

VEHICULAR HYDROGEN STORAGE USING LIGHTWEIGHT TANKS

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Abstract

Lightweight hydrogen storage for vehicles is enabled by adopting and adapting aerospace tankage technology. The weight, volume, and cost are already acceptable and improving. Prototype tankage was demonstrated with 11.3% hydrogen by weight, 1.74 million inch (44.3 km) burst performance factor ($P_b V/W$), and 3.77 kWh/kg specific energy for the tank and hydrogen (LHV).

DOE cannot afford full scale aerospace development costs. For example, it costs many tens of \$M to develop a rocket motor casing with a safety factor (SF) of 1.25. Large teams of experts are required to design, develop, and test new processes. Car companies are buying existing technology with only modest investments in research and development (R&D).

The Lawrence Livermore National Laboratory (LLNL) team is maximizing the leverage from DOE funding by joining with industry to solve technical risks at the component level. LLNL is developing fabrication processes with IMPCO Technologies, Thiokol Propulsion, and Aero Tec Laboratories (ATL). LLNL is creating commercial products that are close to adoption under DOE solicitation. LLNL is breaking ground to achieve greater than 10% hydrogen by weight tankage with safety that exceeds the requirements of NGV2 standards modified for hydrogen.

Risk reduction is proceeding along three axes:

- Commercializable products will be available next year with ~90% confidence.
- R&D progress is pushing the envelope in lightweight tankage for vehicles.
- Integration challenges are being met with partners in industry and DOE demo programs.

This project is a key part of LLNL's effort to develop high cycle life energy storage systems with >600 Wh/kg specific energy for various applications, including: high altitude long endurance solar rechargeable aircraft, zero emission vehicles, hybrid energy storage/propulsion systems for spacecraft, energy storage for premium power, remote power sources, and peak shaving.

Long-Term Goals

There are eight long-term goals in this project:

1. Demonstrate tankage with 12% hydrogen by weight (5,000 psi [34.5 MPa] service, 300 K, safety factor [SF] 2.25) and 700 Wh/liter.
2. Certify tankage for operation on vehicles (e.g., NGV2 standards modified for hydrogen).
3. Modify designs for easy manufacturability and have industry adopt lightweight tankage designs.
4. Work with industry to reduce the cost of hydrogen tankage.
5. Work with industry to develop lightweight hydrogen tankage with service pressure ratings up to 10,000 psi (69 MPa).
6. Work with large auto manufacturers to demonstrate lightweight hydrogen tanks on fuel cell vehicles.
7. Suggest modifications to hydrogen tankage codes and standards (e.g., hydrogen permeation standards for modified NGV2 or ISO/TC 197).
8. Develop fast filling operations, tankage, and fueling infrastructure that mitigate overtemperature/overpressure issues.

This list of eight tasks will appear throughout this Annual Report. It provides a common framework to organize the presentation of many related accomplishments. Expertise and contacts are shared between these tasks, and an accurate picture of present or future activities cannot be obtained from viewing particular tasks in isolation.

The recurrence of these eight tasks reflects the LLNL team's attempt to answer the DOE required project reporting format for the 2000 Hydrogen Program Annual Review Meeting (San Ramon, CA, May 9-11, 2000). All LLNL lightweight tankage research activities have been organized into these eight categories. Within each category, extensive presentations of the LLNL team's activities will be found largely in the Current Year Progress section. All current and planned activities derive from the list of long term goals above, reflecting LLNL's dedication to aggressive pursuit of this pivotal hydrogen storage technology.

LLNL interest in lightweight hydrogen storage derives from several aggressive aerospace vehicle projects (Carter 1999, de Groot 1997, Kare 1999, McElroy 1998, Mitlitsky 2000, Mitlitsky 1999-a,b,c,d,e Mitlitsky 1998-a,b,c,d,e,f, Mitlitsky 1997, Mitlitsky 1996-a,b,c,d, Mitlitsky 1994, Mitlitsky 1993) whose feasibility relied heavily on the availability of advanced pressure vessels to hold gaseous hydrogen and oxygen. Attempts to dramatically improve mass performance of aerospace vehicle tanks had obvious spinoffs in automotive and utility applications of interest to DOE (Mitlitsky 1999-c,d,e, Mitlitsky 1998-b,d,e,f, Mitlitsky 1997,

Mitlitsky 1996-a,c, Mitlitsky 1994). Of the potential applications, automotive fuel tanks is by far the most important to DOE, as written into the Hydrogen Program's enabling legislation (Gronich 2000).

Mass-sensitivity may be reduced in automobiles compared to aircraft or spacecraft, but it cannot be ignored. The mass of fuel a vehicle can afford to carry directly limits its range. The reason why battery-powered automobiles are not capable of the ~380 mile (610 km) range desired for electric vehicles is due to the mass compounding effect of the energy storage system. Each kg of energy storage on the vehicle results in a 1.3-1.7 kg increase in vehicle mass, due to the additional powerplant and structure required to suspend and transport it (Mitlitsky 1999-e). Large mass fractions devoted to energy storage ruin a vehicle design, devoting too much costly hardware to transport a smaller fraction available for passengers and payload. Although the entire power train mass should be minimized to save costly components, fuel mass cannot be pitched overboard without sacrificing vehicle range. Therefore, lightweight tankage is required for vehicular energy storage systems that can store sufficient specific energy in order to achieve a market-acceptable vehicle driving range.

Lightweight vehicular hydrogen tankage has recently advanced to the threshold of application in demonstration vehicles. Competition with other ways to store hydrogen, or to produce hydrogen from other fuels onboard a vehicle, is intense. Various vehicle designs are being fueled by hydrogen stored in various technologies. Only the technology investigated herein, Type IV pressure vessels, is currently capable of furnishing adequate vehicle range with percent hydrogen by mass performance adequate for the ~380 mile range drivers appear to insist on. The research reported herein can essentially double the range performance of this lowest-mass hydrogen storage alternative.

Volume restrictions are an additional constraint on hydrogen-fueled vehicles, but only in the near term when the relatively few demonstration vehicles cannot afford designs that depart from conventional vehicle layouts. Vehicles that are not designed from the ground up to accommodate enough hydrogen can fail to achieve attractive vehicle range. The relatively low density of energy stored in the form of compressed hydrogen requires significant volume devoted to hydrogen tanks. Increasing storage pressure reduces the storage volume required, at the expense of increased compression losses and infrastructure complexity.

These vehicle design issues have been studied extensively by Directed Technologies, Inc. (DTI) and LLNL. Figure 1 (Mitlitsky 1999-e) shows how hydrogen density is related to temperature and pressure, and its impact on the DOE 2000 tankage goals. Three overlays of tank external volumes show the relative sizes of tanks (and insulation) which store 3.6 kg of hydrogen at 34.5 MPa (300 K), 69 MPa (300 K), and low pressure liquid hydrogen (20 K) (Aceves 2000). The non-ideal compressibility of hydrogen at high pressures is shown by the decreasing slopes of the density curves (at constant temperature) and the sag in the weight percent curves (at constant tank performance factor). The DOE goal of 12 weight percent H₂ at 5,000 psi (34.5 MPa), 300 K translates directly into the need for a tank with a performance factor of 1.85 million inch (47.0 km). The team of LLNL and its industrial partners reports achieving almost all of this goal in the Current Year Tasks section below.

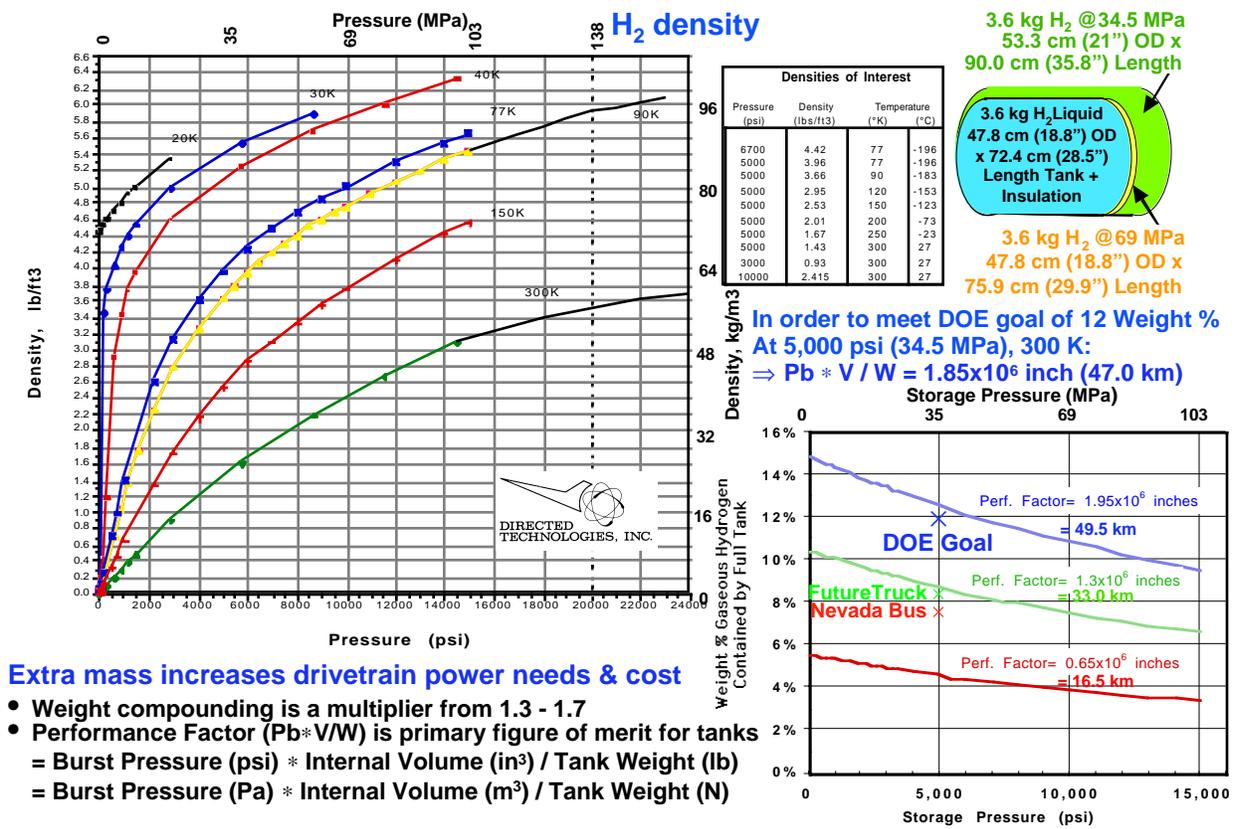


Figure 1 – DOE 2000 Tankage Goals

LLNL has served, and will continue to serve as a conduit for tankage design information between DTI, DOE demonstration programs, and LLNL's industrial partners who are producing high performance hydrogen tanks (IMPCO Technologies and Thiokol Propulsion). Besides technical management of the DOE-funded hydrogen tankage development at IMPCO Technologies (program start May 2000), and Thiokol Propulsion (Haaland-2000), LLNL is funded directly by DOE to develop advanced tankage with significantly better mass performance. Tanks are being built to LLNL specifications, with LLNL design and materials selections, which realize the DOE 2000 Goals. The hydrogen storage mass of 3.6 kg is required for a PNGV-like fuel cell vehicle with a range of 380 miles (610 km) for the EPA Combined Cycle (Mitlitsky 1999-e).

This Year's Objectives and Rationale

1. Prototype tankage with ~12% hydrogen by weight (5000 psi [34.5 MPa] service, 300 K, safety factor 2.25). This will set a tank performance record ($P_b V/W = 1.85$ million inch = 47.0 km) for high cycle life tankage and demonstrate the feasibility of certifying tankage for vehicular operation with >10% hydrogen by weight.
2. Specify a ~1 year program to certify tankage with 7.5-8.5% hydrogen by weight (5000 psi, 300 K, SF 2.25). This will rapidly demonstrate certified hydrogen tankage that has significantly better mass performance (by 50-80%) than industry has demonstrated to date.
3. Develop a lightweight liner fabrication process and permeation reduction coatings that are easily adopted by industry. Variable thickness rotomolded liners are lightweight and easily adopted. Permeation reduction coatings enable lightweight liners to meet proposed permeation specifications, but increase fabrication complexity.
4. Develop lightweight tankage with SF 2.25 and accumulate cost projections from DOE tank solicitations. Lightweight tank liners enable weight goals to be achieved with less expensive fiber. Tankage with SF 2.25 uses ~75% of the fiber required for tankage with SF 3. This is important because fiber cost dominates tankage cost, especially in high volume production.
5. Work with car companies and tank manufacturers to understand prospects for adopting higher pressure (up to 10,000 psi [69 MPa] service pressure). Higher pressure improves storage density of hydrogen at the expense of hydrogen compression cost/inefficiency and infrastructure complexity.
6. Design tankage for Nevada Bus (7.5% hydrogen by weight certified tankage) and FutureTruck 2001 (8.5% hydrogen by weight certified tankage) hydrogen fueled demonstration vehicles. Certify lightweight tankage and illustrate to a wide audience that this technology is ready for adoption. Feasible tank designs can then be developed with and acquired from industry.
7. Scrutinize the proposed specification for hydrogen permeation in the modified NGV2 standard (< 1 standard cc / hr / liter of water capacity at service pressure, room temperature, beginning of life). Overly stringent standards increase cost and preclude attractive technology options without improving safety.
8. Collect data on overtemperature/overpressure issues with fast filling procedures. Overtemperature issues in fast filling are greater than anticipated and might not allow hydrogen tanks to be rapidly filled to capacity without exceeding temperature limits in some cases.

Current Year Tasks

1. Prototype tankage with ~12% hydrogen by weight (5000 psi [34.5 MPa], 300 K, safety factor 2.25). Design hydrogen tanks with lightweight liners, fabricate liners and tanks, burst test tanks, and recommend improvements.
2. Specify a ~1 yr program to certify tankage with 7.5-8.5% hydrogen by weight (5000 psi, 300 K, SF 2.25). Write, modify, and review a solicitation to develop lightweight hydrogen tankage. Technically direct the program that commenced in May 2000 (IMPCO Technologies).
3. Develop a lightweight liner fabrication process and permeation reduction coatings that are easily adopted by industry. Optimize fabrication of variable thickness rotomolded liners. Develop metal and plastic permeation coatings, permeation test coatings, fabricate tanks with coated liners (if funding permits).
4. Develop lightweight hydrogen tankage with SF 2.25 and accumulate cost projections from DOE tank solicitations. Demonstrate plastic lined composite tankage (cylinders and conformable) with safety factor 2.25.
5. Technically direct a 10,000 psi (69 MPa) tankage development effort (pending a DOE/OTT funding award and/or additions to the current DOE/Golden contract DE-AC36-GO10494).
6. Design tankage for Nevada Bus (7.5% hydrogen by weight) and FutureTruck 2001 (8.5% hydrogen by weight) hydrogen fueled vehicles. Design Nevada Bus tankage around existing government tooling for rapid development and demonstration. Design FutureTruck 2001 tankage for a GM Suburban (K 15906) modified for hydrogen fuel.
7. Scrutinize the proposed specification for hydrogen permeation in the modified NGV2 standard and/or the ISO/TC 197 standard. Less than 1 standard cc / hr / liter of water capacity at service pressure, room temperature, beginning of life has been proposed by others. Review the basis for the proposed hydrogen permeation specification and propose a modification if necessary.
8. Collect data on overtemperature/overpressure issues with fast filling procedures. Propose alternatives to mitigate overtemperature/overpressure issues.

Recent progress on each of these eight tasks will be presented in the following section.

Current Year Progress

1. Prototype tankage with ~12% hydrogen by weight (5000 psi [34.5 MPa] service, 300 K, safety factor 2.25). Design hydrogen tanks with lightweight liners, fabricate liners and tanks, burst test tanks, and recommend improvements.

By the beginning of FY00, designs were in hand at LLNL and contracts were in place to realize tank designs at the ~12% hydrogen by weight performance level. Such high performance results were anticipated to set a world record and nearly double the available levels of tank mass efficiency (for high cycle life tankage). All pieces and processes believed necessary to attempt prototyping stood ready to assemble and debug: Special rotational mold tooling had been procured and built in FY99 in an earlier attempt to produce a tank relevant to a Ford demonstration program. Sizing issues for that earlier vehicle program will be discussed in the Task #2 subsection of Current Year Progress (starting on page 17). The availability of this advanced liner production tooling allowed the research this section describes to commence swiftly at the beginning of FY00 under a tank fabrication subcontract with Thiokol that had also been put in place in FY99.

LLNL subcontracted with Thiokol Propulsion to wind tanks on advanced liners produced by ATL. Thiokol's final report to LLNL under that contract has been appended as a major contribution to this report (Appendix 1). The lightweight liners LLNL directed Thiokol to wind around employed a tapered sidewall technology that assists tanks in this design family to achieve the highest percent hydrogen by mass performance levels, using processes that industry might adopt almost immediately.

Tapered thin liners using the technology developed by LLNL at ATL were produced in three batches at the start of FY00. The first batch was employed primarily for process research, and produced several lightweight units that were used in initial winding trials at Thiokol. The second batch completed LLNL's contract with ATL and produced five tanks each in five different wall thickness, ranging in total liner weight from 5 to 10, 15, 20, 25, and 30 pounds. Subsequently a last batch of five liners (6 lb each) with additional processing was built under a small additional LLNL subcontract to ATL. ATL attempted to duplicate a new process step that Thiokol's earliest prototyping attempts found to be necessary on this last batch. Without this thermal preconditioning process step, unacceptable and unexpectedly large liner shrinkage was encountered later in the manufacturing sequence: up to 8% shrinkage occurred during composite cure. By subjecting the liner to thermal cycling similar to its upcoming cure cycling, this shrinkage was found to be stable after just one cycle, so prototypes turned out to be up to ~8% undersized (compared to their designs) and the liner-overwrap interfaces that resulted appeared to be very well adhered (as designed).

Burst tests were performed on four of the five tanks prototyped in this research program. Several attempts to wind on thin liners produced rejects before the process sequence was ironed out early in calendar 2000. The first successful prototype is shown immediately below in Figure 2, next to the thin-wall liner from the same first lot it was wound on. The success of this manufacture in overcoming risky process steps prompted LLNL to reject the entire second batch of fabricated liners in favor of those which duplicated the successful 6 pound prototype liner for

Tank #1, with the additional post-molding thermal process step being applied in the third batch of liners produced and treated at ATL. Prototype Tanks #4 and #5 were wound on parts from this last batch of thermally preconditioned liners.



Figure 2 – First Composite Wrapped Tank Prototype Next to Thin-Wall Liner

Figure 2 above shows a thin-wall liner next to finished Tank #1. The Thiokol boss design employed throughout this research, and built into the tapered thickness rotational mold tooling with 18 inch diameter, is visible at the top of the finished tank in Figure 2, and is sketched in cross section in Appendix 1 (see Appendix C therein). Tank #1 proved to be a useful showpiece, as the LLNL team investigated coating methods in hopes of adopting further process improvements this year. Coating and permeation barrier research is described in Task #3. Although encouraging coating results were obtained with permeation test coupons, neither funds nor time were available to scale candidate coating processes up to pretreat 48 inch long liners. As the Annual Performance Review approached and prospects for scaling up a coating process dwindled, the decision was taken to build more tanks like Tank #1 and obtain burst data confirming the team's P_bV/W performance predictions.

Tank #2 did not pass the leak test prior to proof testing, and its manufacturing failure prompted LLNL to seek ATL help by duplicating Thiokol's preshrinking process step. That prototype

leaked through spiral cracks that formed around just one of its bosses during a Thiokol attempt to heat treat its tank liner horizontally. One boss was driven in the oven by a motor, but the other was retained on a ball bearing fixture which had slight rotational drag. That end developed the spiral cracks, whereas the 'successful' Tank #1 had been heat treated in a vertical orientation. ATL retained the rotational mold tooling and set it up again to perform a heat treat after molding without removing the part from its mold. The rotational mold's motion capabilities provided a superior substitute to horizontal spindle support, avoiding the slumping and hub drag problems encountered at Thiokol in horizontal heat treat process development. Tank #2 was sawed in half perpendicular to its long axis for debugging purposes, and subsequently furnished two demonstration articles (one was shown at the Annual Review).



Figure 3 – Remains of Test Tank #3 after Premature Burst

Figure 3 shows the remains collected after prototype Tank #3 was burst. Tank #3 was built with ATL's first heat treated 6 pound liner, and passed its 7500 psig proof test without any problems. Initially its burst test was thought to be a successful milestone in this research effort's performance goals. It burst at a pressure initially thought to be several percent above predictions. Subsequent recalibration of that test pressure sensor uncovered a test site operator error. The wrong sensor/preamplifier combination was reading pressures at 2/3 of actual. This calibration error triggered a full scale fact finding investigation at Thiokol Propulsion.

Direct evidence from the carcass shown in Figure 3 and the video frame shown below in Figure 4 helped to explain why Tank #3 burst at 70% of design pressure. The accidental canceling of human errors on the test stand and this premature failure mode obscured the dismal results for several days, until the confirmed mismatch of pressure sensor and preamp established the low actual value for burst pressure of Tank #3. Understanding why the burst pressure fell so far below predictions sent Thiokol fact finders in two directions: detailed failure analysis and reverse engineering of what had been built. Winding patterns of the as-built tank were extracted and resubmitted to Thiokol design codes.

Figure 4 shows a video frame captured at the instant when Tank #3 was burst. This evidence agreed with the location of the end dome separation visible in Figure 3, confirming that premature failure occurred near the tangent line between cylindrical section and end dome. Reverse engineering with Thiokol design codes showed great sensitivity to the exact pattern of winding in that region. The Thiokol investigation uncovered a number of problems, including subtle but significant differences between what was modeled and what was actually built in this sensitive region of the tank design. Great detail and a record of this extensive simulation effort that reverse engineered hoop wound fiber termination patterns and their consequences is given in Appendix 1 (see Appendix C therein).



Figure 4 – Video Captures Localized Failure During Tank #3 Burst Test

Additional modeling and oversight was put in place in order to improve the design of Tank #4 and Tank #5, which provided the only affordable attempts to rectify implementation errors presumed to be built into Tank #1 and the sectioned halves of Tank #2. These measures were deemed adequate to assure that the last two prototype tanks to be built would match an improved design. The fate of Tank #1 was decided in the few days before the Annual Review, it was worth more as a burst data point of a design known to be defective than as a showpiece that needed apologies. It burst at 85% of its design burst pressure, confirming the process variability and the sensitivity to details of tank designs with the wrong hoop wound fiber termination locations. This result confirmed the pattern observed at Thiokol by other tank designers – that failures in the tangent line are much less repeatable than failures in the sidewall. Avoidance of premature dome failures and boss failures constrained the improved designs and their prevention appeared to require additional weight, but an added wafer incorporated during winding in the end domes kept the improved design weight growth below 0.1 kg.

Tank #4 was fabricated and burst at 10,464 psi, which is 93% of its 11,250 psi design prediction for burst. The failure mode was not conclusively isolated, although the dome and boss regions did not appear to fail. It was not clear whether failure was in the cylindrical section or the transition from the cylindrical section to the dome. Very little of this tank was left as a helpful carcass, and no dramatic moment was captured on video to indicate where failure initiated. Thirty three

milliseconds between video frames was clearly too long to capture the failure event, one frame showed a complete tank and the next showed no tank left!

Tank #4 had an estimated internal volume at ambient pressure of 8,600 in³ (or 8,800 in³ at 5,000 psi working pressure). The tank weight was 52.8 lb (excluding 1.2 lb of fiberglass used to hold a label). This corresponds to a burst performance factor ($P_b V/W$) of 1.74 million inches (44.3 km) or 11.3% H₂ by weight. However, the safety factor of this design is only 2.09 compared to 2.25, if tanks identical to Tank #4 were operated at 5000 psig. This roughly 7% loss in performance is significant, and the data remaining from the burst test of Tank #4 was insufficient to resolve how the performance was lost or what to do in order to recover it.

The unsatisfactory state of understanding after Tank #4 burst would have left this highly visible research effort inconclusive. IMPCO Technologies agreed to pay for design and test of Tank #5 as a means of helping the entire DOE-funded tankage program and acquiring technology from Thiokol Propulsion, who recently became their strategic partner. Therefore, the final report from Thiokol to LLNL (Appendix 1) only discusses Tanks #1-4.

Design of Tank #5 explored numerous options, but ended up close to the design of Tank #4 to minimize the risk of new problems. A single additional hoop wrap was added to bring the new design towards the desired burst pressure with a slight improvement in projected $P_b V/W$. Higher speed video (~400 frame/second compared to the ~30 frame/second that was affordable for earlier testing of Tanks #3, #1, and #4), redundant pressure and strain sensors, “belly band” sensors at both tangent lines and mid-tank, and standard test video were all used to monitor the burst of Tank #5.



Figure 5 – Condition of Tank #5 before Highly-Instrumented Burst Test

Figure 5 shows the initial condition for the burst test of Tank #5. It was digitized from the early frames captured by one of six high speed cameras employed in this highly instrumented test. These six cameras were set up to record ~400 frames/second and were run in pairs, with slight overlap to ensure that both sides of the tank under test were covered for almost 3000 psi of final filling time at the pumping rate of Thiokol Test Slab #9. These cameras were fast enough to capture the failure event that was missed during the burst of Tank #4. Many frames from the middle pair of cameras show water being ejected and the painted grid (visible on the outside of tank, with 2 inch grid spacing, in Figure 5), but the frame shown in Figure 6 best shows the failure event. As with tank #4, very little for Tank #5 remained after burst, and 30 frame/second video was useless because the tank failed in less than the two frames of the high speed camera (~5 milliseconds). Expert observers were impressed by the speed and thoroughness of the burst.

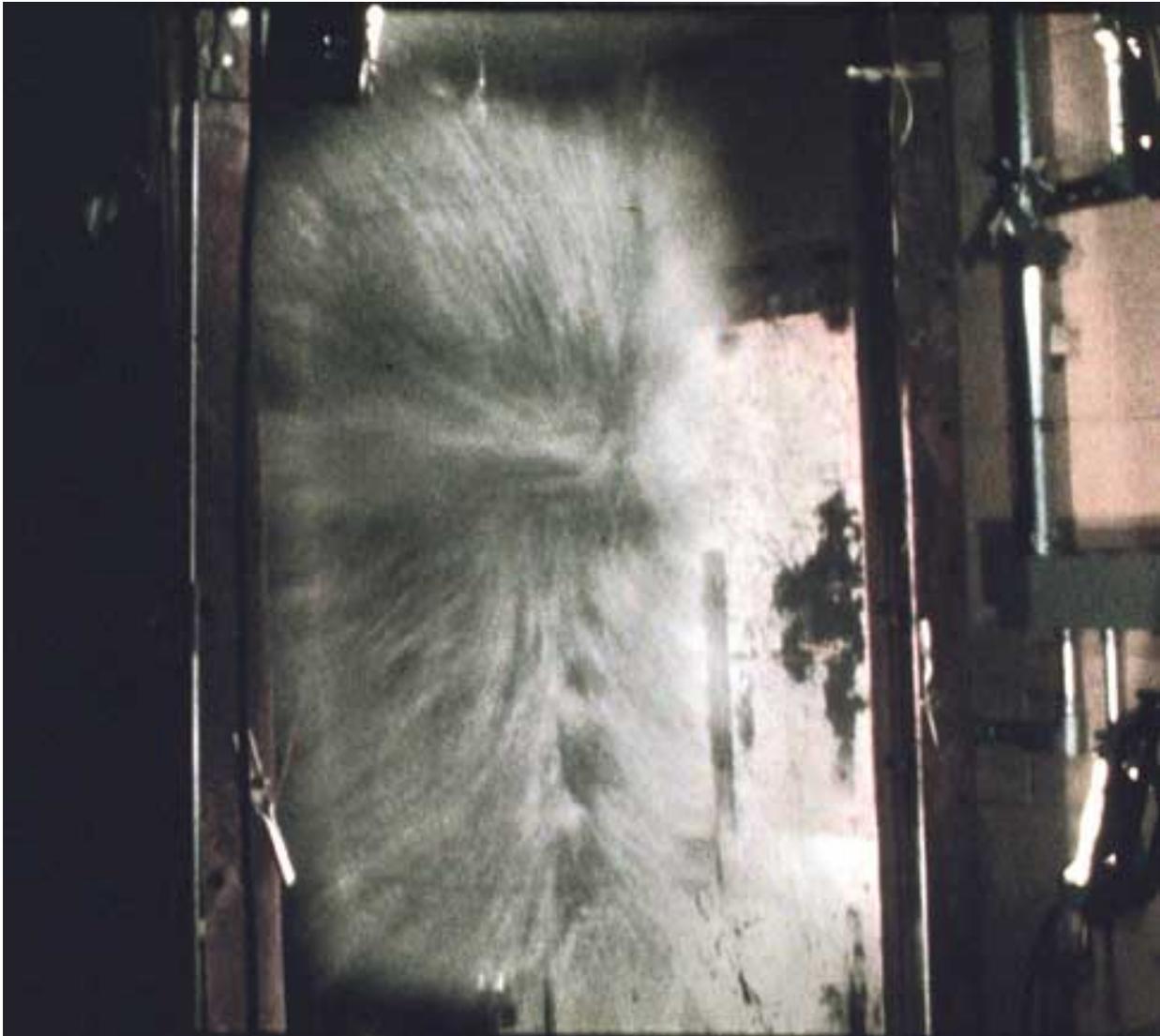


Figure 6 – Best High Speed Camera Frame Captures Tank #5 At Burst

Figure 6 shows the burst of Tank #5 on July 13, 2000. Performance was again ~7% below the performance that was expected for T1000G fiber, quite consistent with the results from Tank #4. Although it is not clear what caused this ~7% performance loss, it may be due to the pressurization sequence used for winding on inflatable mandrels that has not yet been optimized. The additional hoop wrap in Tank #5 delivered increased burst pressure, with comparable volume, and increased weight (compared to Tank #4). The burst pressure was 95.55% of the 11,250 psig design. Tank #5 had a comparable $P_b V/W$ to Tank #4, but the measurement errors are known more precisely (specifically the internal volume measurement).

The importance of this published record, prompted LLNL researchers to seek traceable accuracy on all the measurements reported and inferred in Table 1. If the service pressure for Tank #5 is considered to be 5,000 psig, then the safety factor is only 2.15 (as opposed to the desired 2.25). The percent hydrogen by weight and specific energy shown in Table 1 assumed a service pressure of 4777 psig (32.94 MPa) to keep the safety factor fixed at 2.25.

Table 1. 11.3% H₂ by Weight was Demonstrated for a High Cycle Life Cylinder

Performance Variable	Unit	Measurement	Std. Deviation	3 Sigma Error
Burst Pressure (P _b)	psig	10,749	14	42
Internal Volume at 5000 psig (V)	in ³	8,758	7.6	23
Tank Weight (W)	lb	54.0	0.018	0.054
Performance Factor P _b V/W	inch	1.743 E6	0.0028 E6	0.0083 E6
Specific Energy (tank + H ₂ , LHV)	Wh/lb	1708	2.4	7.3
Percent Hydrogen by Weight	%	11.31	0.016	0.048

Performance Variable	SI Unit	Measurement	Std. Deviation	3 Sigma Error
Burst Pressure (P _b)	Pa	74.112 E6	0.097 E6	0.290 E6
Internal Volume at 34.5 E6 Pa (V)	m ³	0.1435	0.00013	0.00038
Tank Weight (W)	N	240.2	0.080	0.240
Performance Factor P _b V/W	m	44.27 E3	0.070 E3	0.211 E3
Specific Energy (tank + H ₂ , LHV)	Wh/kg	3767	5.2	16
Percent Hydrogen by Weight	%	11.31	0.016	0.048

Considerable discussion and several alternative approaches were considered to calculate tank internal volume at service pressure. IMPCO makes use of a superior and simple technique to measure water volume by weighing additional water before it is pumped into a tank being hydrotested to maximum expected operating pressure (MEOP). Since Thiokol test facilities were not rapidly reconfigured to weigh the water going in to their high pressure pump, the entire tank was weighed by a load cell during its cycle through proof pressure, and weight was noted particularly at MEOP of 5000 psig. Internal volume calculations at 5000 psig were performed using the methodology described by the Compressed Gas Association (CGA 1996). The volume calculations using this methodology were within 1% of volume estimated by Thiokol design codes, and within 1% of geometry models used by LLNL spreadsheets. All the error analysis of measured and derived performance parameters given above assumes Gaussian-distributed independent random error contributions from multiple calibrated error processes in each sensor/instrument employed.

Although the volume of Tank #4 is expected to be very similar to that of Tank #5, it was not measured directly with water. If the volume measurement from Tank #5 (8758 in³ at 5000 psig) is used for Tank #4, instead of the 8800 in³ estimated by geometry and strain, then the P_bV/W for Tank #4 is 1.74 E6 inch (44.1 km). By this criterion, the percent hydrogen by weight for Tank #4 is 11.2%.

Recommendations based on preliminary fact-finding after the Tank #5 burst test are still being developed in detail. A list of suggestions is included in the final section (Conclusion and Recommendations).

More detail on the recent progress in this task is in the report from Thiokol Propulsion to LLNL, which is included as Appendix 1.

2. Specify a ~1 yr program to certify tankage with 7.5-8.5% hydrogen by weight (5000 psi, 300 K, SF 2.25). Write, modify, and review a solicitation to develop lightweight hydrogen tankage. Technically direct the program that commenced in May 2000 (IMPCO Technologies).

The technical advantages of lightweight pressure vessels for vehicular hydrogen storage are not in doubt, but eventual adoption depends on high volume price reductions as well as public acceptance. Industrial partners are vital to the production of near-term tank technologies in quantities sufficient to support demonstration projects. Only tank technologies that can be reduced to commercial manufacturability over the next year or two can advance the entire hydrogen powered vehicle effort through the integration phases that lead to vehicle demonstrations and public acceptance. Two DOE funded demonstration vehicle projects are almost ready to adopt such near-term lightweight pressure vessels for onboard hydrogen storage. DOE/Golden contract DE-AC36-GO10494 that LLNL is directing will demonstrate vehicle ranges acceptable to the public.

Because of the low density of hydrogen, tanks that give acceptable range are difficult to fit within existing vehicle designs. The problem of how to best accommodate hydrogen storage aboard vehicles has generated numerous solutions over the past few years. DTI has extensive experience working on this problem for Ford, and has advocated a single large tank under the rear seat for a Ford demonstration vehicle. Figure 7 illustrates some of the packing issues DTI captured in the specification process for tankage and suggested to LLNL roughly two years ago, which led LLNL to direct its research toward the 18 inch diameter by 48 inch length tankage that was explored by the research effort described in Task #1.

Single Cylinder is Lowest Tankage System Cost & Complexity but Requires Car to be Designed from the Ground Up with that Priority

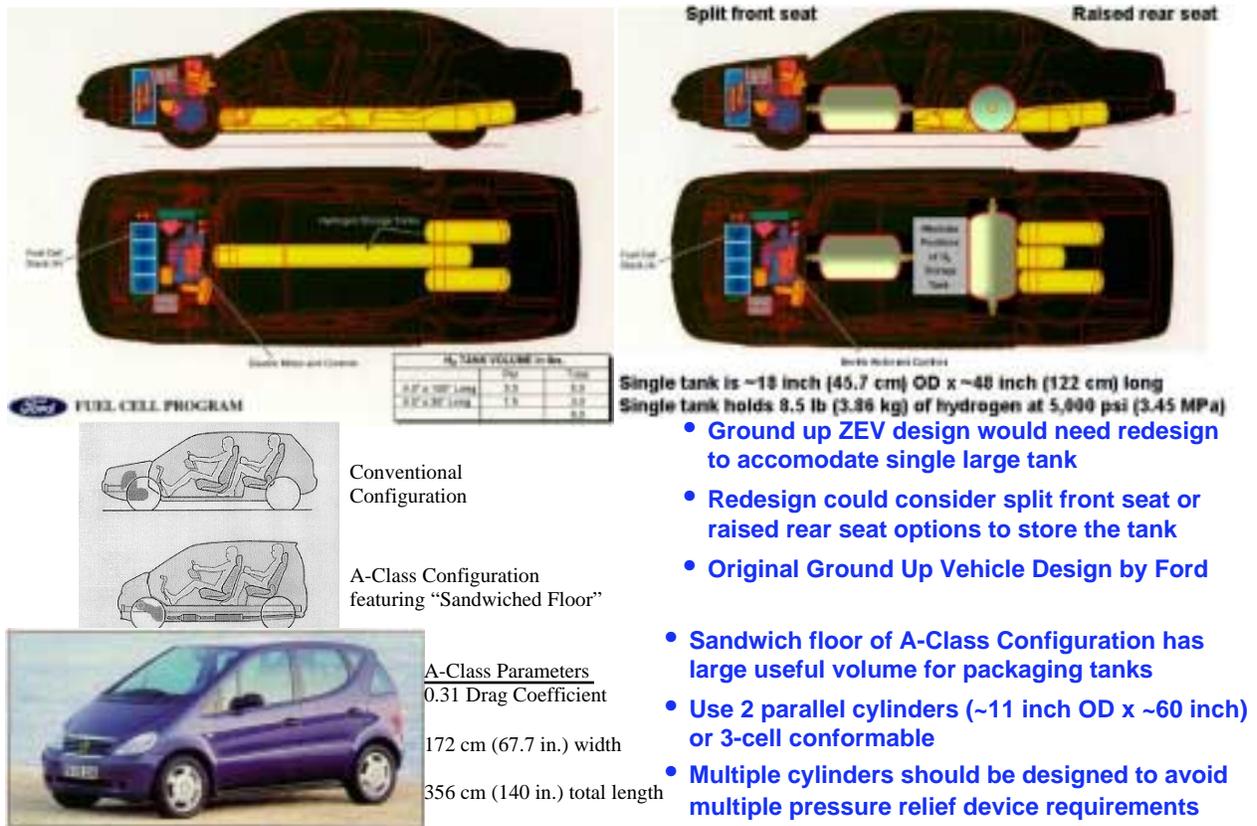


Figure 7 – Tank Size and Location Considerations for Vehicular H₂ Storage

Figure 7 illustrates the problem of packing 3.6 kg of hydrogen at 5000 psi into a passenger car, if sufficient volume has not been designed in from the ground up. A single large cylinder (~46 cm OD x 122 cm long) can be placed under a raised rear seat or between split front seats. Alternatively a ~30 cm OD x ~270 cm long tank can run the length of the car. If the OD is kept to ~30 cm or less, packaging can be done into a sandwich floor construction, like that found in the A-Class configuration. Preliminary design information on GM’s recently unveiled hydrogen-powered Opal appears to use a similar under-the-floor tank configuration.

Besides finding the volume for both tanks and passengers, a complex number of safety and regulation issues remain to be resolved before new hydrogen storage technology can be deployed in widespread applications. The first demonstration vehicle project for lightweight tanks is the Nevada Bus project, which operated as the Savannah River bus in previous years. Any moderate advance in Type IV hydrogen pressure vessels will give this bus a significant range increase.

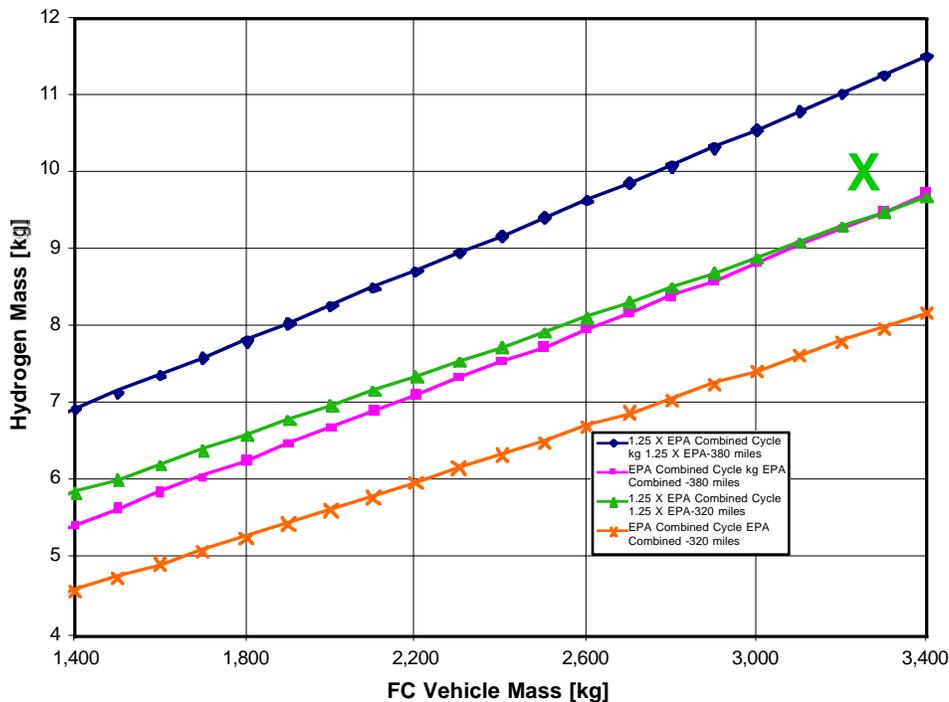
Design of near-term producible tanks for DOE to solicit from industry for the Nevada Bus project was straightforward because the demonstration vehicle project is already underway (linked to a DOE hydrogen infrastructure demonstration in Las Vegas), and because it can accept and benefit from uncertified tanks that provide a modest advance in performance. Plenty of room on the roof of the bus avoided many of the cramped vehicle configuration issues posed by modifying existing automobiles. Various configurations of rooftop tankage this demonstration

vehicle might install are shown in the brief discussion of Task #6. Essentially all of the vehicle installation design for this project was supplied by the Nevada Bus team, once LLNL decided in the interests of delivery time to base early phases of the DOE 2000 tankage solicitation on existing 18 inch tooling.

The other DOE hydrogen vehicle demonstration project that LLNL specified when the DOE tankage solicitation was written in early FY00 is known as FutureTruck, and it involved much more design attention. This SUV is bigger than a car, but still based on an existing automotive design, and its range will be set by to the amount of hydrogen that can be stored onboard. Volume constraints in the underside of the SUV chosen for FutureTruck limit the tank outside diameter to ~11 inches, as opposed to the 18 inch diameter tooling that was already available. Since new tooling will be required, initial lightweight tankage developments will be done for delivery to the Nevada Bus program on existing tooling.

Extensive assistance from DTI, General Motors, and Argonne National Laboratory staff who oversee the FutureTruck competition helped in setting the tank design requirements. Foremost was the specification of the amount of hydrogen such a heavy vehicle (3265 kg ‘curb’ weight) would need to carry. DTI performed the sizing analyses that are shown in Figure 8. Due to volume constraints and a programmatic desire to keep maximum storage pressure to 5000 psi (35 MPa), LLNL chose a configuration with ~10 kg hydrogen storage (marked with a green X on Figure 8). This specification should enable modified SUVs to exceed the minimum requirement of 320 mile range (assuming 1.25 x EPA Combined driving cycle).

DTI analysis of H₂ mass requirement vs. FC vehicle mass



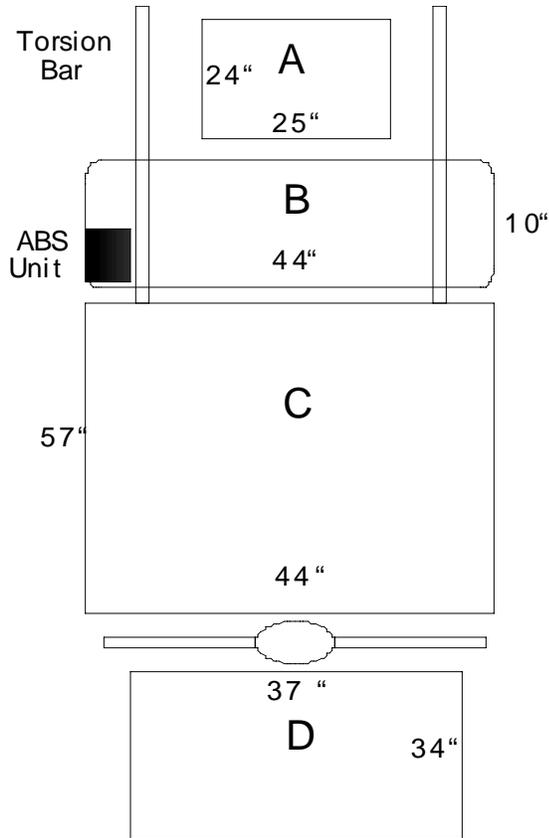
X – Design point for GMT 830 K 15906 (3265 kg GVW prior to retrofit)

Figure 8 – Hydrogen Requirements for FutureTruck

After selecting the amount of hydrogen FutureTrucks ought to carry, a level playing field among solicitation bidders required the detailed specification of tank sizes and masses. Mass specifications were set to percent hydrogen by weight levels that would be truly enabling for hydrogen fueled vehicles, that were still low enough to give multiple bidders high likelihood of developing safety certifiable tanks in the short time available (before the FutureTruck competitions moved on to a different vehicle design in 2002). LLNL's earlier experience with the DOE/Ford demonstration project argued strongly against trying to hit a moving target whose geometric requirements could be very different, and thus technical ambition on these tanks was held to 8.5% hydrogen by weight in order to deliver certified tanks with >90% probability early enough for at least two competing teams to integrate on their vehicles (before May 2001, with the contest scheduled in July 2001).

Because the academic teams modifying FutureTrucks do not have the engineering manpower resources to undertake safety modifications, tanks delivered by the solicitation had to be fully safety certified before delivery to avoid any DOE liability. Although LLNL is advocating modification of the hydrogen safety standard for permeation (as discussed below in Task #7), the success of that advocacy could not be foretold when the solicitation was written, so its terms merely include the possibility of relaxing the required hydrogen loss rate in this one safety test. Similar safety uncertainties precluded installing hydrogen tanks anywhere besides inside a raised vehicle floor, since roll and side-impact safety could not have been developed within available human resources and time. With sufficient installation engineering manpower, some of the experts in LLNL-directed conference calls with ANL and GM would have preferred roof mounted tanks.

Figure 9 summarizes the tank placement issues that constrain possible tank geometries for FutureTruck SUVs modified to store ~10 kg of hydrogen. Although lifting the vehicle with an after-market "lift kit" modification was a straightforward way to gain safely in tank diameter, safe ground clearance and a maximum GM-recommended lift of 3 inch restricted under-the-floor tank outside diameters to ~11 inch. It initially appeared that tanks must occupy most of the crowded volume inside the vehicle frame shown below in Figure 10 in order to store 10 kg of hydrogen at 5000 psi. LLNL experts designing the solicitation sought interest in higher pressure designs from both auto manufacturers and FutureTruck contest staff, but late in 1999 the hydrogen demonstration vehicle community remained skeptical of pressure ratings above 5000 psi. By mid-2000 this situation has changed dramatically, and can only be mentioned briefly in the discussion of Task #5 below because an increased scope for the ongoing solicitation is being prepared as this report goes to 'press'.



Area 'A' would require **removing the Transmission and Transfer case**, but without a prop shaft - what good are they anyway? The assumption here being at least two motors (1 driving each axle). **Redesign of the Transmission crossmember** is required to allow for tanks to pass further forward in the chassis.

Area 'B' would require moving the Torsion bar hanging cross member and the tank shield. These would need to be relocated forward and reattached. This requires **new torsion bars** (due to the change in length). The **ABS unit would need to be relocated** as well, but without the converters from the IC engine, there should be room.

Area 'C' in the Diagram is currently available with **removal of the Prop shaft and Exhaust**. There needs to be some additional dimensional checking to insure that the 11" OD tanks would not protrude beneath the frame line.

Lifting a "Body on Frame" vehicle is not a difficult design task, and after-market companies will most likely have kits available very soon.

Area 'D' would require **relocating the spare tire**. A very simple design change, though probably not as pleasing to the Marketing group. Also the volume in area 'D' may not allow use of the 11" diameter tank.

Figure 9 – Hydrogen Tankage Placement Issues for FutureTruck

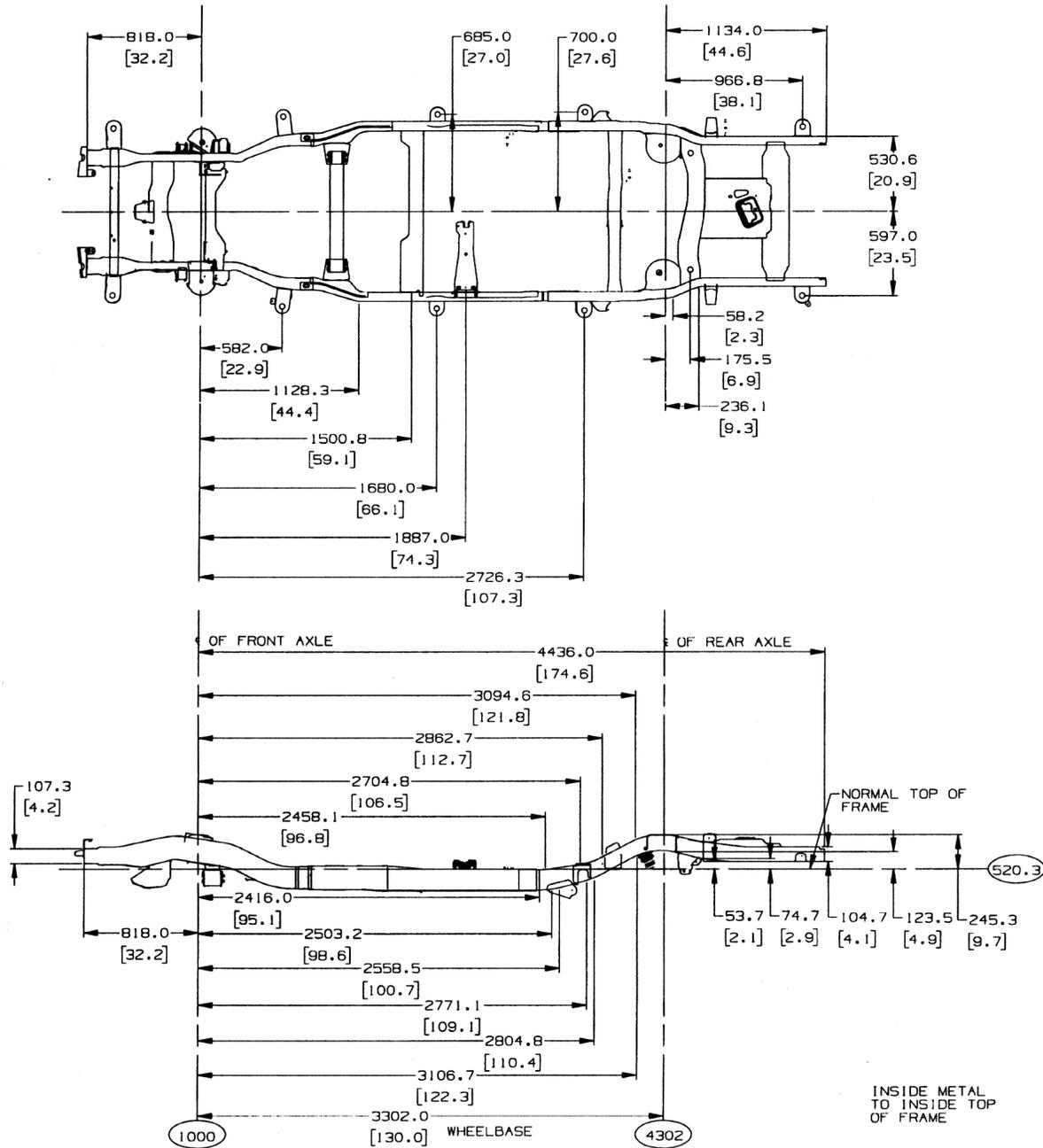


Figure 10 – Preliminary GM Suburban Rail and Crossmember Arrangement

Some cleverness and detailed consideration of the certification requirements of two tank designs that differ only in length allowed two length specifications to join with the 11 inch maximum diameter specification and totally describe the shape of tanks that multiple bidders would be required to quote. This pair of lengths contains the requisite 10 kg of hydrogen in four tanks with MEOP of 5000 psi, in the configuration shown below on Figure 11.

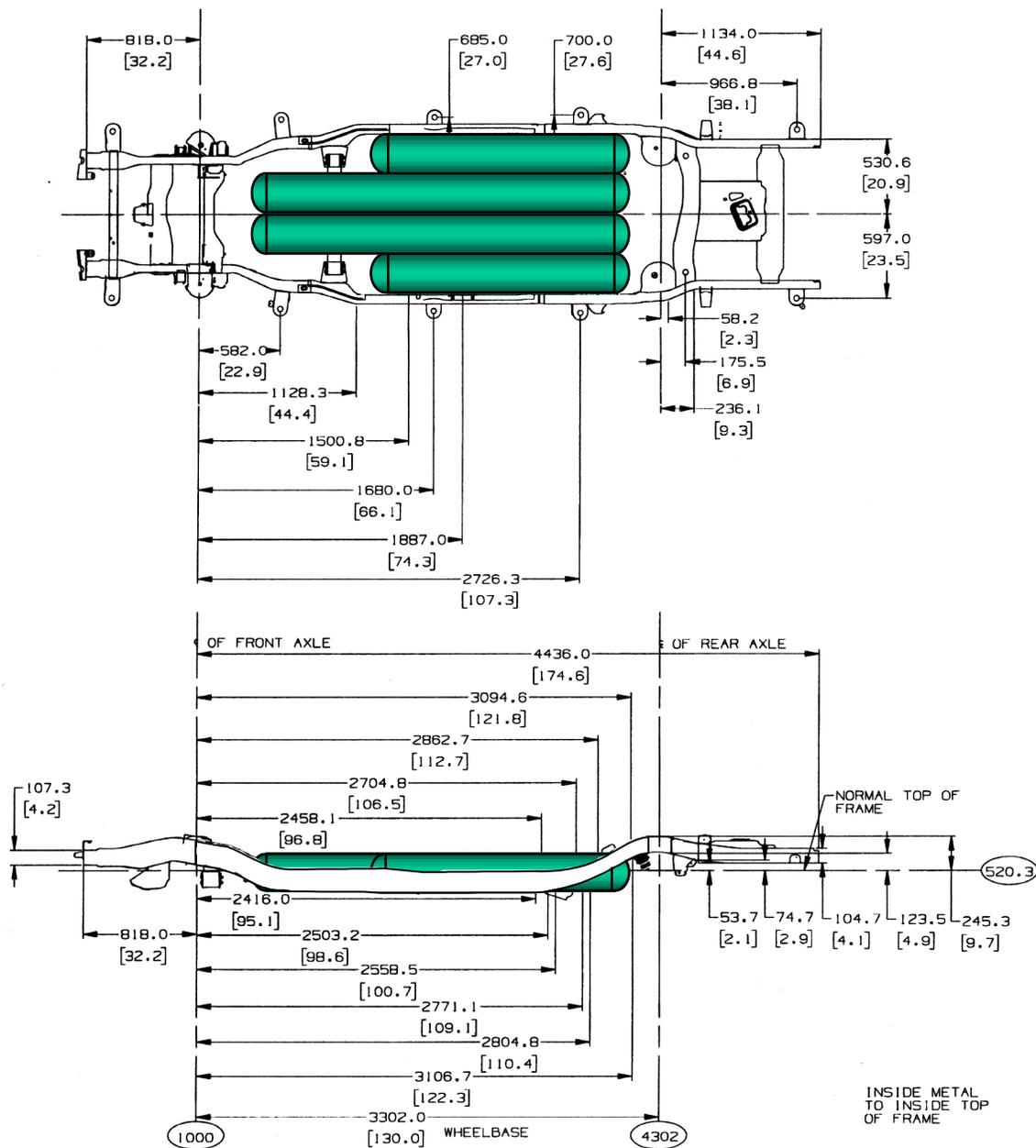


Figure 11 – Proposed H₂ Tank Placement for FutureTruck (GMT 830 K 15906)

Many other considerations applied to the design of tanks and program. These had to be captured in the requirements wording of the Solicitation itself. Figure 12 below reproduces the crucial information from the Solicitation, which assembled all specifications for the four phases chosen to structure this effort. The desire to solicit competing bidders precluded contract language that would have allowed non-cylindrical pressure vessels to occupy the same tank envelopes. Since only Thiokol would be able to deliver such ‘conformable’ tank technology (Golde 1999, Haaland 2000), its advantages for hydrogen storage were specifically not addressed by this solicitation. Thiokol did not respond to the Solicitation directly.

Carbon fiber cost barriers and perceived safety risks and consumers' desire for vehicle ranges typical of gasoline powered vehicles are issues that apply to conformable as well as cylindrical tanks. Although cylindrical tanks could provide lighter weight, less expensive storage, the design traditions and existing practices/tooling/workforce puts significant burdens on the kind of "ground up" vehicle design that could benefit from single large cylinders of the sort DTI advocated in 1998, for which LLNL had already developed advanced, government-owned tooling.

The scheduling of four phases was very tight, allowing the bare minimum of time to innovate on cylindrical tank mass performance, test to reduce risks of failing safety certification, and then produce batches large enough to deliver multiple test articles to the demonstration projects after 14 identically-manufactured tanks pass safety certification tests. The lower performance Nevada Bus phases were designed to accelerate learning and deliver the lessons of manufacturing and testing from the easier first two phases to the more challenging mass performance requirements of the final two (FutureTruck) development phases. Man months of detailed attention, rewritings, and conference calls between LLNL and the DOE/Golden contracting office further stress this contracts schedule with the real work of hammering out a Federal Contract.

	Phase 1	Phase 2	Phase 3	Phase 4
Vehicle requiring tanks	Nevada Bus		FutureTruck	
Acquired Tank Generation	First Generation		Second Generation	
Phase Begins	Apr '00	Oct '00	Oct '00	Jan '01
Mission of Phase	Risk Reduction, Tooling	NGV2 certify for H2, Demo Bus	Risk Reduction, New Tooling	NGV2 certify for H2, Demo Suburban
Tank Diameter (O.D.)	18 inch (46 cm)		11 inch (28 cm)	
Tank Length (approximate)	48 inch (122 cm)		69 inch (175 cm) (A) 93 inch (236 cm) (B)	
Tank Service Pressure	5,000 psi (34.5 MPa)		5,000 psi (34.5 MPa)	
Burst Pressure (minimum)	11,250 psi (77.6 MPa)		11,250 psi (77.6 MPa)	
Hydrogen Contained (34.5 MPa, 300 K)	7.9 lb (3.6 kg)		4.6 lb (2.1 kg) 6.3 lb (2.9 kg)	
Quantity of Tanks Produced	10	22	10 (A) 4 (B)	20 (A) 8 (B)
Quantity of Tanks Tested	10	14	10 (A) 4 (B)	14 (A) 2 (B)
Minimum %H2 by weight (34.5 MPa, 300 K)	7.5%		8.5%	

(A) Applicable to the 69-inch length

(B) Applicable to the 93-inch length

Figure 12 – H₂ Tankage for Nevada Bus Program and FutureTruck Competition

Figure 12 compresses the contractual requirements of four phases into a concise list of technical requirements. Many other contractual clauses had to be adapted from standard government practice. Economic performance of the resulting tankage was not included explicitly in the solicitation, but plays a strong implicit role due to the “modified step-and-half” form of Solicitation that the LLNL/Golden team chose. That form awarded the contract to the lowest bidder only after bidders had been downselected for competence to deliver the very stringent technical requirements. Low cost manufacturers would win this solicitation, but only if they were capable of delivering the requisite high (7.5% and 8.5% hydrogen by weight) mass performance requirements.

This contract was awarded to a team of IMPCO and Thiokol in May of 2000. Since that time, considerable technology transfer has infused IMPCO's future product line with Thiokol's aerospace (high performance) tank fabrication expertise, and the transfer of LLNL thin-wall rotational molded liner technology into Phase 1 efforts is proceeding on schedule. A loan agreement (LLNL Loan Agreement # 101-2485) was generated to enable IMPCO to borrow and modify the government-owned mold, which is being supplied as government furnished equipment. A preliminary design for the boss and shell of tanks in Phase I has been established. Capital equipment for testing and fabrication has been specified and ordered. Process trials with tow-preg materials (Thiokol Propulsion's method of winding) have commenced at IMPCO. The high probability of success plans built into the solicited tank development program production and test schedules are on track, and should soon be generating risk reduction test data such as the extra stringent (hydrogen fill not required by safety certification) bullet test conducted and passed by Thiokol on one of their conformable tanks shown in Figure 13. The Thiokol conformable tank program was technically directed by LLNL and is discussed in (Haaland-2000).



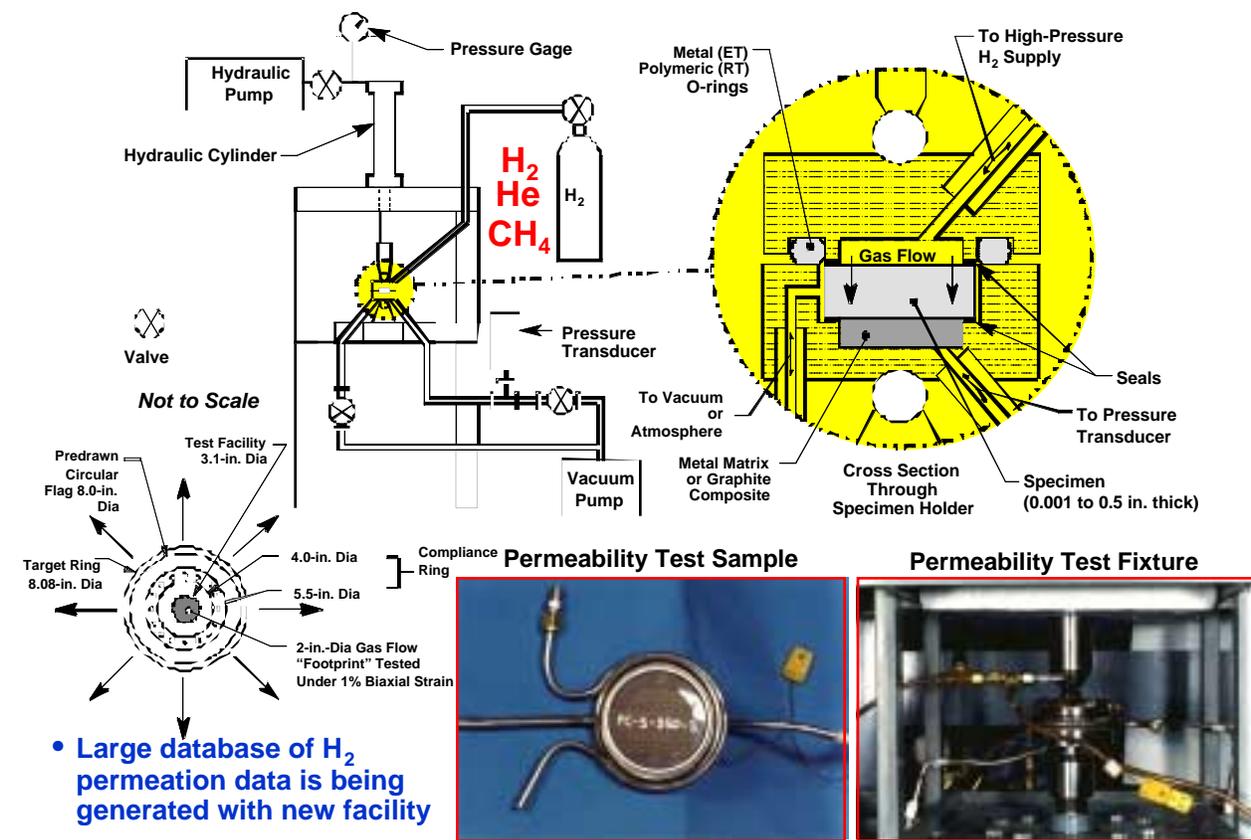
.030 caliber armor piercing bullet impact at 45° to one cell side wall of a conformable tank pressurized to 5000 psi (34.5 MPa) using hydrogen gas caused **no fragmentation failure**

Test with 5000 psi Hydrogen – No Deflagration

Figure 13 – Conformable Tankage Fabricated by Thiokol Passed Bullet-Test

3. Develop a lightweight liner fabrication process and permeation reduction coatings that are easily adopted by industry. Optimize fabrication of variable thickness rotomolded liners. Develop metal and plastic permeation coatings, permeation test coatings, fabricate tanks with coated liners (if funding permits).

The LLNL effort to develop the next generation of advanced hydrogen tankage was able to take advantage of existing SRI capability to further a broader understanding of hydrogen permeability. Thiokol internally funded (in 1998-1999) a new facility at SRI capable of permeation testing at high pressure (up to 5000 psi or 34.5 MPa) with hydrogen, as well as an unprecedented fixturing arrangement that enabled the first-ever collection of permeation test data under controllable biaxial strain at the levels anticipated in tank liner service (Golde 1999, Haaland 2000). This test capability has been made available with Thiokol's permission for LLNL research, and is illustrated in Figure 14.



Tests with gas ΔP up to 5 ksi (34.5 MPa) as a function of temperature & biaxial strain

Figure 14 – Schematic and Photos of Permeability Test Fixture at SRI

LLNL took advantage of an earlier hiatus in Thiokol's testing to procure ASTM-traceable calibration for all subsequent measurements, confirming previous measurements made at LLNL on LLNL-developed liners. LLNL, the USAF, and Thiokol IR&D funded significant additional hydrogen permeation testing on a variety of candidate liner materials, as a function of pressure (Mitlitsky 2000, Mitlitsky 1999-a,b,e, Souers 1986). The graph in Figure 15 not only confirms the hydrogen permeability of several previously employed liner materials, it extends the sparse earlier results to a much wider range of pressures, temperatures, and materials. This new

database, and the literature survey summarized in (Mitlitsky-1999) have been used to assess many relevant materials' acceptability as thin liners for high pressure tankage. Downselection is currently proceeding in parallel with rotational molding process development to produce LLNL's next generation of advanced liners (sufficient to enable DOE 2000 Goals).

A permeability of 1×10^{-14} corresponds to a "half-life" of 6 months for 0.15 inch (3.8 mm) thick liner

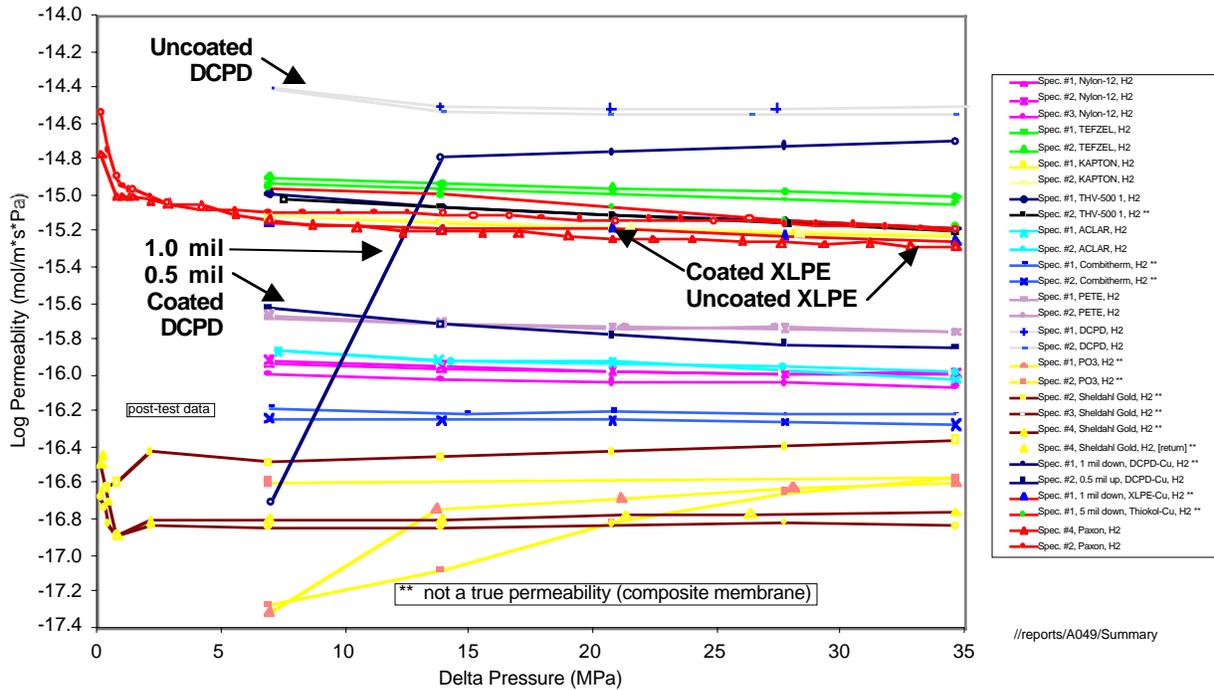


Figure 15 – Measured Hydrogen Permeability of Several Candidate Liner Materials

Metalized samples of dicyclopentadiene (DCPD) and cross-linked polyethylene (XLPE) that were manufactured by Epner Technology were subjected to permeation testing at Southern Research Institute (SRI). Metalized samples of XLPE that were manufactured by Thiokol were also tested at SRI. All samples were screened for pinholes using bright light illumination through the back of the samples. All of the XLPE samples from Epner Technology and two of the DCPD (one of the 0.5 mil thick and one of the 1.0 mil thick metal coated) samples have visible holes/cracks through the coating as detected with back lighting. SRI measured one of the remaining 1.0 mil thick metal-coated DCPD samples that has no visible holes. This sample showed a ~2 order-of-magnitude decrease in H₂ permeation at 1000 psi (7 MPa) delta-pressure compared with an uncoated DCPD sample. However, at 2000-5000 psi (14-35 MPa), the sample showed only modest improvement compared to uncoated DCPD samples. The curve from this sample is labeled in Figure 15 "1.0 mil Coated DCPD" and shows a rapid rise in log permeability as a function of pressure between 7 and 14 MPa.

Examination of the metalized sample when removed from the permeation test rig showed that trapped H₂ bubbles between the DCPD and the film caused delamination and tearing of the film. The tearing is consistent with the 2 order of magnitude increase in permeation measured at 2000 psi (14 MPa) compared to 1000 psi (7 MPa) delta-pressure.

Metal-coated XLPE samples from Thiokol did not appear to have pinholes when examined with bright light illumination through the backside. Permeation testing of metal-coated XLPE samples from Thiokol did not show an improvement compared to uncoated XLPE at any delta-pressures that were measured (7-35 MPa). Examination of the metalized XLPE sample when removed from the permeation test rig showed that trapped H₂ bubbles between the XLPE and the film caused delamination and tearing of the film, which must have occurred at delta-pressure below 1000 psi (7 MPa). Both of these metalized substrates were measured with the metal coating toward vacuum (high pressure applied from the uncoated side of the substrate).

LLNL and SRI decided to test the 0.5 mil thick metalized DCPD substrate with the metal oriented towards the high pressure H₂ supply (uncoated side of the substrate towards vacuum). This sample showed > 1 order-of-magnitude reduction in H₂ permeation at all pressures from 1000-5000 psi (7-35 MPa). There were no signs of film delamination or tearing when this sample was removed from the permeation test rig. This sample serves as an existence proof that metalization of plastic samples (such as DCPD) can achieve sufficiently low H₂ permeation at 5000 psi (35 MPa) to enable thin liners (< 3 mm thickness) to meet the stringent permeation specifications that have been proposed for hydrogen pressure vessels. This experiment also shows that initial pressure cycling of such a metal coated plastic sample does not fail the permeation reduction capability of the coating.

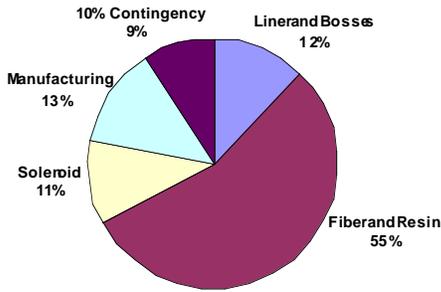
Designs of lightweight tankage using metal-plated molded plastic liners were further refined using the latest results from coating experiments and permeation tests. Methods for addressing issues of liner shrinkage during cure were successfully. Specifically, a post-annealing step of the liners prior to metalization was performed on an additional set of liners procured by LLNL and fabricated by ATL. A cross-linked polyethylene (XLPE) liner (12 inch diameter x 20 inch length) was prepared and coated with a permeation reduction coating by Thiokol Propulsion in May. This work will be put on hold until additional funds are available.

4. Develop lightweight hydrogen tankage with SF 2.25 and capture cost projections from DOE tank solicitations. Demonstrate plastic lined composite tankage (cylinders and conformable) with safety factor 2.25.

Lightweight tank liners enable weight goals to be achieved with less expensive fiber. Tankage with safety factor of 2.25 uses ~75% of the fiber required for tankage with safety factor of 3. Since fiber cost dominates tankage cost, it is important to keep the safety factor low, while improving storage system safety in other ways. Revised economic evaluations are summarized in Figure 16, and show that high volume tankage (>500,000 unit per year) will cost ~\$640/unit for tanks that hold 3.6 kg H₂ at 5,000 psi service pressure using T700S carbon fiber (assuming \$6/lb cost of fiber). This result is significant because tankage now appears to be both less expensive and lighter weight when using T700S (at \$6/lb fiber), compared to Panex-33 (at \$5/lb fiber) because the strength-to-weight advantage of T700S requires sufficiently less fiber per tank to more than compensate for its higher cost per pound.

- Economic evaluations have been performed in conjunction with Thiokol Propulsion, Directed Technologies Inc. (DTI), Toray Composites, and colleagues at LLNL.
- The major cost driver is the cost of various grades of carbon fiber; large uncertainties remain in the projected fiber costs that may be achievable with high volume production, especially for T1000G (currently the highest strength-to-weight carbon fiber commercially available).
- Although use of T1000G will result in the lightest weight tanks, its current cost of ~\$70/lb (\$154/kg) must be compared to lower strength/less expensive carbon fibers, such as M30SC with current cost of ~\$28/lb (\$62/kg), or even lower strength/less expensive fibers, such as T700S with current cost of \$9-14/lb (\$20-31/kg) or Panex-33 with current cost of ~\$6-6.5/lb (\$13-14/kg).
- High volume cost projections for these fibers (500,000 units/yr) have been estimated by DTI (with new input from manufacturers) to be \$15-25/lb (\$33-55/kg) for T1000G (<\$40/lb will be very difficult according to Toray), \$6-7/lb (\$13-15/kg) for T700S, and ~\$5/lb (\$11/kg) for Panex-33; M30SC would have an estimated high volume cost of \$15-20/lb (\$33-44/kg).
- Assuming Panex-33 at \$5/lb (\$11/kg) with a high density polyethylene (HDPE) liner, the cost of a tank that is capable of storing 8.0 lb (3.6 kg) of hydrogen has been estimated by DTI to be \$841 (\$78 for liner and bosses, \$500 for fiber & resin, \$69 for solenoid, \$117 for manufacturing, and a 10% cost contingency).
- These assumptions project costs of \$105/lb (\$231/kg) of hydrogen stored or \$6.93/kWh.
- Repeating this analysis for a tank with similar capacity using new cost estimates for T700S, assuming a fiber cost of \$6/lb (\$13/kg), the estimated tank cost in high volume production would be ~\$640/unit (~\$80/lb or ~\$176/kg of hydrogen stored or ~\$5.28/kWh).

Tank Cost Breakdown (T700S)



Direct H2 Fuel Tank Subsystem

3.58 kg (7.9 lb) of H2 at 34.5 MPa (5,000 psi) stored in carbon fiber tank, integral solenoid and pressure relief device (PRD)

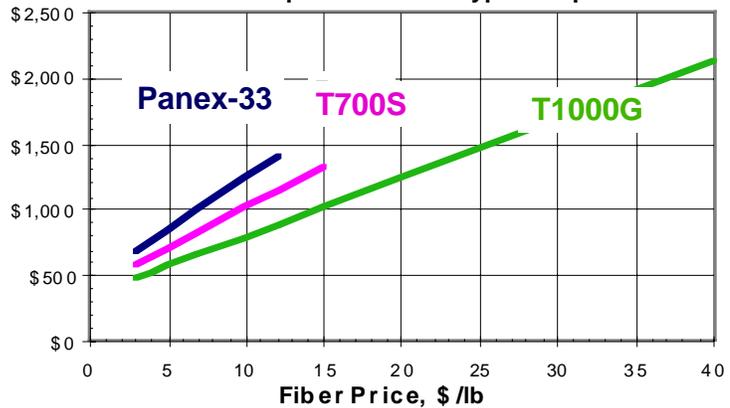
Liner and Bosses	\$78
Fiber & Resin	\$352
Solenoid	\$69
Manufacturing	\$83
10 % Cost Contingency	\$58

Tank System Cost **\$640**

Major Assumptions

- Use Toray T700S carbon fiber with manufacturer projected future price of **\$6/lb**
- Pressure vessels overwrapped on HDPE liner
- High production volume manufacturing (500,000 units/year)

Tank System Cost (pressure vessel & solenoid) as a function of fiber price with fiber type as a parameter



Fiber Type	Current Price \$/lb	Projected Price \$/lb
Panex-33	6-6.5	5
T700S	9-14	6-7
M30SC	28	15-20
T1000G	70	15-25 (40)

- Tank System Cost of **\$640** corresponds to **\$5.28/kWh** (DOE 2000 goal was **\$20/kWh**)
- Tank System Cost of **\$1015** corresponds to **\$8.24/kWh** using similar assumptions for **T1000G** with a future price of **\$15/lb**

Figure 16 – Economic Analysis of Carbon Fiber Tanks in High Volume Production

5. Technically direct a 10,000 psi (69 MPa) tankage development effort (pending a DOE/OTT funding award and/or additions to the current DOE/Golden contract DE-AC36-GO10494).

Addition of phases 5-8 to the four phase DOE/Golden contract DE-AC36-GO10494 has been discussed with the sponsor. Modifications to the technical specifications for that contract's Phases 1 through 4 allow timely development of 10,000 psi, safety-certifiable tanks that can drop in to a modified FutureTruck vehicle, and either extend the projected range by up to 2/3 or allow a 3-tank configuration (instead of 4), with all tanks chosen to be the same ~69 inch length (instead of the 2 different lengths chosen for the configuration shown in Figure 11). Design of the new phases includes the development and certification of in-tank regulator/valve/pressure-relief devices rated for 5,000 psi and 10,000 psi service. These ancillaries will contribute significantly to the safety of demonstration projects by sparing project team members the rigors of designing and protecting such high pressure plumbing. A draft Statement Of Work for phases 5-8 of the ongoing contract has been written, and is being seriously considered for DOE funding.

A demonstration vehicle for tankage fabricated under this extended program is being sought. FutureTruck 2002 is one possible candidate. The vehicle for this year-after-next competition will not be the same GM Suburban SUV, but 11 inch diameter tankage is likely to be applicable to it, as well as other demonstration vehicles by large auto manufacturers. Length modifications are relatively inexpensive to safety certify at a fraction of the cost of a diameter retooling. The near-term provision (perhaps within a year of Phase 4 completion) of commercial products derived from DOE funded developments under this solicitation by IMPCO makes the 11 inch diameter envelope very attractive for future vehicle integrators and for the development of "drop-in upgrade" tanks with up to 10,000 psi service pressure.

6. Design tankage for Nevada Bus (7.5% hydrogen by weight) and FutureTruck 2001 (8.5% hydrogen by weight) hydrogen fueled vehicles. Design Nevada Bus tankage around existing government tooling for rapid development and demonstration. Design FutureTruck 2001 tankage for a GM Suburban (K 15906) modified for hydrogen fuel.

Four possible configurations for mounting hydrogen tanks on the Nevada bus rooftop are shown in Figure 17 (Boehm 2000). The rightmost configuration is preferred because there are nominally 6 tanks available for this program (plus 2 spares) and all 6 tanks in this configuration can be plumbed easily through a single manifold. Design efforts are ongoing in collaboration with the Nevada bus team.

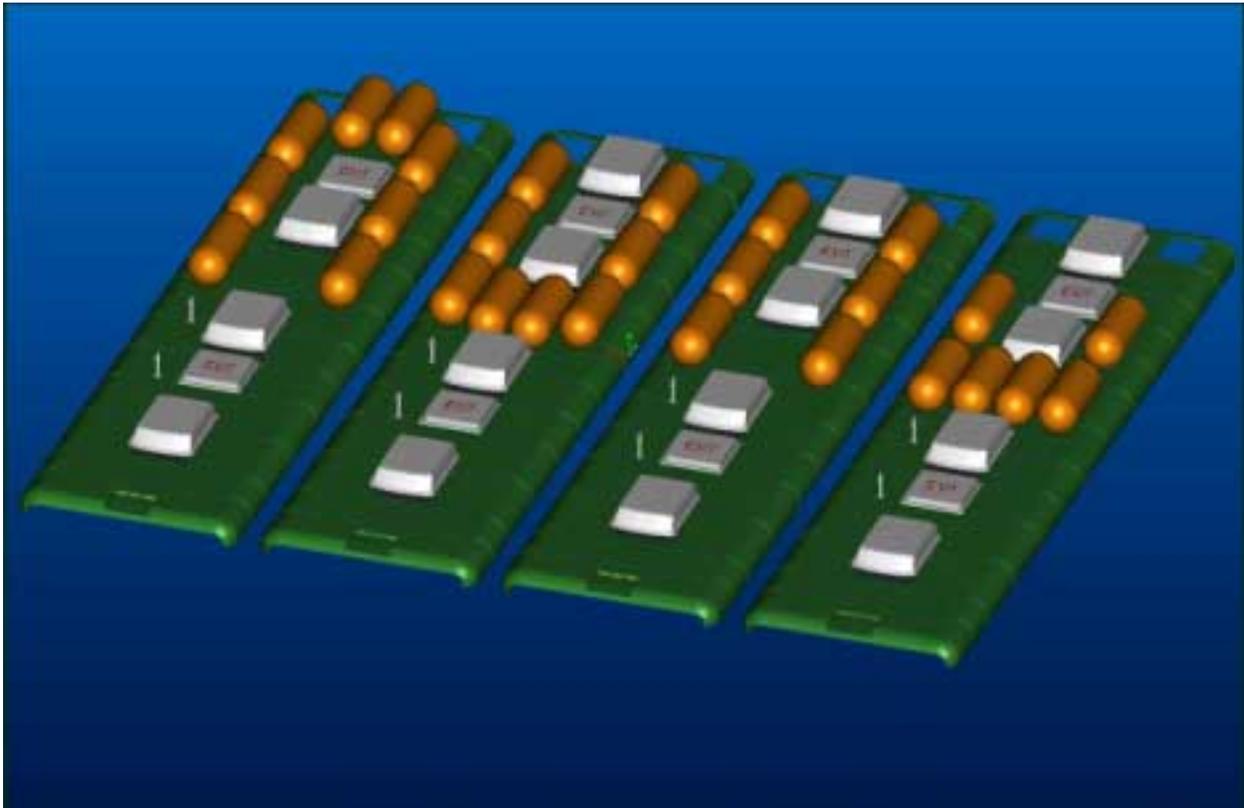


Figure 17 – Possible Tank Configurations for Nevada Bus

7. Scrutinize the proposed specification for hydrogen permeation in the modified NGV2 standard and/or the ISO/TC 197 standard. Less than 1 standard cc / hr / liter of water capacity at service pressure, room temperature, beginning of life has been proposed by others. Review the basis for the proposed hydrogen permeation specification and propose a modification if necessary.

LLNL and a team of experts reviewed a Committee Draft version of ISO/TC 197 N 148 and ISO/TC 58/SC 3 N 907. The following information was sent to the ISO/TC 197 Committee for comment:

Thank you for sending the Committee Draft version of ISO/TC 197 N 148 and ISO/TC 58/SC 3 N 907. After review of this document by a team of experts from LLNL and industry, we are proposing two changes to this document before it becomes an accepted standard.

The first change relates to the permeation test (A.20). Specifically, we propose that the permeation rate shall be less than 10.0 standard ml of hydrogen per hour per liter water capacity of the tank at room temperature, instead of the draft value of 1.0 ml of hydrogen per hour per liter water capacity of the tank. We further propose that verification of this permeation rate can be accomplished by monitoring for 50 hours instead of 500 hours for the test apparatus that is already used for verification. Documentation for this proposed change is provided below.

The second change relates to the stress ratio and burst pressure ratios for Type IV carbon fiber tanks, as described in Table 9 (of the Committee Draft). Although the team agrees that a stress ratio of 2.25 (burst pressure of 2.25 times the working pressure) should be chosen (as it was for CNG), we did not have adequate time to further justify this position with calculations. The team would be very interested to hear the rationale for why a draft value of 2.35 is being considered, since this will result in more expensive tanks without improving safety.

Documentation for proposed change to “A.20 Permeation test” of the Committee Draft:

It is the opinion of the review group that the draft specification for the A.20 permeation test is overly stringent, which will result in increased tank weight and cost without improving safety. LLNL has constructed a worst case scenario for safety issues resulting from excessive permeation of hydrogen through a tank wall. We evaluated the permeation level that could create a hazard under such a scenario and have proposed a specification which offers a safety margin adequate to prevent a hazardous situation even in such an extremely rare worst case scenario.

Background information is required to understand the basis for the worst case scenario. Permeation losses can be modeled as a very slow leak, which can cause a hazardous situation in a closed residential garage without forced ventilation or catalytic conversion of hydrogen. Hydrogen permeation rates through a tank wall are roughly linear with pressure, exponential with temperature (Souers 1982), and may be marginally worse near

the end of life of a tank, compared to the “as built” tank. Hydrogen concentration in a garage can increase most rapidly in small garage volumes with limited air exchange rates.

The worst case scenario results when a large vehicle (e.g., an SUV) which has a very large hydrogen capacity, is completely fueled (e.g., 12 kg hydrogen), is parked in a very small one car garage (e.g., 30 m³), the garage has poor ventilation (0.18 air changes per hour), the garage is very hot (180 degrees F) for sustained periods, and the tank is near end of life. We would like to keep the hydrogen concentration in this worst case garage safely below the lower flammability limit for hydrogen in air (4.1%) with a margin of safety of 2.

For reference on garage ventilation rates, the American Society of Heating, Refrigerating, and Air Conditioning Engineers, Inc. (ASHRAE) Standard 62-1989, “Ventilation for Acceptable Indoor Air Quality”, Atlanta, (1989), recommends an air exchange rate of 3.73 air changes per hour. Assuming a Poisson distribution of air exchange rates for 1 billion garages, the worst case would be $3.73/\text{LN}(1 \text{ billion}) = 0.18$ air changes per hour. Coincidentally, 0.18 air exchanges per hour is also the worst case air exchange rate for a residential garage that was measured in a study “Modeled and Measured Infiltration: a Detailed Study of Four Electrically Heated Homes”, prepared by ECOTOPE, Inc., Seattle, WA, EPRI Report No. CU-7327, May 1991 and “Air Infiltration and Interzonal Airflow Measurements in Research Houses”, prepared by GEOMET Technologies, Inc., Germantown, MD, EPRI Report No. EM-5968, August (1988), as referenced in “Addendum to Hydrogen Vehicle Safety Report: Residential Garage Safety Assessment”, analysis conducted by M.R. Swain, under subcontract to Directed Technologies, Inc., August (1998).

We assumed a 5000 psi working pressure for the hydrogen tank, which has 540 liter water capacity. In order to achieve the 4.1% lower flammability limit, the 30 m³ (30,000,000 ml) garage must have 1,230,000 ml (30,000,000 ml * 4.1%) of hydrogen present. This level requires a hydrogen permeation rate of 221,389 ml/hr (1,230,000 ml * 0.18 air change/hr). This requires a permeation rate from the hydrogen tank of 410 ml/hr/liter water capacity (221,389 ml/hr / 540 liter water capacity). Note that this is the permeation specification at the elevated temperature, for the “near end of life” tank without a margin of safety.

Hydrogen permeation experiments at elevated temperatures (180 degrees F) have been performed on plastic liners for tank walls and have shown a factor of 10 increase in permeation compared with similar measurements at room temperature (75 degrees F), F. Mitlitsky, A.H. Weisberg, and B. Myers, “Vehicular hydrogen storage using lightweight tanks (Regenerative fuel cell systems),” DOE Hydrogen Program Annual Review, Lakewood, CO, May 4-6 (1999); UCRL-JC-134540. In order to account for this difference (assuming that tank permeation qualification measurements will be done at room temperature), then the permeation specification must be reduced by this factor. This requires a permeation rate from the hydrogen tank of 41 ml/hr/liter water capacity (410 ml/hr/liter water capacity / 10). Note that this is the permeation specification at room temperature, for the “near end of life” tank without a margin of safety.

Hydrogen permeation experiments have been performed on plastic liners for tank walls at room temperature and compared with permeation tests for entire tanks that have undergone the 500 hour tests as described in the draft specification for A.20 permeation test. Entire tanks at beginning of life have hydrogen permeation that is approximately a factor of 2 lower than what would be expected for the permeation rate of the liner only. This implies that the composite overwrap is responsible for a permeation reduction of approximately a factor of 2 near the beginning of life. Near the end of life the composite overwrap matrix material will generally have a large density of microcracks. Although this will not effect the structural integrity of the tank, it will provide little (if any) permeation barrier to hydrogen. In order to account for this difference (assuming a worst case where the composite overwrap supplies no barrier to hydrogen permeation near end of life, and assuming that permeation measurements will be done at “beginning of life”), then the permeation specification must be reduced by a factor of 2. This requires a permeation rate from the hydrogen tank of 20.5 ml/hr/liter water capacity ($41 \text{ ml/hr/liter water capacity} / 2$). Note that this is the permeation specification at room temperature, for the “beginning of life” tank without a margin of safety.

Finally, the permeation specification should be reduced to account for a margin of safety below the lower flammability limit if the worst case as described above is achieved. A margin of safety of 2 requires a permeation rate from the hydrogen tank of 10.2 ml/hr/liter water capacity ($20.5 \text{ ml/hr/liter water capacity} / 2$). Note that this is the permeation specification at room temperature, for the “beginning of life” tank with a margin of safety.

Based on these calculations we believe that 10 ml/hr/liter water capacity is a very conservative, but reasonable specification for qualifying hydrogen tanks. Arbitrarily setting the specification any tighter does not appear to have any merit and will result in heavier, more expensive tanks, that are not any safer. Qualification of these tanks could be done as specified in the draft standard using a 500 hour test. However, the rationale for such a long test time was based on the sensitivity of detecting low permeation rates (0.25 ml/hr/liter water capacity as specified for CNG). Since we are proposing a permeation specification that is a factor of 40 higher than this rate, it makes sense to decrease the test time by an order-of-magnitude in order to reduce testing time and cost. We therefore suggest a 50 hour test.

8. Collect data on overtemperature/overpressure issues with fast filling procedures. Propose alternatives to mitigate overtemperature/overpressure issues.

Some initial work has been done to suggest an efficient way to efficiently precool the hydrogen during fast fills in order to keep the tanks from exceeding overtemperature limits. Discussions with Directed Technologies Inc. (DTI) on the subject have occurred (Daney 1995, James 1999, Jasionowski 1992, Kountz 1994, Mitlitsky 1996-c).

The facilities that might collect such data are currently being competitively costed out at IMPCO. Within the next few months, fully decided and approved tests plans should be in place at IMPCO, which include the downselection of this facility from among 3 subcontractors and potential in-house facility construction efforts.

Further LLNL efforts to collect thermal data independent of IMPCO are contingent on FY01 funding, and would require sample tanks of a relevant design to explore this issue with hardware. Computer modeling methods are considered unlikely to resolve the real heat transfer issues of particular tank designs and materials. Experimental characterization of tank fill thermodynamics would be possible with portable instrument accompanying research-instrumented tanks into one of the test cells at LLNL's High Pressure Laboratory, but spare pressure vessels suitable for instrumentation will probably not be available in calendar 2000.

Plans for Next Year

1. Design and test hydrogen tanks with >10% hydrogen by weight (5000 psi [34.5 MPa] service, 300 K, safety factor 2.25) and 700 Wh/liter.
2. Direct a ~1 year program to deliver certified tankage with 7.5-8.5% hydrogen by weight (5000 psi, 300 K, safety factor 2.25).
3. Test lightweight liners with the best permeation reduction coatings that offer easily adopted fabrication processes.
4. Demonstrate lightweight tankage with safety factor 2.25 and improve cost projections based on input from industry.
5. Work with car companies and tank manufacturers to advance the adoption prospects of 10,000 psi H₂ tankage. Technically direct 10,000 psi tankage development effort (pending a DOE/OTT funding award and/or additions to the current DOE/Golden contract DE-AC36-GO10494).
6. Oversee operation of delivered tankage on Nevada Bus (7.5% H₂ by weight) and FutureTruck 2001 (8.5% H₂ by weight) hydrogen fueled vehicles.
7. Recommend alternative specifications (e.g., H₂ permeation) to NGV2 and/or ISO/TC 197 standards committee.
8. Construct models to demonstrate reduction of fast filling overtemperature and overpressure transients.

Objectives for Next Year

1. Demonstrate a prototype hydrogen tank that can be certified to modified NGV2 standards while achieving >10% hydrogen by weight (5000 psi [34.5 MPa] service, 300 K, safety factor 2.25) and 700 Wh/liter.
2. Demonstrate certified tankage with 7.5-8.5% hydrogen by weight (5000 psi, 300 K, safety factor 2.25) for delivery to Nevada Bus and FutureTruck 2001 programs.
3. Work with industry to adopt lightweight liners with the best permeation reduction coatings.
4. Demonstrate that safety factor of 2.25 is adequate for plastic lined composite hydrogen tankage and publish improved cost projections.
5. Demonstrate 10,000 psi hydrogen tankage (pending a DOE/OTT funding award and/or additions to the current DOE/Golden contract DE-AC36-GO10494).
6. Support Nevada Bus and FutureTruck 2001 hydrogen fueled vehicle demonstration programs that use onboard compressed H₂ storage.
7. Persuade NGV2 and/or ISO/TC 197 standards committee to adopt safe but not overly stringent specifications for hydrogen.
8. Recommend a procedure to reduce fast filling overtemperature and overpressure transients.

Conclusions and Recommendations

Hydrogen storage with Type IV pressure vessels is advancing rapidly, and is approaching adoption by automotive demonstration vehicles over the next year. Last year DTI concluded that, a fuel cell powered vehicle fueled with compressed H₂ (at 5,000 psi) was the system to beat (James 1999). Such vehicles offer: low weight (with >10% hydrogen by weight feasible), while storing hydrogen in an acceptable volume, at an acceptable cost. The other advantages of advanced Type IV hydrogen tanks include high system simplicity, high safety, the potential for faster refills than their competitors, as well as expected support by a feasible H₂ infrastructure (in both start-up and mature phases). Over the next year, LLNL lead efforts are poised to turn this prediction into reality.

Efforts at LLNL have made progress toward significant weight and cost improvements over the last year in two directions: research leading to fundamental improvements and facilitating the commercialization of recent advances. Thin liner technology pioneered by LLNL is already being folded into commercializable tank designs, while permeation barrier coatings have been pushed into the preliminary-encouraging regime that might lead to near-term adoption. Safe hydrogen tankage is already commercially available at ~5 % hydrogen by weight. By the conclusion of the LLNL-directed DOE/Golden contract DE-AC36-GO10494, 8.5% hydrogen by weight tankage should be at the threshold of commercialization in 2001. Research prototyping efforts by LLNL and its industrial partners have proven performance levels above 11% hydrogen by weight are feasible.

Further research on the weight frontier could establish manufacturing processes capable of >12% by weight hydrogen storage, and/or modify record-breaking designs for high confidence of meeting safety certification with >10% hydrogen by weight. Technical direction of expert industrial contractors under LLNL subcontracts remains a viable option for pursuing progress further than one year from revenue generation on this crucial frontier.

LLNL technical direction of ongoing and planned DOE solicitations involves technology much closer to adoption than innovating directly on the weight frontier. This effort is part diplomatic and highly technical, and is vital to rapid adoption of hydrogen fueled vehicles. It must execute the glorious demonstration projects that have already been planned by developing, delivering, and competently installing proven tanks. Those tanks are currently numbers converging on a detailed set of designs. Over the next few months, stabilized designs will be manufactured in batches, subjected to risk reduction testing, manufactured in more batches, and safety certified. Success in meeting the solicitations aggressive weight goals depends on careful attention to mass allocation, design decisions, test plans, and every decision based on test conclusions.

In late 1999, the LLNL team crafting technical specifications of the 7.5-8.5% hydrogen by weight DOE solicitation currently underway sought industrial interest in higher pressure tank technology. Higher pressure designs would be easier to package aboard demonstration vehicles (e.g., FutureTruck), and could dramatically increase (by ~2/3) the range of vehicle already fitted with 5,000 psi tanks. After initial skepticism, increasing interest has been circulating among automobile companies, DTI, and other interested parties across the hydrogen community. Thus

there is a new constituency for progress on the density frontier. This new constituency encourages DOE funding to extend LLNL-directed, near-commercializable tank technology development to deliver 10,000 psi tankage.

Understanding of the engineering and economic issues on the weight, cost, safety, and density frontiers is rudimentary at present. The world's experts can barely account for the failure phenomena that have emerged on the LLNL-subcontracted weight frontier research. Aerospace expertise has spent few man years on the frontiers of hydrogen tankage, and DOE can't afford much of this expense. Commercial expertise has largely chased aerospace out of the non-aerospace tankage business, offering lower costs but relying on empirical methods and much less sophisticated engineering. Neither commercial nor aerospace experts can model many of the phenomena that LLNL-lead research has encountered.

Academic researchers should be capable of debugging many of the process and material phenomena that advanced tank development has and will uncover, but they have seldom been able to afford the refined state of the art in composite manufacturing. Neither solid rocket motor cases, nor aircraft wings, nor automotive driveshafts have much of an academic research community despite being frontiers for composite materials. This leaves only national laboratories with the research means and incentives to make progress that won't show a profit in the next two years. The alternative is to let industry declare artificial performance limits, deliver 'research' that turns into products that were already possible without government funding, and to employ non-innovators to disburse taxpayers funds that entrench premature monopolies.

Industrial policy ought to be off limits for purely technical efforts at National Laboratories, yet recent progress in the hydrogen community makes it nearly impossible to ignore interest groups. DOE and the taxpayers and hydrogen technologies in general will be well served by LLNL-lead initiatives to remove confusion in international safety regulations for hydrogen, especially those related to hydrogen permeation. The fate of liquid hydrogen and natural gas infrastructures in Europe could determine the outcome of LLNL's recent regulatory initiative. Without some experts' time and ability to travel, such important 'diplomatic' frontiers will wither before many months elapse, and long before accidents of regulatory history (that heavily penalize a near-monopoly for the U.S. in Type IV tanks) can be reversed.

Without real research to extend our understanding of what progress to pursue next, staff devoted to contracts between DOE and industry must follow rather than lead. In the absence of a research community dedicated to understanding the utmost that industry can do (and what might profitably lie just beyond that 'utmost'), industry's arguments that they already know their business best are true.

If leadership on any of these frontiers is justified, there is already a strong case to be made for process research on smaller (perhaps 5 inch diameter) pressure vessels. Only affordable process research can sort out the unknown failure mechanisms encountered recently on the weight performance frontier. Small tanks may need to be produced and integrated-performance tested in statistically significant quantities (batches of at least 6 identical units). It probably makes sense to attempt correlating failure modes with microstructure, but much more expert advice should be tapped to determine how best to probe composite microstructures. More permeation barrier and

coating research could be very cost-effective in speeding adoption of advanced liners. The first exploration (at small scale) of blow molding could discover the superior cost and quality and performance liner options many experts anticipate. Permeation should also be studied as a function of cycling, to know rather than guess the permeation consequence of the relatively huge cycle lives required by safety certification. It is also not premature to begin design studies of 10,000 psi ancillaries.

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List of Figures

- Figure 1 – DOE 2000 Tankage Goals
- Figure 2 – First Composite Wrapped Tank Prototype Next to Thin-Wall Liner
- Figure 3 – Remains of Test Tank #3 after Premature Burst
- Figure 4 – Video Captures Localized Failure During Tank #3 Burst Test
- Figure 5 – Condition of Tank #5 before Highly-Instrumented Burst Test
- Figure 6 – Best High Speed Camera Frame Captures Tank #5 At Burst
- Figure 7 – Tank Size and Location Considerations for Vehicular H₂ Storage
- Figure 8 – Hydrogen Requirements for FutureTruck
- Figure 9 – Hydrogen Tankage Placement Issues for FutureTruck
- Figure 10 – Preliminary GM Suburban Rail and Crossmember Arrangement
- Figure 11 – Proposed H₂ Tank Placement for FutureTruck (GMT 830 K 15906)
- Figure 12 – H₂ Tankage for Nevada Bus Program and FutureTruck Competition
- Figure 13 – Conformable Tankage Fabricated by Thiokol Passed Bullet-Test
- Figure 14 – Schematic and Photos of Permeability Test Fixture at SRI
- Figure 15 – Measured Hydrogen Permeability of Several Candidate Liner Materials
- Figure 16 – Economic Analysis of Carbon Fiber Tanks in High Volume Production
- Figure 17 – Possible Tank Configurations for Nevada Bus

List of Tables

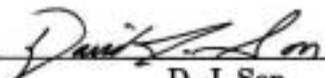
- Table 1. 11.3% H₂ by Weight was Demonstrated for a High Cycle Life Cylinder

Appendix 1

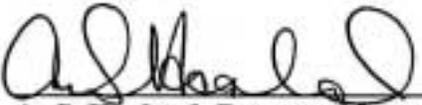
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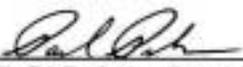
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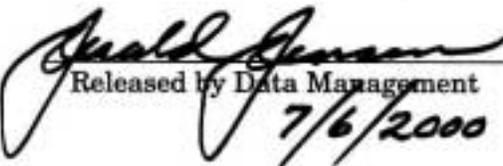
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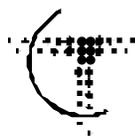

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Appendix 1

TABLE OF CONTENTS

1.0	INTRODUCTION	47
2.0	OBJECTIVES	47
3.0	SUMMARY.....	47
4.0	TANK DEVELOPEMENT	48
4.1	Requirements.....	48
4.2	Tank Design and Analysis.....	48
4.3	Liner Tests.....	49
4.4	Fabrication and Testing.....	50
4.4.1	Tank #1	50
4.4.2	Tank #2	50
4.4.3	Tank #3	51
4.5	Failure Analysis	51
4.5.1	Tank #1 Hydroburst Test	56
4.6	Redesign	56
4.6.1	Hoop Termination Pattern Design	56
4.6.2	Wafer Design.....	60
4.6.3	Tank #4 Fabrication and Testing.....	62
5.0	CONCLUSION	63

FIGURES

Figure 1:	Tank #3 after hydroburst test	51
Figure 2:	Tank #3 failure initiation.....	52
Figure 3:	Finite element model geometry.....	53
Figure 4:	Fiber Strain Plot.....	54
Figure 5:	Modeled vs. as-built hoop termination.....	55
Figure 6:	Modeled vs. as-built fiber strain plot	55
Figure 7:	Measured vs. estimated fiber strain plot (Tank #1)	56
Figure 8:	Redesigned hoop termination pattern.....	57
Figure 9:	Redesigned hoop pattern fiber strain plot.....	57
Figure 10:	FEM with hardware.....	58
Figure 11:	Fiber strain plot with hardware	58
Figure 12:	FEM with smoothed composite.....	59
Figure 13:	Fiber strain plot with smoothed composite	60
Figure 14:	Wafer FEM	61
Figure 15:	Wafer design fiber strain plot	61
Figure 16:	Tank #4 before hydroburst test.....	62
Figure 17:	Tank #4 after hydroburst test.....	63

Appendix 1

TABLES

Table 1: Test Results.....	47
Table 2: Design Requirements.....	48
Table 3: Tank Design Estimates.....	48
Table 4: T1000G Properties	49
Table 5: CLT Stress/Strain Estimates.....	49
Table 6: Measured vs. CLT strains for Tank #3.....	52
Table 7: CLT vs. isotropic strain estimates	52

APPENDICES

- APPENDIX A: Tank Drawings
- APPENDIX B: Hoop Termination Design Iterations
- APPENDIX C: Wafer Design Iterations
- APPENDIX D: Test Plan TTP528

Appendix 1

1.0 INTRODUCTION

Thiokol designed, fabricated and tested 18-inch diameter by 48-inch length gaseous hydrogen cylinders for Lawrence Livermore National Laboratory (LLNL) under sub-contract #B503790. The program was conducted from 15 Mar 99 to 30 June 00. Plastic liners for the tanks were supplied by LLNL as government furnished material and was manufactured by Aero Tec Laboratories Inc. (ATL) of Ramsey, NJ.

2.0 OBJECTIVES

The goal of the program was to fabricate high performance tanks for the storage of hydrogen for vehicular applications. Main design issues to be addressed by this program included maximum volume and minimum weight. The designed service pressure for the tank was 5,000 psig, with a safety factor of 2.25. The primary performance metric for the tank was expressed as burst pressure * internal volume / total tank weight. The design goal for this metric was a value of 1.8 million inches.

3.0 SUMMARY

Four tanks were fabricated using T1000G TCR-prepreg because of its high strength to weight ratio. The tanks used plastic liners and aluminum polar bosses. One tank leaked following cure and was subsequently sectioned prior to delivery to LLNL. Three tanks were hydroburst tested at Thiokol. Results are given in Table 1. Tanks 1-3 employed the same design; tank 4 was a redesign. An annealed liner was used to fabricate tank 4, which lead to a volume decrease. Tank 4 reached 94.4% of the performance goal of 1.8 million inches.

Table 1: Test Results

Tank #	1	2	3	4
Test Date	5/8/00	--	4/28/00	6/16/00
Burst Pressure (psig)	9,503	--	7,872	10,464
Meas. Hoop Strain (in./in.)	.01444	--	.01192	.01518
Ambient Internal Volume (in ³) ¹	9,240	9,240	9,240	8,600
Total Tank Weight (lbs.)	53.5	56.8	55.7	54.0
Adjusted Tank Weight (lbs.) ²	53.5	54.4	54.5	52.8
Liner Weight (lbs.)	5	6	6	6
Polar Boss Weight (lbs.)	1.5	1.5	1.5	1.5
P _b V/W (X 10 ⁶ in.)	1.64	--	1.33	1.70

¹ Water volume measured for Tank 1. Tanks 2 & 3 similar to Tank 1. Tank 4 (annealed liner) estimated with CAD program (conservative value).

² Total tank weight minus glass overwrap weight (1.2 lbs./ply). Tank 2 had two glass hoop plies, Tanks 3 & 4 had one ply.

Appendix 1

4.0 TANK DEVELOPMENT

4.1 Requirements

The requirements for the hydrogen tank are listed in Table 2. The main goal was to obtain a greater than 1.8 million inches $P_b V/W$, where P_b is the burst pressure, V is the internal volume, and W is the total weight of the tank including the composite, liner, and polar bosses.

Table 2: Design Requirements

Parameter	Requirement
Outside Diameter (in.)	18.0
Length, boss to boss (in.)	48.0
Liner Material	Plastic
Service Pressure (psig)	5,000
Safety Factor	2.25
Minimum Burst Pressure (psig)	11,250
H ₂ Storage Capacity (lbs.)	8.5
$P_b V/W$ (in.)	$> 1.8 \times 10^6$
H ₂ by Total Tank Weight (%)	12

4.2 Tank Design and Analysis

The 18-inch tank design was scaled up from standard IR&D 12-inch diameter cylinders. The polar boss to liner interface was unchanged except for the boss being thickened for the higher design pressure (see Appendix A for boss drawing). The first tank was made with an plastic liner. The design parameters for the tank are listed in Table 3.

Table 3: Tank Design Estimates

Liner (Plastic)	
Liner O.D. (in.)	17.25
Liner Length (in.)	46.0
Internal Volume (in ³)	9482
Liner Weight (lbs.)	5.0
Polar Boss (AL6061-T6)	
Total Boss Weight (lbs.)	1.6
Composite (T1000G)	
Total Composite Thickness (in.)	.352
Composite O.D. (in.)	17.95
Stress Ratio	.70
Composite Weight (lbs.)	47.5
Total Tank Weight (lbs.)	
	54.5
$P_b V/W$ (in.)	1.96×10^6

Appendix 1

The composite case was designed using classical lamination theory (CLT). Lamina properties are given in Table 4 (ref. TR10958), and the stress and strain estimates based on CLT are provided in Table 5. The lower 3σ value was used for the allowable fiber stress.

Table 4: T1000G Properties

Property	Value
E_{11} (Msi)	22.70
E_{22} (Msi)	1.05
ν_{12}	.337
G_{12} (Msi)	.606
Allowable Fiber Stress (psi)	740,000

Table 5: CLT Stress/Strain Estimates

Laminate Strain	5,000 psig	11,250 psig
Hoop (in./in.)	.00859	.0193
Axial (in./in.)	.00586	.0132
Fiber Stress		
Hoop (ksi) / Safety Factor	330 / 2.24	743 / .996
Helical (ksi) / Safety Factor	234 / 3.16	525 / 1.40

The finite element model for the composite was generated using FWIND and STACKER, both in-house codes. Hardware was added using I-DEAS MS 7.0 and ABAQUS 5.8 was used as the solver. In the initial analysis, much time was taken to determine a suitable helical step-back pattern to avoid dome failure. After numerous iterations, a winding sequence was chosen. The analysis is covered in Section 4.5.

4.3 Liner Tests

The first liner from ATL made of plastic material weighed 6 pounds and had a thin area in the sidewall. The liner was pressurized to 2 psi with air at Thiokol's I-10 test facility without significant deformation. The liner was being pressurized to 4 psi when it ruptured in the thin area and resulted in a longitudinal split down the entire cylindrical length.

The second liner from ATL made of plastic material weighed 5 pounds and had more uniform thickness distribution. The liner was threaded and polar bosses with smaller diameter flanges were installed and torqued to a level of 15 ft-lbs. (180 in-lbs.). The liner was pressure tested with air at M-9 on June 11, 1999 to 4 psi for five minutes without any significant deformation. It was then considered safe to wind with an operator next to the liner with an internal pressure of 2 psi.

The correctly sized polar bosses for the second liner were installed and torqued, but not to the full 180 in-lbs. It seemed that the torque was not getting increasingly higher. It was decided to do some additional torque evaluations with the first liner that burst. The closed end of the burst liner was torqued to 180 in-lbs., and then increased to 270 in-lbs. with an additional 1/2 revolution. The open end was torqued to 180 in.-lbs. After a few minutes, it was torqued again to 180 in-lbs. with an additional 1/2 revolution.

Appendix 1

The results indicated that additional applications of the same torque could cause significant revolutions between the polar boss and the plastic liner. It was decided to not do any additional tightening, except as would be performed by the winding operations.

The second liner was installed in the winding machine. Four tows at 6 lbs./tow tension were attempted in a hoop winding mode. The polar boss continued to turn so the hoop winding was terminated. Helical layers were applied with 4 tows per winding band. There was no more turning of the polar boss on the liner. After part of the first helical layer was applied, it was noted that the polar boss to polar boss length was reducing. The compressive force of the helical layer was more than the internal pressure (2 psig) would withstand. Additional helicals were wound using two tows per bandwidth. There was no further reduction in length. It was also noted that during the helical winding operations, the right side axis of the liner that is held by the tail stock in the winding machine was raising about 3/4 to 1 inch when the winding was being performed at this end of the liner. The support rod in the tail stock is much longer than what is needed, and there was some "play" between the support rod and the tail stock live center. These features allowed the right end of the liner to be able to move. It was decided to wind with two tows/band for first helical layer, then determine if additional winding can be accomplished with four tows per band without anymore turning of the polar boss on the liner.

Scotch-Weld DP-8005 was evaluated for use as an adhesive between the polar boss and liner. The material was mixed and applied to the threads and flat recess for the polar boss on one of the ends of the first liner. After the adhesive had cured overnight, the polar boss was torque tested. A torque of 600 in-lbs. was achieved without movement between the polar boss and the plastic liner.

4.4 Fabrication and Testing

4.4.1 Tank #1

The first tank was used as a demonstration piece for LLNL. The liner weighed 5 lbs. and the outside diameter measured 17.12 inches (2 psig). The tank was cured horizontally with rotation. No glass hoop plies were applied to the tank, but shrink-wrap was used. The tank weighed 53.5 lbs. and the internal volume was measured to be 9,240 cu. in. The tank was successfully proof tested to 7,500 psig, with a two-minute hold, on June 23, 1999. The tank was then delivered to LLNL.

4.4.2 Tank #2

Tank two was fabricated on April 14, 2000. The liner weighed 6 lbs. and the outside diameter measured 17.21 inches (2 psig). The tank was built and cured using the same process as the first tank, with the exception that two glass hoop plies were applied to secure a label. The total tank weight was 56.8 lbs. The tank failed to hold pressure, with air leaking around the polar boss. Dissection revealed cracks in the liner in the polar boss flange region. The liner also showed radial folds, most likely caused by the rotation of the tank during cure. It was decided to cure vertically with no rotation for subsequent tanks. A section of the tank was delivered to LLNL for further evaluation.

Appendix 1

4.4.3 Tank #3

Tank three was fabricated on April 27, 2000. The tank was vertically cured with no rotation. A heavier 6-lb. liner was used and the outside diameter measured 17.06 inches (2 psig). One glass hoop ply held the label, and the total tank weight was 55.7 lbs. The tank was hydrostatically tested on April 28, 2000 according to test plan TTP528 (attached in Appendix D). The burst pressure was 7,872 psig, 70% of the required minimum burst pressure. The measured fiber strain at burst was 1.192%. The tank after burst is pictured in Figure 1.



Figure 1: Tank #3 after hydroburst test

4.5 Failure Analysis

Following the low burst pressure of Tank #3, a failure analysis ensued. The tank was inspected by the manufacturing engineers for possible fabrication problems. The correct material was used, no wrinkled fibers were observed, and the winding drawing had been followed. In reviewing the video footage taken of the burst test, it was determined that failure initiated at the tangent line. Figure 2 shows the tank setup and the frame from the video showing water sprays from the tangent line.

Appendix 1



Figure 2: Tank #3 failure initiation

The design was also reviewed. The measured hoop strains was compared to the CLT hoop strains in Table 6. In a prior rocket motor program, a fiber direction modulus of 24.8 Msi was used. This value gave better CLT estimates compared to the measured strain values.

Table 6: Measured vs. CLT strains for Tank #3

Pressure (psig)	Measured Strain (in./in.)	CLT Strain with E1=22.7 Msi	Ratio	CLT with E1=24.8 Msi	Ratio
5000	0.00793	0.00859	0.92	0.0079	1.00
3333	0.00532	0.00573	0.93	0.00527	1.01
7500	0.0115	0.0129	0.89	0.0119	0.97
7872	0.0119	0.0135	0.88	0.0124	0.96

As a quick check of the CLT numbers, simple isotropic calculations were done using the hoop thickness in Table 7. This verified the right material thickness was used.

Table 7: CLT vs. isotropic strain estimates

Pressure (psig)	CLT		Isotropic		Ratio Stress	Ratio Strain
	Stress (ksi)	Strain (in./in.)	Stress (ksi)	Strain (in./in.)		
11250	743	0.0193	793	0.0186	0.94	1.04
7500	495	0.0129	528	0.0124	0.94	1.04
5000	330	0.00859	352	0.00825	0.94	1.04

The focus then shifted to the finite element analysis. Upon reviewing the axisymmetric model results, a problem was discovered in the tangent line. In the initial analysis, only the first five helical layers were looked at. The focus of the analysis was the step-backs to avoid a dome failure, where the highest strains occurred in the inner helical layers. Similar to previous cylinders, the tangent line was not thoroughly analyzed. Standard 12-inch cylinders have been used extensively for burst tests, cycle tests, etc. without any problems in the tangent line area. Because of this history, the 18-inch tank followed the 12-inch cylinders in design. Unfortunately in the 18-inch tank, the highest strained helical was at the tangent line of the outside layer, which was above the level of the hoops. The

Appendix 1

higher 0.7 stress ratio design, compared to 0.55 for M30S cylinders, further attributed to the problem. The model geometry is pictured in Figure 3 and the fiber strain plot for the 18-inch tank is shown in Figure 4.

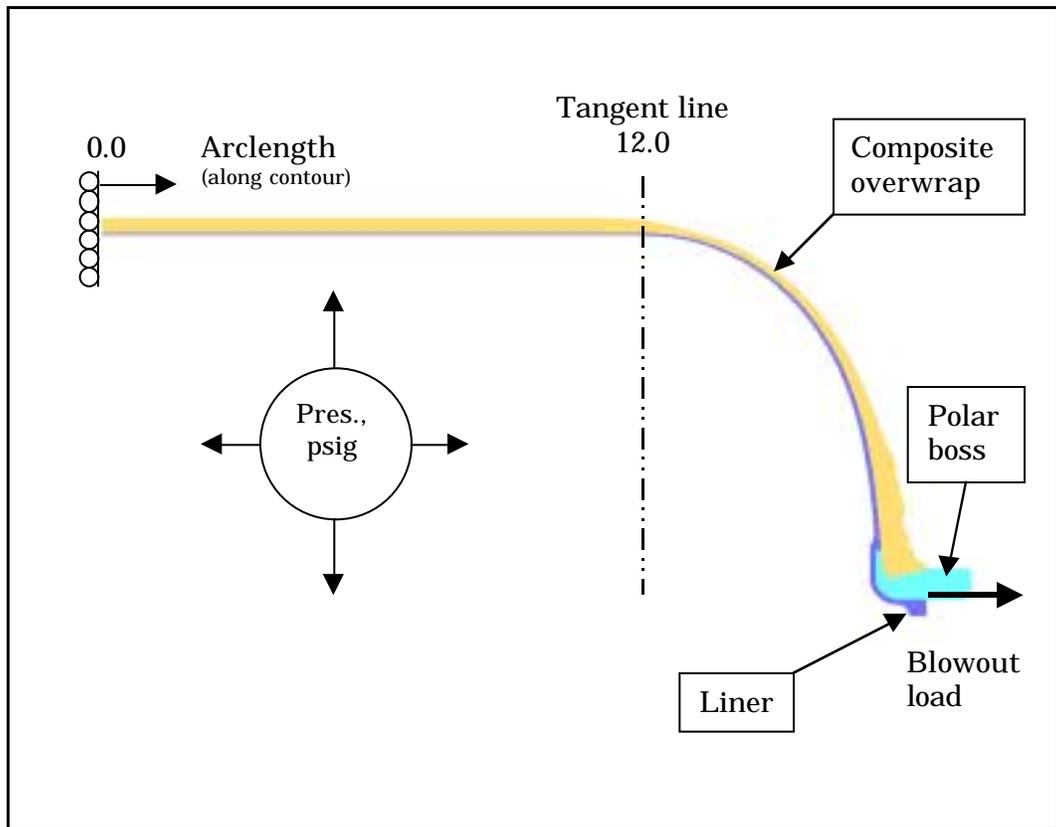


Figure 3: Finite element model geometry

Appendix 1

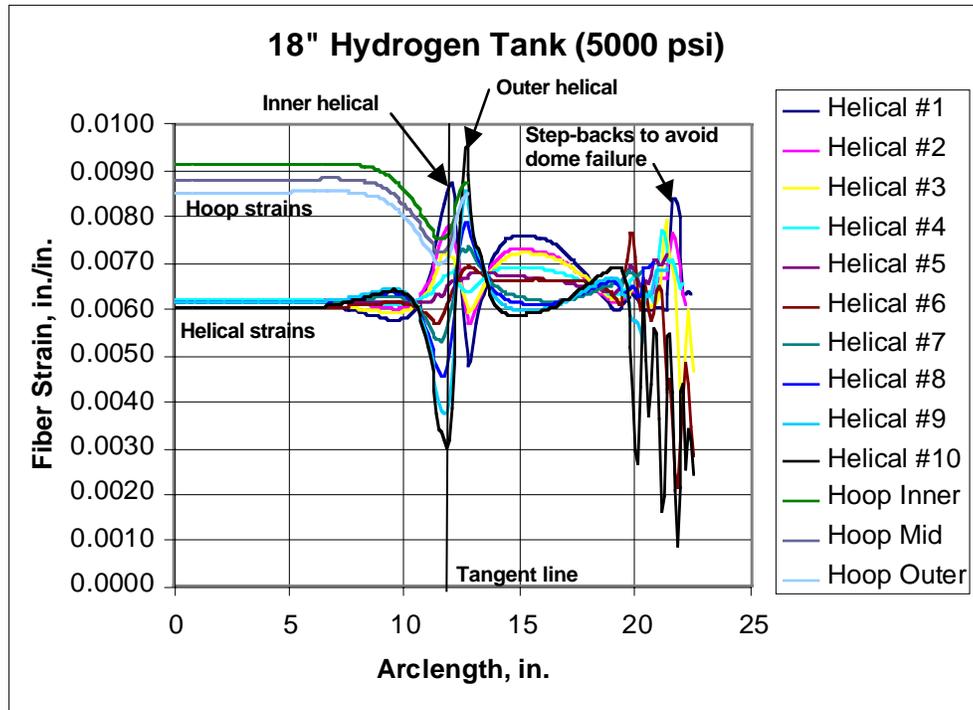


Figure 4: Fiber Strain Plot

Since the failure mode was determined to be the tangent line, the hoop terminations were analyzed. As in previous drawings, the hoop terminations were not specified, and establishing the position for the terminations was left to the manufacturing engineer. The hoop termination in the model is compared to the as-built in Figure 5. The hoop terminations were modeled as a "V", extending 0.5 inches past the tangent line down the dome. The as-built tank did not have hoops extending past the tangent line. The fiber strain plot comparison between the two is given in Figure 6.

Appendix 1

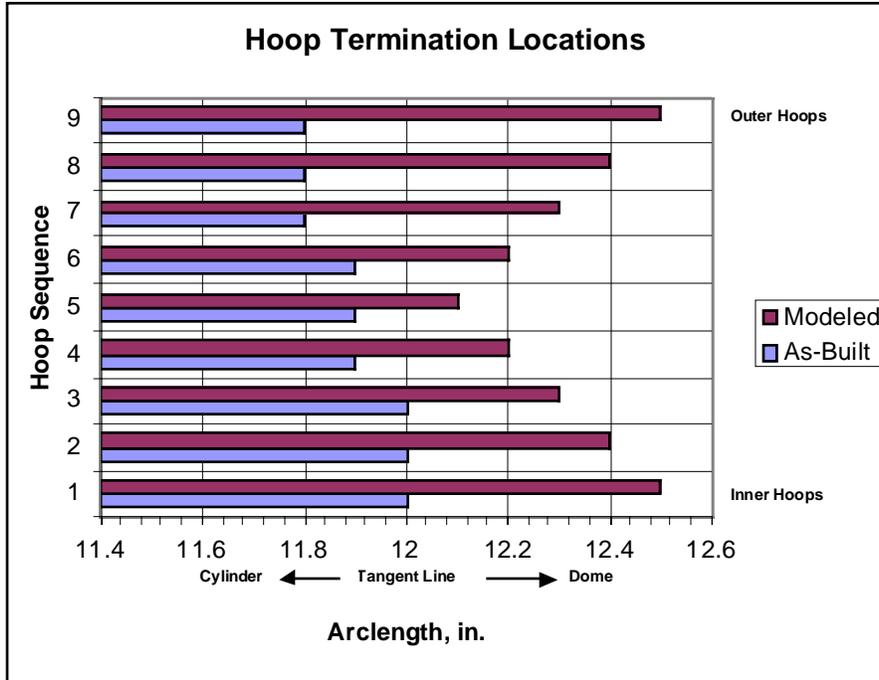


Figure 5: Modeled vs. as-built hoop termination

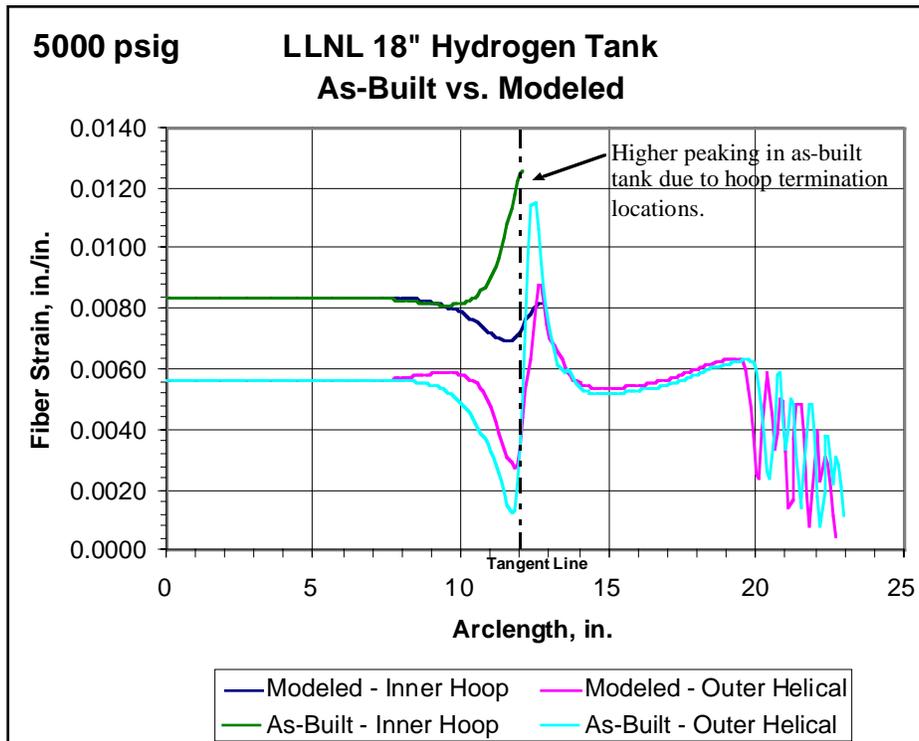


Figure 6: Modeled vs. as-built fiber strain plot

Appendix 1

4.5.1 Tank #1 Hydroburst Test

Tank #1 was returned to Thiokol for burst testing to address variability of the burst pressure and to verify the failure analysis findings. The tank was fitted with six strain gages, three along each tangent line over a 1-inch length. The tank was burst tested on May 8, 2000 at a pressure of 9,503 psig, 20.7% higher than the first tank, with a measured fiber strain of 1.444%. The tank also failed in the tangent line. The high variation is typical with bending failures. The recorded strains at 5,000 psig are plotted with the estimated strains in Figure 7. The measured strains agreed well with the estimates, although the peak helical strains were missed.

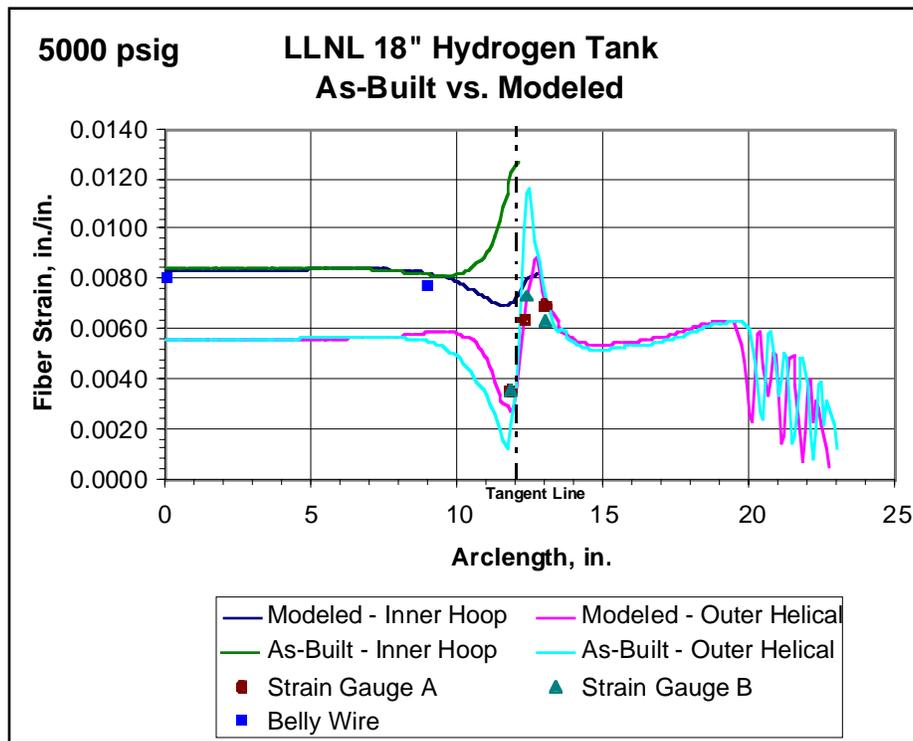


Figure 7: Measured vs. estimated fiber strain plot (Tank #1)

4.6 Redesign

The redesign effort first focused on finding a hoop termination pattern that dropped helical strains at the tangent below the level of the hoop strain. Some iterations were done to investigate the sensitivity of the termination points. After a suitable pattern was determined, the polar boss and liner was added to the model to see whether dome failure would be an issue.

4.6.1 Hoop Termination Pattern Design

In trying to find the optimum hoop termination pattern, only the composite was modeled. The target was to get a peak helical strain at the tangent line to be 10-15% lower than the hoop strains. The pattern in Figure 8 achieved the target (see Appendix B for the complete iteration summary). The fiber strain is plotted in Figure 9.

Appendix 1

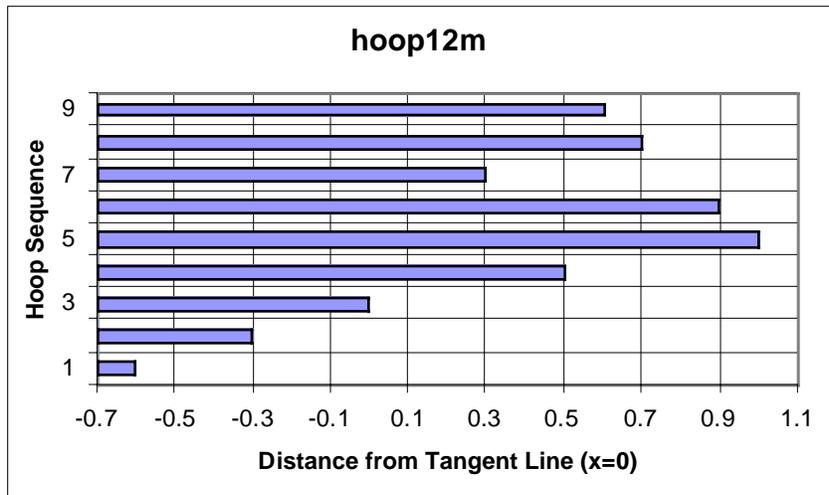


Figure 8: Redesigned hoop termination pattern

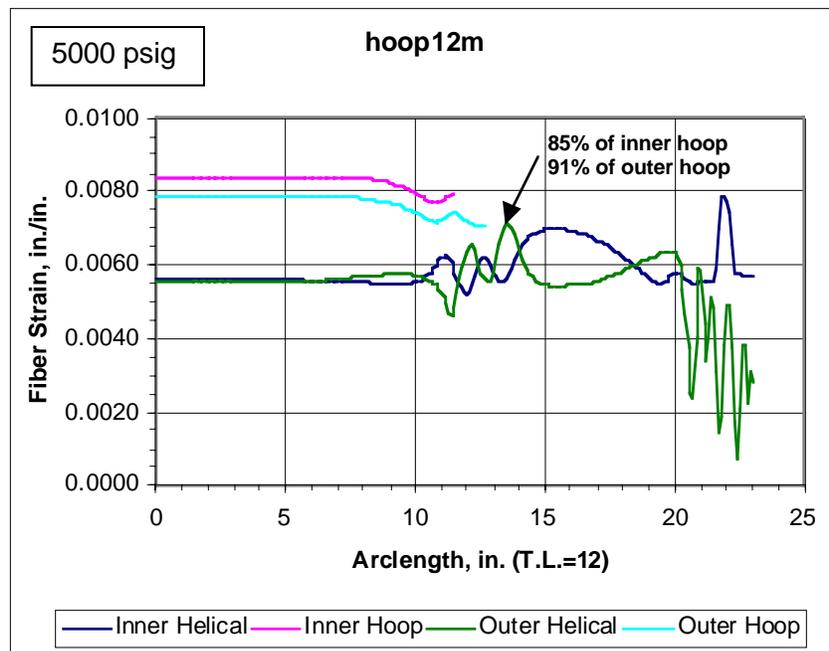


Figure 9: Redesigned hoop pattern fiber strain plot

Sensitivity was also looked at, where all the hoop terminations were offset ± 0.3 inches from the tangent line. An offset forward of the tangent line had minimal impact, however, offsetting aft of the tangent line increased helical strains considerably. The drawing was dimensioned to err on the side of the dome.

The tangent line strains were judged to be acceptable based on experience with rocket motor designs employing similar stress ratio. However, the peak strain in the inner helical layer near the boss was a concern. Generally, when the polar boss is added into the model,

Appendix 1

the peaks decrease. To check this, the liner and polar boss were added into the FE model. The close-up of the boss region of the model is pictured in Figure 10. The model failed to converge at ultimate pressure (11,250 psig) but reached 10,940 psig. The fiber strain plot at this pressure is given in Figure 11.

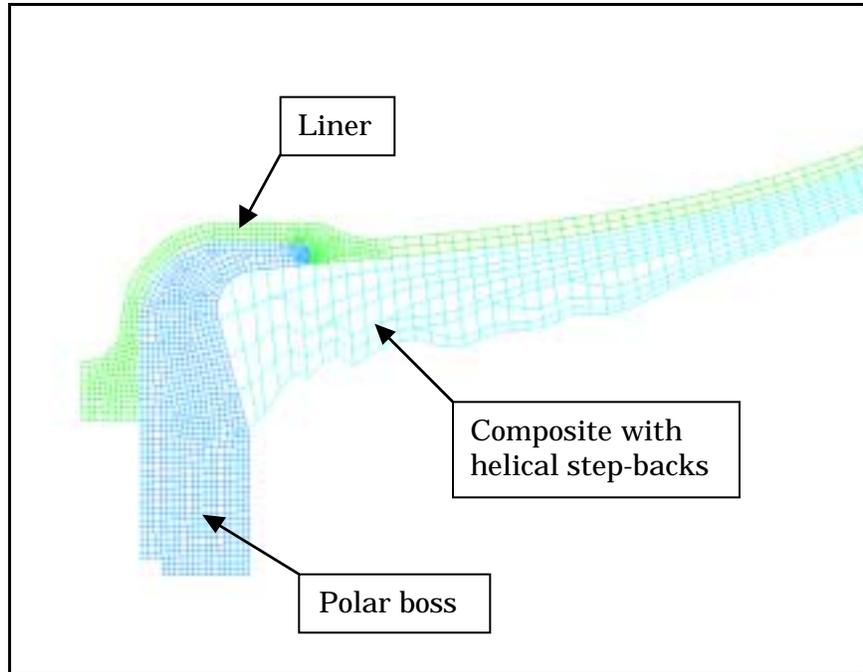


Figure 10: FEM with hardware

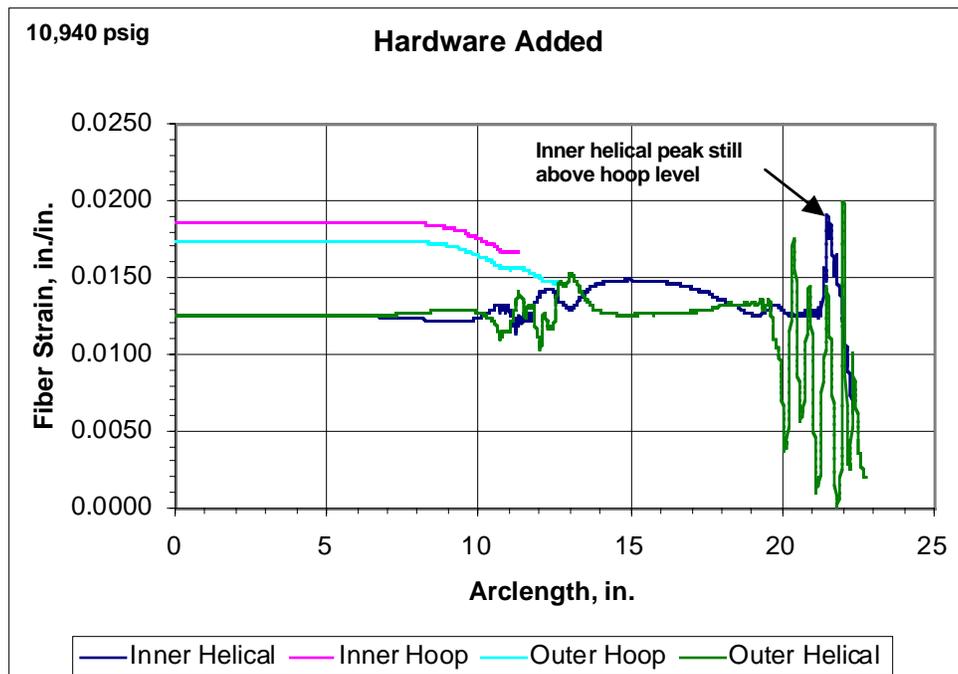


Figure 11: Fiber strain plot with hardware

Appendix 1

The addition of the boss did not bring down the peak inner helical strain. The level is above the hoop strain level, which would indicate a dome failure upon burst testing. The peaks in the outer helical layer were also high, however, it was felt that those peaks were artificial and were a function of the element mesh. As can be seen in Figure 10, the composite mesh is quite jagged on the outer helical layer. To verify this, the composite mesh was manually smoothed in the polar boss region, from about where the furthest step-back is to the boss. The element smoothing was done by moving nodes to simulate an actual build-up, and then degrading properties where resin pools would be located. The smoothed model is shown in Figure 12. The buildup is smoother, but the outer helical is still rougher than what would actually be wound. The fiber strain plot at ultimate pressure is shown in Figure 13. Comparing the fiber strain in the outer helical to the un-smoothed plot in Figure 11, the peaks have drastically decreased. Thus, the outer helical peaks were deemed artificially caused by the mesh geometry.

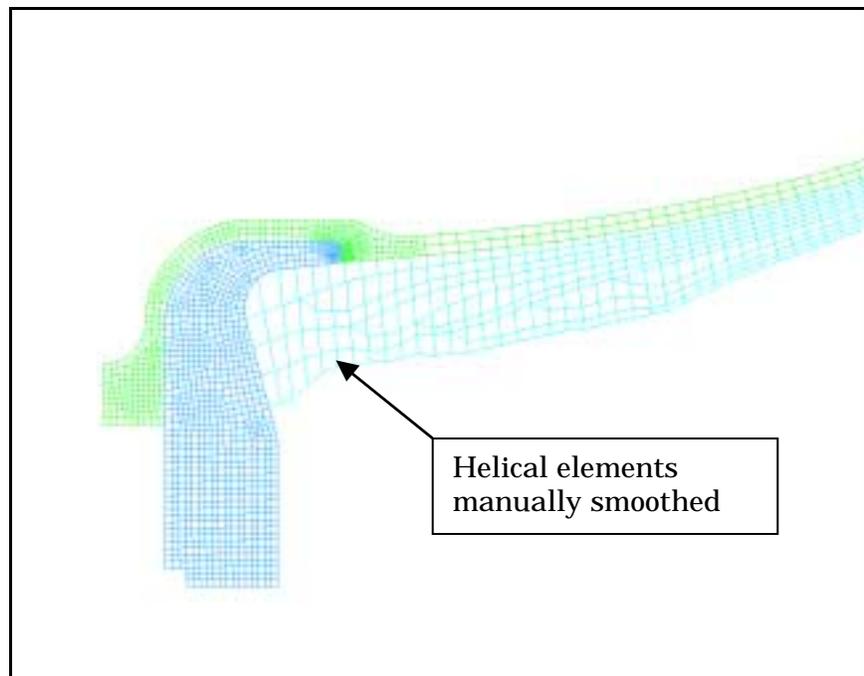


Figure 12: FEM with smoothed composite

Appendix 1

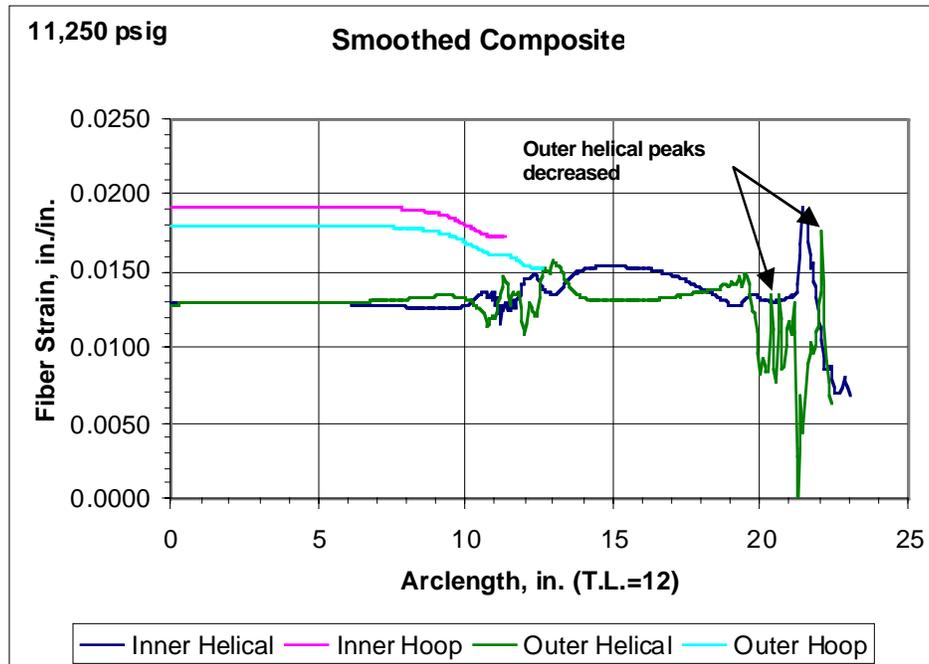


Figure 13: Fiber strain plot with smoothed composite

4.6.2 Wafer Design

The full analysis with all the hardware indicated a dome failure was a high possibility. Several design options were considered to minimize this failure mode.

1. Build a tank with just the new hoop termination pattern – the helical peak could not be as high as predicted.
2. Add one more helical layer to decrease the stress ratio.
3. Use a wafer to decrease the helical peak.

Since the next tank to be built required a high probability of meeting the burst strength target, option 1 was dismissed. Likewise, it was felt that option 2 would not bring the strain peak low enough. Also, the extra weight was undesirable since performance was key. Option 3 seemed to be the best choice. The weight would be minimally impacted by the addition of a wafer, plus, it had the most potential to bring the helical peak down.

The finite element model was modified to include a wafer. Analyses were done with various wafer dimensions, number of wafers, and locations of wafers. The design iterations are attached in Appendix C. The final design used one wafer between the 1st and 2nd helical layers. This location proved optimal in that the wafer would be positioned on a smooth surface contour compared to an outer layer where step-backs would cause bumps under the wafer. Also, the location greatly impacted the helical strain. The wafer model is pictured in Figure 14. The wafer is made of T1000 prepreg, same as the case, and includes an adhesive backing to hold it in place. The basic dimensions are a 2.9-inch inside diameter and a 6.6-inch outside diameter. The thickness increases from the inside diameter to the full 0.1-inch thickness over 0.25 inches, remains constant for 0.6 inches, then tapers down

Appendix 1

to the outside diameter. The model was run at ultimate pressure, and the fiber strain plot is given in Figure 15.

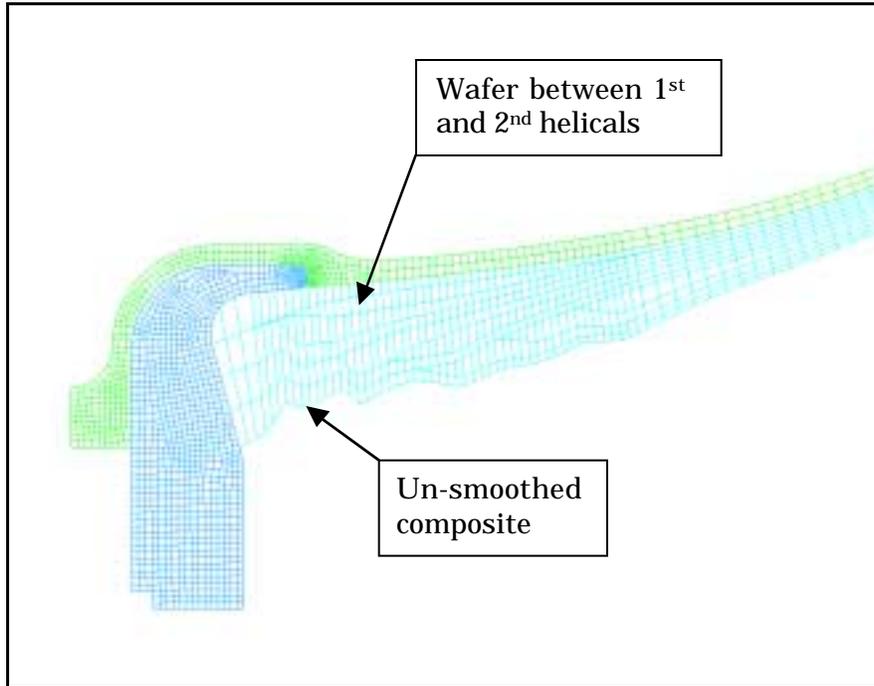


Figure 14: Wafer FEM

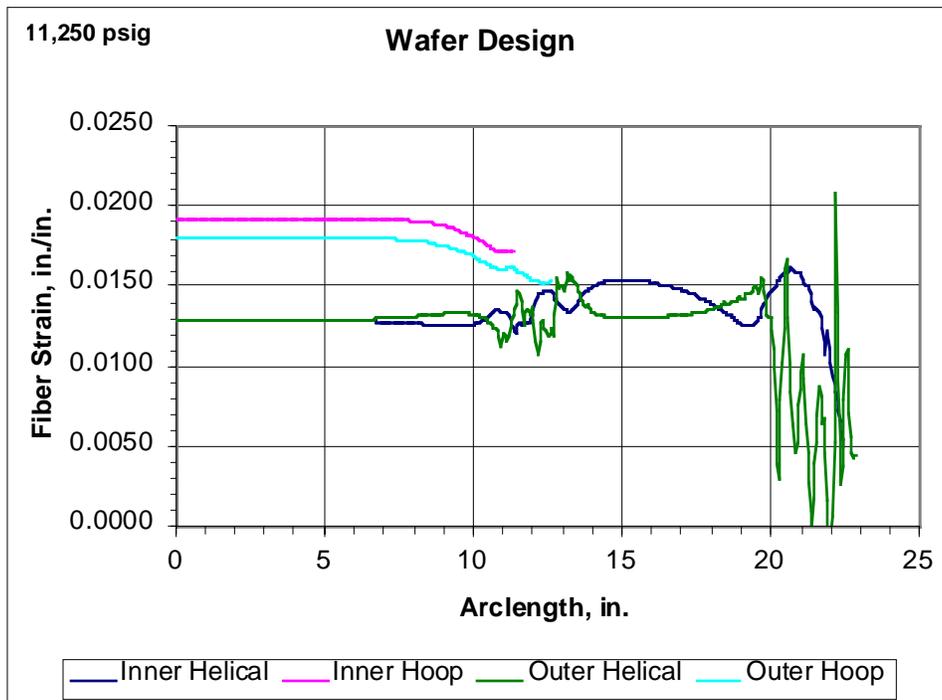


Figure 15: Wafer design fiber strain plot

Appendix 1

Compared to Figure 10, the wafer drastically decreased the fiber strain in the inner helical layer. As before, the outer helical peaks were deemed artificial.

With the new hoop termination pattern and the addition of the wafer in the dome, the design looked promising. The original winding drawing was revised to include the changes and is attached in the appendix.

4.6.3 Tank #4 Fabrication and Testing

Tank four was built incorporating the new design aspects. An annealed liner from ATL was used. The liner weighed 6 lbs. with an outside diameter measuring 16.54 inches (2 psig), about a half-inch smaller than the first three liners. The cylinder length measured 32.51 inches. Each wafer weighed 0.11 lbs., and the total tank weight was 54 lbs. One glass hoop ply secured the label. The internal volume was conservatively estimated to be 8,600 cu. in.

The tank was hydroburst tested on June 16, 2000 per TTP528 Rev. A. The burst pressure was 10,463 psig, 93% of the minimum burst pressure. The measured fiber strain at burst was 1.518%. Figure 16 shows the tank before burst, and Figure 17 shows the tank following the test. The failure was much more catastrophic compared to the previous bursts. The tank failed along the entire length of the cylinder. Whether the failure initiation location was mid-cylinder or at the tangent line is unknown. The domes in the polar boss region looked good with no evidence of fiber breakage except due to secondary impacts.



Figure 16: Tank #4 before hydroburst test

Appendix 1



Figure 17: Tank #4 after hydroburst test

5.0 CONCLUSION

Tanks 1-3 had a design flaw that made the tangent line the failure mode. Tank 4 was redesigned to fix the problem at the tangent line and improve the dome characteristics by adding a wafer; the stress ratio remained unchanged. The redesigned tank reached 93% of the minimum burst pressure requirement (11,250 psig), and 94.4% of the performance goal (1.8×10^6 inches). The fiber strain did not meet expectations. The lower than expected burst pressure was not resolved as a part of this effort.

If tank 4 failed in the cylinder region, more hoops could be added to the design to increase the burst pressure and performance goal, but this would increase the stress ratio and may move the failure mode to the domes. The cause of the low burst needs to be investigated so that the design can be modified or the material property allowables can be changed accordingly. Also, if liners are to be annealed, the mold needs to account for the shrinkage so that the designed internal tank volume is not decreased.

Appendix 1

APPENDIX A Tank Drawings

TD102286 Rev A - Polar Boss
TD102288 Rev A - Plastic Liner
TD102287 Rev B - Tank Drawing

Appendix 1

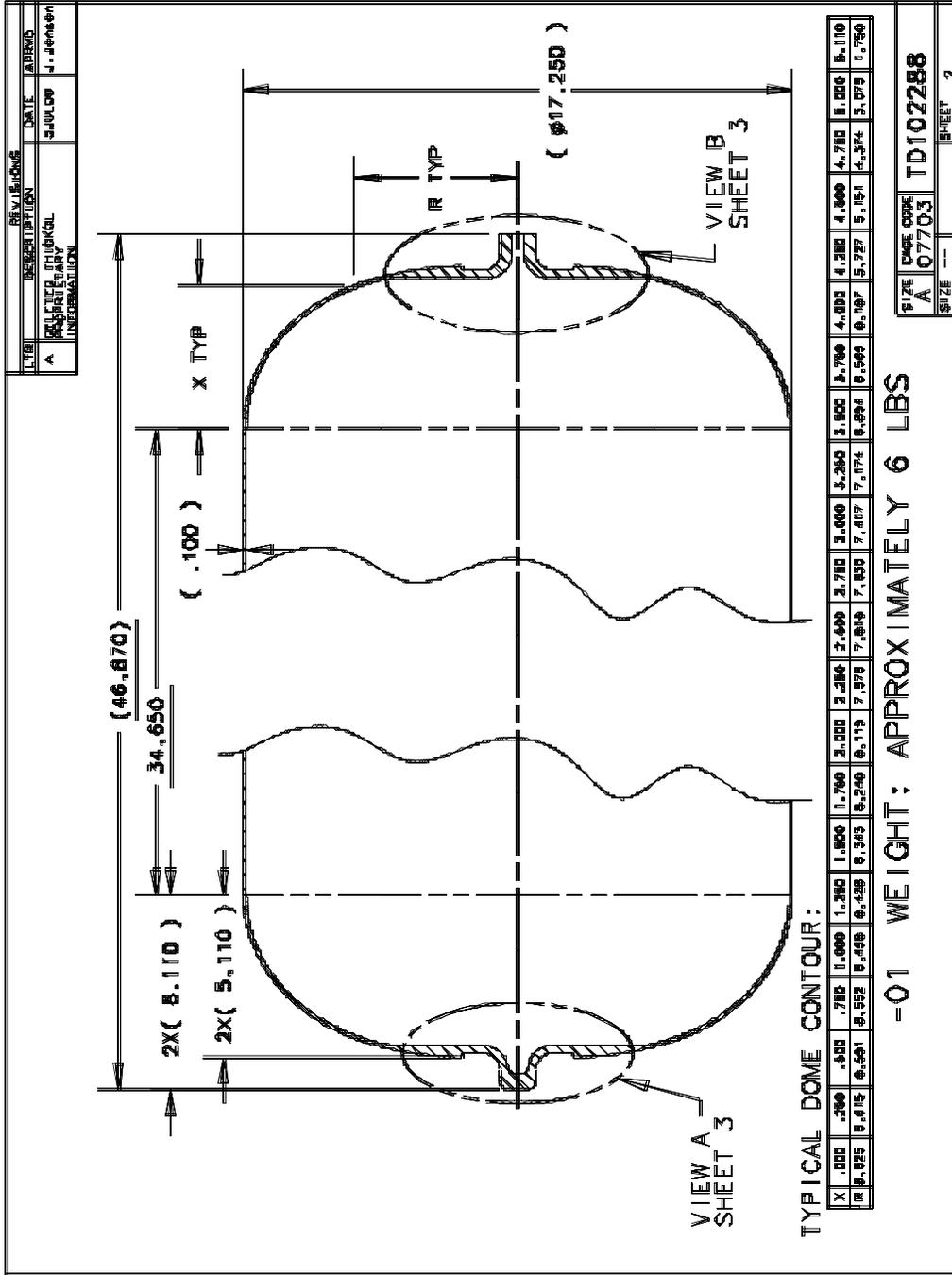
REV. NO.	DESCRIPTION	DATE	BY
A	DELETED TECH. CHGAL PROGRAM LEGACY INFORMATION	EAPLEO	J. JENSEN

NOTES:

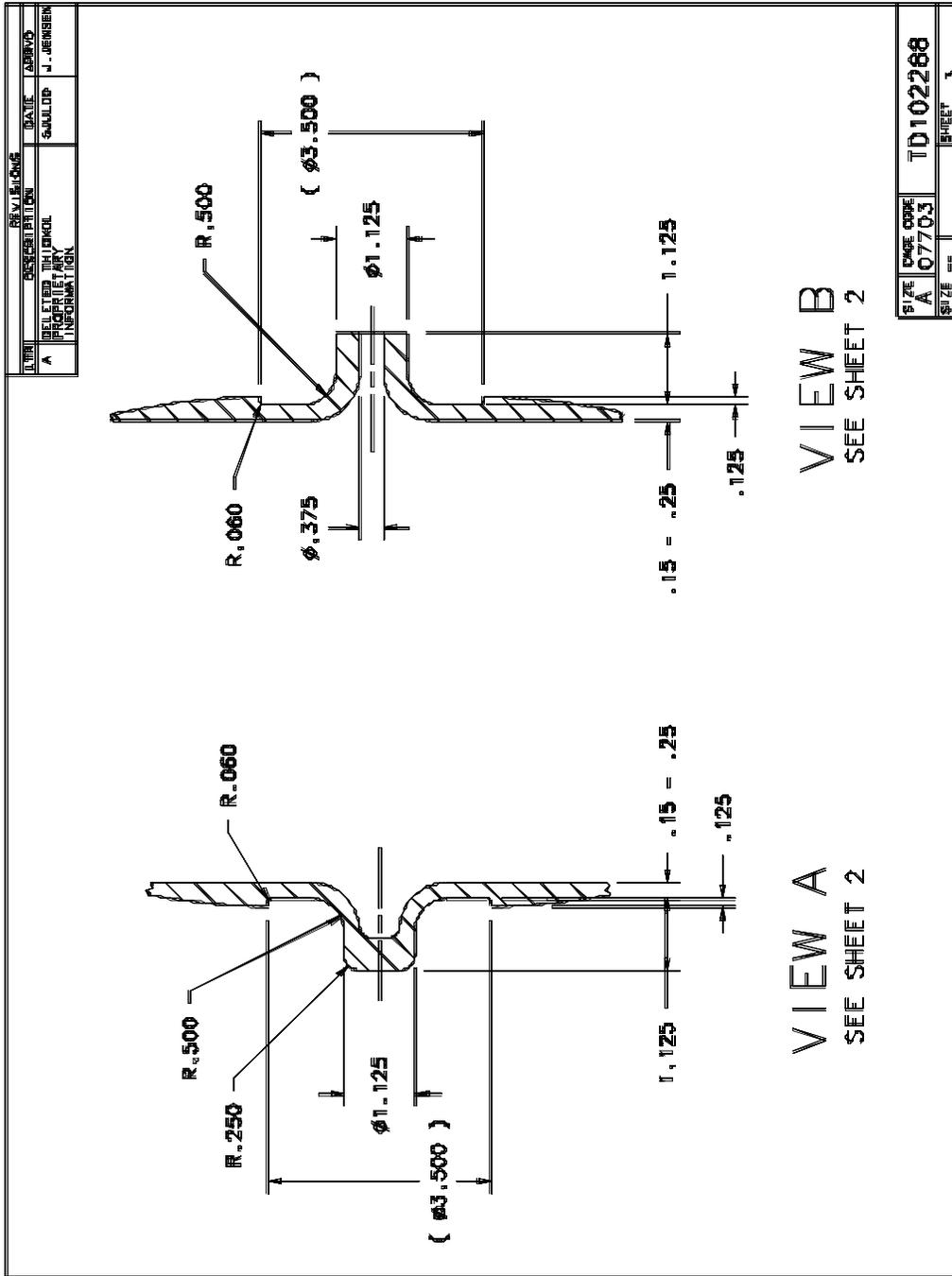
- 1 ACCEPTABLE MATERIAL: FOR -01: TBD.
- 2 OUTSIDE CONTOUR DIMENSIONS ARE FOR TOOLING INSPECTION ONLY. THICKNESS DIMENSIONS APPLY TO MOLDED PARTS.
- 3 MARK CASE CODE, PART NUMBER SERIAL NUMBER, AND MFG DATE USING .25 MIN HEIGHT LETTERS WITH BLACK PERMANENT INK.

CITY	REQD	UNIT	FIND	PART NUMBER	DESCRIPTION	MATERIAL SPECIFICATION	NOTE
<p>CONTRACT: -- UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES. TOLERANCES: .XX ± .005 .XXX ± .010 .XXX ± .015 ANGLES: --</p> <p>FINISH: -- ✓</p> <p>ALL DIMENSIONS APPLY TO THE COATING OR FINISH INTERIOR SURFACE UNLESS OTHERWISE SPECIFIED.</p>							
PARTS LIST							
D. A. BEKALBY		DATE: 22DEC08		FINISHED		BIRMAN CITY, MISSISSIPPI	
CHECKED:		R. K. KUNZ		LINER, 18-INCH DIA X 48-INCH LENGTH			
DRAWN BY:		J. J. JENSEN		SIZE: CASE CODE A 07703		TD102288	
CHECKED:		J. J. JENSEN		SIZE: --		SHEET 1 OF 3	

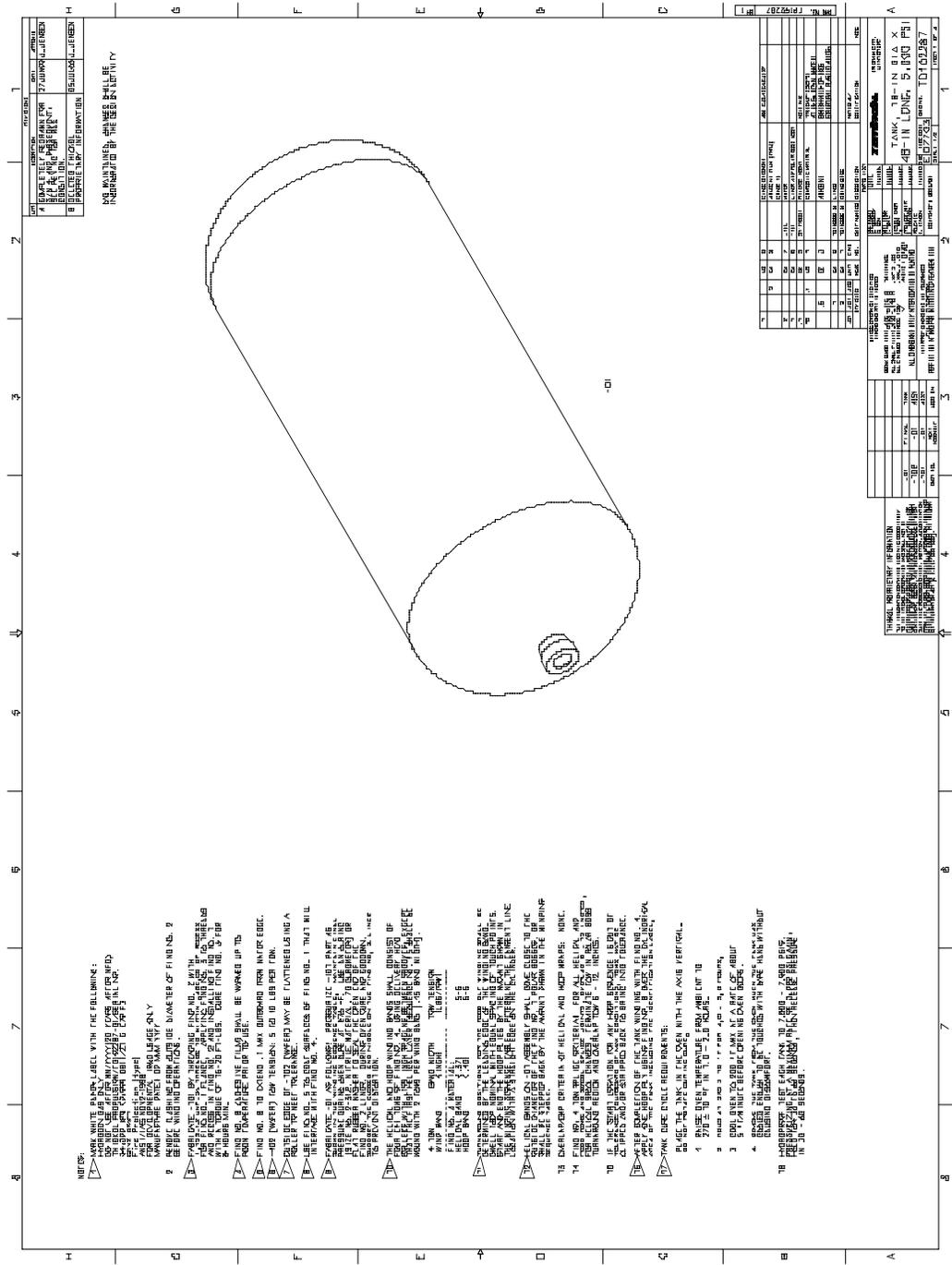
Appendix 1



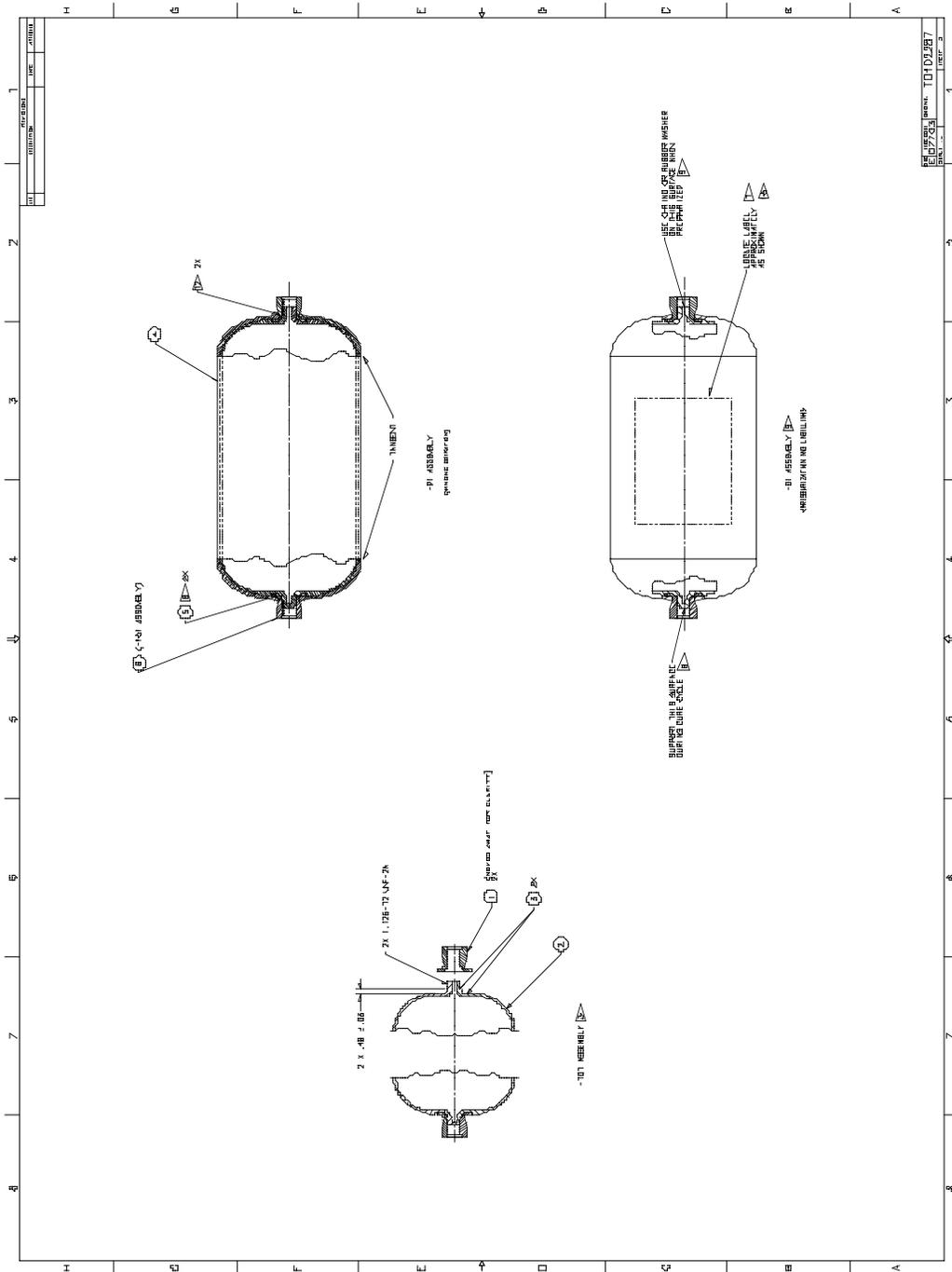
Appendix 1



Appendix 1



Appendix 1



Appendix 1

APPENDIX B Hoop Termination Design Iterations

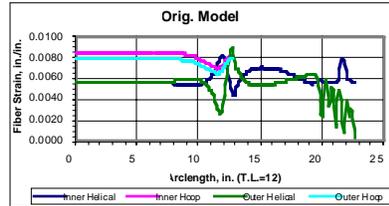
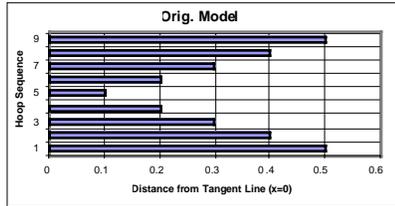
Appendix 1

LLNL 18" Hydrogen Tank Hoop Termination Design Iteration

Pressure = 5000 psi

Orig. Model

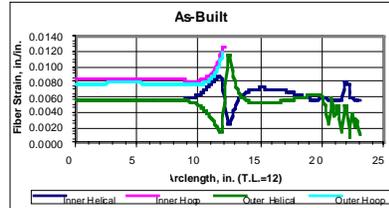
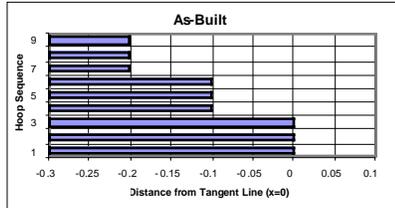
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4	0.2
5	0.1
6	0.2
7	0.3
8	0.4
9	0.5



inner hoop	0.0084	outer hoop	0.0078
T.L. max. hel.	0.00882		
hel./l.hoop	1.05	hel./o.hoop	1.13

As-Built

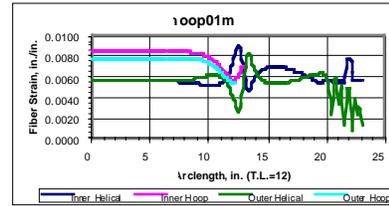
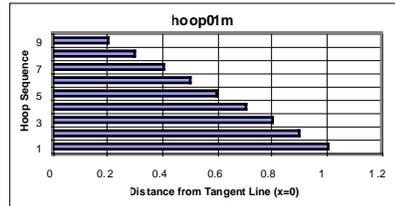
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5	-0.1
6	-0.1
7	-0.2
8	-0.2
9	-0.2



inner hoop	0.0084	outer hoop	0.0078
T.L. max. hel.	0.01157		
hel./l.hoop	1.38	hel./o.hoop	1.48

hoop01m.stk

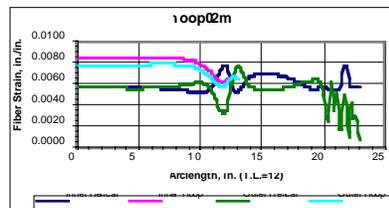
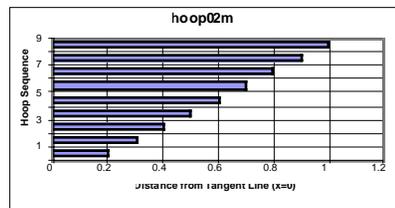
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9	0.2



inner hoop	0.0084	outer hoop	0.0078
T.L. max. hel.	0.00910		
hel./l.hoop	1.08	hel./o.hoop	1.16

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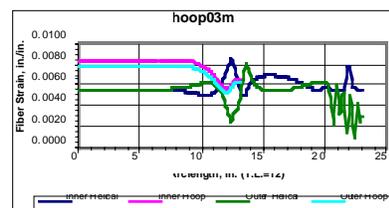
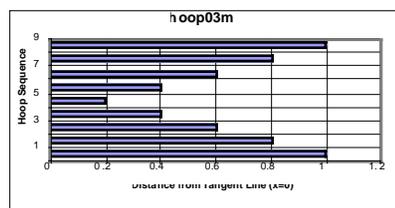
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6	0.7
7	0.8
8	0.9
9	1



inner hoop	0.0084	outer hoop	0.0078
T.L. max. hel.	0.00778		
hel./l.hoop	0.93	hel./o.hoop	0.99

hoop03m.stk

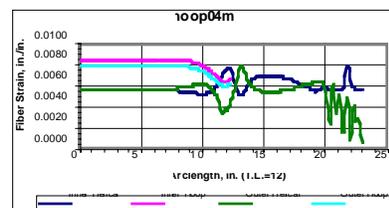
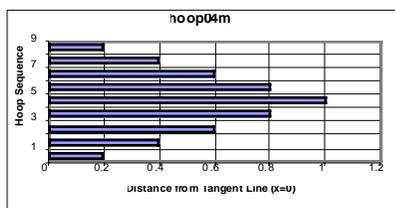
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3	0.6
4	0.4
5	0.2
6	0.4
7	0.6
8	0.8
9	1



inner hoop	0.0084	outer hoop	0.0078
T.L. max. hel.	0.00846		
hel./l.hoop	1.01	hel./o.hoop	1.08

hoop04m.stk

Seq.	Term.
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8	0.4
9	0.2

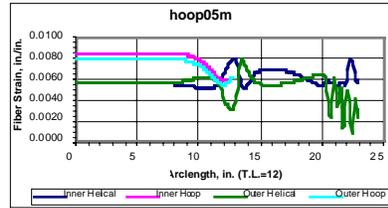
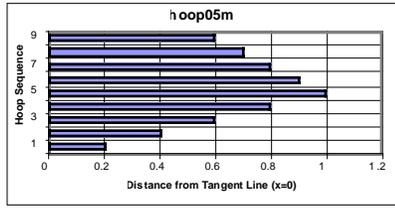


inner hoop	0.0084	outer hoop	0.0078
T.L. max. hel.	0.00779		
hel./l.hoop	0.93	hel./o.hoop	0.99

Appendix 1

hoop05m.stk

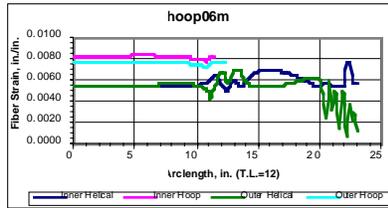
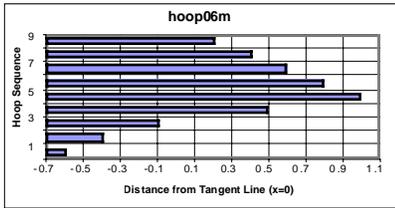
Seq.	Term.
1	0.2
2	0.4
3	0.6
4	0.8
5	1
6	0.9
7	0.8
8	0.7
9	0.6



inner hoop	0.0084	outer hoop	0.0078
T.L. max. hel.	0.00795		
hel./l.hoop	0.95	hel./o.hoop	1.02

hoop06m.stk

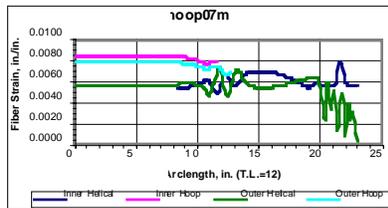
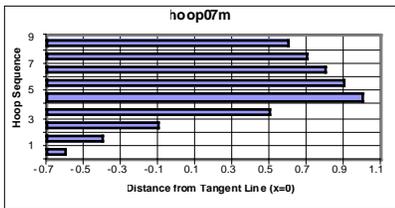
Seq.	Term.
1	-0.6
2	-0.4
3	-0.1
4	0.5
5	1
6	0.8
7	0.6
8	0.4
9	0.2



inner hoop	0.0084	outer hoop	0.0078
T.L. max. hel.	0.00710		
hel./l.hoop	0.85	hel./o.hoop	0.91

hoop07m.stk

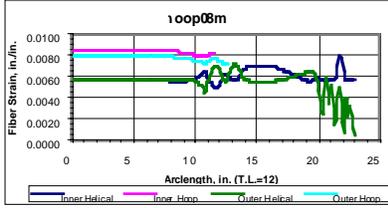
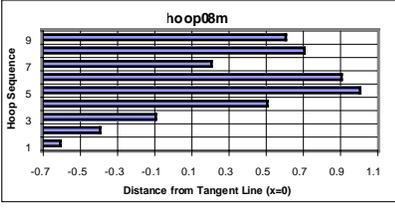
Seq.	Term.
1	-0.6
2	-0.4
3	-0.1
4	0.5
5	1
6	0.9
7	0.8
8	0.7
9	0.6



inner hoop	0.0084	outer hoop	0.0078
T.L. max. hel.	0.00727		
hel./l.hoop	0.87	hel./o.hoop	0.93

hoop08m.stk

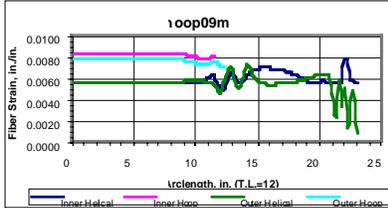
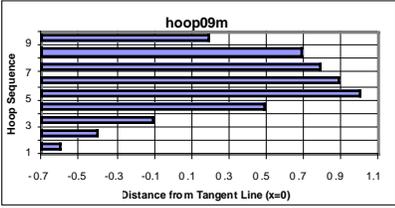
Seq.	Term.
1	-0.6
2	-0.4
3	-0.1
4	0.5
5	1
6	0.9
7	0.2
8	0.7
9	0.6



inner hoop	0.0084	outer hoop	0.0078
T.L. max. hel.	0.00709		
hel./l.hoop	0.84	hel./o.hoop	0.90

hoop09m.stk

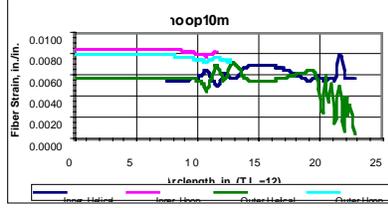
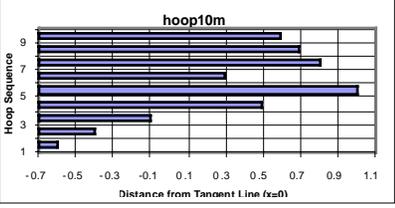
Seq.	Term.
1	-0.6
2	-0.4
3	-0.1
4	0.5
5	1
6	0.9
7	0.8
8	0.7
9	0.2



inner hoop	0.0084	outer hoop	0.0078
T.L. max. hel.	0.00720		
hel./l.hoop	0.86	hel./o.hoop	0.92

hoop10m.stk

Seq.	Term.
1	-0.6
2	-0.4
3	-0.1
4	0.5
5	1
6	0.3
7	0.8
8	0.7
9	0.6

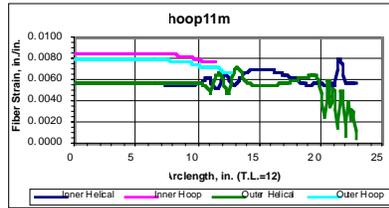
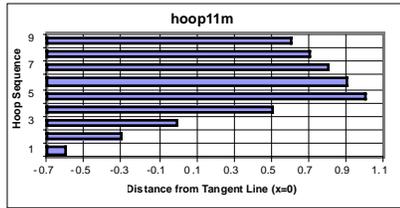


inner hoop	0.0084	outer hoop	0.0078
T.L. max. hel.	0.00710		
hel./l.hoop	0.85	hel./o.hoop	0.91

Appendix 1

hoop11m.stk

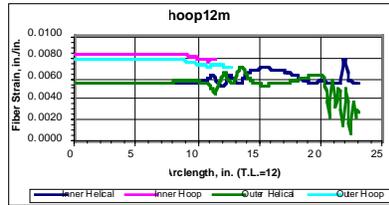
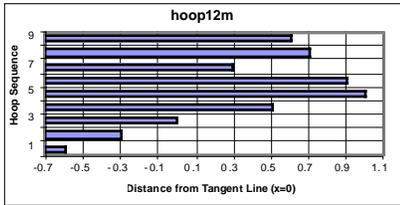
Seq.	Term.
1	-0.6
2	-0.3
3	0
4	0.5
5	1
6	0.9
7	0.8
8	0.7
9	0.6



inner hoop	outer hoop
0.0084	0.0078
T.L. max. hel.	
0.00721	
hel./l.hoop	hel./o.hoop
0.86	0.92

hoop12m.stk

Seq.	Term.
1	-0.6
2	-0.3
3	0
4	0.5
5	1
6	0.9
7	0.3
8	0.7
9	0.6



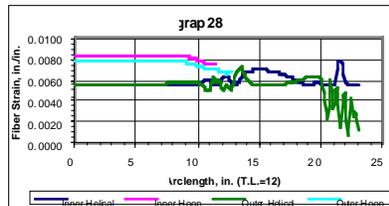
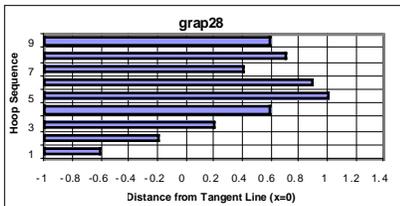
inner hoop	outer hoop
0.0084	0.0078
T.L. max. hel.	
0.00713	
hel./l.hoop	hel./o.hoop
0.85	0.91

NOTE: hoop12m chosen over hoop08m because here is more margin between the hoop strain in the cylinder and near the termination where it peaks up (arclength 11-12).

Sensitivity for hoop12m.stk

grap28.stk

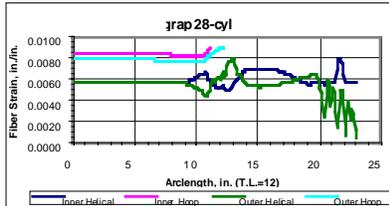
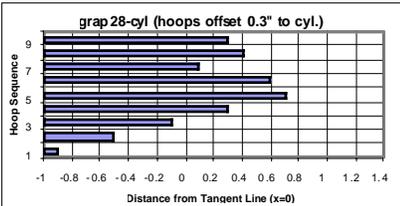
Seq.	Term.
1	-0.6
2	-0.2
3	0.2
4	0.6
5	1
6	0.9
7	0.4
8	0.7
9	0.6



inner hoop	outer hoop
0.0084	0.0078
T.L. max. hel.	
0.00719	
hel./l.hoop	hel./o.hoop
0.86	0.92

grap28-cyl.stk

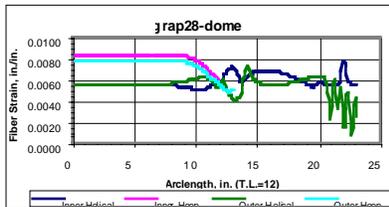
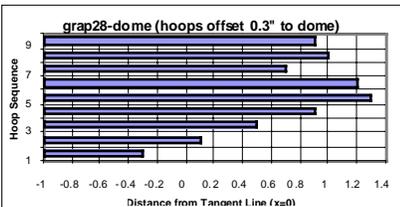
Seq.	Term.
1	-0.9
2	-0.5
3	-0.1
4	0.3
5	0.7
6	0.6
7	0.1
8	0.4
9	0.3



inner hoop	outer hoop
0.0084	0.0078
T.L. max. hel.	T.L. max. hoop
0.00783	0.00902
hel./l.hoop	hel./o.hoop
0.93	1.00

grap28-dome.stk

Seq.	Term.
1	-0.3
2	0.1
3	0.5
4	0.9
5	1.3
6	1.2
7	0.7
8	1
9	0.9



inner hoop	outer hoop
0.0084	0.0078
T.L. max. hel.	
0.00731	
hel./l.hoop	hel./o.hoop
0.87	0.93

hoops cannot vary towards the cylinder!

Appendix 1

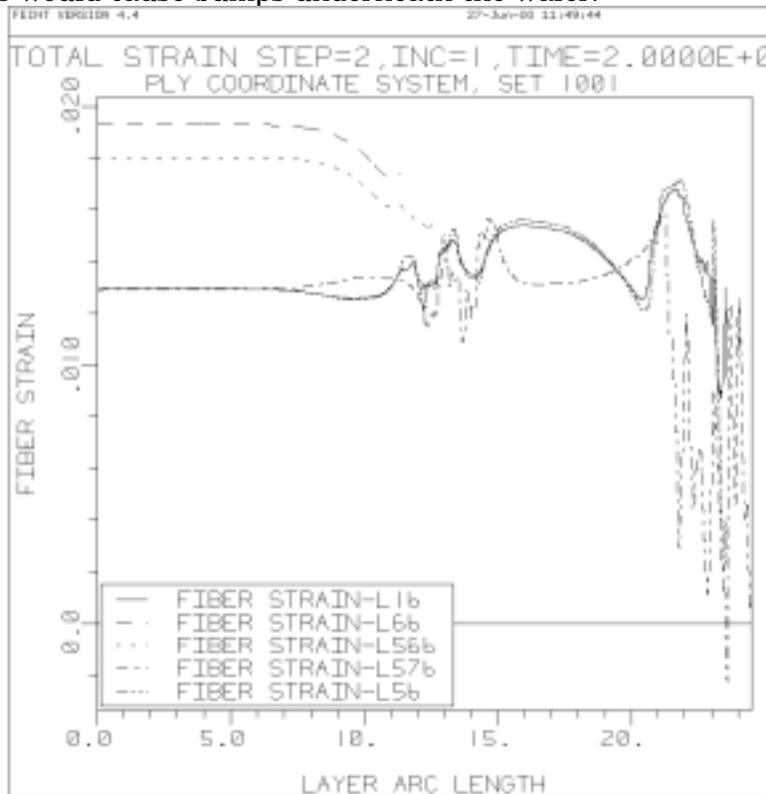
APPENDIX C Wafer Design Iterations

Appendix 1

Wafer Design Iteration

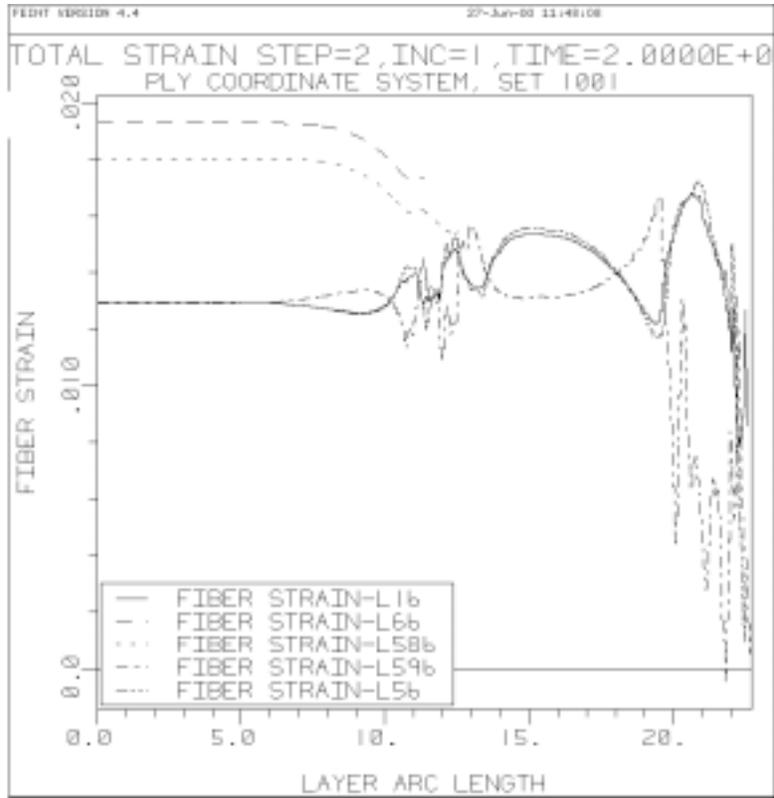
Fiber Strain Plots at 11,250 psig (Composite only)

Run 6 was used as the baseline design. Runs 4 and 7 were comparable to Run 6 and showed that the design could tolerate dimensional changes with little impact to the peak helical fiber strain. Runs 1,2, and 8 showed slightly higher strain peaks, while Runs 3 and 5 were unacceptable. The two wafer design in Run 2 did not give better performance compared to using just one wafer. Placing the wafer between the 1st and 2nd helical layers was desirable since the surface contour was smoother compared to outer layers where helical step-backs would cause bumps underneath the wafer.

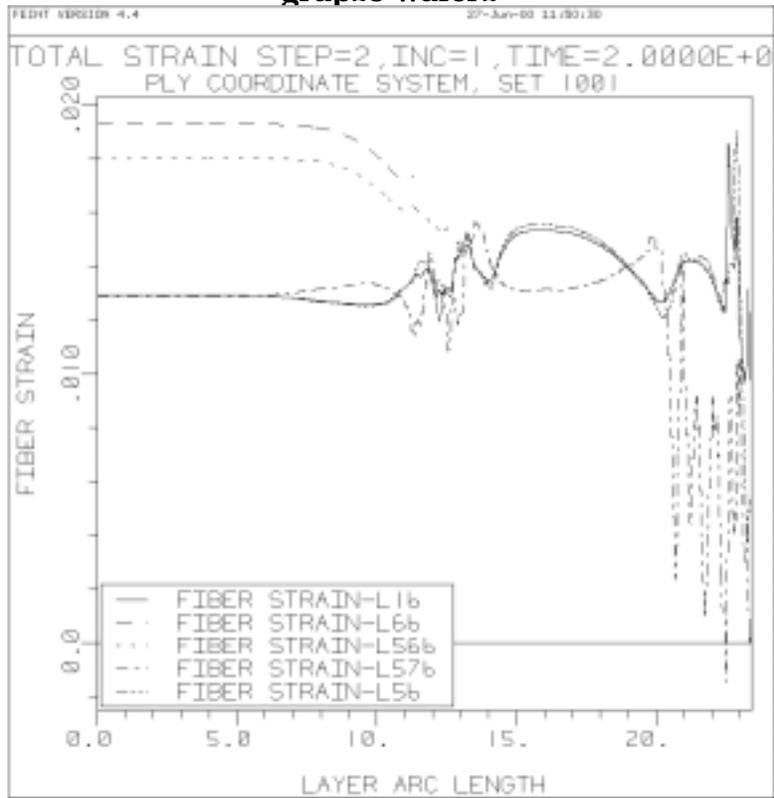


grap28-wafer1

Appendix 1

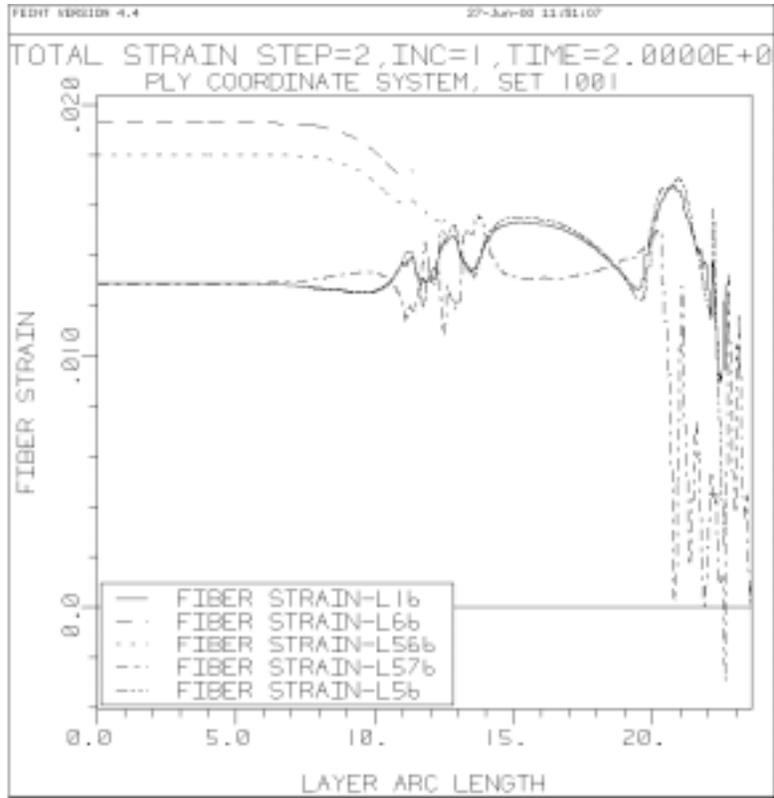


grap28-wafer2

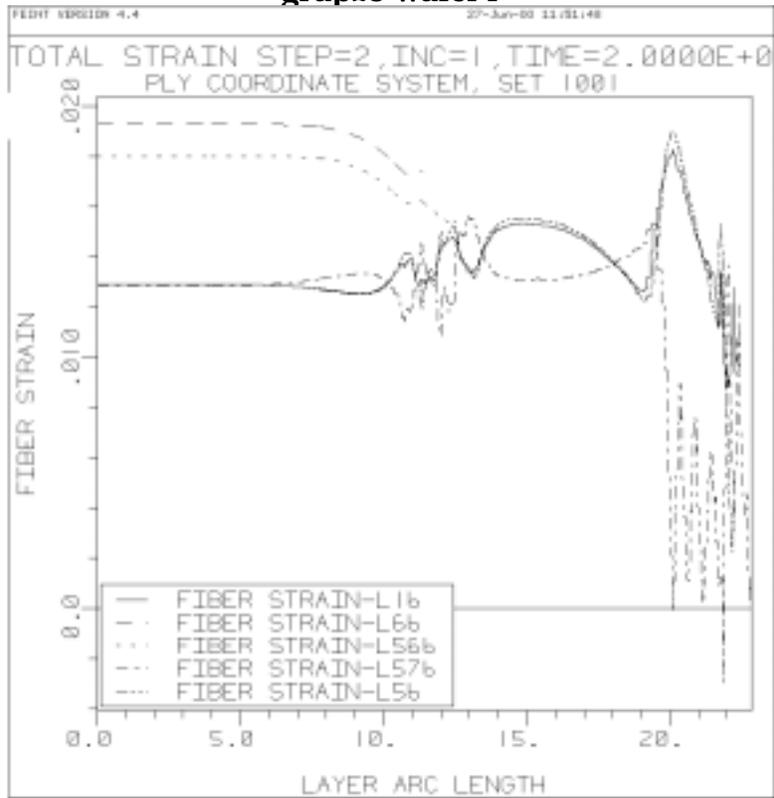


grap28-wafer3

Appendix 1

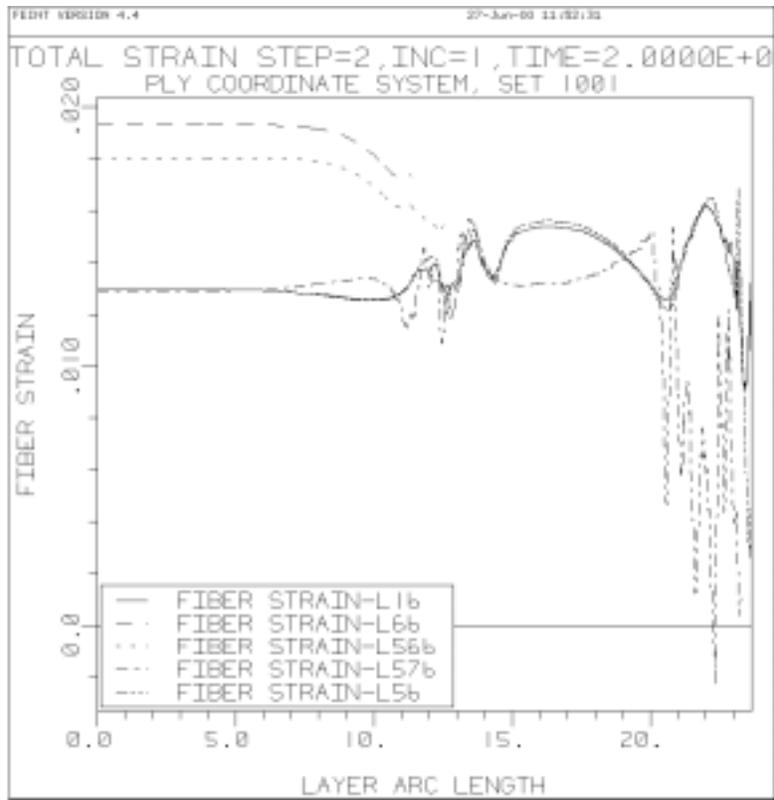


grap28-wafer4

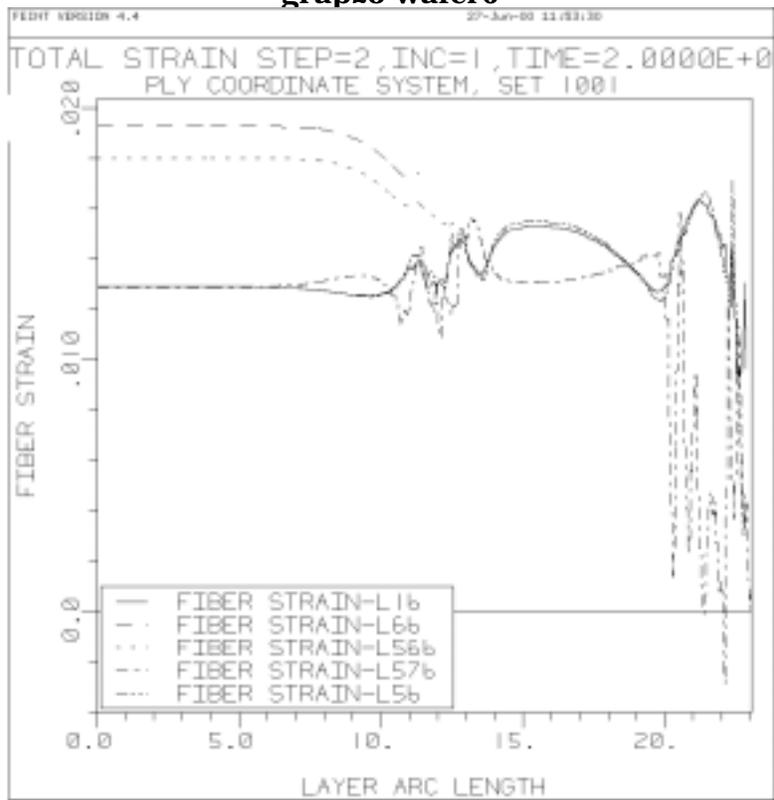


grap28-wafer5

Appendix 1

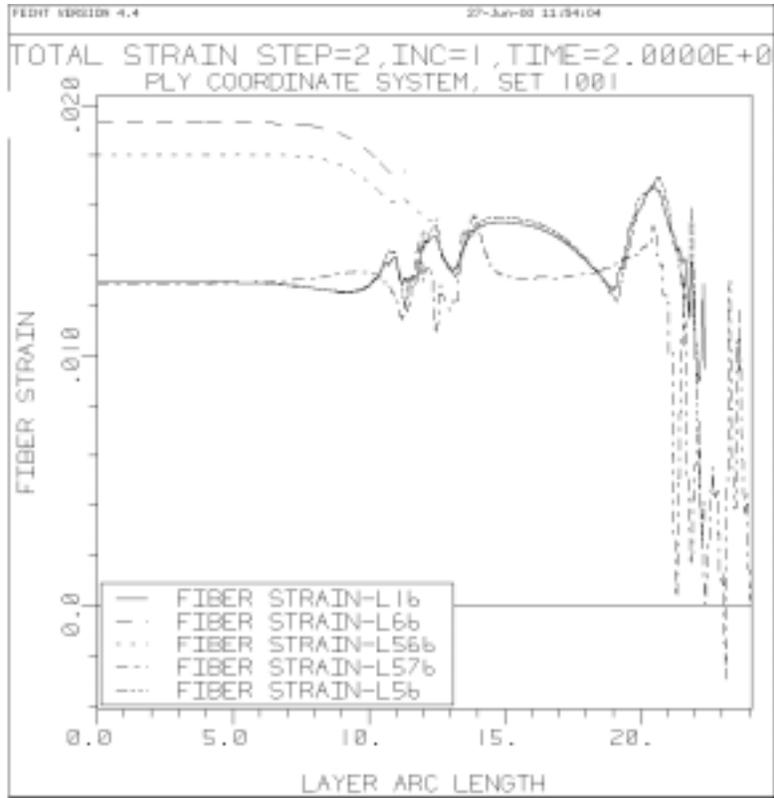


grap28-wafer6



grap28-wafer7

Appendix 1



grap28-wafer8

Appendix 1

APPENDIX D Test Plan TTP528

Appendix 1

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