FINAL REPORT

**Development Of Carbon-Carbon Composites
From Solvent Extracted Pitch**

June 24, 1996

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Report Prepared by

FIBER MATERIALS, INC.

5 Morin Street

Biddeford, Maine 04005

under

Subcontract 86X-SR795C

for

OAK RIDGE NATIONAL LABORATORY

Oak Ridge, Tennessee 37831

Managed by

LOCKHEED MARTIN ENERGY RESEARCH CORP.

for the

U.S. DEPARTMENT OF ENERGY

under contract DE-AC05-96OR22464

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INTRODUCTION

There are several methods used to fabricate carbon-carbon composites. One used extensively in the fabrication of aerospace components such as rocket nozzles and reentry vehicle nosetips, as well as commercial components for furnace fixturing and glass manufacturing, is the densification of a woven preform with molten pitch, and the subsequent conversion of the pitch to graphite through heat treatment. Two types of pitch are used in this process; coal tar pitch and petroleum pitch. The objective of this program was to determine if a pitch produced by the direct extraction of coal could be used as a substitute for these pitches in the fabrication of carbon-carbon composites. The program involved comparing solvent extracted pitch with currently accepted pitches and rigidizing a carbon-carbon preform with solvent extracted pitch for comparison with carbon-carbon fabricated with currently available pitch.

BACKGROUND

Carbon-carbon composites have been manufactured with pitch for over 20 years. From the early 1970's, two pitches have typically been used in this process, Allied Signal's 15V coal tar pitch and Ashland Chemical's A-240 petroleum pitch. Both pitches are the by-product of other processes; coal coking for coal tar pitch and petroleum distillation for petroleum pitch.

The process for fabricating carbon-carbon composites consists of assembling a fiber preform, through consolidation, lay-up, weaving, or some other method, and densifying the preform with pitch. The densification is a series of repetitive process steps, each step consisting of impregnation, carbonization, and graphitization, as depicted in Figure 1.

Figure 1

The number of process cycles depends on required composite density, processing parameters, pitch coking value, etc. The resulting carbon-carbon composite is a very strong, very high temperature material suitable for demanding aerospace and commercial application.

Over the years, several problems have arisen with the use of Allied 15V and Ashland A-240 pitches. Both use the residue of other processes and consistency of such residues cannot be counted on.

There are significant environmental issues, particularly with coal tar pitches. Coking ovens are regularly closed down as the steel industry switches to new methods of steel manufacture, overcoming the significant pollution generated by such ovens. With no coking ovens, there will be no supply of coal tar. Even today, as ovens are closing, new sources of tars must be found which affect the resulting pitch.

As the majority of the petroleum used in the U.S. is imported, the availability of petroleum pitch may not be assured. In addition, the properties of crude oil are changing, often resulting in higher sulfur content and this can significantly affect the properties of petroleum pitch and the resulting carbon-carbon.

What is required is a clean process for making pitch from a very large source of raw material available in the U.S. This will ensure a continuous supply of material

without the worry of environmental issues affecting supply. In addition, if the process is flexible enough, pitches could be manufactured having different properties or, similar pitches could be manufactured from different raw material sources.

West Virginia University has developed a process of extracting the usable hydrocarbon content from coal while leaving the ash behind. This solvent extraction process, using solvent N-methyl pyrrolidine (NMP), has shown that pitches suitable for making coke for smelting electrodes can be produced. It has also been shown that the properties of the pitch can be varied so that the resulting coke can range from isotropic on one end of a scale to anisotropic on the other, depending on the degree of hydrogenation applied to the coal before extraction. Being able to vary the properties of the pitch can have several advantages:

- Adjust the process so different raw coals can produce equivalent pitch products.
- Adjust the process so one coal can produce a variety of pitches useful in many applications.
- Tailor a pitch to have specific properties.

For the past eight years, workers in the Chemical Engineering Department at West Virginia University have been developing a technique based on solvent extraction of coal whereby significant quantities of the organic material in coal can be separated from the inorganic contaminants^(1,2,3,4). This process employs a novel class of organic compounds known as dipolar aprotic solvents. When bituminous coal is treated with these solvents at their normal boiling point, much of the carbonaceous material in the coal is solubilized and the inorganic matter remains as an undissolved solid. Through simple filtration the solution is separated from the residual inorganic solids and the organic material is reconstituted by solvent evaporation. Through efficient use of

evaporative technologies, virtually all of the solvent is recovered and recycled in the process. A typical concentration of inorganic matter in the extracted product (as measured by ASTM ashing techniques) is as low as 0.1%. Depending on the starting coal, significant reduction in sulfur species can also be achieved. It has been found that extract yields in the range of 30 to 50% by weight of the original coal can be realized for most bituminous coals indigenous to the Appalachian region. Moreover, if the raw coal is first treated by mild hydrogenation, extract yields as high as 80% can be achieved. Most importantly, by adjusting processing conditions and/or blending extracts from the hydrogenated and raw coals together, desirable properties of the product can be enhanced. It has been shown that the solvent extract can serve as an excellent precursor for a wide range of carbon product feedstocks. Indeed, a successful program has just been completed for the U.S. Department of Energy in which cokes produced from the solvent extraction process were utilized as feedstocks for the production of fine-grained, isotropic graphite⁽⁵⁾. The current program was able to draw from WVU's success to date in the fabrication of carbon-carbon composites.

Evaluation Procedure

To determine whether a solvent extracted pitch is capable of producing carbon-carbon composites having properties similar to currently produced composites, it is necessary to determine if the pitch properties are similar to properties of pitches currently in use. This was accomplished by testing a pitch produced by the Chemical Engineering Department of West Virginia University processed by the solvent extraction process, and comparing it to other pitches. The WVU pitch was processed from WVGS 13421 coal by first hydrogenating the ground in a proton donor, tetralin, at 450°C for 2 hours. This was followed by extraction with n-methyl pyrrolidine (NMP), after which the solution is filtered and the NMP removed by rotary evaporation. A second extraction was carried out using toluene to remove toluene insolubles, thus reducing the softening point. The resulting pitch was identified as WVU D026 pitch.

For this program, a 500 gram sample of WVU D026 pitch was tested with samples of Ashland A-240 and Allied 15V pitches. The test matrix presented in Table 1 was used for these evaluations.

TABLE 1
Pitch Test Matrix

PROPERTY	TEST PROCEDURE
Coking Value	ASTM D-2416
Ash Content	ASTM D-2415
Quinoline Insoluble	ASTM D-2318
Toluene Insoluble	ASTM D-4072
Sulfur	ASTM D-1552
Softening Point	ASTM D-36
Specific Gravity	ASTM D-71

Samples of WVU, Ashland, and Allied pitches were also coked so the grain structure could be evaluated. The coked samples were impregnated with epoxy resin and polished to a metallographically smooth surface, the final polishing done with 0.05 μ alumina powder slurry. Samples of each were then observed under a light microscope using polarized light, with significant observations documented with photomicrographs. Polarized light allows the qualitative analysis of the anisotropy of the pitches.

The next step was to compare how the WVU pitch would impregnate a carbon fiber preform as the first step toward fully processing a carbon-carbon composite. It typically takes four or five cycles of impregnation, carbonization, and graphitization to densify a carbon-carbon billet. As this requires considerable pitch, on the order of a minimum of 25 to 30 kilograms, only one cycle could be completed in this program. Once pilot plant quantities of WVU pitch can be produced, the densification can be completed.

The usual process for impregnating a preform involves melting the pitch in one container by heating. In a second container, connected to the first by a tube and valve, the preform is heated to the same temperature as the pitch. A vacuum is pulled on the vessel holding the preform to remove all gas. When the valve between the two vessels is opened, the molten pitch flows from the container holding the pitch under atmospheric pressure into the evacuated container holding the preform. A considerable amount of extra pitch is required to ensure the transfer vacuum is not lost by air entering the transfer tube from the pitch melter.

Because the quantity of available WVU pitch was minimal, a different technique was utilized. Preforms measuring 13 cm by 13 cm by 5 cm were placed in containers. Each preform was covered with 4.5 kilograms of pitch, one with WVU pitch, the other with Allied 15V pitch. The WVU pitch consisted of two lots, 1 kg of D054 and 3.5 kg of D087. Both lots were manufactured using the same process used for the previously tested lot D026. The two containers were placed in an impregnator which was sealed and evacuated. The impregnator was heated to 250°C under vacuum and held for 4 hours. The vessel was then backfilled with nitrogen to atmospheric pressure, forcing the molten pitch into the preform, filling the voids. Although not as efficient as the hot pitch transfer method described earlier, this method works well with small parts and limited quantities of pitch. The impregnator was allowed to cool and the two cans removed for further processing.

The cans were next placed in a carbonization oven and heated to approximately 800°C under a nitrogen blank at heating rates of approximately 20°C per hour from ambient to 350°C, 5°C per hour from 350° to 600°C per hour, and 10°C per hour from 600°C to 800°C. This process slowly converts the organic pitch to carbon, driving off nitrogen, oxygen, hydrogen, sulfur, and other elements which make up the organic pitch. The resulting carbon, or coke, is amorphous in nature with almost no crystallinity.

Once the pitch has been coked, the rigidized preform is removed from its container and excess coke removed, in preparation for converting the amorphous coke to crystalline graphite. This is accomplished by heating the part to 2525°C, again under a nitrogen blanket to prevent oxidation. This heat treatment takes about 30 hours to accomplish, followed by a 1/2 hour hold prior to cooldown.

Upon completion of rigidization, the carbon-carbon parts were machined to remove excess coke. The parts were then measured and weighed for density determination. Samples extracted from each billet were mounted in epoxy, polished metallographically, and observed under a light microscope to compare the microstructures of the WVU and Allied pitches in the carbon-carbon composite.

RESULTS

Evaluation of the WVU pitch resulted in the properties shown in Table 2.

TABLE 2
WVU Solvent Extracted Pitch Lot D026

PROPERTY	VALUE
Coking Value - %	49.3
Ash Content - %	0.02
Quinoline Insoluble - %	0.39
Toluene Insoluble - %	1.12
Sulfur - %	0.4
Softening Point - °C	117
Specific Gravity	1.22

A comparison was made of this data to a data base of pitch properties established over the last 20 years for Allied 15V pitch and Ashland A-240 pitch. This comparison is shown in Table 3.

TABLE 3
Comparison Of Pitch Analyses

PROPERTY	WVU D026	ASHLAND A-240	ALLIED 15V
Number Of Lots	1	14	30
Coking Value - %	49.3	50.2	46.7
Ash Content - %	0.02	0.03	0.17
Quinoline Insoluble - %	0.39	0.15	4.3
Toluene Insoluble - %	1.12	6.52	14.8
Sulfur - %	0.4	2.0	0.54
Softening Point - °C	117	117	88
Specific Gravity	1.22	1.26	1.28
Viscosity - Sec	--	16	19

It can be seen that the WVU pitch compares very favorably to both Ashland A-240 petroleum pitch and Allied 15V coal tar pitch. With the exception of sulfur, and toluene insoluble contents, D026 is nearly identical to the Ashland pitch. The lower toluene insoluble content is due to the toluene extraction carried out in the pitch manufacturing process to reduce the softening point to an acceptable value. The lower sulfur is also a reflection of the solvent extraction process, as well as the low sulfur coal used as a raw material. In addition to the toluene insoluble content, the 15V pitch also exhibits higher quinoline insoluble content and a lower softening point.

Coked samples of the three pitches were observed under a light microscope to qualitatively evaluate the isotropy/anisotropy of the pitch. Two types of crystal structure

were observed for each material, a finer grain structure and a coarser grain structure. These are shown in Figures 2 and 3 respectively. Figure 2 is the finer grain structure. It can be seen that the Allied 15V pitch has the finest grain structure of the three. This is due to the higher quinoline insoluble fraction acting as nucleation sites in the pitch. The Ashland A-240 and WVU D026 pitches have lower quinoline insoluble, so do not have the nucleation sites available for the fine grain crystallization. The grain size of the Ashland and WVU pitches compare very favorably.

In Figure 3, areas of each sample having larger grains were observed. Again the grains of the Ashland and WVU pitches compare very well. Although the grains in the 15V pitch do not appear as free flowing as the other two materials, the observed grain size compares favorably.

The key observation is that all three pitches produce essentially on anisotropic coke. The coal tar pitch may have localized area of isotropy due to local pockets high in insoluble matter acting as nucleation sites, but in general the structure of this material is also anisotropic.

During the rigidization of a carbon fiber preform, the density was measured after each step in the process. The results are shown in Table 4.

TABLE 4
In Process Composite Densities

PROCESS STEP	ALLIED 15V MATRIX	WVU PITCH MATRIX
Preform	0.75	0.75
After Carbonization	1.07	1.04
After Graphitization	1.07	1.06
After Machining	1.11	1.07
Density Increase	0.36	0.32

COKES FROM SOLVENT EXTRACTED, COAL TAR, AND PETROLEUM PITCHES

Figure 2a: WVU D026 HiPIC Coke

Figure 2b: Ashland A240 HiPIC Coke

Figure 2c: Allied 15V HiPIC Coke

COKES FROM SOLVENT EXTRACTED, COAL TAR, AND PETROLEUM PITCHES

Figure 3a: WVU D026 HiPIC Coke

Figure 3b: Ashland A240 HiPIC Coke

Figure 3c: Allied 15V HiPIC Coke

Samples from each carbon-carbon composite were mounted in epoxy and polished for observation under a light microscope. Significant observations are shown in the photomicrographs in Figures 4 and 5. Figure 4 compares the microstructure of a matrix pocket in the woven structure of fibers. The WVU solvent extracted pitch appears more anisotropic than the coal tar pitch, as was seen in the coked pitch samples discussed earlier. The difference in isotropy is not so great so as to be of concern. Variations of this magnitude are seen throughout normal carbon-carbon processing.

Figure 5 compares fiber bundles which have been impregnated with the two pitches. It can be seen that both pitches impregnate the fiber bundles well, and become well crystallized. The photomicrographs are taken under polarized light, with the matrix showing considerable optical activity, as did the matrix materials in the pockets between fibers.

DISCUSSION

The results of the WVU pitch analysis and comparison to pitches currently used in the fabrication of carbon-carbon composites indicate that the WVU solvent extraction process can produce a pitch which is similar to currently used pitches. Not only are the physical properties of the pitches similar, the microstructure of coked samples of the pitches are also similar. Another criteria of how similar the WVU pitch is, is to compare it to other pitch specifications.

Allied 15V pitch and Ashland A-240 pitch are made to specifications peculiar to each. Although different, there are many similarities, as shown in Table 5.

CARBON-CARBON COMPOSITE MATRIX POCKET

Figure 4a: WVU D026 Pitch Impregnated C/C

Figure 4b: Allied 15V Pitch Impregnated C/C

CARBON-CARBON COMPOSITE FIBER BUNDLE

Figure 5a: WVU D026 Pitch Impregnated C/C

Figure 5b: Allied 15V Pitch Impregnated C/C

TABLE 5
Pitch Specifications

PROPERTY	ALLIED 15V SPECIFICATION	ASHLAND A-240 SPECIFICATIONS	WVU D026 ACTUAL
Coking Value - %, min	35	40	49.3
Ash Content - %, max	0.25	0.15	0.02
Quinoline Insoluble - %, max	6	1	0.4
Toluene Insoluble - %, max	22	10	1.1
Sulfur - %, max	0.7	3	0.4
Softening Point - °C	95 max	130	117
Specific Gravity	1.24-1.32	1.20-1.30	1.22
Viscosity - Sec, max	23	--	--

It is readily evident that WVU pitch D026 meets all the requirements of the Ashland A-240 specification. In addition, it meets all the requirements of the Allied 15V specification with the exception of the softening point and specific gravity. One would therefore conclude that using the WVU pitch in a carbon-carbon composite should result in a composite similar to composites currently being manufactured.

To demonstrate this, two carbon fiber preforms were rigidized, one using Allied 15V pitch, the other with WVU solvent extracted pitch. A comparison of the density increase between the two parts, 0.36 g/cc for the 15V part and 0.32 g/cc for the WVU pitch part, show that, in fact, the WVU pitch should yield carbon-carbon composites have properties similar to composites currently being fabricated with Allied and Ashland pitches.

The minor density difference between parts may be attributed to the density difference in the pitch itself, or may be an artifact of processing. Continued processing

through four or five subsequent densification cycles would aide in clarifying this, as well as provide composite material for preliminary mechanical and thermal property testing. Unfortunately, follow-on programs are not currently planned to either produce additional pitch, nor perform additional processing, even though the work performed to present is very promising.

The photomicrographs taken of the two carbon-carbon composites further confirm their similarity. Both samples show well impregnated fiber bundles, important for the mechanical properties of the composite. Impregnation of the matrix pockets was equivalent for both materials. Some pockets showed more pitch than others in both samples. This is due to the fact that the preform had only undergone one densification cycle. Further densification cycles will result in all the matrix pockets being equally densified. Pitch typically fills the pocket from the walls inward, which was observed in the samples evaluated. The photomicrographs shown in Figure 4 are at the bottom of a pocket, near the fiber bundle, so a large area of matrix is visible. Again the matrix in both materials is observed to be very similar, although the WVU pitch is somewhat more anisotropic than the Allied 15V coal tar pitch. However, this was to be expected from the observations of the coked pitches conducted earlier in the program.

Having worked with one WVU pitch in pitch analysis and carbon-carbon processing, it is believed this material shows great promise in providing a long term, stable, cost effective supply of pitch for producing carbon-carbon composites. It has also led to a number of recommendations for continued work in this area.

First, and foremost, is the need to complete the densification of the carbon-carbon composites which are currently rigidized so preliminary mechanical properties can be determined for a more thorough evaluation of the pitch's ability. This will provide the basis for a more in-depth material characterization program to establish a data base of information.

Second, further work needs to be done to modify the properties of WVU pitch, and then evaluate how these changes will affect carbon-carbon properties. One change would be to increase the density and lower the softening point so a WVU pitch will meet the Allied 15V pitch specification. Another change would be to develop a pitch with a higher density and higher coking value. Increasing the density to 1.5 g/cc and the coking value to 80% will reduce the number of process cycles required for a carbon-carbon composite to reach a density of 1.9 g/cc from five to two. This would reduce carbon-carbon costs on the order of 50% or greater, making it more cost competitive in high temperature applications.

A third area of further research is the development of a higher strength, high strain carbon foam from the WVU pitch. This foam can then be used as a very low cost preform, replacing the costly woven fiber preform, in a carbon-carbon composite. This type of carbon-carbon processing is promising in the mass production market for such products as pistons in internal combustion engines or low cost tactical missile nozzles.

As more is learned about these solvent extracted pitches, new applications will be developed. The clean process has alleviated environmental concerns of both coal tar and petroleum pitch processes. The huge supply of domestic coal assures a ready domestic supply of these pitches. The economics of the process ensure the pitches will be cost competitive with current pitches. The ease of varying pitch properties will allow the production of a variety of pitches for a variety of composite materials. These parameters will lead to robust carbon-carbon manufacturing once the needed research is concluded.

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