NARC Rayon Replacement Program for the RSRM Nozzle, Phase IV Qualification and Implementation Status

M. Reed Haddock*, Gary M. Wendel†, Roger V. Cook‡, ATK Thiokol Inc., Brigham City, Utah 84302

Abstract

The Space Shuttle NARC Rayon Replacement Program has down-selected Enka rayon as a replacement for the obsolete NARC rayon in the nozzle carbon cloth phenolic (CCP) ablative insulators. Full qualification testing of the Enka rayon-based carbon cloth phenolic is underway, including processing, thermal/structural properties, and hot-fire subscale tests. Required thermal-structural capabilities, together with confidence in erosion/char performance in simulated and subscale hot fire tests such as Wright-Patterson Air Force Base Laser Hardened Materials Evaluation Laboratory testing, NASA-MSFC 24-inch motor tests, NASA-MSFC Solid Fuel Torch – Super Sonic Blast Tube, NASA-MSFC Plasma Torch Test Bed, ATK Thiokol Forty Pound Charge and NASA-MSFC MNASA justified the testing of the new Enka-rayon candidate on full-scale static test motors. The first RSRM full-scale static test motor nozzle, fabricated using the new Enka rayon-based CCP, was successfully demonstrated in June 2004. Two additional static test motors are planned with the new Enka rayon in the next two years along with additional A-basis property characterization. Process variation or “corner-of-the-box” testing together with cured and uncured aging studies are also planned as some of the pre-flight implementation activities with 5-year cured aging studies over-lapping flight hardware fabrication.

Nomenclature

NARC = North American Rayon Corporation
CCP = carbon cloth phenolic
RSRM = reusable solid rocket motor
LHAMEL = laser hardened materials evaluation laboratory
PTTB = plasma torch testbed
FPC = forty pound charge motor
MSFC = Marshall Space Flight Center
SFT = solid fuel torch
SSBT = super sonic blast tube
SGL = SGL Polycarbon
NECP = National Electrical Carbon Products (formerly NSP)
NSP = National Specialty Products
UK = United Kingdom

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I. Introduction

U.S. export laws prevent the public release of certain CCP test results. This paper will be limited to a discussion of yarns, weaving, carbonization processes and general program test activities. Prepreg, composite and thermal performance data has been intentionally left out of this paper.

NARC rayon is a precursor to the CCP used in the RSRM nozzle ablative liners. Figure 1 shows a nozzle with the CCP lining material. NARC discontinued production of aerospace-grade rayon in September 1997 for financial reasons, which required NASA to purchase a stockpile of NARC rayon prior to their shutdown to support RSRM production. NARC was the second rayon vendor to go out of business during the RSRM program. The Rayon Replacement Program began testing candidates in 1999.

II. Objectives

The objectives of the Rayon Replacement Program are to find the best replacement fiber for NARC rayon used in the RSRM nozzle CCP, reduce variability and ensure predictable performance by incorporating knowledge gained in previous programs, and not reduce performance or structural margins of safety in the RSRM nozzle CCP. In finding a replacement fiber for NARC, the following criteria were considered:

- Thermal/ablative performance
- Structural performance
- Processability
- Variability
- Long term availability
- Cost
- Drop-in replacement
III. Program Plan

The Rayon Replacement Program was laid out in four phases as shown in Figure 2.

<table>
<thead>
<tr>
<th>Phase I - Candidate Screening</th>
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<tr>
<td>Objective: Evaluate fiber candidates using RSRM baseline processes</td>
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<tr>
<th>Phase IA - Process Optimization Pathfinder</th>
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<tr>
<td>Objective: Better understand the process parameters for the most promising candidates by performing optimization studies</td>
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<tr>
<th>Phase II - Process Optimization</th>
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<tr>
<td>Objective: Verify the results of Phase IA by evaluating the four most promising fibers under several processing parameters.</td>
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<tr>
<th>Phase II A - Lyocell Optimization</th>
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<tr>
<td>Objective: Improve the performance of the lyocell candidates before proceeding with Phase III</td>
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<tr>
<th>Phase III - Variability Study</th>
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<tr>
<td>Objective: Determine the level of lot-to-lot and within lot variability of the best candidates. Characterize these four candidates and select the NARC replacement</td>
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<tr>
<th>Phase IV - Qualification</th>
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<tr>
<td>Objective: Complete characterization of the selected material and demonstrate acceptable performance in three full scale static test motors</td>
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Figure 2: Rayon Replacement Program Plan.

IV. Rayon Down-Selection

The first phase of the plan was candidate screening with the objective to screen prospective fiber candidates using baseline RSRM processes. Polyacrylonitriles (PAN) and other commonly used carbon precursors were not included in the screening process. There were 21 cellulose (pulp-based) fiber candidates tested. The following list includes the fiber family and the overall findings:

- Staple rayon – low in-plane tensile
- Tire cord rayon – poor interlaminar properties
- Textile rayon – acceptable properties, industry needs very uniform properties for dye-ability
- Aerospace-grade rayon – Cydsa’s copy of NARC looked good, but they closed the business
- Staple Lyocell – no environmental issues (i.e., carbon disulfide emissions from spin machine)
- Continuous Lyocell – pilot plant in Germany that has since shut down

Candidate materials were woven into fabric and carbonized by one of two different carbonizers, SGL Polycarbon or National Electrical Carbon Products (NECP) formerly National Specialty Products (NSP). Carbon fabric was pre-impregnated with phenolic resin and then used in the fabrication of composite panels and parts for subsequent testing. Testing included lot acceptance of fabric and prepreg, composite thermal and mechanical properties and hot-fire testing in simulated hot-fire testbeds and subscale motors. The most promising candidates from each fiber family were selected for optimization and evaluation in Phase IA.

The objective of Phase IA was to optimize the CCP fabrication process for each of the most promising fibers. Adjustments to the processes were sometimes necessary, because the RSRM material fabrication
processes were not optimum with respect to maximum thermal and mechanical properties. Optimization studies were performed at every level of the fabrication process, from fiber production to resin impregnation processes trying to improve critical properties. Over 700 iterations of fibers were tested in the Optimization Phase. The most promising fibers from Phase I (including control), that were optimized, are shown in Table I.

### Table I: Most promising fiber candidates from Phase I.

<table>
<thead>
<tr>
<th>Fiber Description</th>
<th>Fiber Designation</th>
<th>Vendor</th>
<th>Yarn Denier</th>
<th>Fiber Denier</th>
<th>Filament Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aerospace Grade Rayon</td>
<td>NARC</td>
<td>NARC</td>
<td>1650</td>
<td>2.3</td>
<td>Continuous</td>
</tr>
<tr>
<td>Tire Cord Rayon</td>
<td>Candidate G</td>
<td>Acordis</td>
<td>1650</td>
<td>1.65</td>
<td>Continuous</td>
</tr>
<tr>
<td>Tire Cord Rayon</td>
<td>Candidate W</td>
<td>Acordis</td>
<td>2200</td>
<td>1.65</td>
<td>Continuous</td>
</tr>
<tr>
<td>Tire Cord Rayon</td>
<td>Candidate Y</td>
<td>Acordis</td>
<td>3300</td>
<td>1.65</td>
<td>Continuous</td>
</tr>
<tr>
<td>Textile Rayon</td>
<td>Candidate N</td>
<td>Acordis</td>
<td>1800</td>
<td>5.0</td>
<td>Continuous</td>
</tr>
<tr>
<td>Aerospace Grade Rayon</td>
<td>Candidate S</td>
<td>Cydsa</td>
<td>1650</td>
<td>2.3</td>
<td>Continuous</td>
</tr>
<tr>
<td>Aerospace Grade Rayon</td>
<td>Candidate Xa</td>
<td>Cydsa</td>
<td>2200</td>
<td>3.0</td>
<td>Continuous</td>
</tr>
<tr>
<td>Aerospace Grade Rayon</td>
<td>Candidate Xb</td>
<td>Cydsa</td>
<td>3300</td>
<td>3.0</td>
<td>Continuous</td>
</tr>
<tr>
<td>Lyocell</td>
<td>Candidate V</td>
<td>Acordis</td>
<td>1750</td>
<td>3.0</td>
<td>Staple</td>
</tr>
</tbody>
</table>

Testing in Phase I included lot acceptance of the fabric and prepreg, composite thermal/structural properties, simulated hot-fire test-beds, and subscale motors.

The objective of Phase II was to demonstrate the acceptable performance of the most promising four fibers in conjunction with their associated optimized processes. Acordis tire cord and Cydsa aerospace-grade rayon fibers were dropped in Phase II. The tire cords were dropped for poor structural performance and the Cydsa candidates were dropped due to closure of their rayon plant. The remaining fibers were Enka and Lyocell, each fiber carbonized by both carbonizers, NECP and SGL. There were 36 process variations evaluated with the four selected fibers in Phase II. Parameters evaluated with the Enka Textile Rayon candidates were:

- Fiber plant - Ede, Netherlands, or Obernburg, Germany
- Fiber production process - spool spun or continuous spun
- Pre-carbonization process - post-woven or pre-woven
- Weave pattern - 5 or 8 harness satin weave
- Use of a catalyst in carbonization process
- Carbonization temperature

Parameters evaluated with the staple Lyocell candidates were:

- Staple length
- Carbonization of fabric compared to yarn
- Pre-carbonization process
- Carbonization temperature

Testing in Phase II included lot acceptance of the fabric and prepreg, composite thermal/structural properties, simulated hot-fire test-beds, and subscale motors. A couple of candidates were wrapped as partial rings into some fullscale static test motors.
Since Phase II Lyocell candidates were only marginally acceptable, the decision was made to run more optimization trials in Phase IIA with the Lyocell fibers. Significant problems with the Phase II Lyocell candidates were:

- Poor processability (weak and brittle carbon fabric)
- Poor composite properties
- Lack of maturity of the SGL post-woven and NECP (catalyst) processes

Two Lyocell-based carbonization processes were studied during Phase IIA, a proprietary carbonization process using pre-woven Lyocell at NECP and a proprietary carbonization process using post-woven Lyocell yarn at SGL Polycarbon. Post-woven fabric was carbonized yarn woven into fabric after the carbonization process.

Acordis Enka textile rayon fabric, carbonized by both SGL Polycarbon and NECP, was selected for further evaluation in Phase III. Process parameters selected included:

- Fiber plant – Enka fiber from Ede, Netherlands, and Obernburg, Germany, was found to have similar performance
- Fiber production process - Spool spun process was found to produce a better carbon precursor
- Pre-carbonization process - Pre-woven carbonization was selected, primarily due to simplicity and RSRM history
- Weave pattern - 5-harness satin weave pattern was selected over the existing 8-harness satin pattern because of improved fabric stability and no overall loss in composite properties
- Catalyst to aid the carbonization process - No catalyst will be used on the Enka textile rayon
- Carbonization temperature - Mid-range carbonization temperatures were selected at both carbonizers

In addition to the Enka rayon, Acordis Staple Lyocell, carbonized by both SGL Polycarbon and NECP, was selected for further evaluation in Phase III. Selected process parameters for the NECP pre-woven candidate included:

- Fiber - 3.0 denier/filament and 60 to 90 mm staple length
  - 3.0 fiber denier was selected because of a processing preference in the carbon form and lack of experience with the 1.5 denier fiber
  - 60 to 90 mm staple length was selected because of better interlaminar performance
- Ply configuration - 6/2 ply - sized for 1650 denier (6 is # of 840 yds/lb.)
- Precarbonization process - pre-woven proprietary process produces the most supple and strongest carbon fabric of any of the NECP Lyocell candidates
- Carbonization - mid-range temperature best balance of composite properties and pocketing sensitivity

The objective of Phase III was to evaluate the lot-to-lot and within-lot variability of the four selected candidates from Phase II and IIA. Five lots of Enka textile rayon were carbonized by SGL Polycarbon, three Obernburg lots and two Ede lots. One lot of high carbonization temperature Enka rayon material was added mid-Phase III to evaluate a corrective action for a static test pocketing-erosion episode. Five lots of Enka textile rayon were also carbonized by NECP, three lots from Obernburg and two lots from Ede. An additional high carbonization temperature Enka lot processed by NECP was also added mid-Phase III. Five lots of post-woven Lyocell were carbonized by SGL Polycarbon. Five lots of Lyocell from Grimsby, UK were also tested, three lots woven by Sigmatex and two lots woven by Albany International, followed by carbonization at NECP. Of those five lots, four lots were tested, one lot from Mobile and three from Grimsby, UK. Testing included lot acceptance of fabric and prepreg, composite thermal/structural properties, process simulations, simulated hot-fire test-beds, and subscale motors.
Thermal/structural property testing was increased over what was done in previous phases due to the down-selection to four materials.

The candidate that was down-selected in Phase III and is being characterized in Phase IV is Enka rayon-based CCP. The Enka rayon fiber is a 5.0-denier/filament, 60 continuous filaments per yarn (300-denier yarn), 6-plied into an 1800-denier yarn. NARC is a single 1650-denier yarn with 2.3-denier/filament. The rayon yarn is exclusively from Obernburg, Germany, and the yarn is plied by Middleberg. Highland weaves the yarn into a 5-harness satin cloth (NARC is a 8-harness satin weave), which improved fabric stability with no overall loss in composite properties. The noted Enka yarn is carbonized by NECP. The NARC carbonizer is SGL Polycarbon, so NECP was a change from the current RSRM program material. The Enka carbonization temperature is similar to the NARC temperature. The phenolic resin prepregger is Cytec Engineered Materials (CEM) using SC1008 Borden Phenolic resin, no change from the current material. A 600X photomicrograph of Enka fiber in the phenolic resin matrix is shown in Figure 3.

![Figure 3: Enka-based rayon in CCP at 600X.](image)

The objective of Phase IV is to complete characterization and testing of the Phase III, down-selected candidate, Enka, and qualify the material. Enka-based CCP characterization/testing includes:

- Lot acceptance testing of whitegoods, carbon cloth and CCP
- Thermal/mechanical property characterization
- Process studies
- Aging – long term composite and uncured shelflife
- Full thermal/structural analyses using A-Basis properties
- Hot-fire Enka-based CCP in simulated test beds and subscale motors
- Demonstration and certification on fullscale static test motors

Examples of simulated and subscale hot-fire testing are shown in Figures 4-7.
Figure 4: PTTB test set-up.

Figure 5: LHMEL test bed for heating materials.

Figure 6: Solid Fuel Torch with CCP in blast tube region.
Examples of subscale hot-fire test beds are shown in Figures 8-9.

Enka-based CCP was also demonstrated/evaluated in a fullscale RSRM nozzle for erosion and char performance, an example of a static test demonstration is shown in Figure 10.
One third of the Phase IV qualification testing has been completed. The FSM-11 static test was all Enka except one small component. Previous to FSM-11, portions of individual components on two static tests were evaluated. Char and erosion performance of the Enka material is equivalent to the NARC material in all forward nozzle components. The AEC had equivalent virgin material remaining post-test to current material. Plans going forward include completion of the qualification testing and certification on at least two additional static test motors. The Phase IV activities are shown in Figure 11.

Figure 11: Phase IV program flow diagram since Phase III down-selection.
V. Program Concerns

There are a few program concerns with the Enka material, beginning with the FSM-11 Enka AEC experiencing ply-lift late in burn. The ply-lift is considered benign due to typical virgin material remaining at post-test inspection. Nevertheless, since ply-lift is not totally understood, a team was formed to determine ply-lift mechanisms and implement preventative measures. In addition, a design solution is being evaluated as a secondary measure. A subtle lift was also observed in the char region of the FSM-11 nose cap. Ply-lifts have been seen previously on RSRM and SRM programs in Avtex material, a material that became obsolete prior to NARC. Another concern is the spin finish on the Enka textile rayon is obsolete, the spin finish company that supplied Enka for 45 years shut down in the spring of 2002. The change is viewed as low risk since the finish is removed before carbonization. Carbonization is expected to manifest any problems if spin finish is not completely removed in the prior cleaning operation. Another concern is a change in the pulp supplier at Acordis (Enka). The pulp supplier was previously Bahia Pulp (formerly Klabin Bacell) in Camacari, Brasil, while the new pulp supplier is Domsjo Fabriker AB (formerly MoDo) in Ornskoldsvik, Sweden. It is believed that the pulp supplier change will not affect performance, as it was common practice to switch pulp suppliers earlier in the RSRM program. At any rate, the new pulp will be used on an upcoming static test. Another concern is with the carbonizer fabric scouring process chemicals. Restrictions on scouring units at both NECP and SGL Polycarbon will probably continue to tighten and may force a change to another cleaning agent in the future. This is a long-term concern that the team is monitoring. The last concern is that of Enka longevity, rayon plants continue to close, most recently Fabelta Ninove in Belgium (January of 2005). The plan, if Enka is implemented, is to conduct a fiber aging study, if required, to allow stock-piling. This would preclude a shortage if Acordis (Enka) shuts down.

VI. Conclusion

The Rayon Replacement Program for the Space Shuttle has been successful in evaluating cellulose fibers (pulp-based), optimizing them for comparison, and down-selecting a viable candidate to replace NARC rayon. Enka rayon offers similar thermal and structural performance to NARC rayon in carbon cloth phenolic used in the Space Shuttle Solid Rocket Motor Nozzle. Enka has been tested in numerous thermal/structural tests, subscale hot-fire tests, and fullscale nozzle tests. Many of the same processes used to fabricate Enka were retained from NARC-based CCP production. Although ply-lifting was seen on a fullscale static test AEC, it has historically been a benign condition on the Space Shuttle RSRMs and ATK Thiokol is working to understand/eliminate it in low ply-angle nozzle liners.

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References