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Rapid Production of Composite Prototype Hardware

(MSFC Center Director's Discretionary Fund Final Report, Project No. 96–02)

T.K. DeLay
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March 2000

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LIST OF ACRONYMS

ASTP	Advanced Space Transportation program
CDDF	Center Director's Discretionary Fund
CNG	compressed natural gas
CTE	coefficient of thermal expansion
ETFE	ethylene-tetrafluoroethylene
IR&D	Independent Reasearch and Development
lox	liquid oxygen
FEP	fluorinated ethylene propylene
IR&D	Independent Research and Development
MSFC	Marshall Space Flight Center
NRA	NASA Research Announcement
PBO	polybenzoxazole
PI	Principal Investigator
PVDF	polyvinylidene fluoride
R&D	research and development
RLV	reusable launch vehicle
UV	ultraviolet
SLA	StereoLithography

TECHNICAL MEMORANDUM

RAPID PRODUCTION OF COMPOSITE PROTOTYPE HARDWARE
(MSFC Center Directors's Discretionary Fund Final Report, Project No. 96–02)

1. INTRODUCTION

The composites industry has been changing at a very rapid pace in many areas, including aerospace, defense, sporting goods, and consumer products. The newer launch vehicle programs being pursued by NASA and industry partners are pushing aggressively toward cheaper access to space. This often requires the use of composite materials to make the overall structure lighter and stronger. The design changes of the reusable launch vehicles (RLV's) are rather frequent; hence, costs are incurred in many areas, especially tooling. The need for producing cost-effective prototypes to test new ideas and clarify design issues before new programs become too mature has become much more of a necessity. Subscale prototypes are also very useful in the development phase of new manufacturing processes while the design is still changeable and before a large investment in full-scaled tooling has been made. The author's intent was to remain flexible for rapid changes in the needs of future programs and produce composite prototypes to benefit these programs.

2. METHOD

The initial phase of this research was to identify some of the most pressing needs of the RLV projects and search for cost-effective methods to test new designs and manufacturing processes. The approach of this research was intended to be highly “hands-on” and create innovative manufacturing methods that push the state of the art. Prototype composite structures are then produced and later refined for potential use in mainstream projects. The new manufacturing methods are offered to these projects for further development and testing as the Principle Investigator (PI) moves on to other prototypes. An effort is made to keep in tune with what is being developed in the nonaerospace/commercial and recreational community. This approach was chosen to foster an environment for developing creative ideas.

3. TASKS AND PROJECTS DEVELOPED

3.1 Composite Lines and Ducts

One of the first areas that required attention was the need to save weight for the X–33 and VentureStar programs. Several projects were already in place to develop large airframe structures; however, little was being done to address the potential weight savings of small components. It became apparent that a tooling method was needed to fabricate prototype composite feed lines and manifold structures. Industry has mature methods for producing large, straight runs of ducting. However, smaller complex structures with flanges are still costly to produce and heavy, due to the weight of the flanges. A method was developed to produce modular urethane foam duct components that could be fitted to the needed configuration. This method began with a scrap stainless steel pipe elbow that served as a mandrel for a composite mold. This mold was later filled with a urethane tooling foam (MARCORE) that was developed by Lockheed Martin Corporation. The elbow fittings were fitted with straight sections of the same tooling foam to form complex piping structures (figs. 1 and 2). The foam mandrel was encapsulated with multiple layers of graphite/epoxy prepregged fabric, vacuum bagged, and autoclave cured. After the cure was complete, the tooling foam was extracted by mechanical means and a high-pressure (3,500 psi) water sprayer (fig. 3). This same method was used to produce manifold structures and "T" fittings. Urethane tooling foams also proved to be useful in the development of coatings for the inside of composite ducting.

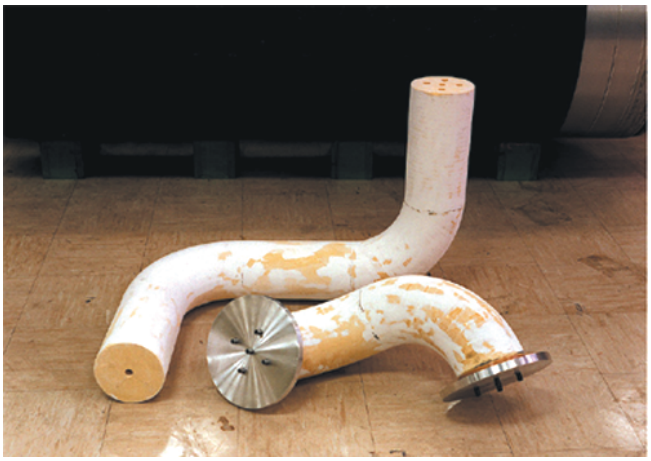

Figure 1. Complex foam mandrel.

Figure 2. Prototype composite duct with integral flanges.

Figure 3. Extraction of foam mandrel using high-pressure water sprayer.

3.2 Foam Tooling Methods

Many of the mainstream RLV projects are focusing on unlined composite structures. However, it became apparent that few composite materials are compatible with fluids such as liquid oxygen (lox), and that microcracking often results in the composite structures due to low temperatures. A method was developed that involved thermally spraying (fig. 4) the foam mandrel cast in a mold (fig. 5) with a thin layer of aluminum (service provided by Plasma Processes, Inc.), followed by an overwrap of composite materials (fig. 6). This technique initially appeared to be promising. However, it was later found that the liner was brittle and cracked as the foam expanded slightly when heated during the cure cycle of the composite overwrap. The PI performed some crude tests to help characterize or control the foam expansion. This included a secondary source of a similar tooling foam (Alpha Products, Inc.). Multiple 6-in. cubes of different tooling foams were exposed to different temperature ranges and autoclave pressures. Oven temperatures varied between 250–350 °F and autoclave pressures ranged between 25 and 100 psi. The formulations of the tooling foam were adjusted to control the density from 8 to 20 lb/ft^3. Basically, it became apparent that the foam expansion can be controlled by changing variables. The most reliable temperature and pressure combination was 250 °F and 50 psi.

Figure 4. Thermal spraying of aluminum liner.

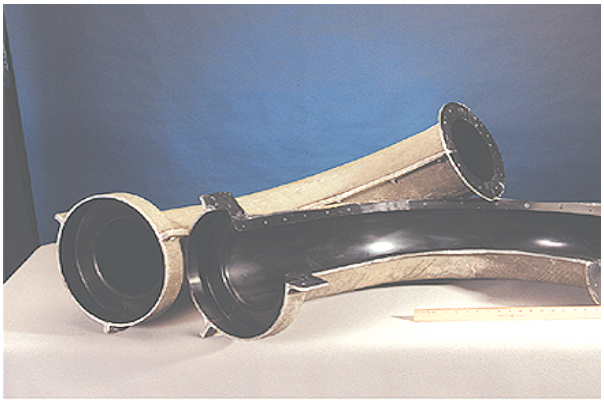

Figure 5. Mold tooling for casting foam mandrels.

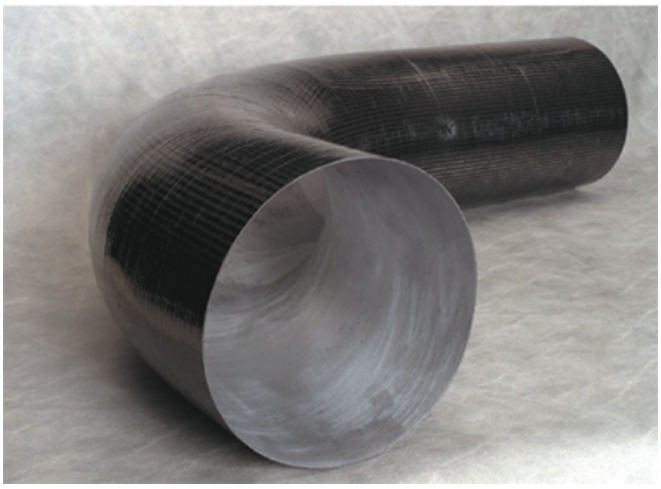

Figure 6. Aluminum-lined composite duct.

3.3 Metal Coating of Composites

Numerous possibilities were considered to provide a metal liner in composite ducting such as vacuum vapor arc deposition (developed by Rocketdyne and the University of Alabama in Huntsville) or electrodeposition. The former method is restricted to rather small articles. Electroformed Nickel, Inc. has developed a method for depositing layers of metal on disposable mandrels with great reliability. The PI modified this approach by curing and adding a composite overwrap structure on the metal liner before the mandrel is removed. This may prove to be an excellent way of producing metal-lined composite ducts. The author later decided to go further with this concept with pressure vessels.

3.4 Low-Cost Tooling

A small company, Heidelburg, International, located in the rural hill country of northern Georgia, was developing an all-composite race car body. They suggested an innovative, low-cost tooling approach for composite ducting. The method involved the use of a spiral-formed metallic heating, ventilation, and air conditioning duct which serves as the mandrel. The mandrel would be bent to the desired configuration and its grooves filled and finished. This tool would then be covered with multiple layers of graphite/epoxy fabric and oven cured. The mandrel was extracted from within the composite duct by unraveling the spirals of the metallic form. This method did not have a high tolerance for dimensional stability but shows promise for providing a quick-turnaround prototype. This method may be useful in areas such as test stands where it is difficult to fit a complex duct, or perhaps when prints of the configuration are not available.

3.5 Braided Fibers and Resin Infusion

The initial ducts produced by previous methods required multiple cuts in the fabric, compromising the strength of the structure. Braided sleeves of carbon fiber (fig. 7) were incorporated in the fabrication process of later ducts. Preimpregnated braiding was not very usable because of the difficulty of the fibers to conform to the complex geometry of the mandrel. A method was developed that utilized dry carbon braiding that was later infused with a layer of filmed epoxy resin. This method, with a little trial

and error, also allows for control of the resin content by the weight of the filmed resin and the number of layers. An advantage of this approach was that resin vendors can produce custom blends of epoxy resins that could be tested for new programs with little cost. The cost was minimized because the material suppliers did not need to interrupt the product runs of prepregged fabric for small trial samples. Another benefit of the braiding was that it could also be incorporated with prepregged fabric. This was especially useful in the buildup of material in flange areas of the ducts. Composite ducts with integral flanges require multiple cuts of the woven fabrics. The fibers of braided sleeves conform to the complex curvatures without the need to cut the carbon fibers.

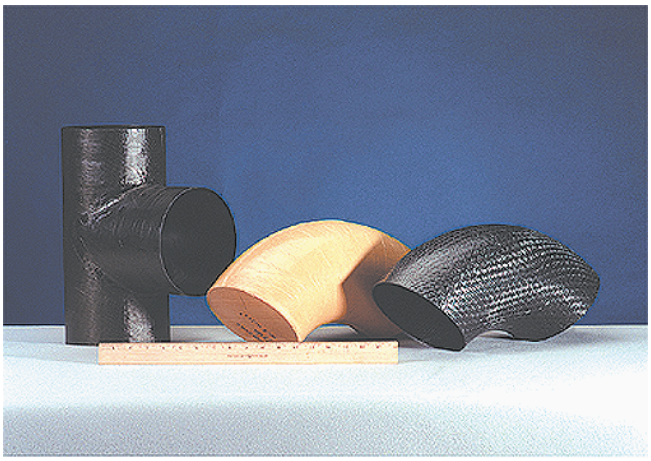

Figure 7. Ducts using braided carbon.

NASA Research Announcement (NRA) 8–21 (composite lines and ducts) was awarded to Marshall Space Flight Center (MSFC) under the Advanced Space Transportation program (ASTP) in the later part of 1998. The methods produced under this Center Director's Discretionary Fund (CDDF) research effort proved to be very useful in the jumpstart of this project. This NRA utilized the urethane and eutectic salt tooling method (fig. 8) for production of the initial ducts and was later used along with a cast and machined segmented aluminum tool produced by a patternmaker. The prototype tooling methods were utilized to test the initial concepts before a much larger investment was made in more permanent tooling.

Figure 8. Eutectic salt mandrel for NRA 8–21.

3.6 Composite Bat Structure

The use of braided fibers and filmed resins were also useful for the development of prototypes supporting the commercial sector. A method was produced that involved the use of several fiber types and thermoplastic and thermoset matrix resins to fabricate a composite softball bat. It has been estimated that this is potentially a $150M/yr market. Patents were filed for two of the more promising concepts. A bat structure with a nylon-6/aramid fiber barrel section and an epoxy/carbon handle performed well. A foam filler was also utilized to tune bat performance. The tooling utilized for the composite bat structures involved pneumatic mandrels and expandable foams trapped in molds. Technology will sometimes travel in a circle—from aerospace, to commercial goods, and back. The author is currently modifying one of the softball bat processes to be used for aerospace structures.

3.7 Ultraviolet Curing

A small effort was made to test the feasibility of using ultraviolet (UV) curing additives to stage epoxy resins. Loctite developed a UV-sensitive additive (Loctite 303) that can be added to epoxy resin systems. The additive, when exposed to UV light, initiates a chemical reaction that gels the resin. A preimpregnate fabric is not available with the additive, so a rudimentary impregnator was produced in order to wet small batches of fabric. This method became quite difficult for fabric, but it works for single tows in filament winding of composite cylinders. An advantage of the additives was the considerably less waste of epoxy resin on wet-wound structures. The additive was offered to the Fastrac composite combustion chamber program as a method to speed production, but it was decided that it would be too costly to validate any changes in the chosen processes.

3.8 Stereolithography Tooling

Rapid prototyping was sampled as a means of producing mandrels and support tooling directly from the StereoLithograhy (SLA) machine. The resin used for SLA produces a fine finish; however, it softens and loses dimensional stability at ≈200 °F. A lower temperature tooling prepreg had to be used on the SLA master first. The tooling prepreg was cured at 120 °F, removed from the master, and postcured at 350 °F. This additional step produced a satisfactory end item but time could be saved if the SLA resin could handle temperatures at ≈300 °F.

3.9 Epoxy Tooling

The PI received additional toolmaking experience through a Ciba Chemical, Inc.-sponsored class at Georgia Tech. The class focused on a new epoxy tooling system developed for the production of molds for resin-injected plastic automotive body and interior components. This method can be adapted to other tooling applications. The automotive industry in Detroit has benefited greatly from prototyping and has innovated many new processes. The PI is currently designing a mold that can be used to cast mandrels for composite pressure vessels.

One of the advantages of the CDDF program is it provides a mechanism to rapidly respond to the needs of other programs. Another example of this is the support of the x-ray mirrors for the Constellation-x program. The project is looking for new methods to save weight and provide a backup structure for replicated optics mirrors. To produce a prototype structure, the author used some materials

made available from this CDDF project. Since then, the project has funded an optical company to study the use of composites for some of the structures needed for the telescope. Composites for optical support structures look rather promising, but more work is needed.

3.10 Hydrogen Peroxide Tanks and Cryogenic Tanks for Upper Stages

After completion of the above tasks (Nov. 1998), the author was preparing to finalize the research. However, several of the composite tank concepts produced for RLV's by outside contractors developed noticeable leaks. There has also been a sudden increase in the interest of using high-test hydrogen peroxide (90- to 98-percent concentration) as an oxidizer in some of the upper-stage propulsion flight experiments (X–37, X–34, etc.). Very few materials are compatible with high-test peroxide, especially >90-percent concentration. The MARS ascent vehicle program has long-term storage needs for cryogenic oxygen. Because of the above needs, the PI chose to modify some of the developed tooling methods to produce lined and unlined composite tank concepts that show promise in being compatible with these fluids. Fortunately, the Technology Transfer office provided supplemental funding through the Technology Investment program. The extended research also led to the development of several patentable concepts for composite tanks and ducts.

3.11 Tank Liner Development

3.11.1 Tooling Methods

Several manufacturing processes were mated to allow fabrication of the composite structures. This required considerable experimentation with the choice of liner materials, tooling materials for the mandrels, and composite materials suitable for the overwrap structure. Tooling used for the tank mandrels involved water-soluble eutectic salts, urethane foams, cast aluminum, and mild steel outfitted with the appropriate metallic end-fittings. Eutectic salt has the advantage of being water soluble and performs quite well as long as the material remains below its melt temperature (450 °F). Urethane foams have the advantage of being lightweight, castable, and machineable but are difficult to remove if the ends of the composite vessels are relatively small. A cast aluminum segmented mandrel costs a bit more to fabricate but has the advantage of being fairly light and reusable. The segmented steel mandrel has a lower coefficient of thermal expansion than the aluminum but is rather heavy.

3.11.2 Thermoplastic Liners

Several liners were chosen for this effort. Table 1 shows the basic liner properties. The nonmetallic liners include polyvinylidene fluoride (PVDF), fluorinated ethylene propylene (FEP), ethylene-tetrafluoroethylen (ETFE), polyethylene, nylon 11, and nylon 6. The fluorinated polymers were chosen because of their compatibility with high-test peroxide. Polyethylene and nylon were selected for ease of processability and compatibility with kerosene-based fuels. Roto-molded thermoplastic liners are now commercially available for the compressed natural gas (CNG) market; however, they are too heavy to be considered for aerospace applications. Thermal spraying of the nonmetallic liners was initially chosen for its ability to change the processing variables and raw materials. Thermally sprayed metals also have the potential of being scaled to a very large structure and of being very lightweight. The nonmetallic liner development required the careful selection of the appropriate mold release system and surface preparation technique. A series of 4-in. by 4-in. aluminum panels were utilized to perform the initial

screening to avoid damaging the expensive tooling. The thermoplastic-lined composite tank concept requires that the liner material stay on the heated mandrel during the coating process but does not become bonded to the surface after cooling. A tool surface that is too smooth and/or has a chemical mold release that works too well causes the molten thermoplastic material to slip off the mandrel before it has time to cool or be fully coated. A surface that is too rough can create a mechanical bond with the coating, and a poor performing mold release can also result in a bond to the tooling material. For this particular application, it was found that the ideal combination was a smooth mandrel surface (not highly polished) coated with an initial layer of a fluorinated release (Freekote 700nc or Chemlease 70), followed by a thin film of a water-based wax (Chemlease 58R). The liner material must also be chosen to be compatible with the curing process of the composite overwrap structure. The melt temperature of the liner material and the cure temperature of the epoxy resin of the composite overwrap can be paired to ruin or enhance the effectiveness of the liner. It was found that in one particular case, the melt temperature of a polyethylene/acrylic copolymer (figs. 9 and 10) was very close to the cure temperature of the epoxy resin, resulting in an improvement in the flow and densification of the liner and increasing the bond to the carbon/epoxy overwrap.

Table 1. Basic liner properties.

Polymer	Tensile Strength (kPa)	Specific Gravity	CTE /C×10^{-5}	Melting Point (°F)	Process Temperature (°F)
Polyethylene co-polymer	5,515–27,580	0.934	10–12	221	200–400
Nylon 6	62,000	1.13	8	215	300–500
Nylon 11	50,000	1.05	10	185	180–400
PVDF	55,160	1.76	4.2–8.5	315	300–400
ETFE	48,260	1.7	5–7	500	325–500
FEP	20,000	2.15	8–10	518	350–600

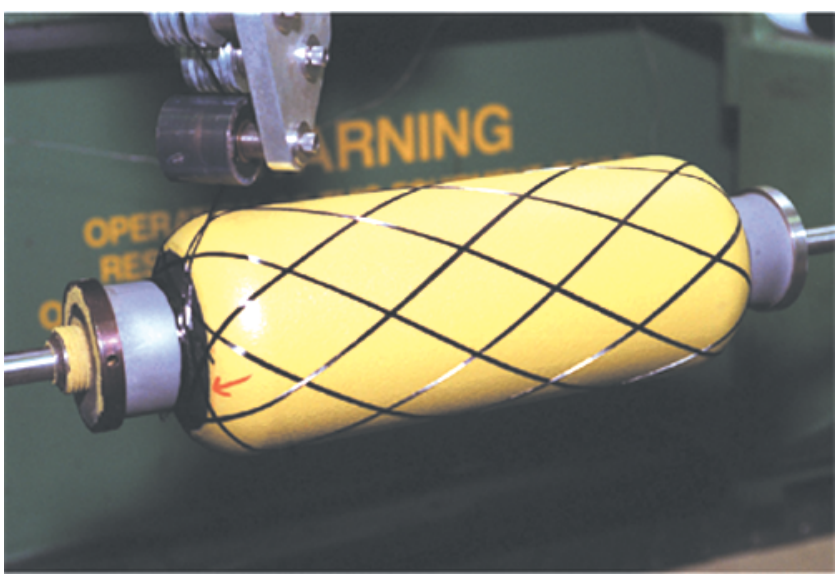

Figure 9. Polyethylene liner on steel mandrel being overwrapped with graphite/epoxy.

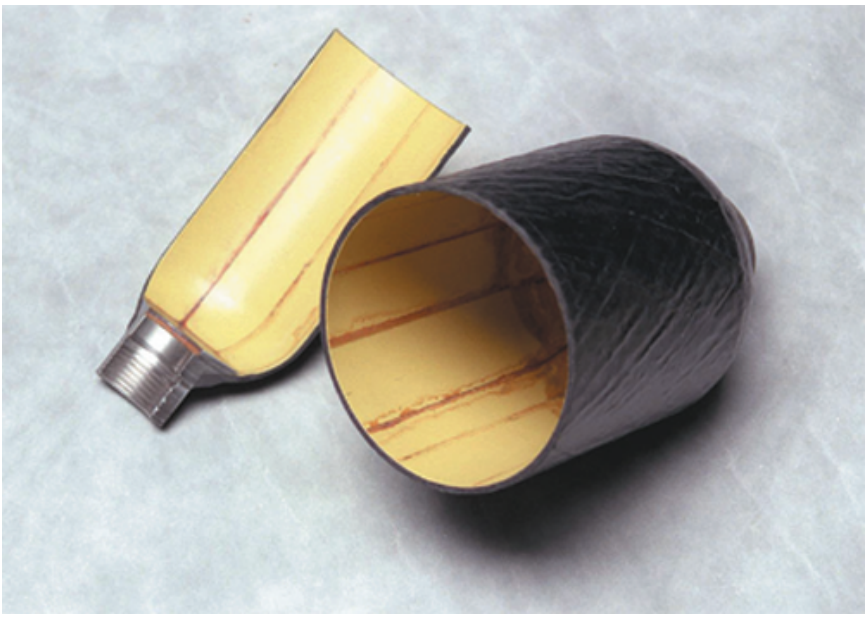

Figure 10. Destructive inspection of polyethylene-lined tank.

The thermally sprayed fluorinated polymers required a much higher mandrel temperature in order for the material to flow properly, resulting in some coefficient of thermal expansion (CTE) mismatch problems with the liner and mandrel combinations. The liners tended to develop cracks (fig. 11) due to the stresses produced as the hardware cooled. There may be a way to include some filler or reinforcement material to the liner material to reduce the cracking.

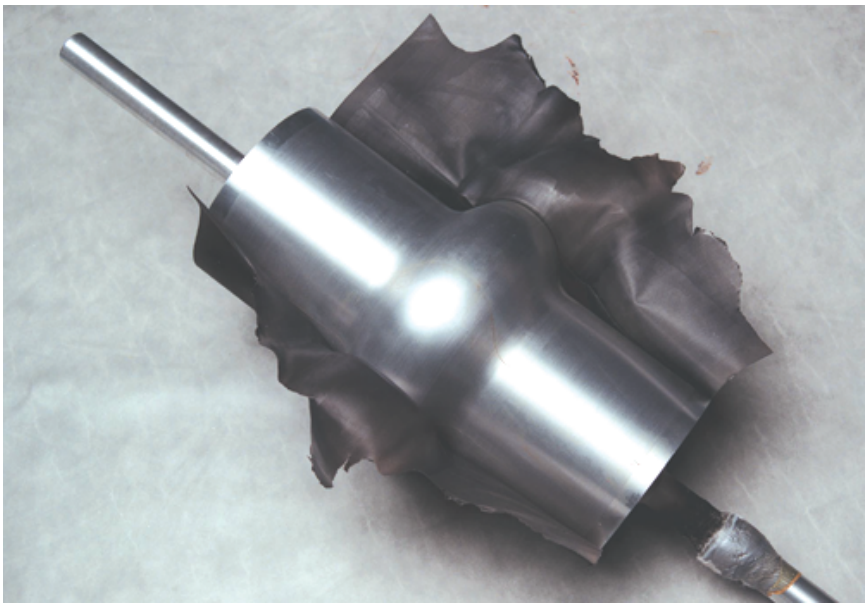

Figure 11. FEP liner on aluminum mandrel.

3.11.3 Compatible Overwrap

The unstable nature of the high-test hydrogen peroxide was strong motivation to investigate other materials and processes to develop a composite tank for the long-term storage of this oxidizer. Additional research in the MSFC Redstone Scientific Information Center revealed that there was substantial effort to identify peroxide-compatible materials in the 1950's and 1960's. This was due to the efforts of the Germans in early rocketry development and the needs of the X–15 program. The data found in the archives reinforced the use of fluorinated polymers and pure aluminum for long-term storage of highly concentrated hydrogen peroxide. Some of the remaining eutectic salt mandrels and multisegmented steel mandrels were used to produce candidate vessels. The metallic boss end-fittings for the tanks were machined from aluminum 1100 to ensure the highest purity. Extruded fluorinated sheet film (plasma-coated on both sides to enhance bonding) was used as an alternate method of producing a liner. The advantages of using films for a liner are that the method is scalable, the liner thickness is highly controllable, and the mandrel materials do not require a severe preheat temperature. An epoxy resin system was identified to be compatible with hydrogen peroxide and was found to work well with the liner material and the fibers of the composite overwrap structure. The use of the above resin with a new fiber (Zylon, a polybenzoxazole (PBO) fiber produced by Toyobo, Japan) can also be utilized to produce a liner for redundancy. The fluorinated polymer liner is most compatible with the peroxide; however, the somewhat compatible overwrap adds some robustness to the system. A tank was produced that uses the PBO/epoxy material as the first layer of the tank. This was followed by carbon and PBO fiber with a different resin; filament-wound layers that are rotisserie cured in an oven provide more of a structural member for a pressure vessel.

3.11.4 Metal Liners (Spun Formed, Electroformed, and Thermally Sprayed)

Spun-formed aluminum tanks and liners are currently mass-produced for commercial applications; however, the same liners have proven to be useful for research and development (R&D) purposes.

The small aluminum tanks are rather inexpensive and can be used to express new tank technology concepts and to test new materials. The aluminum tanks are produced from Al 6061 and can be anodized to enhance peroxide resistance. The PI is currently preparing to produce a similar tank from Al 5254 and Al 1100 to further the peroxide containment technology under an (Independent Research and Development (IR&D) task. The spun-formed tanks are useful but they are not scalable to a size needed for launch vehicles.

Several composite tanks were produced at the end of this research effort that contained very thin (5–17 mil), metallic permeation barriers. The liners included electroformed copper, electroformed nickel, and thermally sprayed Al 1100. The electroformed tank liners were produced by sealing a water-soluble eutectic salt mandrel with a conductive paint then plating the mandrel with metallic end pieces in place (figs. 12 and 13). The selection of the liner material depends on processability and the environment of the application. Nickel and Al were chosen for their compatibility with lox and copper for its resistance to hydrogen embrittlement. The electroformed liners are rather ductile and can respond to changes in temperature and pressure. The thermally sprayed aluminum had porosity and brittleness problems and may not be suited for this application. Thermal spraying; however, proved to be useful as another method for creating a conductive coating for the electroforming process.

Figure 12. Electroformed nickel-lined tank.

Figure 13. Graphite/epoxy overwrap of liner.

A follow-on activity is needed to test and verify the results of the lined tanks. This could be accomplished through another CDDF, or perhaps mainstream support from one of the ASTP vehicles. Preliminary commercial interest is currently being demonstrated. The composite processing facilities in the Productivity Enhancement Complex are being used to develop new tank concepts, with some development from vendors.

3.11.5 Mockup Tooling

The urethane tooling foam approach was useful on another application: the prototype production of larger scaled composite tanks. There are several upper-stage flight experiments on the horizon: X–37, X–34, spaceliner–100, as well as the upcoming MARS ascent vehicle project. The design of these vehicles is rather preliminary at this point. One of the characteristics they have in common is the need for lightweight fuel tanks that can adequately store fuels and oxidizers for longer periods of time. The ability to store these fluids could dramatically reduce operational costs of the new launch vehicles while enhancing their performance. Tank sizes chosen by the PI were 28-in.-diameter, 32-in.-length, $\sqrt{2}$ elliptical domes. This size was approximately half scale of the X–37 hydrogen peroxide tank and near full-scale of the MARS tank. The urethane tooling foam, provided by Alpha Foam Products, was initially cast as a cylindrical form and machined on a lathe at MSFC. The tooling was produced in 2 days. This prototype tool (fig. 14) proved to be very useful in several ways. It allowed the verification of the operational functions of the polar filament winding machine, the tolerances of the support tooling for the mandrels were checked, new composite materials for the projects were tested for processability, and the winding patterns for producing the tank were determined in advance (fig. 15). An added advantage of the mockup tooling was it allowed changes to be made to the tooling design as the actual tooling was being produced by the vendor. (In this case, cast aluminum, break-apart tooling was being fabricated by Smith Pattern and Tooling, Inc., Kanab, UT). The PI was also fortunate to receive filament winding training from the manufacturer of the machine (ENTEC, Inc., Salt Lake City, UT). This particular winder is a one-of-a-kind machine that is ideally suited for tanks of this size.

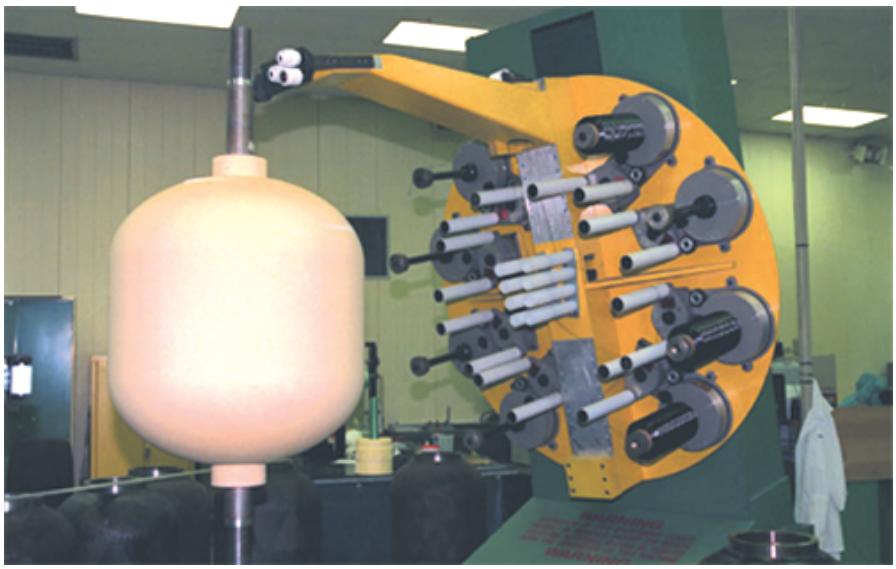

Figure 14. Urethane foam prototype tool.

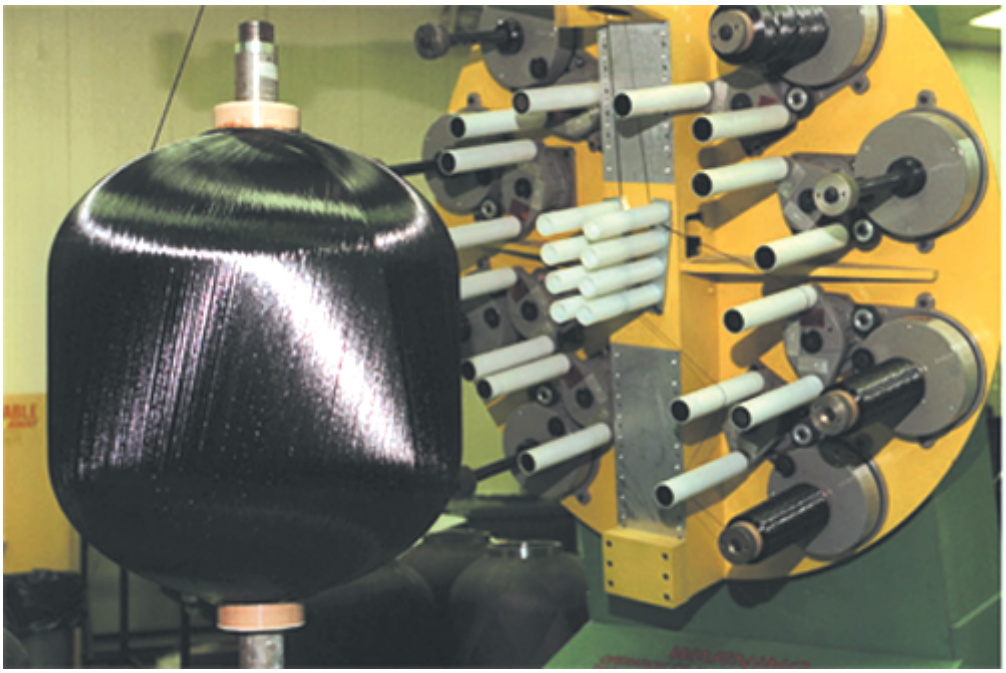

Figure 15. Polar winding of one-half scale X–37 tool.

4. CONCLUSIONS AND RECOMMENDATIONS

The rapid production of composite prototypes has proven to be a very beneficial and cost-effective means for developing new manufacturing methods and for clarifying the designs of new concepts. This investigation brought about an array of future projects and developed several patentable new technologies. The prototypes have minimized waste due to design changes and reduced the amount of rework for tooling. The new manufacturing methods and design need more refinement and testing to validate their usefulness. It is hoped that one of the ASTP projects will be able to utilize and fund some of the technologies. A secondary follow-on CDDF would be very useful in enhancing the development. One of the areas recommended by the author is the further development of the lined composite and unlined tanks. A series of standardized tanks could be produced with chosen materials and tested for strength and chemical compatibility. The composite tanks could also be used to validate end-fittings, interfaces, and seals. This research has produced methods that can rapidly deliver cost-effective composite hardware.

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The objective of this research was to provide a mechanism to cost-effectively produce composite hardware prototypes. The task was to take a hands-on approach to developing new technologies that could benefit multiple future programs.

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