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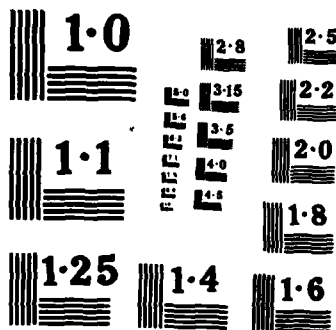
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Mesophase Mechanisms in the Formation of Graphitic Microstructures

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31 December 1984

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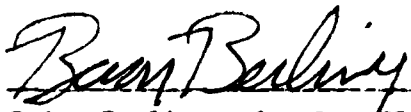
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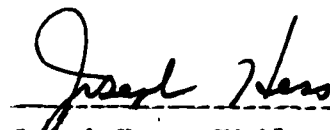
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This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.


Brian Berliner, 1st Lt, USAF
Project Officer


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coalescence, disclination reactions, and orientation fluctuations. However, the mesophase usually congeals to coke while under mechanical deformation, e.g., by bubble percolation in the delayed coker, so that the acicular and lamellar constituents of needle coke are nonequilibrium microstructures locked into place as the mesophase hardens. Mesophase carbon fibers, formed by the drawing and quenching of mesophase pitch, represent extreme examples of deformed mesophase morphologies. Disclination models can be constructed for the internal structures of mesophase carbon fibers.

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PREFACE

We thank the Office of Naval Research for support in the preparation of this review and Dr. L. H. Peebles, Jr., for his encouragement and criticism. We also acknowledge support by the Space Division of the U. S. Air Force, the Naval Surface Weapons Center, and The Aerospace Corporation in various phases of the experimental work reviewed here.

Most of the information in this report was included in a preprint for a presentation to the Second Symposium on Petroleum-Derived Carbons, sponsored by the Division of Petroleum Chemistry as part of the 187th National Meeting of the American Chemical Society, held in St. Louis, Missouri, 9-11 April 1984.

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I. INTRODUCTION

Cokes and manufactured graphites are unique among structural materials, because the liquid crystalline (mesophase) state plays the dominant role in the formation of their microstructures. The lamelliform morphologies of the discotic nematic liquid crystal¹ are locked into place as the carbonaceous mesophase hardens, and the microconstituents thus produced are fundamentally different from those of conventional polycrystalline metals or ceramics; e.g., mesophase-based materials lack grain boundaries, and disclinations are prominent features in their microstructures. Most mesophase products of practical importance form while under deforming stresses, and the microstructures trapped by hardening are often in deformed nonequilibrium states that would relax if hardening had not intervened. Structurally, therefore, most graphite materials may be regarded as heterogeneous assemblies of mesophase fossils with nonequilibrium microstructures.

In the previous symposium, we reviewed mesophase mechanisms involved in the formation of petroleum coke,² whose microstructures are sufficiently coarse for resolution by polarized-light micrography. Two significant developments since 1975 are the application of hot-stage microscopy to observe the dynamic behavior of the carbonaceous mesophase in its fluid state,^{3,4} and the emergence of carbon fibers spun from mesophase pitch^{5,6} as effective competitors in applications for which high elastic modulus or good graphiticity are important. This report reviews information obtained by hot-stage techniques, particularly on disclination reactions and the response of mesophase to deformation, and then considers the deformed and highly oriented lamelliform morphologies in mesophase carbon fibers.

The micrographic study of mesophase fiber is more difficult than that of coke because the extensive deformation involved in fiber-spinning reduces the disclination structures to scales beyond resolution by optical techniques. However, electron micrographic techniques now being applied to the study of carbon fibers^{7,8} indicate that the same basic mesophase morphologies are present, leading us to speculate on the disclination structures in mesophase

carbon fibers. Micrographic evidence for the disclination structures in the carbonaceous mesophase as well as in coke and graphite has been the subject of a recent review.¹

II. THE CARBONACEOUS MESOPHASE

The carbonaceous mesophase usually appears in the pyrolysis of tars and pitches at about 400°C, where aromatic polymerization reactions are active in producing molecules with weights of 800 mol wt or more. To identify the types of molecules most conducive to the formation of needle coke or the spinning of mesophase fiber, numerous investigations are being conducted to characterize the molecular structures. Japanese workers have been particularly active in this work,⁹ and Fig. 1 is a sketch of the molecular architecture based on the results obtained by Mochida et al.¹⁰

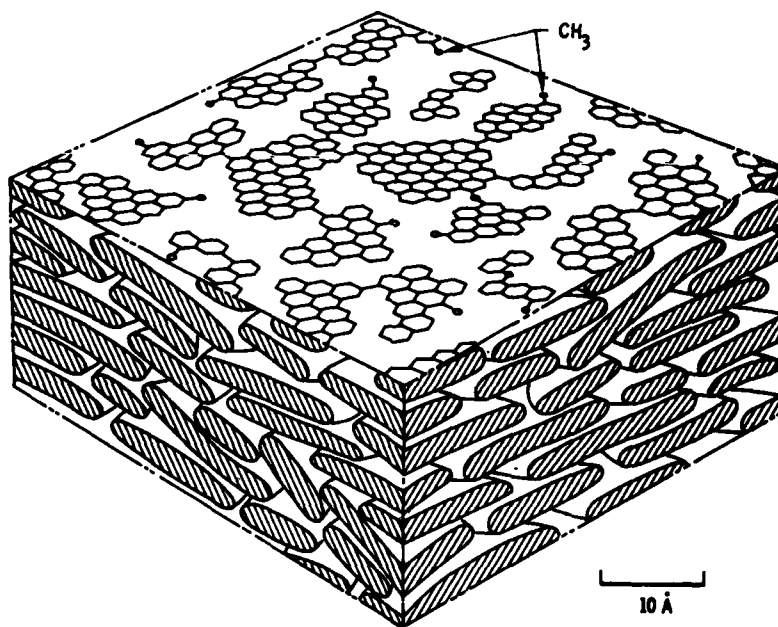


Figure 1. Schematic Model of the Carbonaceous Mesophase; after Mochida et al.¹⁰

In contrast to conventional nematic liquid crystals, the basic molecular units of the carbonaceous mesophase are disk shaped and range widely in size, even when the mesophase is produced by pyrolysis of pure compounds.¹¹ The molecules are usually reactive in the temperature range at which the mesophase

is still fluid, so that polymerization reactions continue to evolve gaseous products and the mesophase usually undergoes deformation by bubble percolation before it congeals to a solid semi-coke.

The disk-shaped molecules are not rigorously oriented to parallel arrays; the mesophase state represents a preferred orientation, with the molecular layers generally lying within $\pm 15^\circ$ of the director representing the average orientation. Thus, the mesophase tolerates easily the bend, twist, and splay involved in the formation of disclinations, and both wedge and twist disclinations are readily identified in petroleum coke by polarized-light microscopy. Figure 2 illustrates why disclinations do not occur commonly in ordinary crystalline materials. The distortions at the core of a crystal

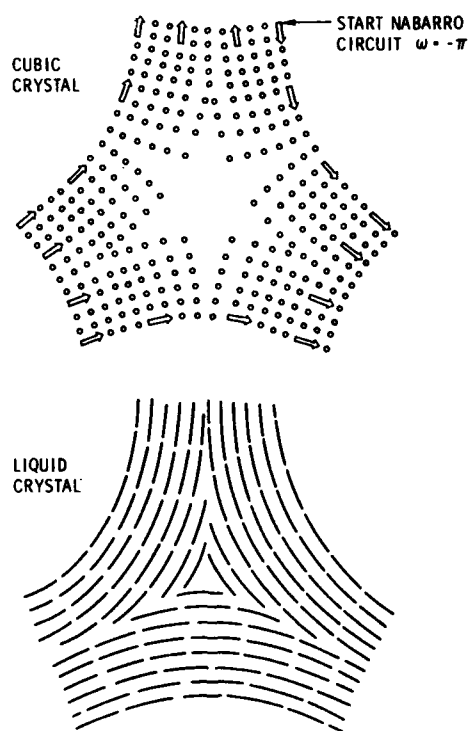


Figure 2. Schematic Structures of Wedge Disclinations (Rotational Strength = $-\pi$) in a Cubic Crystal and a Liquid Crystal

disclination involve energies so large as to prohibit the formation of disclinations except by entrapment mechanisms such as the hardening of a liquid crystal. Figure 2 also illustrates how a Nabarro circuit (analogous to a Burgers circuit for a crystal dislocation) can be followed to define the rotational strength of a disclination.

Models of the wedge and twist disclinations commonly observed in the carbonaceous mesophase are diagrammed in Fig. 3. Their appearance in the fibrous morphology of needle coke is depicted in Fig. 4; here the disclinations are primarily wedgelike in character because the mesophase layers, and the disclination lines and their rotation vectors, all tend to align with the axis of the needle-coke particle.

The flow or deformation characteristics of the mesophase are a fundamental part of mesophase-fiber spinning and needle-coke formation; however, published results of deformation studies have advanced little beyond stating such elementary points as that uniaxial deformation produces fibrous morphologies (exemplified by Fig. 4), whereas biaxial deformation produces lamellar morphologies.² Viscosity measurements are now appearing in the literature,¹²⁻¹⁴ but the studies to date tend to focus on partially transformed mesophase pitches for which the measurements are less complicated by

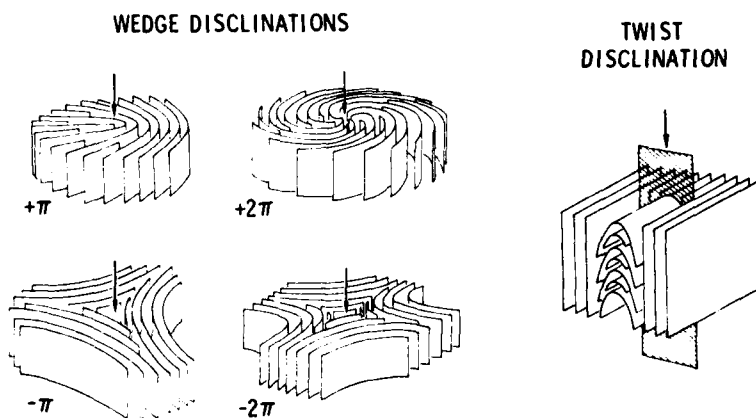


Figure 3. Schematic Models of Wedge and Twist Disclinations in the Carbonaceous Mesophase

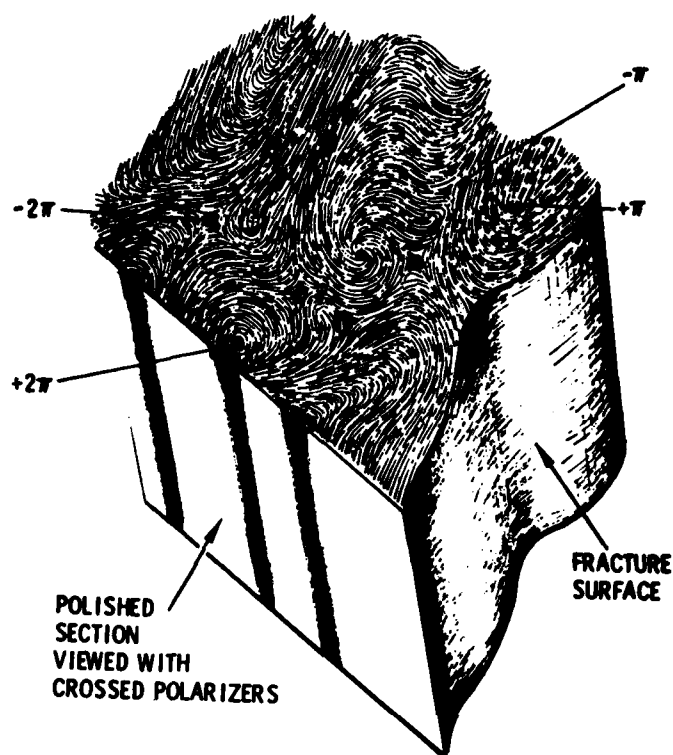


Figure 4. The Fibrous Morphology of an Acicular Region of a Needle-coke Particle. Four wedge disclinations are indicated in terms of their rotational strength.

ongoing pyrolysis reactions and bubble percolation. It is noteworthy that the mesophase pitches studied by capillary rheometry usually manifest viscoelastic behavior by die swell, i.e., an increase in diameter after passage through the rheometer capillary¹⁴—a further indication of the unstable microstructures produced by mechanical deformation. Despite the lack of basic studies of mesophase hardening, practical observations with industrial rheometers (see Fig. 5) show that the mesophase hardens by a progressive increase in viscosity as aromatic polymerization is driven by continued pyrolysis.¹⁵

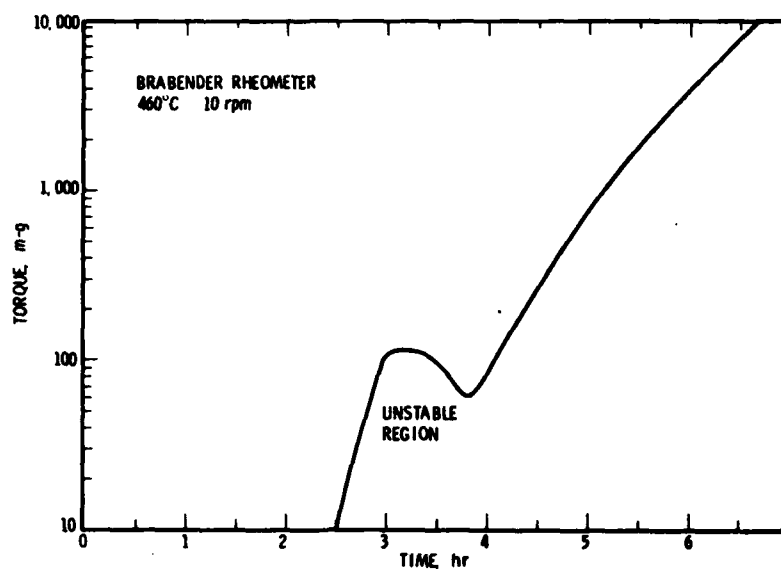


Figure 5. The Increase in Mesophase Viscosity during Pyrolysis within a Rheometer. A240 petroleum pitch.

III. HOT-STAGE OBSERVATIONS

Since Hoover et al.³ demonstrated that polarized light reflected from the free surface of a pyrolyzing liquid could be used to directly observe mesophase behavior, hot-stage techniques have been applied to study the pyrolysis of a number of coke precursors. Provided that excessive volatilization does not interfere by fogging the observation window or by forming a viscous mesophase skin,¹⁶ such methods enable qualitative evaluation of the mesophase's dynamic behavior.

To relate free-surface observations to the three-dimensional morphology of bulk mesophase, we constructed a hot-stage with quenching capability. With it, a specimen can be pyrolyzed to a point of interest observed on the free surface, then quenched to a solidified body that can be sectioned for detailed micrographic study.¹⁸ The hot-stage (Fig. 6) was designed with a simple probe to permit deformation of the mesophase at various stages of pyrolysis. A 32 $\times$ objective with 6-mm working distance was adequate to resolve orientational fluctuations similar to those observed in nematic liquid crystals.¹⁷ The optical system remained stable during quenching; fine cracks could be seen to develop after the temperature dropped below the softening point of the mesophase.

Petroleum pitch (Ashland A240) was well suited to hot-stage microscopy, particularly after it was thermally treated to reduce evolution of volatiles. As long as the mesophase was quite fluid, the coalescence behavior and disclination reactions were similar to those of conventional nematic liquid crystals.¹⁷

The micrographic sequence of Fig. 7 illustrates five disclination reactions observed within 2 min. In region A, two 2π disclinations of opposite sign appear to be spontaneously generated by a "pinch-off" reaction; those disclinations then separate, with the right-hand disclination moving toward region B, where it annihilates another 2π disclination. In region C the reaction is $(+\pi) + (-2\pi) \rightarrow (-\pi)$; the disclination signs were identified by rotating the plane of polarization. The various disclination reactions have

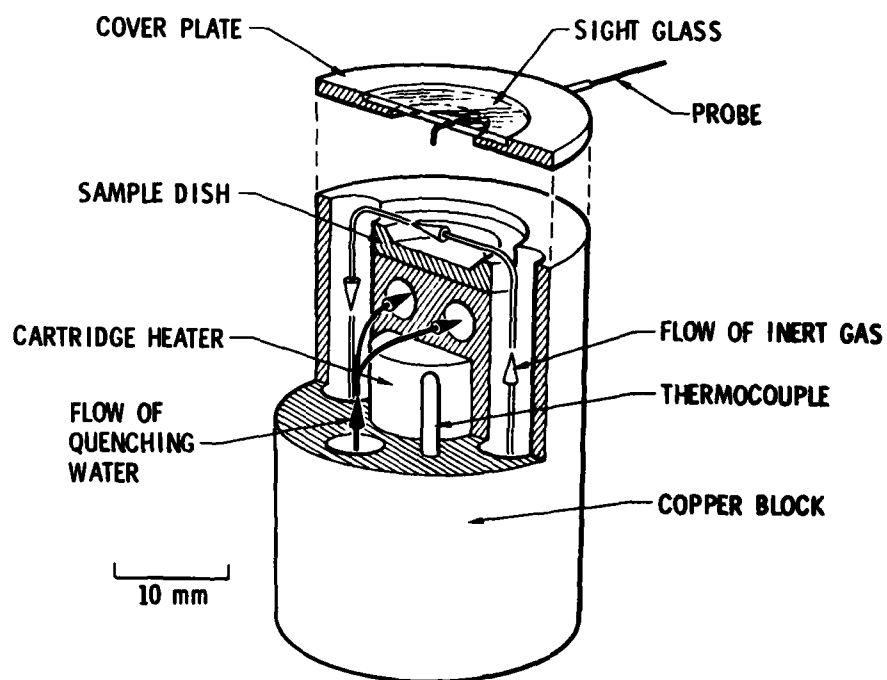


Figure 6. A Microscope Hot-Stage Designed for Quenching and for Deformation of a Liquid Specimen by a Horizontal Probe



Figure 7. Disclination Interactions Observed by Crossed Polarizers. A, generation; B, annihilation; C, reaction $(+\pi) + (-2\pi) = (-\pi)$.

been observed to take place in both forward and reverse directions,¹⁸ suggesting that the energies of disclination structures are small relative to the work of deformation by mechanisms like bubble percolation. Detailed study of the pinch-off reaction revealed that the freshly formed disclinations can have nonequilibrium structures¹⁸ that represent substantial distortions of the relaxed structures shown in Fig. 3. As pyrolysis is continued, the disclination reactions slow well before the mesophase loses its deformability.

Mesophase specimens were deformed with the hot-stage probe to form fibrous microstructures, and observations of structural coarsening have been made on the free surface.¹⁹ Vertical sections of quenched specimens (Fig. 8) exhibit arrays of wedge disclinations. At higher levels of pyrolysis, the extent of recovery decreases, leaving larger residual densities of disclinations in the hardening mesophase. The mesophase's ability to be deformed well beyond the pyrolysis condition at which disclinations can interact accounts for the dense arrays of disclinations in products fabricated from the carbonaceous mesophase.

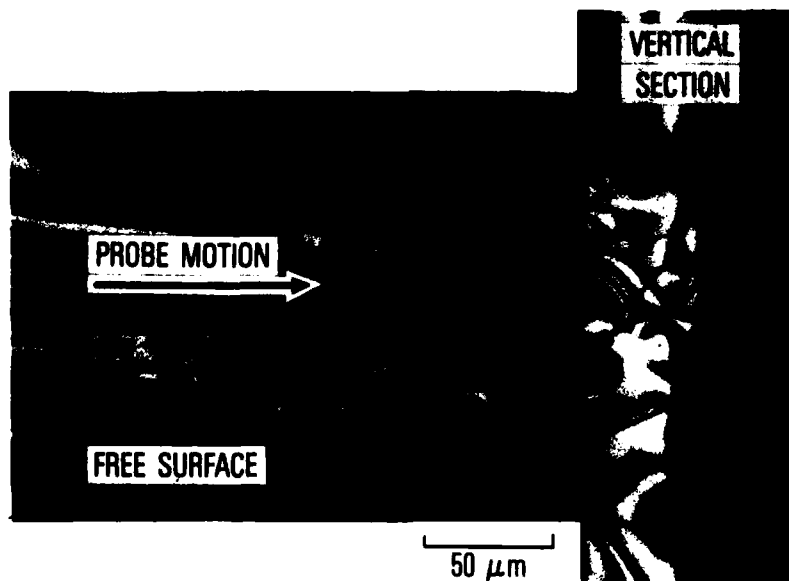


Figure 8. Mesophase Deformed by Horizontal Probe Motion, Observed by Crossed Polarizers on Free Surface and on Vertical Section

IV. CARBON FIBER SPUN FROM MESOPHASE PITCH

The schematic of Fig. 9 illustrates the spinning of a mesophase monofilament according to the Singer patent.⁵ After extrusion through the spinnerette hole, extensive draw-down imparts a strong preferred orientation to the mesophase layers. This morphology is "frozen" into the filament by rapid cooling and then stabilized by a controlled oxidation treatment. The fiber can then be heat-treated to 1500°C or higher to attain high elastic modulus. Fibers with tensile moduli from 25 to 100 Mpsi (170 to 700 GPa) have been developed commercially,²⁰ and fibers with moduli in excess of 120 Mpsi (800 GPa) have been produced experimentally.

Typical fracture surfaces from the three structural types commonly found in commercial mesophase carbon fiber²¹ are depicted in Fig. 10. They may be distinguished by their shape as open-wedge, round, and oval filaments. Detailed micrographic studies using fractured surfaces and oxidation etching⁸ reveal that the graphitic layers are extensively wrinkled, with the wrinkles running parallel to the filament axis, and that the layers tend to stand in radial arrays in the outer rim. Disclination models for the internal structures of the three types of filament²¹ are sketched in Fig. 11.

Analysis of the flow dynamics of an anisotropic liquid may be required to explain the radial orientation of mesophase layers in the outer rim; however, the hot-stage observations provide a reasonable rationale for the core structures of the mesophase carbon fibers. Figure 12 shows how disclination loops that enter the spinnerette channel can be converted by uniaxial deformation in the draw-down region to a pair of closely spaced parallel wedge disclinations of opposite sign.²² An array of such disclinations must be expected to interact to varying degrees of completion according to the viscosity of the mesophase and the rate of cooling of the filament. If fiber is spun from fluid mesophase and the cooling conditions permit the disclination reactions to run to completion, a round and totally radial filament is expected; upon heat treatment, the open wedge develops because the mesophase shrinks more perpendicular than parallel to the layers. If fiber is spun from viscous

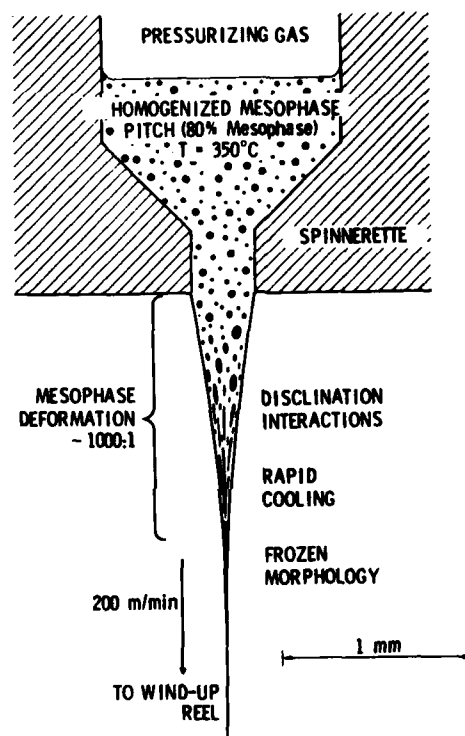


Figure 9. Schematic of the Spinning of a Monofilament from a Homogenized Mesophase Pitch



Figure 10. Fracture Surfaces for Three Types of Mesophase Carbon Fibers Tested in Tension

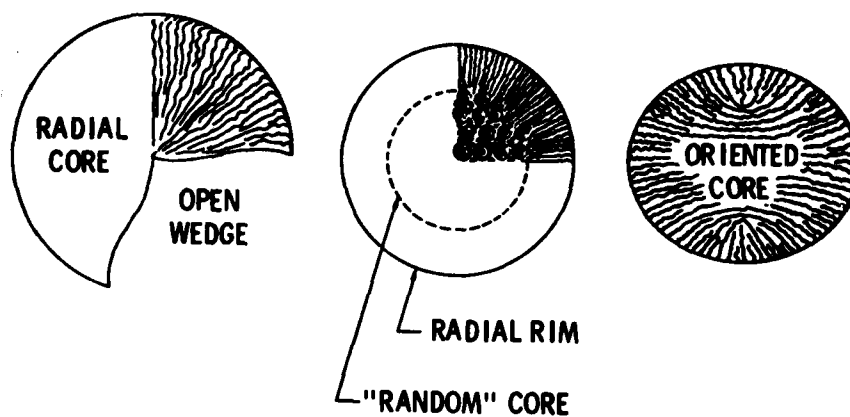


Figure 11. Structural Models for the Morphology of Open-wedge, Round, and Oval Filaments Spun from Mesophase Pitch

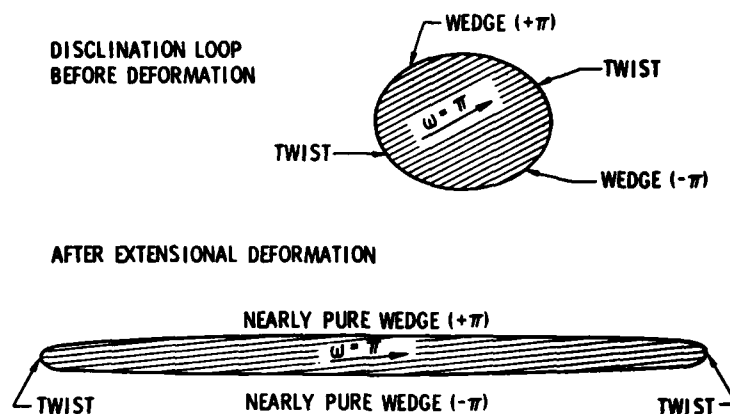


Figure 12. Schematic of the Uniaxial Deformation of a Disclination Loop

mesophase with rapid cooling after spinning, the disclination reactions may be interrupted, leaving the random core structure with the round filament shape. The oval fiber, then, represents an intermediate state of disclination reaction in which just two $+\pi$ wedge disclinations remain with an oriented core; the oval shape results from the anisotropic shrinkage of the oriented core upon heat treatment.

On the basis of the preceding explanation, the morphologies of mesophase fibers are closely related to those of needle coke (cf., Fig. 4). The principal differences appear to lie in the extents to which the deformation and relaxation mechanisms are able to act. Spinning involves large draw ratios that produce dense arrays of disclinations, whereas the long dwell times in the delayed coker afford opportunities for extensive disclination annihilation and more complete microstructural relaxation. By comparison, the cooling after fiber-spinning constitutes a rapid quench, and the disclination structures and partially reacted arrays may deviate appreciably from the equilibrium structures of Fig. 3 and the needle-coke disclination arrays of Fig. 4.

V. CONCLUSIONS

Hot-stage observations of mesophase behavior provide insights into structure. From those insights, we have established patterns for the formation of the morphology of deformed mesophase products, e.g., needle coke and carbon fibers spun from mesophase pitch. The critical mechanisms are (1) the creation of strong preferred orientations by deformation, (2) the interactions of disclinations brought into proximity by the deformation process, and (3) the retention of the oriented and disclinated microstructures by cooling or by chemical reaction. The observations also indicate that, at least for the fluid mesophase state, the highly oriented fibrous and lamellar morphologies are not thermodynamically stable, and that relaxation to less oriented, less distorted, and less disclinated structures will occur if the viscosity is sufficiently low. The extents to which the disclination-reaction mechanisms might apply to heat-treated cokes, graphites, and carbon fibers present interesting areas for investigation.

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