and Garcia, F., "Thermal expansion of TATB-based explosives from 300 to 566 K" *Thermochimica Acta,* Vol. 384, pp. 71-83, 2002.