# ORIGIN OF MICROSTRUCTURES FOUND IN MESOPHASE PITCH-BASED CARBON FIBER

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## Introduction

Mesophase pitch-based carbon fibers have been recognized as one of the strategic materials for energy-saving and thermo-management because of their excellent mechanical strength and thermal conductivity. It is very necessary to clarify the meso and microstructure at the nanoscale and establish its correlations to found the properties and procedures to built in such a structure. The present authors have observed pleat and microfibril structures exclusively in the graphitized mesophase pitch-based carbon fibers and suggested their significances on the properties.

In the present study, the authors intended to confirm such structures in the mesophase pitch-based carbon fibers and to clarify how the structure was built in the fibers through the fiber preparation from mesophase pitch by observing the mesophase pitch and its insoluble fraction, as spun fiber and its insoluble component, stabilized and carbonized fiber, using a high resolution SEM.

## Experimental

The naphthalene derived mesophase pitch was prepared by Mitsubishi Gas Chemical Company using HF/BF3 as a catalyst.

Mesophase pitch was melt-spun at 310°C through a spinneret with diameter 0.3 mm, L/D=3, using the laboratory scale mono-filament spinning apparatus. The spun fibers were oxidatively stabilized at 270°C for 30 minutes in air with a heating rate of 0.5°C/min. The carbonization was performed at 1000°C for 1 hour in Ar gas with a heating rate of 10°C/min.

Mesophase pitch and its spun fiber were extracted with toluene, tetrahydrofuran, and pyridine in a Soxhlet apparatus at their boiling point.

Surface structures of the pitch, spun fiber and their insoluble fractions were observed by a high resolution scanning electron microscope (HR-SEM). All samples were observed after the platinum coating. The fiber samples were observed in two directions as shown in Figure 1 where the surface of the sample fiber was placed either perpendicular or tilt against the electron beam.

## Results

Figure 2 shows HR-SEM photographs of the mesophase pitch and its pyridine-insoluble fraction. The mesophase pitch as-received exhibited featureless flat surface of flaky grains, no particular grain unit being detected. The extracted spun fiber with solvent revealed that the insoluble fraction consisted of rod-like micrograins of diameter 10 nm and length 50 nm, respectively.

Figure 3 shows the HR-SEM photographs observed from low angle to the fiber axis of as-spun, pyridine-insoluble fraction of the as-spun, and carbonized fibers. As-spun fiber exhibited wavy surface, consisting of randomly distributed ripples, however, no definite fibril and pleat was observed in this fiber. At the surface of as-spun fiber extracted with pyridine, the pleat structures, existing in perpendicular direction to the fiber axis, obviously appeared at the surface of the spun fiber, as appeared in similar shape observed in its carbonized fiber. Fibrils formed by lamination of pleats also appear to be aligned along the fiber axis and the thickness of fibril was similar to those in the carbonized fiber (Figure 3 (c)). In contrast, the carbonization produced definite fibrils and pleats which run in the parallel and perpendicular directions, respectively, to the fiber axis.

## Discussion

The height of pleat unit was ca 5 nm, hence tilt observation was very necessary to observe their regular alignment, while its perpendicular observation showed up the microdomain, their ordered alignment being difficult to be detected.

The mesophase pitch showed the textureless feature of its as-received surface. However, the solvent extraction closed up the rod-like microdomains in the insoluble fraction, of which size and shape were rather uniform. Such insoluble microdomains are arranged by the spinning with the soluble fraction in the fibers, forming the basic structure of fibril and pleat as observed in the extracted as-spun fiber. The spinning appears to arrange the microdomains of the insoluble fraction parallel along the fiber axis, maintaining basically its the shape and size. The soluble fraction appeared to coat the insoluble fraction at spinning to hide the alignment of the insoluble fraction.

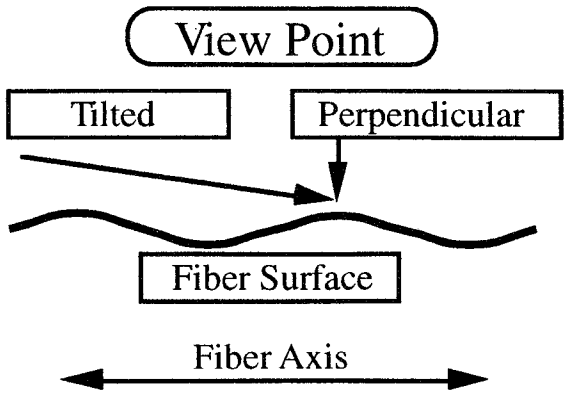


Fig.1 Schematic diagram of two view points examined the surface structure of fiber by HR-SEM.

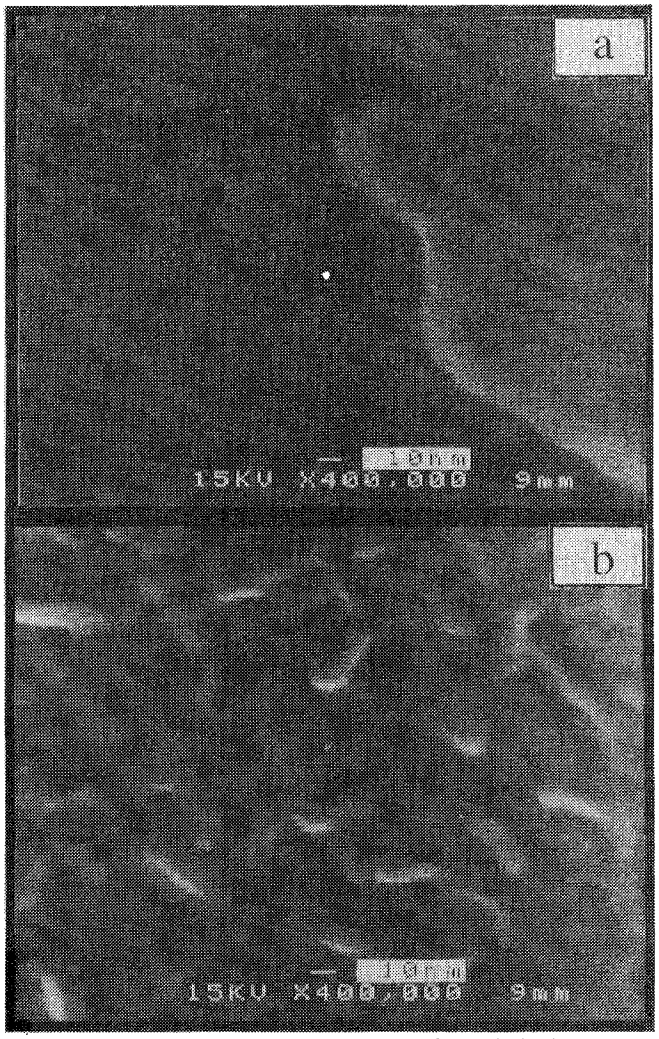


Fig. 2 HR-SEM photographs of naphthalene-based mesophase pitch (a) and its insoluble fraction of pyridine (b).

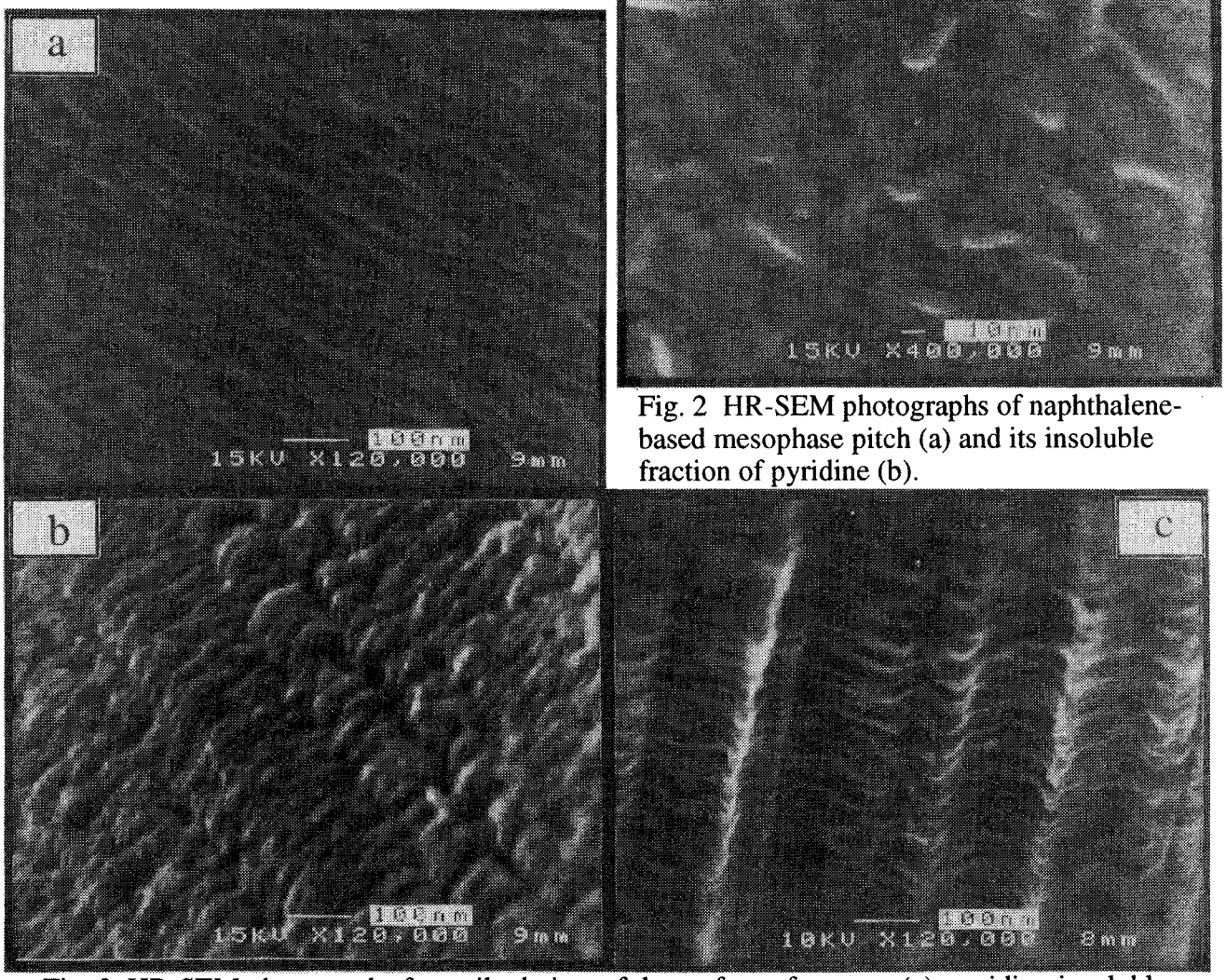


Fig. 3 HR-SEM photographs from tilted view of the surface of as-spun (a), pyridine-insoluble fraction of the spun (b), and carbonized fiber (c).