

LOW COST CARBON FIBERS FROM COAL TAR PITCH

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Introduction

Carbon and graphite fibers inherent properties of low density, high strength, high stiffness, good electrical and thermal conductivity, and electrochemical properties make them attractive for a wide variety of applications that has lead to their commercial commodity status. The cost of carbon/graphite fibers is typically related to their properties, which relates to the processing requirements. The basic cost of carbon/graphite fibers is governed by the cost of the precursor, capital for the processing plant and the timing/difficulty of the various processing steps. One of the most cost sensitive processing steps is the stabilization/oxidation of the thermoplastic spun green fiber to convert the structure to one that will not fuse during the pyrolysis/carbonization step.

Even though carbon/graphite fibers are a commodity product and the cost has dramatically been reduced over the past decade, the cost has not decreased to the point to meet automotive requirements, which is under \$5/lb in 2000 dollars. There is a very large incentive to meet the low cost desired by automotive, which can reduce weight dramatically, and which translates to fuel efficiency and savings. For example, graphite fiber composites have densities in the 1.25 to 1.6 g/cc (0.045 lbs/in³ – 0.058 lbs/in³) compared to aluminum at 0.099 lbs/in³ and steel at 0.28 lbs/in³.

If it is assumed the fiber spinning cost is a constant, then any cost reduction to produce carbon/graphite fibers would be precursor cost and the conversion of the green spun fiber to the carbon state. Polyacrylonitrile (PAN) has been the primary precursor to produce carbon fibers. PAN has been extensively investigated for several decades without any major cost reduction and no suitable resin/polymer has been demonstrated to provide any measurable savings. Petroleum pitch is the only other precursor from which commercial fibers have been produced. Preparation cost due in part to the variability of the pitch that stems from the wide variations in crude has not resulted in a lower cost carbon/graphite fiber. Further, at least in part petroleum pitch is not a domestic source precursor since the U.S. imports 50% or more of its crude. A good refined grade of petroleum pitch, which can be used to produce reasonable quality carbon/graphite fiber is in the \$1000 - \$8000 per ton, which is not conducive to low cost fiber.

Heretofore, no commercial fibers have been produced from coal-tar pitch. However, for some of the same reasons coal-tar is an excellent and preferred binder precursor to produce anodes for aluminum refining and carbon-carbon composites, it is a good precursor to produce carbon/graphite fiber, and it is a domestic source. Coal-tar pitch is quite economical in the cost range of \$200-\$350/ton. A program was undertaken to demonstrate a low cost domestic source, coal-tar pitch, could be utilized to produce high quality low cost carbon/graphite fibers.

Experimental

In order to eliminate the time consuming costly stabilization/oxidation step in carbon/graphite fiber production, an oxidizing source additive was added to the coal-tar pitch precursor. The coal-tar pitch was processed to provide a material suitable for fiber spinning. In one processing scheme, the pitch is dissolved in 1-methyl-2-pyrrolidinone (NMP), which extracts the insolubles including any inorganic ashes. The coal-tar pitch (CTP) is mixed with a select oxidant and heated under a temperature protocol to polymerize the molecular structure and achieve a softening point in excess of 200°C. If mesophase is desired appropriate thermal cycling is performed to produce a mesophase for subsequent fiber spinning. Thus the CTP can be processed to produce an isotropic or mesophase precursor.

The treated/processed pitch precursor is spun into a green fiber using standard processing. Because of the internal molecular oxidant added to the pitch precursor the green fibers can be passed directly into pyrolysis/carbonization thus eliminating the time consuming costly stabilization/oxidation step. Green, as-spun pitch fiber containing the molecular oxidant were pyrolyzed/carbonized using standard conductive heat transfer tube furnaces and separately microwave heating. The strength of the fibers were established as a function of the heat treating temperature.

Results and Discussions

Pitch processing schemes coupled with the oxidant additive was worked out to permit the spinning of high quality fiber. The cost goal was that pitch preparation

processing would not exceed the basic cost of the pitch. That is if pitch cost \$300/ton to result in a precursor cost no greater than \$0.30/lb. Another goal was that to achieve fiber strength equivalent to commercial quality carbon fibers.

To meet the low cost goal of carbon/graphite fibers at less than \$5/lb, the heat treating cost must be low. The most economical furnace systems utilize metallic alloy heating elements whose maximum temperature is slightly less than 1300°C. Therefore, it was desirable to produce high strength fibers at a heat treatment of 1200°C or less. The strength of fibers as a function of temperature is shown in Table I. An SEM of the fibers is shown in Figure 1. As can be seen excellent carbon/graphite fiber can be produced from coal-tar pitch. While only limited experimental quantities of fibers have been produced, there is a high probability the less than \$5/lb goal can be met. A commercial demonstration is scheduled to be conducted in the latter part of 2001 that will further verify properties and cost.

Conclusions

An economical process for processing coal-tar pitch has been demonstrated which eliminates the requirement of a separate step for stabilizing/oxidizing spun green fibers before pyrolysis/carbonization. Fiber strengths equivalent to commercial carbon/graphite fibers have been demonstrated with a projected cost of less than \$5/lb.

Acknowledgement

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Table I Properties of Carbon Fibers Prepared From Coal Tar Pitch Precursor

Fiber Pyrolysis Temp °C	Fiber Tensile Strength, GPa		Commercial Fiber Strength, GPa	
	Isotropic	Anisotropic	Petroleum Pitch	Polyacrylonitrile (PAN) T300
600	1.8	--	--	--
1000	2.3	--	--	--
1200	3.8	2.5	--	--
1800	4.2	--	--	--
			2 – 3	3.5

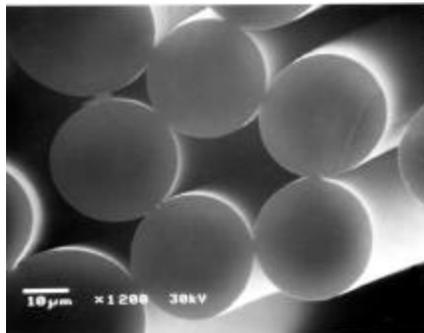
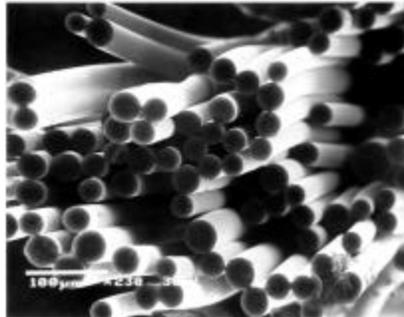
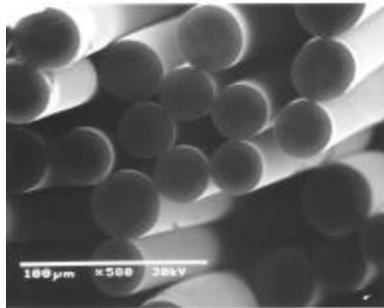


Figure 1. Fibers spun from coal-tar pitch precursor