# STRUCTURE AND PROPERTIES OF FIBERS DERIVED FROM PITCH

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## Introduction

The primary objective of this study was to evaluate various available pitch-base fibers as a practical replacement for carbon fibers derived from continuous filament rayon precursors for use in advanced carbon composites, such as carbon phenolic for heat-shield application and 3-D carbon-carbon composites for nosetip application. On the basis of cost and projected utilization, such fibers will replace the present generation of fibers produced from rayon. The purpose of this study was to evaluate the characteristics of the various grades of pitch fiber available and to fabricate composite hardware for comparison of properties with reference fibers scheduled to be phased out because of pending unavailability.

## Fiber and Composite Evaluation

The fibers were evaluated for density by a density gradient technique, for moisture pickup at 100% relative humidity as an indication of pore accessibility, for microstructure by optical and scanning electron microscopy, and for crystallite size and d-spacing by x-ray diffraction. Measured composite tensile properties consisted of tensile strength, modulus, and elongation.

## Results of Fiber Evaluation

At the present time there is considerable variation in fiber morphology and physical properties. Filaments may vary in shape, size, density, and hygroscopicity depending upon heat-treatment condition and lot. Split or crescent-shaped fibers appear randomly throughout the various types of pitch fiber studied. This effect is reportedly related to the spinning operation and is independent of heat-treatment condition. The internal texture of the fibers as inferred from phase contrast microscopy and scanning electron microscopy of tensile fracture surfaces is radial; i.e., the basal planes radiate from the fiber center.

Two types of pitch fiber were studied: (a) high-fired pitch fibers for nosetip applications, and (b) low-fired pitch fibers for heat-shield application. The high-fired pitch fibers exhibited high densities of 2.002 to 2.024 g/cc. The moisture adsorption was low, from 0.27 to 2.27%, indicating a low intrinsic pore structure. The crystallite size, $L_c$, ranged from 112.0 to 144.0 Å with a d-spacing of 3.43 to 3.44 Å.

The low-fired pitch fibers were heat-treated to varying temperatures, cycles A, B and C. With increasing temperature, the density increased from 1.614 to 1.773 g/cc, the moisture pickup decreased from 7.44 to 0.92%, the fiber diameter decreased from 13.22 to 11.22 microns, the crystallite size increased from 14.59 to 17.44 Å, while the interlayer distance remained invariant at 3.53 Å. Clearly, with increasing temperature, the fiber is shrinking and pore closure takes place.

## Nosetip Composites

Four 3-D preforms (2 in. × 2 in. × 5 in.) using Union Carbide VS-0022 high-fired pitch were woven by Fiber Materials Incorporated (FMI). Two preforms were densified at FMI using the high pressure (10,000 psi) process, and two were densified at Union Carbide (UCC) using both low pressure, 500 psi autoclave, and medium pressure, 5000 psi. Preliminary data as summarized in Table 1 indicate that the composite densities are greater with pitch fiber and that the strengths approach the standard T-50 value. Lower pressures can be used to achieve values comparable to those obtained with high-pressure impregnation.

Table 1

NOSETIP COMPOSITE PROPERTIES

| Type | Specific Gravity (g/cc) | Ultimate Tensile Strength (psi) | Modulus ($psi \times 10^6$) |
|---|---|---|---|
| T-50 (Standard) | 1.91 | 31,000 ±1,800 | 19 |
| Pitch UCC (Low Pressure | 1.95 | 25,000 | 28 |
| Pitch UCC (Med Pressure) 5000 psi | 2.04 | 23,500 | 23 |
| Pitch FMI (High Pressure) 10,000 psi | 1.95 | 27,600 | 23 |

## Heat-Shield Composites

One each (1), 12 in. × 12 in. × 0.3 in. flat panels were fabricated by Hitco for the following systems: (a) UCC pitch fiber A/SC-1008, (b) UCC pitch fiber B/SC-1008, (c) UCC pitch fiber C/SC-1008, and (d) Hitco staple rayon /91-LD. Systems (a) through (c) were prepregged by Fiberite, and system (d) was prepregged by U.S. Polymeric. Table 2 shows preliminary tag end data in comparison with standard rayon material. These data indicate that the A system looks very promising; the density approaches that of the rayon base material, the ultimate tensile strength is higher, and the elongation is greater.

## Conclusions

The highlights of these data are: (a) high-fired pitch fibers have been used to produce 3-D composites with properties comparable to those obtained with T-50; (b) densities of 3-D composite structures using pitch fibers have increased without using high-pressure impregnation; and (c) low-fired pitch-base fibers have been used in carbon phenolic to achieve properties that compare favorably with the reference rayon base material.

Table 2

HEAT-SHIELD COMPOSITE PROPERTIES

| Type | Specific Gravity (g/cc) | Ultimate Tensile Strength (psi) | Elongation (%) |
|---|---|---|---|
| (Standard) CCA-3-1641 Rayon | 1.43 | 7,500 | 0.35 |
| Staple Rayon | 1.405 | 9,600 | 0.71 |
| Pitch A/SC-1008 | 1.465 | 10,946 | 0.59 |
| Pitch B/SC-1008 | 1.519 | 9,993 | 0.63 |
| Pitch C/SC-1008 | 1.521 | 6,861 | 0.47 |