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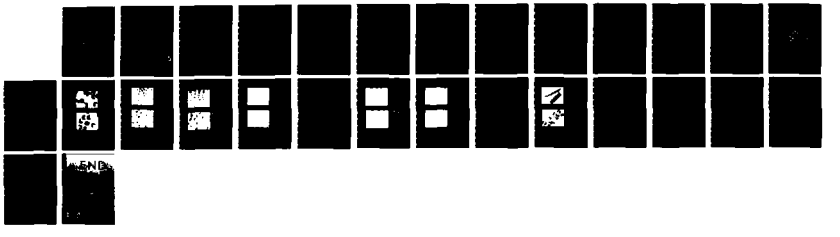

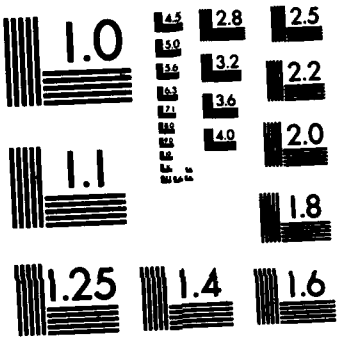

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Mesophase Behavior Fundamental to the Processing of Carbon-Carbon Composites

J. L. WHITE
Materials Sciences Laboratory
The Aerospace Corporation
El Segundo, Calif. 90245

December 1982

Interim Technical Report for Period 1 October 1981 through 30 September 1982

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This report has been reviewed by the Public Affairs Office (PAS) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nationals.

This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

James C. Garcia, Captain, USAF
Project Officer

Joseph Hess, GM-15, Director
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Carbonaceous mesophase	

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Investigated are the behaviors of the carbonaceous mesophase that significantly influence the processing of carbon-carbon composites. An understanding of those behaviors will provide insights for more effective and economical fabrication of such composites. This first annual report summarizes micrographic findings obtained during the development of experimental procedures for observing how mesophase morphologies are formed within carbon fiber bundles at various stages in pyrolysis.

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19. KEY WORDS (Continued)

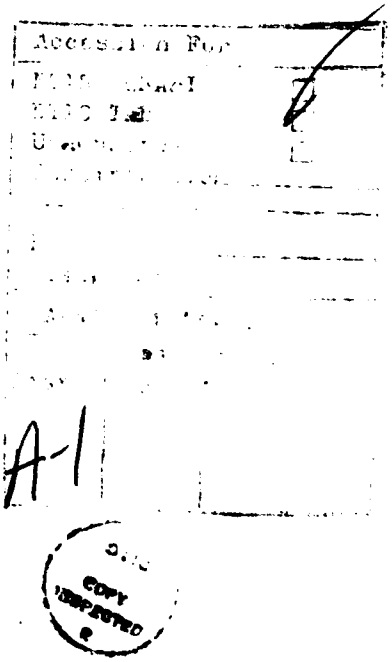
20. ABSTRACT (Continued)

Studies of room-pressure pyrolysis identified the following patterns of mesophase formation within fiber bundles. The mesophase transformation proceeds more slowly within a bundle, and without the extensive deformation that characterizes mesophase pyrolyzed in bulk. In the early stages of transformation, the fiber is wetted by both mesophase and pitch, a behavior that appears to be independent of fiber type, at least for the four fibers tested (PAN-based fiber: T300; mesophase-based fiber: VSA-11, P55, and P100). The coarseness of the porosity developed within a bundle seems to be determined by the extent to which the fiber bundle is restrained. Individual filaments in open, unconstrained bundles can apparently move locally in response to interfacial tensions, to produce regions in which the filaments are more closely packed. Two impregnant pitches, A240 petroleum pitch and 15V coal-tar pitch, were studied and observed to differ principally in that insoluble particles were filtered from the coal-tar pitch, the smaller-diameter fibers being the more effective filtering agents. After pyrolysis through mesophase hardening, the mesophase semi-coke is a fragile solid easily fractured by local stresses within the fiber bundle.

For preparing specimens representative of various stages in high-pressure pyrolysis, a method of quenching by self-cooling of a low-thermal-inertia furnace within a cold-wall autoclave appears to be adequate. Heat transfer rises so rapidly with pressure that some furnace insulation will be required at pressure levels above 7 MPa (~1000 psi). One set of fiber bundles was processed under A240 petroleum pitch to final conditions of 447°C and 7 MPa; micrographic examination revealed that the wetting behavior, the formation of mesophase sheaths, and pore agglomeration appear much the same as those observed in room-pressure pyrolysis.

PREFACE

This is the first annual report on a program to study the fundamentals of mesophase behavior that pertain to the processing of carbon-fiber-reinforced carbon-matrix composites. The work is supported by the Office of Naval Research (ONR) under Contract No. N00014-81-MP0006, work unit number NR 039-183. The principal investigator is J. L. White. The ONR scientific officer is L. H. Peebles, Jr.; his interest and support are gratefully acknowledged. We also wish to thank P. M. Sheaffer, M. Buechler, C. B. Ng, and F. B. Sinsheimer for their contributions to the experimental work; G. S. Rellick and J. S. Evangelides for technical consultation; and J. E. Zimmer of the Acurex Corporation for extensive technical collaboration.



CONTENTS

FIGURES

I. INTRODUCTION

Carbon-fiber-reinforced carbon-matrix composites have been established by their high-temperature properties as leading candidate materials for many structural applications demanding resistance to thermal shock, strength at temperature, and resistance to erosion by hot high-velocity gas streams. Most fabrication processes for such composites form a pregraphitic matrix by impregnating a carbon fiber bundle with coal-tar or petroleum pitches that pass through a mesophase (liquid-crystal) state upon carbonization. Thus the carbonaceous mesophase plays a key role in fabrication because its behavior appears to determine what the microstructure of the composite matrix will be, as well as how many impregnation and graphitization cycles will be required to reach desired levels of density.

That the mesophase transformation has an essential part in determining the graphitizability of carbonaceous materials was first recognized by Brooks and Taylor in 1965,[1,2] and has since been pursued by carbon scientists interested in how the lamelliform morphology of graphitic materials occurs in the carbonization of organic precursors. The principal microstructural features of coke and graphite have now been established as originating during the brief plastic lifetime of the carbonaceous mesophase before it congeals to a solid semi-coke.[3,4,5]

In previous work we have quenched specimens from various stages of pyrolysis, using several different methods, to learn how microstructures form in petroleum coke.[6] Applying such methods to composite processing, however, requires techniques for quenching at high pressure and for micrographic preparation of specimens whose microconstituents differ widely in hardness. Thus the present studies focused initially on developing and testing experimental techniques; a technical report[7] was prepared upon conclusion of that preliminary work, because the micrographic results carried forward observations recently reported by Cranmer et al.[8]

In work currently in progress, the method of interrupted pyrolysis is being used to explore significant process variables, including the types of

pitch and fiber, the pyrolysis temperature, and the processing pressure. This report summarizes structural observations from the preliminary experimental work,[7] documents some points of potential significance emerging from pyrolysis studies currently in progress, and describes briefly two background studies completed during the project year.[5,9]

II. BACKGROUND STUDIES

The fields of research on the carbonaceous mesophase and conventional liquid crystals have developed with little contact or interaction, presumably because of the disparity in applied interests. Two review studies were completed within 1982 to summarize results of potential interest to the respective technical communities.

In collaboration with J. E. Zimmer of the Acurex Corporation, we prepared an invited paper entitled "Disclination Structures in the Carbonaceous Mesophase,"[9] which reviewed microstructural information on the carbonaceous mesophase for the liquid-crystal community. The layer- or plate-like molecules of the carbonaceous mesophase place it in the newly recognized discotic class of nematic liquid crystals. Although analyses of disclination structures in nematic and smectic liquid crystals are quite advanced,[10,11] some practical observations [e.g., the continuous core of -2π wedge disclinations[12]] appear to have been made first in studies of the carbonaceous mesophase. Nevertheless it is not surprising to find that, as long as the carbonaceous mesophase remains quite fluid, its disclination structures and interactions are largely predictable from previous work on nematic liquid crystals.

The second invited review[5] was prepared for presentation to the International Symposium on Carbon, held at Toyohashi, Japan. Based on the foregoing review plus recent observations by a quenching hot-stage microscope,[13-15] the paper focused on disclination structures in coke and graphite and emphasized that such materials may be viewed as mesophase fossils in which the microstructures have been frozen into place as the mesophase hardened. Since the mesophase is often being deformed by bubble percolation or mechanical loading as it hardens, the morphology of materials such as needle coke or mesophase fiber can include nonequilibrium structures, e.g., wrinkled layers[16] and unsymmetrical disclinations,[15] that would not be expected in an equilibrium mesophase. Such features could significantly influence the mechanical properties of the fabricated graphite or mesophase carbon fiber.

III. PYROLYSIS AT ROOM TEMPERATURE

The initial pyrolysis studies were conducted at room pressure to address three questions of experimental technique: (1) the suitability of a device designed to hold unidirectional fiber bundles under well-defined constraints, (2) the micrographic preparation of the fiber-pitch-mesophase specimens, and (3) the possible effect of the presence of fibers on the kinetics of the mesophase transformation. The further purpose was to establish the patterns of mesophase formation in fiber bundles under the more economical conditions of specimen preparation at room pressure; the results should be useful in reducing the number of high-pressure pyrolysis runs required to ascertain the formation pattern at high pressure.

The metal fixture illustrated in Fig. 1 is designed to hold four separate fiber bundles under three conditions of exposure to the impregnant pitch: restrained within a well-defined rectangular cavity (section B), open to penetration by the pitch from the sides only (section C), and open to pitch penetration from both the sides and the splayed end (section A). Only four micrographic sections are required to observe open, constrained, and splayed fiber bundles in longitudinal and transverse sections. The fixture fits within a 19-mm-diam pyrolysis cell standardized in our laboratory to permit simultaneous thermal treatment of as many as 19 separate cells.[17]

For the initial pyrolysis runs, the fixture was submerged in A240 petroleum pitch. A mesophase-based carbon fiber (VSA-11) was selected to permit comparison of the results with recently published work;[8] that fiber lot was composed primarily of radial filaments with graphitic layers exposed in the open wedge. The pyrolysis levels were selected to correspond to an early stage in the mesophase transformation (430°C) and to full transformation (505°C). The results from the relevant topical report[7] are summarized here.

Mesophase transformation within the fiber bundle proceeds differently than that expected from observations of bulk pyrolysis. In the early stages, both pitch and mesophase wet the fiber, and the mesophase tends to occur preferentially within the open wedge of the radially structured filaments.

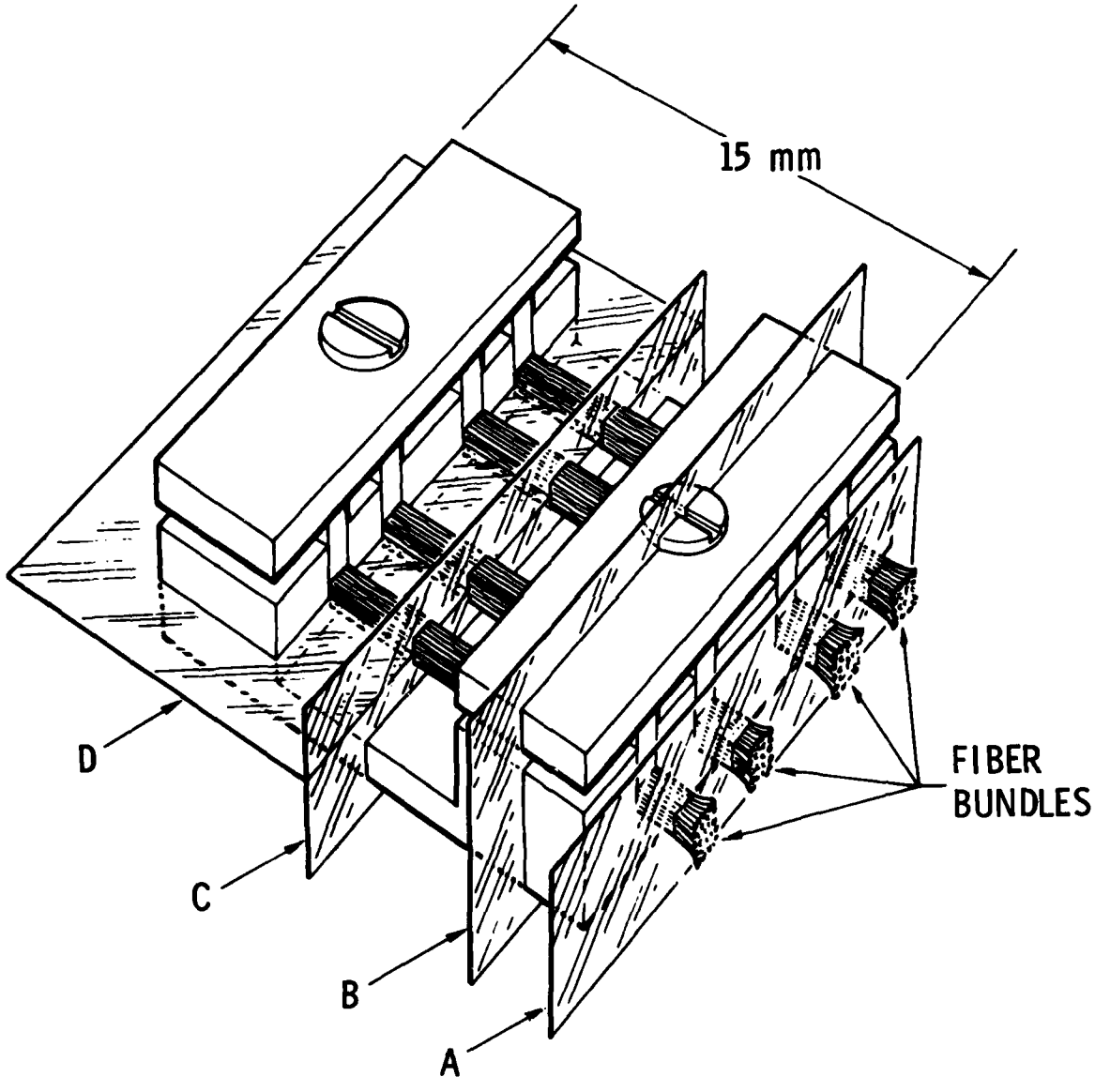


Fig. 1. Fiber Bundle Holder. The aluminum fixture is designed to hold four fiber bundles under well-defined conditions of restraint during pyrolysis within a pool of impregnant pitch. Planes A, B, and C define transverse sections for splayed, constrained, and open bundles, respectively, and plane D defines longitudinal sections for the four fibers.

The sheath effect, i.e., the alignment of mesophase layers parallel to the filament substrate,[18] is present in both the open wedge and on the circular periphery of the radial filaments. The coarseness of the matrix porosity is governed by the bundle constraint. The open bundle appears to permit more local motion of filaments, thus allowing denser packing and contributing to the pore size. When transformation is complete, the mesophase displays good wetting of the fibers. Figure 2 is reproduced from the topical report to illustrate how fragile the mesophase is at 505°C.[7]

On the basis of the initial studies, a more comprehensive study has been undertaken to explore the effects of varying the fiber and the pitch, as well as the pyrolysis level. Three mesophase fibers (P55, P100, and VSA-11) are being compared with a PAN-based fiber (T300) for two types of impregnant (15V coal-tar pitch and A240 petroleum pitch); the pyrolysis levels extend from 425 to 490°C. The micrographic results offered below represent tentative conclusions emerging from the study in progress.

Figures 3 and 4 illustrate competitive wetting by pitch and mesophase on constrained fiber bundles (section B) immersed in A240 petroleum pitch. All fibers were heat-treated in argon to 500°C (5 min) to remove sizing. The pyrolysis temperature was 438°C. The wetting behavior for the four fibers is quite similar despite the differences in filament morphology. Although the pitch-mesophase interface displays a range of angles relative to the fiber substrate, the average angle appears to lie near 90 deg. Where pores are present, the wetting angle for pitch or mesophase is near 0 deg.

The coal-tar pitch 15V is often used for composite impregnation; it contains about 7 wt% quinoline insolubles,[19] primarily in the form of soot-like particles measuring about 1 μm.[20] Figure 5 depicts the filtration effect observed for all fibers on the open bundle section (section C) after pyrolysis to 464°C. Despite their small size, the insolubles do not readily enter the fiber bundle. The filtration becomes more effective at finer fiber sizes; in the case of T300 fiber, few insoluble particles are present within the bundle matrix. Filtration is sufficient in all cases to produce a layer of insoluble-enriched mesophase surrounding the fiber bundle. Figure 5 also illustrates the coarse porosity in the open fiber bundles.

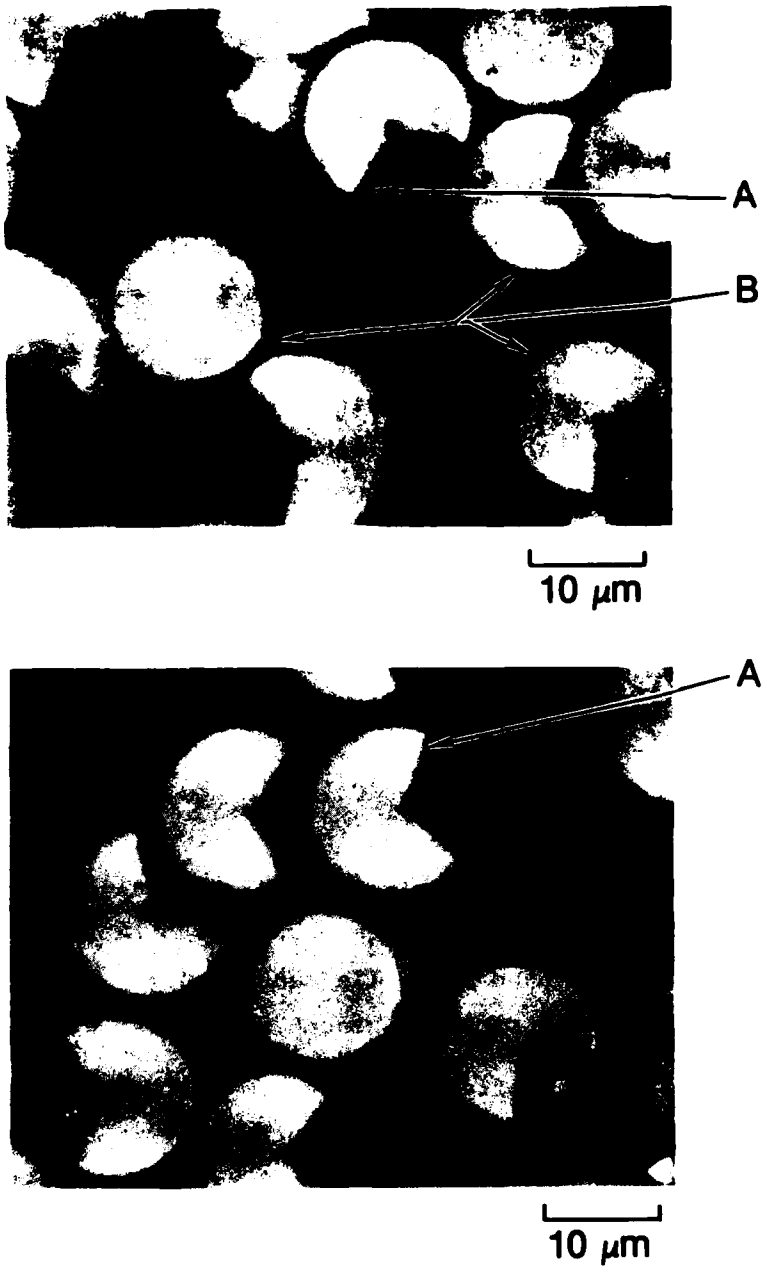


Fig. 2. Mesophase Wetting and Fracture at Pores in Open Fiber Bundle. Pyrolysis condition: 505°C. Near-zero wetting angles appear both on open-wedge surfaces (A) and on the periphery (B) of the mesophase fiber. Immersion oil, polarizer only.

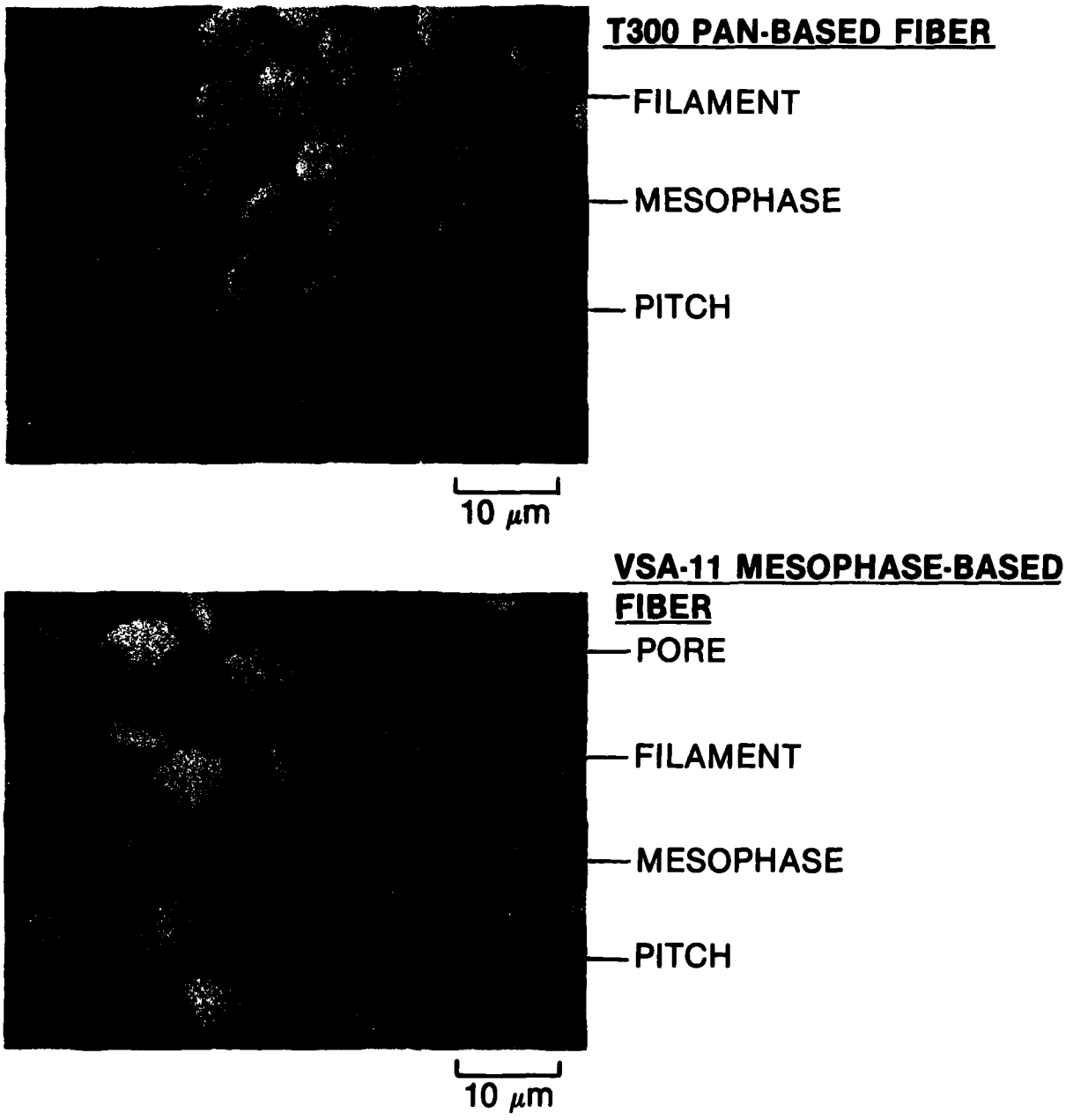
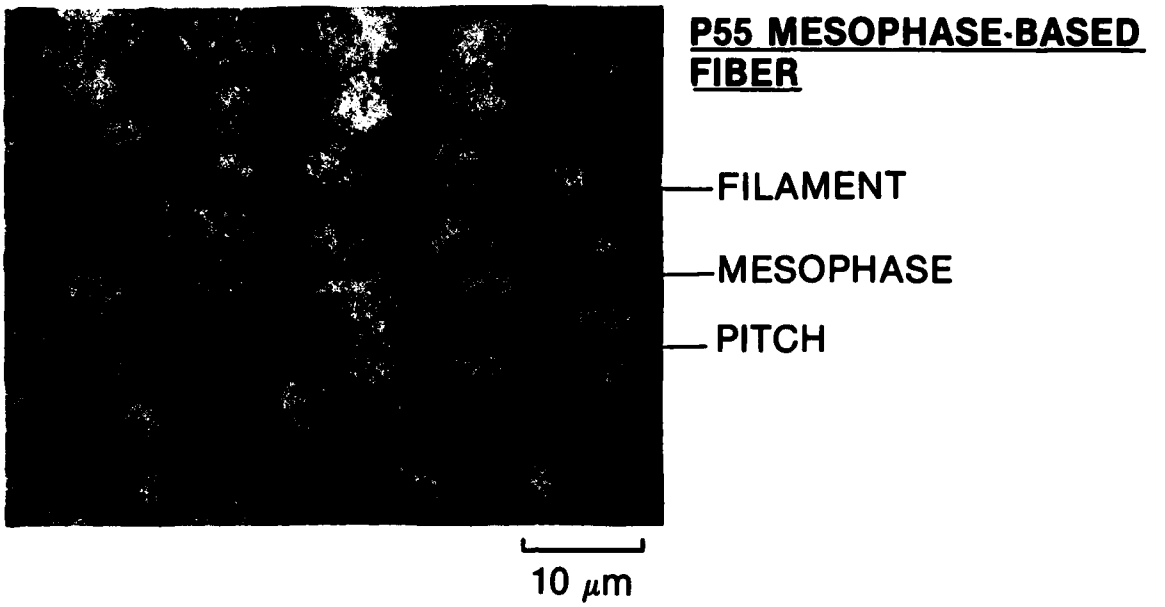


Fig. 3. Wetting by Pitch and Mesophase within Carbon Fiber Bundles. Pyrolysis condition: 438°C. Restrained fiber bundles, section B. Polarizer only, immersion oil.



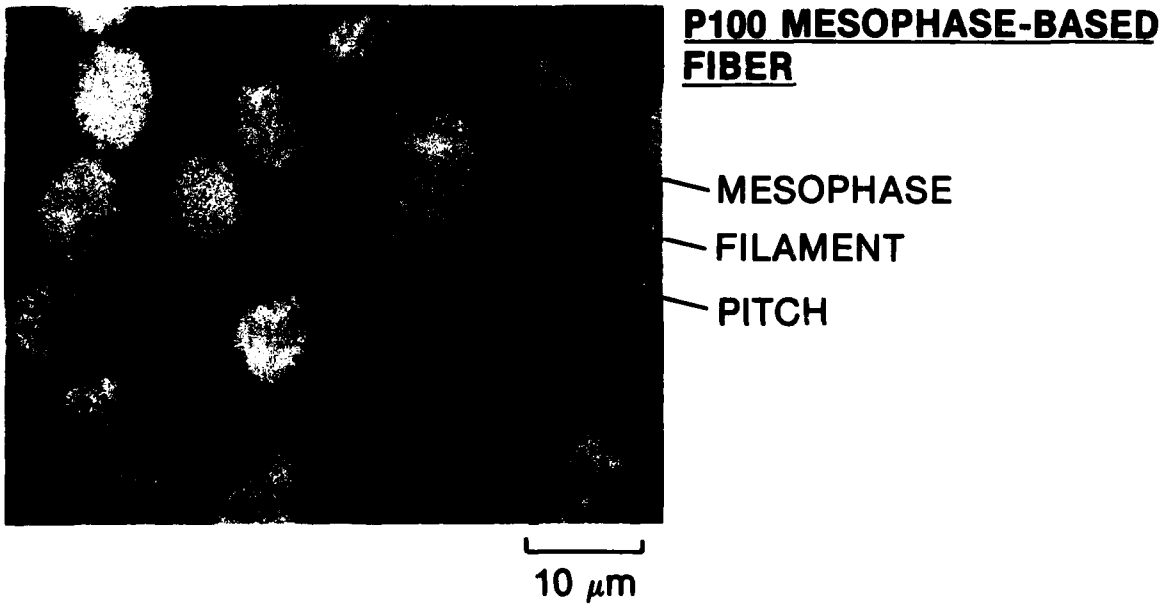


Fig. 4. Wetting by Pitch and Mesophase within Carbon Fiber Bundles. Pyrolysis condition: 438°C. Restrained fiber bundles, section B. Polarizer only, immersion oil.

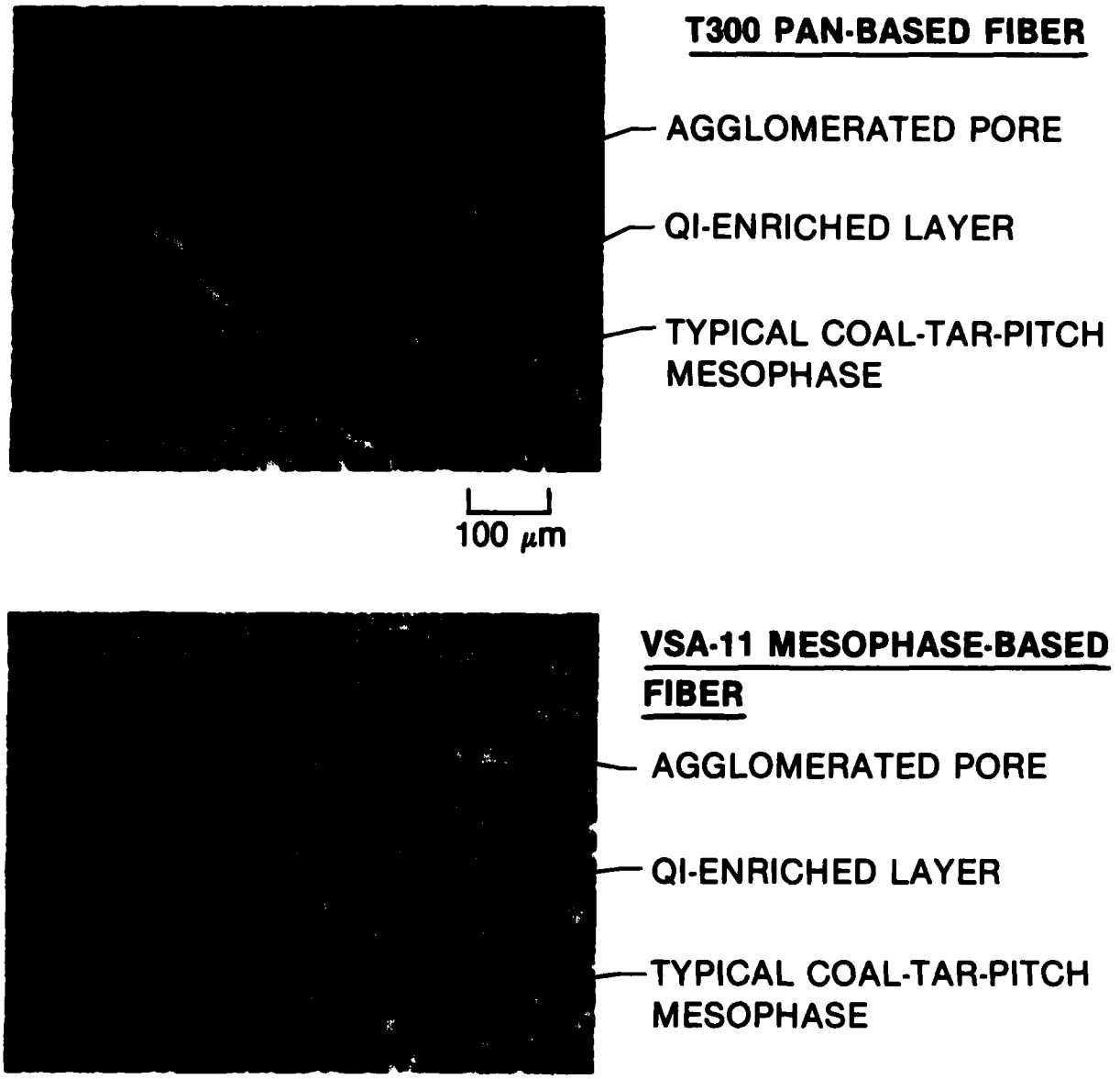


Fig. 5. Carbon-Fiber-Bundle Filtration of Quinoline-Insoluble (QI) Particles from 15V Coal-Tar Pitch. Pyrolysis condition: 464°C. Open fiber bundles, section C. Crossed polarizers.

IV. PYROLYSIS AT HIGH PRESSURE

Reasonable quenching rates must be achieved for specimens containing partially transformed mesophase, so that the liquid-crystal microstructures existing at the point of quench can be frozen.[21] However, rapid cooling is necessary only to about 250°C; hot-stage observations[14] indicate that most mesophase-bearing specimens are frozen solid by that point. Since it is impractical to quench the mass of a hot-wall autoclave, we have tested the idea of self-cooling of a small furnace within a cold-wall autoclave. A furnace of low thermal inertia was designed to fit within the 5-cm-diam autoclave at our disposal and to hold a single 19-mm pyrolysis cell. Test runs demonstrated that cooling rates were adequate even at room pressure and that convective heat transfer increased so rapidly with pressure that power lead-throughs of increased capacity and some insulation will be required to reach 500°C at pressures above 7 MPa (1000 psi).

During those autoclave tests, we pyrolyzed one set of four fiber bundles in A240 petroleum pitch to a minimum temperature of 447°C under nitrogen at 7 MPa. The micrographic observations described below were made on that specimen.

Figure 6 provides a low-magnification comparison of the microstructures of mesophase formed around open bundles of VSA-11 fiber by pyrolysis of A240 petroleum pitch at two different pressure schedules. For both schedules, the final pyrolysis temperature was 447°C, sufficient to induce near-100% transformation in the bulk pitch outside the fiber bundle. The relative spacing of polarized-light extinction contours reflects the major difference between the two: the greater level of deformation by bubble percolation[6] sustained by the mesophase formed under room pressure. The fiber bundles display similar coarseness in porosity; this similarity holds also for the other fiber types not illustrated here.

The higher-magnification views of P55 fiber bundles in Fig. 7 show that the transformation within the fiber bundle is much less advanced than that in the external regions. The wetting conditions at the modest pressure level

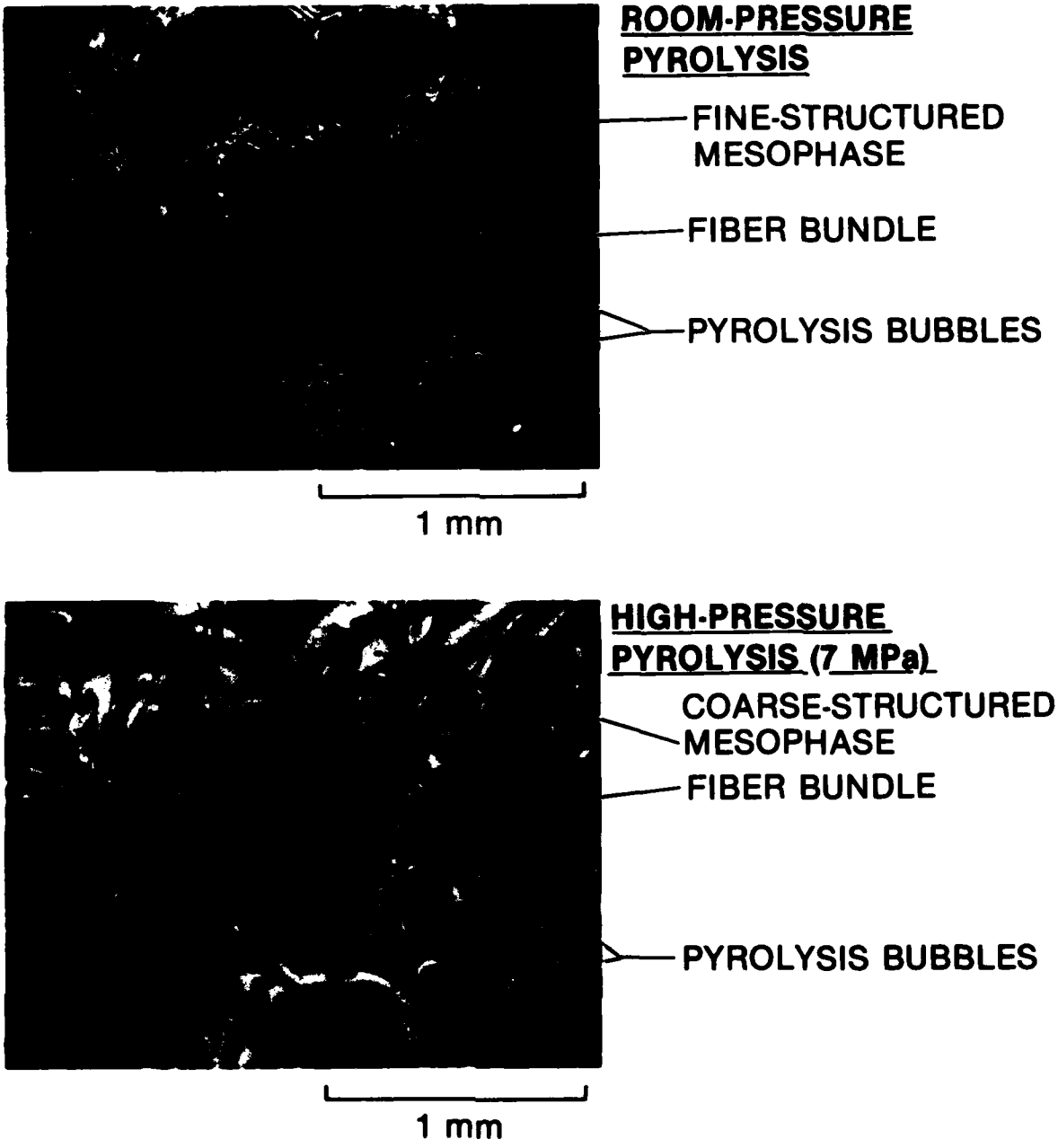


Fig. 6. Effect of Pressure on Pyrolysis to 447°C of VSA-11 Mesophase-Based Fiber in A240 Petroleum Pitch. Open fiber bundles, section C. Crossed polarizers.

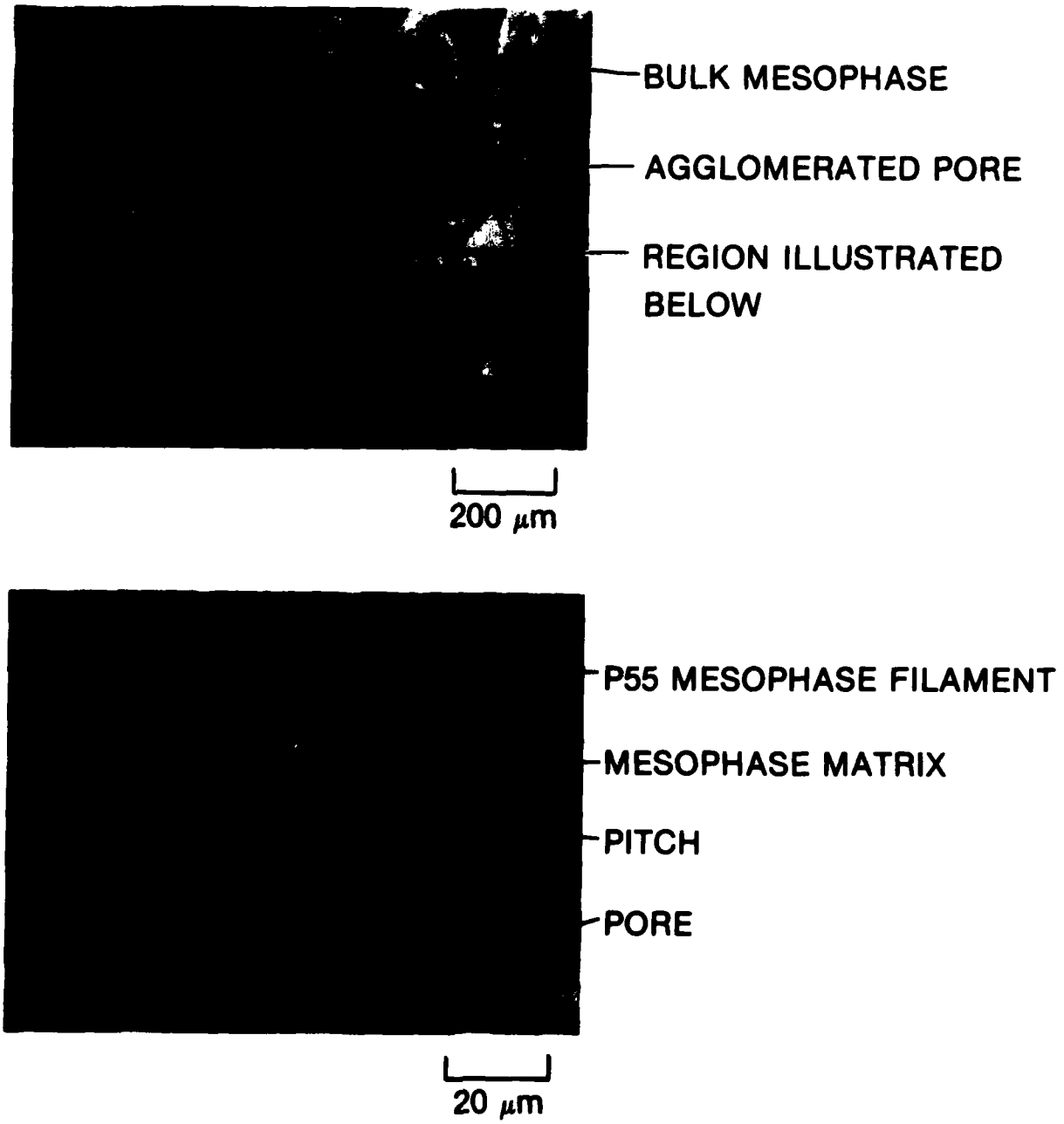


Fig. 7. Mesophase Carbon Fiber (P55) Pyrolyzed at High Pressure in A240 Petroleum Pitch. Open bundle (section C). Final pyrolysis condition: 447°C at 7 MPa (1000 psi). Polarizer only.

evidence no change from room pressure (cf., Figs. 3 and 4). The mesophase appears to contain more closely packed clumps of filaments, indicating that the coarseness of the pore structure may be augmented by the action of interfacial tensions on filaments having some freedom to move locally. This type of particle agglomeration has been observed previously[22] for coke particles and natural graphite flakes, Fig. 8.

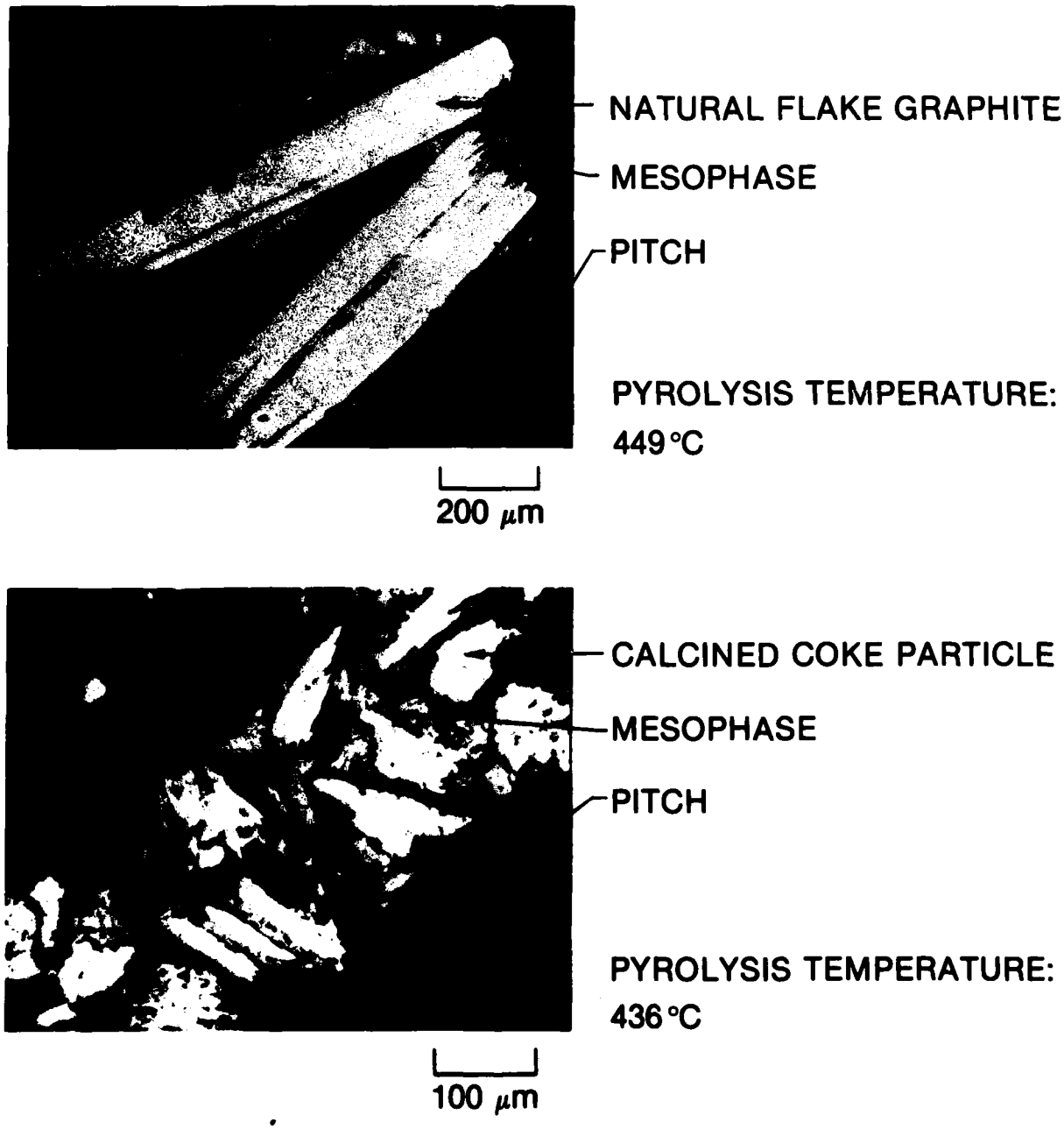


Fig. 8. Agglomeration of Graphite and Coke Particles by Mesophase Transformation in an Extracted Coal-Tar Pitch.[22] Crossed polarizers.

V. FURTHER INVESTIGATIONS

The work to date indicates that the experimental methods are adequate to define the development of mesophase microstructures within fiber bundles. The results demonstrate the probable significance of such phenomena as the competitive wetting behavior of mesophase and pitch, the alignment of mesophase layers on substrate fiber, the effect of bundle constraints on filament rearrangement and matrix porosity, and the filtering action of a fiber bundle on the insoluble particles of a coal-tar pitch. The initial results of current studies also indicate that the mesophase behavior is largely independent of fiber variety, the principal exception being the apparent geometrical effects of fiber size on filtering action and filament packing. This apparent independence of fiber variety may be an important simplifying factor in additional experimentation and will be examined further to test its general applicability to all carbon fibers of practical importance.

Thus the immediate objective in on-going investigations is to complete the description of mesophase formation within fiber bundles, first at room temperature, then at pressures typical of composite processing. Such experiments will also provide specimens for the next phase of study on mesophase hardening and the effects of heat treatment on dimensions, microstructure, and mechanical properties.

VI. REPORTS, PUBLICATIONS, AND PRESENTATIONS

The following publications and presentations were completed during fiscal year 1982 with partial support from this research program:

1. J. E. Zimmer and J. L. White, "Disclination Structures in the Carbonaceous Mesophase," invited review for Adv. Liq. Cryst. 5 (in press).

2. J. L. White, C. B. Ng, P. M. Sheaffer, and M. Buechler, Mesophase Behavior in Carbon Fiber Bundles, TR-0082(2728-01)-1, The Aerospace Corporation, El Segundo, Calif. (1 June 1982).

3. J. E. Zimmer and J. L. White, "Mesophase Alignment within Carbon Fiber Bundles," Carbon (in press).

4. J. L. White, C. B. Ng, G. W. Henderson, and M. Buechler, "Structural Characteristics of Mesophase Carbon Fiber," Extended Abstract for AFWAL/ONR Workshop on Matrix Properties in Carbon-Carbon Composites, Monterey, California, 12-13 May 1982.

5. J. L. White, "Mesophase Mechanisms in Graphite Formation," Ext. Abstr., Int. Symp. Carbon, Toyohashi, Japan (November 1982), pp. 149-152; invited speaker.

6. Related publications and presentations, supported by USAF Space Division:

   a. J. L. White, M. Buechler, and C. B. Ng, "Microscopic Observations on the Carbonaceous Mesophase by Means of a Quenching Hot Stage," Carbon (in press).

   b. M. Buechler, C. B. Ng, and J. L. White, "Observations of Mesophase Behavior by a Quenching Hot-Stage Microscope," Ext. Abstr., Int. Symp. Carbon, Toyohashi, Japan (November 1982), p. 143.

   c. M. Buechler, C. B. Ng, and J. L. White, "Nonequilibrium Disclinations in the Carbonaceous Mesophase," Carbon (in press).

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LABORATORY OPERATIONS

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Aerophysics Laboratory: Launch vehicle and reentry aerodynamics and heat transfer, propulsion chemistry and fluid mechanics, structural mechanics, flight dynamics; high-temperature thermomechanics, gas kinetics and radiation; research in environmental chemistry and contamination; cw and pulsed chemical laser development including chemical kinetics, spectroscopy, optical resonators and beam pointing, atmospheric propagation, laser effects and countermeasures.

Chemistry and Physics Laboratory: Atmospheric chemical reactions, atmospheric optics, light scattering, state-specific chemical reactions and radiation transport in rocket plumes, applied laser spectroscopy, laser chemistry, battery electrochemistry, space vacuum and radiation effects on materials, lubrication and surface phenomena, thermionic emission, photosensitive materials and detectors, atomic frequency standards, and bioenvironmental research and monitoring.

Electronics Research Laboratory: Microelectronics, GaAs low-noise and power devices, semiconductor lasers, electromagnetic and optical propagation phenomena, quantum electronics, laser communications, lidar, and electro-optics; communication sciences, applied electronics, semiconductor crystal and device physics, radiometric imaging; millimeter-wave and microwave technology.

Information Sciences Research Office: Program verification, program translation, performance-sensitive system design, distributed architectures for spaceborne computers, fault-tolerant computer systems, artificial intelligence, and microelectronics applications.

Materials Sciences Laboratory: Development of new materials: metal matrix composites, polymers, and new forms of carbon; component failure analysis and reliability; fracture mechanics and stress corrosion; evaluation of materials in space environment; materials performance in space transportation systems; analysis of systems vulnerability and survivability in enemy-induced environments.

Space Sciences Laboratory: Atmospheric and ionospheric physics, radiation from the atmosphere, density and composition of the upper atmosphere, aurorae and airglow; magnetospheric physics, cosmic rays, generation and propagation of plasma waves in the magnetosphere; solar physics, infrared astronomy; the effects of nuclear explosions, magnetic storms, and solar activity on the earth's atmosphere, ionosphere, and magnetosphere; the effects of optical, electromagnetic, and particulate radiations in space on space systems.

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