

A microscopic view of fiber-reinforced composites, showing a matrix of light blue and yellowish fibers with several large, reddish, spherical inclusions. The fibers are oriented in various directions, some parallel to the top and bottom edges of the image.

Optical Microscopy of Fiber-Reinforced Composites

Brian S. Hayes
Luther M. Gammon



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and
Luther M. Gammon**



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Preface

This book is designed as an instructional reference for preparing fiber-reinforced polymeric-matrix composite materials for examination by optical microscopy and the techniques of optical microscopy used for analysis. It is also meant to be a teaching tool for those who want to learn more about the microstructure of these heterogeneous and anisotropic materials. The content is also appropriate for experienced microscopists or metallurgists who become involved in the preparation and analysis of polymeric composites. This book begins with an introduction to fiber-reinforced polymer-matrix composite materials that focuses on the microstructure and morphology of these unique materials. In the following chapters, the authors explain the materials, equipment, and procedures of how to prepare composite samples, followed by the illumination and contrast techniques of optical microscopy. Included in these chapters are the methods and reagents that are used to bring out distinct features in composite materials, such as different phases and areas of degradation or damage. Also included are details of how to prepare special composite materials having vast differences in hardness and material properties. The remaining chapters present various topics of studies of fiber-reinforced polymeric composite materials that have been performed by using optical microscopy.

These studies include a majority of the microstructural information that is of primary interest when working with composite materials. Insight into processing effects, toughening approaches, damage mechanisms, and environmental effects on the microstructure of composite materials is presented. Through these chapters and the micrographs throughout this book, it will be evident that optical microscopy is one of the most valuable tools for analyzing fiber-reinforced polymeric-matrix composites. Unfortunately, with all of the nonoptical methods of analysis available today, optical microscopy is often overlooked or not used to the fullest extent. The contents of this book were developed after many years of the authors presenting on this subject and teaching short courses about optical microscopy of composite materials. It is hoped that this text is found useful to everyone who desires to further increase their knowledge of optical microscopy and the microstructure of fiber-reinforced polymeric composite materials.

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CHAPTER **1**

Introduction— Composite Materials and Optical Microscopy

Composite Materials

The unique and diverse characteristics of composite materials have caused an increase in their utilization worldwide. From featherweight fly fishing rods to high-performance airplane parts, the use of fiber-reinforced composite materials is becoming more popular due to their high strength-to-weight ratio combined with easy manufacturing methods. Fiber-reinforced polymeric-matrix composites consist of reinforcing fibers and a polymer resin. The fibers are considered as the principal load-carrying constituent of the composite, while the role of the polymer matrix is to transfer the load between fibers as well as provide corrosion resistance, damage tolerance, and thermal and environmental stability (Ref 1). Fiber-reinforced polymeric composites are developed from thermoplastic or thermoset resins combined with either discontinuous or continuous unidirectional fibers or woven fabrics (Fig. 1.1a to 1.1c). Typical reinforcements consist of glass, carbon, or aramid fibers, but other materials, such as boron, ceramic, and thermoplastic fibers, may also be used for specific applications (Fig. 1.2a to 1.2c). Some fiber types may be the same chemical makeup and have similar mechanical properties but are vastly different in structure, depending on the manufacturer (Fig. 1.3a, b).

Many methods are used to manufacture fiber-reinforced composites, including hand lay-up of prepreg materials, automated tape lay-up of prepreg materials, resin transfer molding, vacuum-assisted resin transfer molding, resin film infusion, wet lay-up, filament winding, pultrusion, and compression molding of sheet molding or bulk molding compound. While

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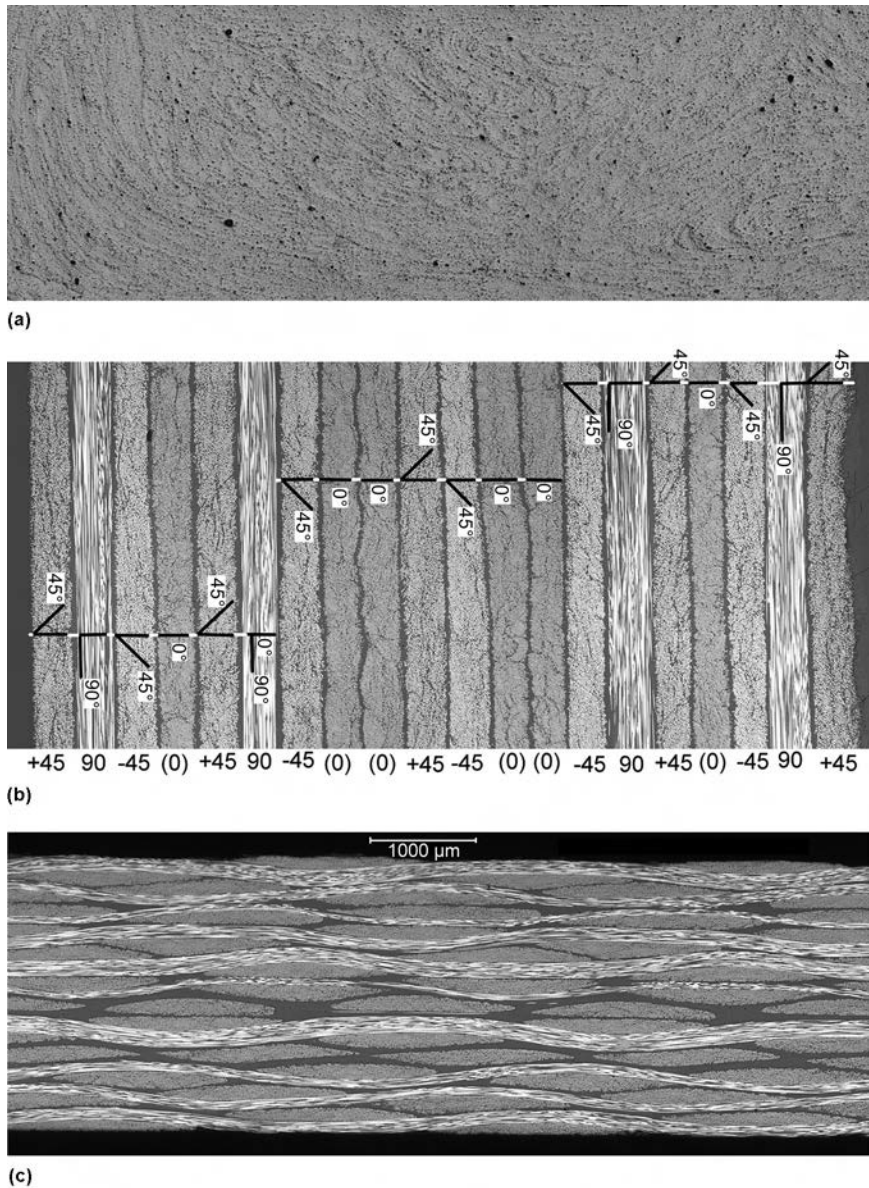


Fig. 1.1 Composite cross sections. (a) Sheet molding compound made from carbon-black-filled epoxy resin and chopped glass fiber. Bright-field illumination, 65 mm macrophotograph montage. (b) Quasi-isotropic unidirectional prepreg laminate. Slightly uncrossed polarized light, 5 \times objective, montage. (c) 3k-70 woven carbon fabric laminate. Bright-field illumination, 5 \times objective, montage

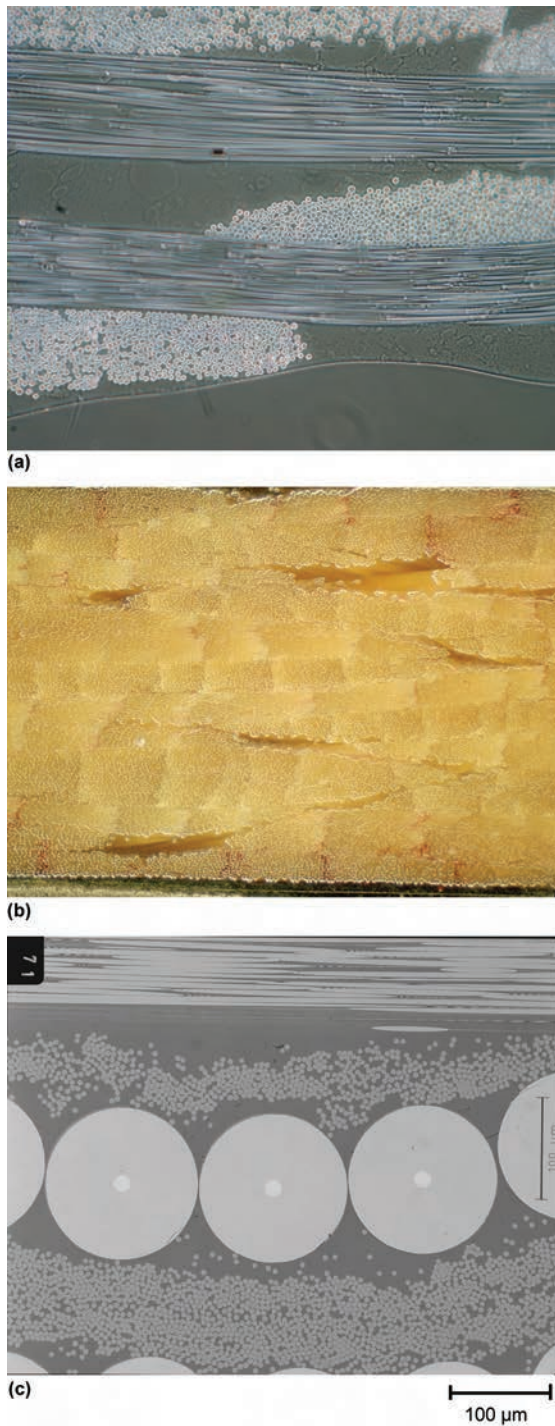


Fig. 1.2 Composite materials made from different types of fibers. (a) Woven glass fiber fabric composite revealing a multiphase-matrix morphology. Ultrathin section, transmitted-light phase contrast, 20× objective. (b) Kevlar (E.I. du Pont de Nemours and Company) fabric composite cross section. Dark-field illumination, 25× objective. (c) Boron fiber polymeric-matrix composite cross section. Bright-field illumination, 50× objective (200× original magnification)

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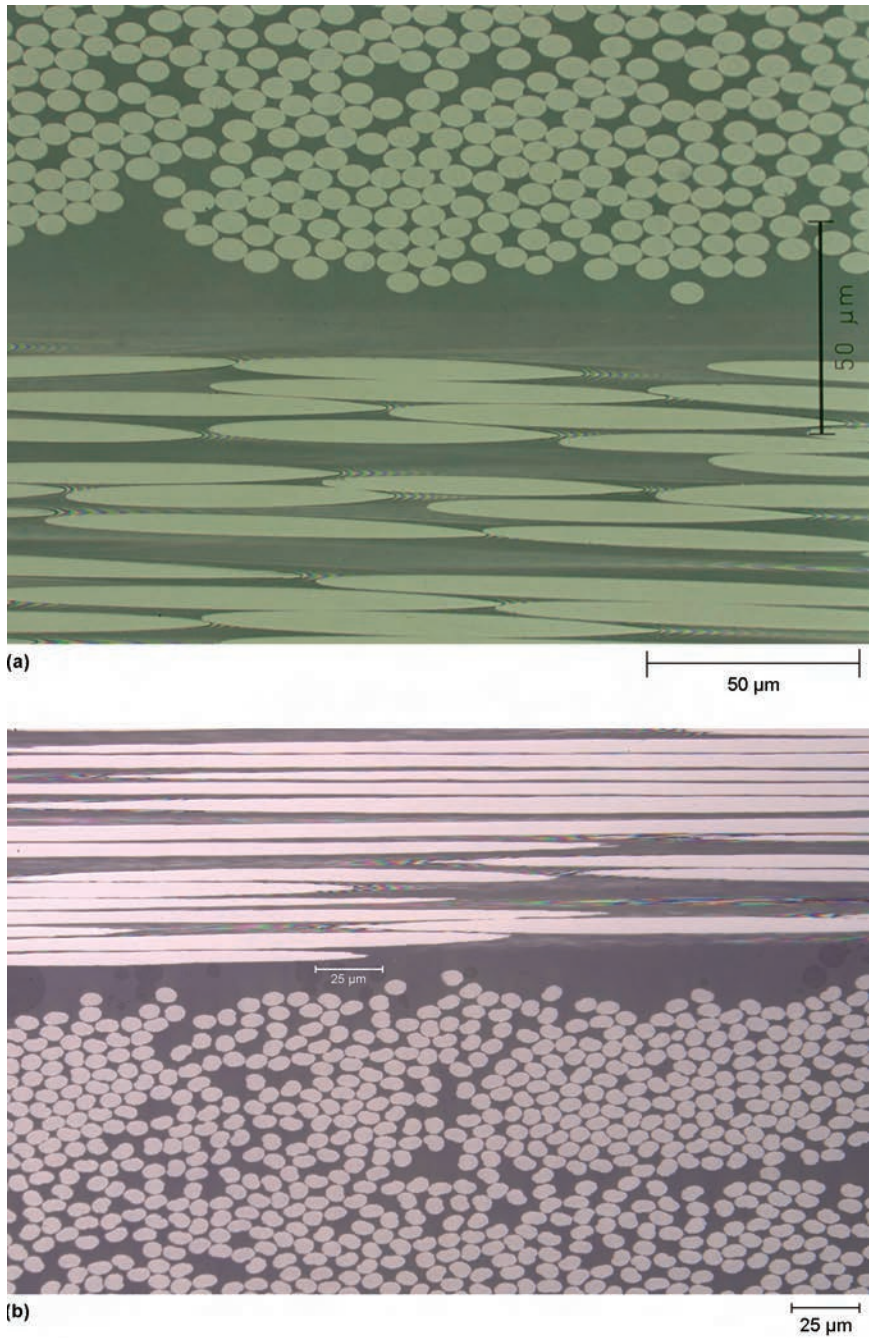


Fig. 1.3 Unidirectional carbon fiber composite cross sections displaying carbon fiber types of similar strength and modulus but differing in fiber shape. (a) Cylindrical carbon fiber shape. Bright-field illumination, 50× objective. (b) Irregular bean-shaped fibers. Bright-field illumination, 25× objective

these processes are general in description, the actual process of combining the fibers and matrix resin is unique and can be different with each resin-fiber system and between manufacturers. The processes described and the manufacturing parameters can lead to differences in morphology of the uncured and cured composite structures.

Polymer Matrices

Polymer matrices used for composite materials can be generally described as either thermoplastics or thermosets. Thermosets are polymer resins that crosslink and form a three-dimensional structure when cured. Once cured, the network structure is irreversible and cannot be reshaped or made to flow below its decomposition temperature (Ref 2). In contrast, thermoplastics, which consist of high-molecular-weight linear or branched polymer chains (not crosslinked), can be reshaped with the application of heat and pressure (Ref 2). In relation to composite materials, the distinction between these types of matrices is that they are reactive (thermosets) and nonreactive (thermoplastics) polymers. Most thermoplastic-matrix composites are developed with their polymerization complete. As a result, thermoplastic fiber-reinforced composites are generally more difficult to produce due to high viscosity resulting from the high-molecular-weight polymer chains. These materials usually require high temperatures, pressures, or the use of solvents for processing that must be removed after manufacturing. An added complexity of processing thermoplastics exists in the ability for some thermoplastics to form a semicrystalline structure (Fig. 1.4a to 1.4c) (Ref 3). This is very important to realize, if the material used is amorphous or semicrystalline, because the cooling and heating rates can affect the crystallinity of the matrix and hence the final composite properties (Ref 4). Current commercial high-performance composite matrices may contain an engineering thermoplastic in combination with a thermoset, thereby taking advantage of the different properties.

There are many types of thermosetting and thermoplastic materials that are used as matrices in fiber-reinforced composites. The selection of the matrix material and fiber type is dependent on the physical and mechanical properties that are required for the designed part. Thermosetting matrices commonly used in composite materials include polyesters, epoxy vinyl esters, epoxies, cyanate esters, bismaleimides, and other higher-temperature resins. Some common thermoplastic matrices used in composites include polyethylene terephthalate, polyamides (nylon), polyetheretherketone, and polyphenylene sulfide.

Prepreg Materials

Prepregs are the most widely used materials for manufacturing high-performance composites (Fig. 1.5). The manufacturing of prepreg is usu-

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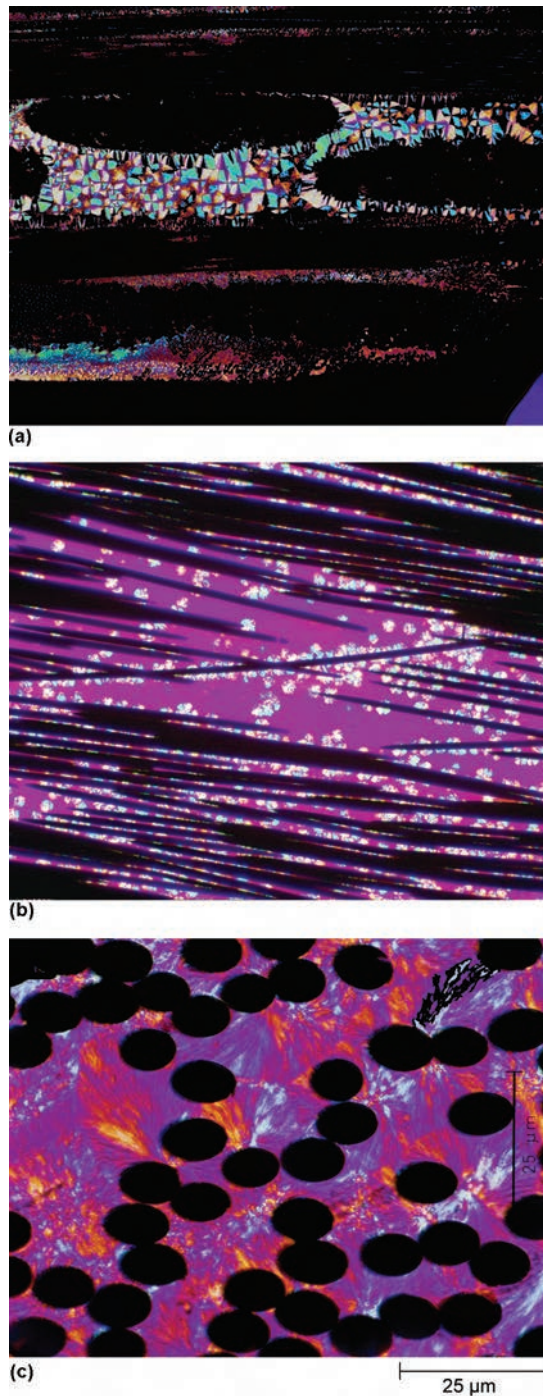


Fig. 1.4 Crystallinity in thermoplastic-matrix carbon fiber composites. (a) Crystalline region in the center area of a woven carbon fabric composite cross section. Ultrathin section, transmitted polarized light with a full wave plate (540 nm), 20× objective. (b) Fiber-induced spherulite growth. Ultrathin section, transmitted polarized light with a full wave plate (540 nm), 20× objective. (c) Large spherulitic growth in a carbon fiber composite. Ultrathin section, transmitted polarized light with a full wave plate (540 nm), 100× objective

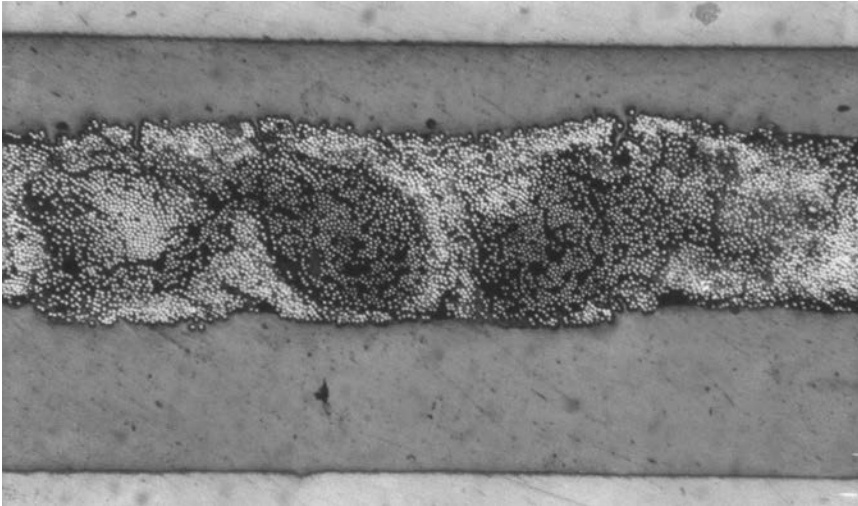


Fig. 1.5 Cross section of uncured prepreg material showing unimpregnated areas (dark) and impregnated areas (gray). Bright-field illumination, 25 \times objective

ally accomplished through either solvent or hot-melt impregnation of the matrix resin into the continuous unidirectional or woven fiber fabric materials (Ref 5, 6). These pre-engineered laminating materials have a discrete resin/fiber ratio that requires further lay-up of the continuous fiber plies to achieve the final composite. The prepreg lay-up is subjected to elevated temperature and pressure to cure and consolidate the part. The method of pressure application is dependent on the part and configuration, but the use of an autoclave is most common for high-performance parts.

The physical difference between thermosetting and thermoplastic polymers imparts an extreme differentiation in the handling characteristics of prepreg materials in the form of tack and drape. Tack can be defined as the ability of prepreg plies to stick together, while drape is the ability of the prepreg to conform to different contours (Ref 7). The high-viscosity characteristic of thermoplastic-matrix prepreps provides limited tack-and-drape capabilities. While this limits the use of thermoplastic materials for some applications, the inherent toughness of these materials is superior in most cases when compared to unmodified thermosets. This desirable aspect, along with short manufacturing times, has provided an increasing market for these materials. However, the compaction pressure and temperature required for consolidation of thermoplastic prepreps are usually substantially higher than what is typically used for thermosetting-matrix prepreg materials. Thermoset prepreg materials are attractive because they have desirable handling characteristics and a wide range of properties. If the tack of thermoset prepreg material is too high, it may enable greater air entrapment between the prepreg plies. Entrapped air in composites that is

not removed during manufacturing can lead to voids in the cured composite (Fig. 1.6). Also, care must be taken to remove water and solvent contained in the matrix materials and prepregs, which may cause voids in the cured composite, resulting from an increase in the solvent vapor pressure with cure temperature (Fig. 1.7) (Ref 8 to 10).

Infusion Processes

Fiber-reinforced composites can be developed by omitting the intermediate impregnation or “prepreg” step and directly infusing the dry fabric

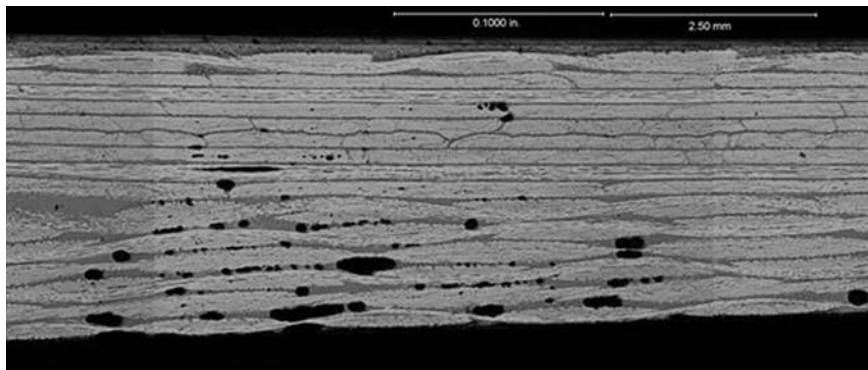


Fig. 1.6 Entrapped air in a composite part made from unidirectional carbon fiber prepreg and woven fabric prepreg. Voids (dark areas) are shown mainly in the interply regions of the cross section. Bright-field illumination, 65 mm macrophotograph montage

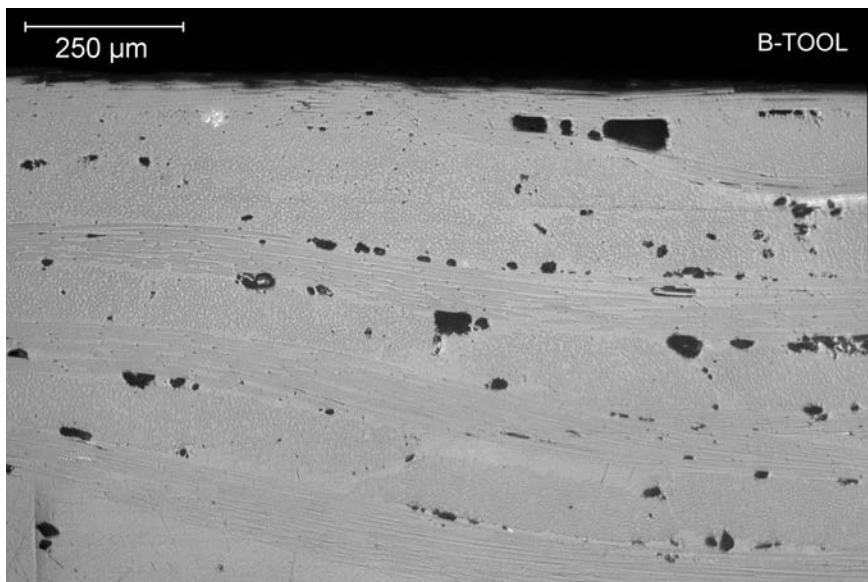


Fig. 1.7 Voids found in a glass fiber composite cross section due to solvents from manufacturing. Bright-field illumination, 10× objective

reinforcement or fibrous preform with the matrix resin. Infusion processes are low-cost composite manufacturing methods and are being readily adopted for replacement of some prepreg composite parts. Of the many infusion methods and processes that have been developed, the major techniques are variations of resin transfer molding (RTM), vacuum-assisted resin transfer molding (VARTM), and resin film infusion (RFI). In comparison to prepreg matrices, infusion resins must be homogeneous and single phase before impregnation, or the particles may become filtered by the fibers. This limits the use of some standard prepreg curing agents and modifiers, because many are in particle form (Fig. 1.8). Also, because the

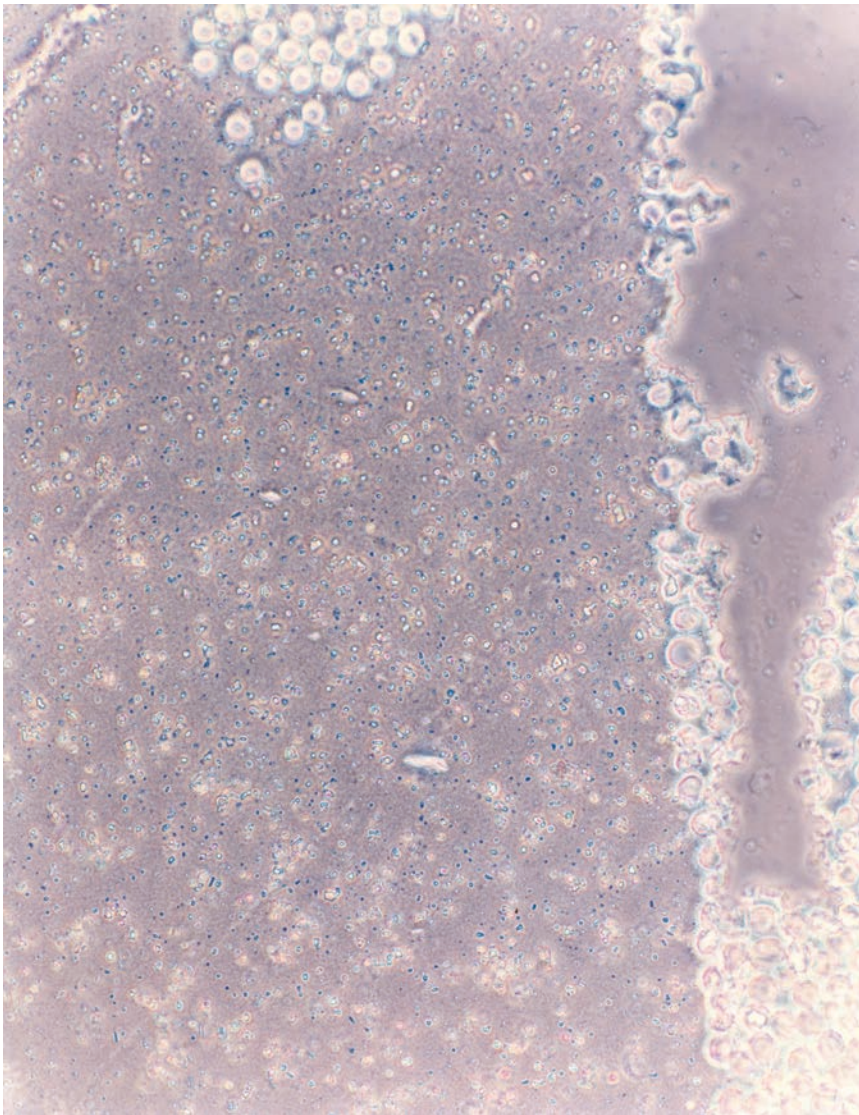


Fig. 1.8 Residual curing agent particles in a thermoset-matrix glass fiber composite. Reflected-light phase contrast, 40× objective

resin is not preimpregnated, many conventional toughness modifiers, such as thermoplastics and high-molecular-weight elastomers, cannot be used in these systems, since the viscosity is increased to an unacceptable level. If the viscosity of these systems is too high, long infusion times may be required, and the resin may gel before complete infusion. This may also result in a thicker part and improper fiber wet-out. The physical difference of the resins used for RFI versus VARTM or RTM is that the resin is usually in sheet or film form. Resins for VARTM or RTM may be one-component systems, containing the resin and catalyst/curing agent combined from the supplier, or, more generally, are two-part systems where the catalyst/curing agent is separate from the resin.

Although it is not defined as an infusion process, hand wet lay-up of continuous fabrics or preforms is very cost-effective and highly controllable if performed correctly. These wet prepreg materials commonly are consolidated under vacuum or compression cured with or without the application of heat to create the final part. Many of the parts that are developed with wet prepreg materials also include wood or foam cores to enhance the stiffness. These types of processes are currently being used for developing many high-performance composites.

Composite- and Matrix-Toughening Methods

The desire to use fiber-reinforced composites in more damage-prone environments has created a need for tougher, more damage-tolerant polymer-matrix composites. One method to increase composite toughness is through the use of more damage-tolerant fibers made from aramid or glass; however, other performance requirements may not allow the use of these materials for the design. Work also continues on the development of stitched fibrous preforms (Fig. 1.9) to improve toughness and provide easier manufacturing. While stitching may work very well for some applications, problems may arise, such as microcracking, due to the differences in the properties of the stitching materials.

Dispersed-Phase Toughening

There are many methods used to increase the toughness of thermosetting resin systems. However, the methods used must be selected so as not to reduce the processing, handling characteristics, or the mechanical and/or physical properties. Extensive research has been conducted over the past several decades involving the modification of high-performance thermosetting resins and fiber-reinforced composites. As a result of this effort, many materials and techniques have been and are currently being developed for improving the toughness of these materials. One of the most widely researched techniques involves the development of a multiphase structure with the use of either rubber or thermoplastic modifiers (Fig.

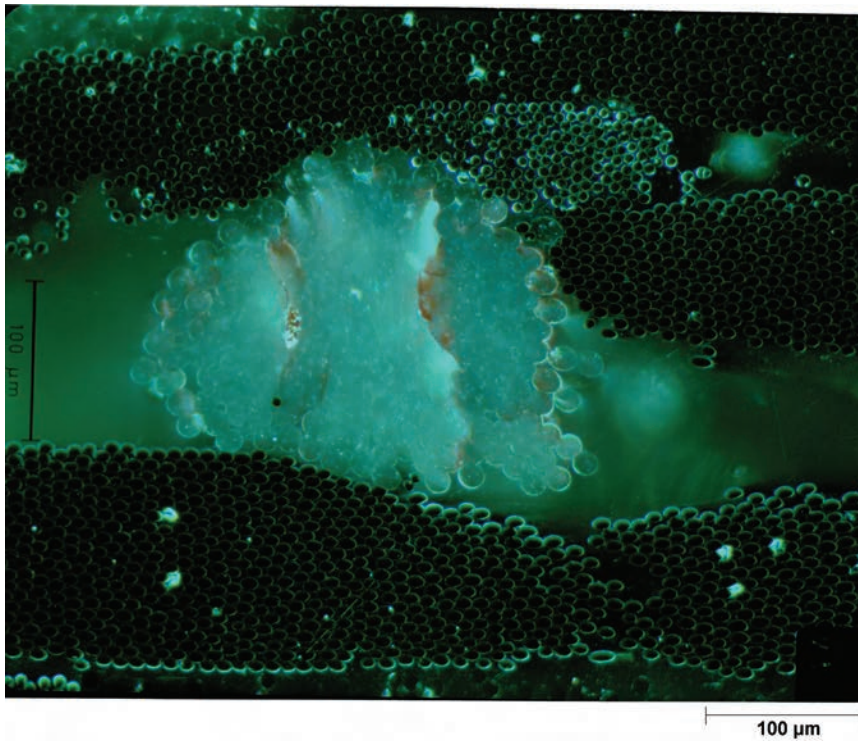
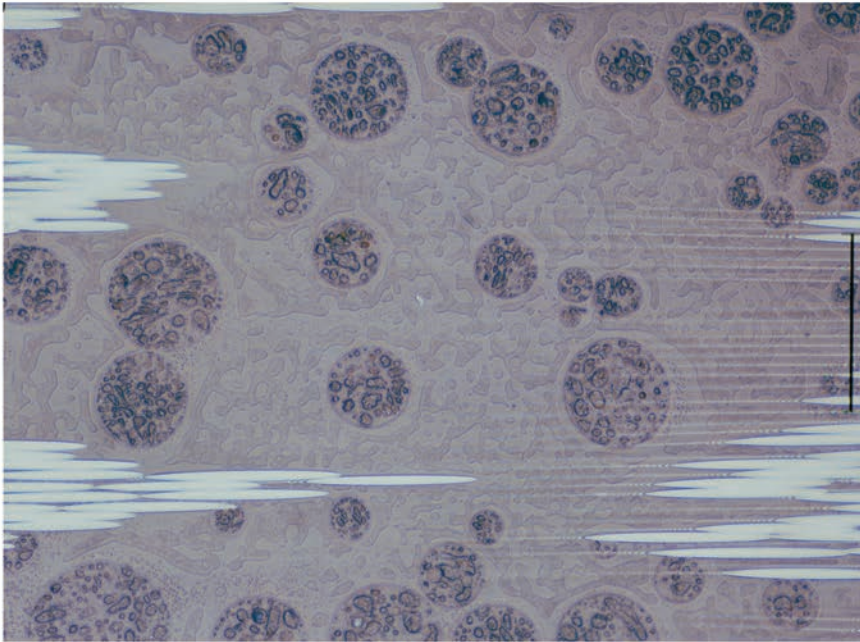


Fig. 1.9 Thermoplastic stitch in carbon fiber composite material. Note the microcracks in the center of the stitch. Epi-fluorescence, 390–440 nm excitation, 25× objective

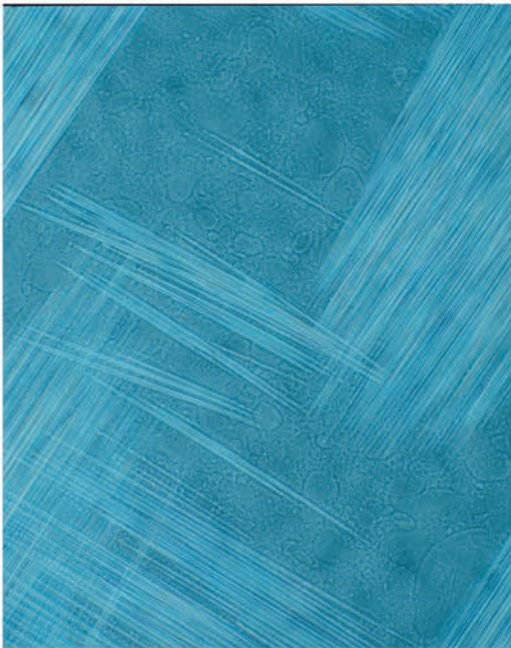
1.10a to 1.10c) (Ref 11, 12). The most common rubber materials used for toughening thermoset matrices are based on butadiene-acrylonitrile and are often functionalized for co-reacting with the thermoset matrix (Ref 13, 14). Thermoplastic materials have also found significant use as modifiers for thermosetting-matrix resins. Engineering thermoplastics that commonly are used in high-performance prepreg matrices include polyether-sulfone and polyetherimide (Ref 15 to 17). Some other thermoplastics that have been used for modifying epoxy matrices include polyvinyl formal, polyvinyl butyral, and polyether-blockamides (Ref 18). As with the rubber modifiers, phase separation is a complex function of the matrix formulation and processing conditions. The dispersed phases for these particles generally range from 0.3 to 10 μm and may be spherical or have an irregular shape. In some cases, the modifier may be designed to remain in the continuous phase of the resin as opposed to phase separation.

There are essentially two methods used to generate a multiphase morphology within a thermosetting resin, with a third method leading to true nanostructured phase separation. The first method involves the formation of an initially soluble solution of the modifier and uncured thermoset in which phase separation of the modifier occurs upon cure, forming distinct domains (Ref 19). The second technique involves the addition or forma-

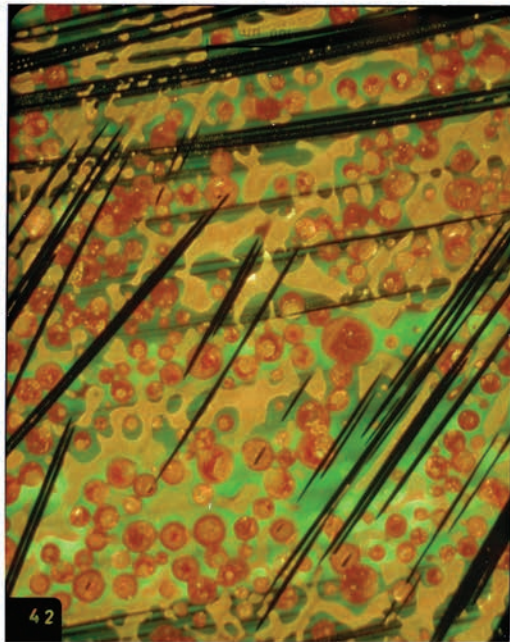
12 / Optical Microscopy of Fiber-Reinforced Composites



(a)



(b)



(c)

Fig. 1.10 Multiphase thermosetting-matrix composites. (a) Carbon fiber composite cross section. Etched and viewed using reflected-light phase contrast, 25× objective. (b) Glass fiber composite ultrathin section. Transmitted light, 20× objective. (c) Carbon fiber composite cross section having a multiphase-matrix structure. Rhodamine B dyed. Epi-fluorescence, 390–440 nm excitation, 25× objective

tion of preformed particles in the uncured resin that are initially distinct particles and remain so after cure (Ref 20). These materials, often core-shell modifiers, have a matrix-compatible shell with a rubber or thermoplastic core. Common particle sizes range from 50 nm to 3 μm . Dispersed nanostructured phases in thermosetting resins have been developed using block copolymers in which phase sizes less than 100 nm have been found in many resin systems (Ref 21 to 23). Due to the resolution of optical microscopy, these nanomorphologies cannot be observed, because these phases are smaller than the wavelength of light. Therefore, nanometer-scale phases or inclusions are best viewed using a technique such as transmission electron microscopy.

Interlayer-Toughened Composites

The need to improve high-performance thermosetting composite toughness further than is possible through chemistry has resulted in an engineering approach to toughen composite materials (Fig. 1.11a, b). Without improving the toughness of the resin itself, the addition of a modified interlayer can impose large increases in final composite damage tolerance (Ref 24). This modified interlayer reduces the interply delamination, which has been found to be the life-limiting factor in most composites (Ref 25, 26). To date, the most efficient and highest damage-tolerant composites arise from the use of interlayer-toughening concepts. Interlayer toughening comprises the placement of a thin toughened resin between each ply of the composite structure. The first interlayer-modified composites consisted of two resins of different composition, with the interlayer resin being much tougher than the resin used in the intraply region (3M patent) (Ref 27). Materials frequently used in the interlayer were thin thermoplastic sheets. However, prepreg handling problems, including tack and drape as well as hot-wet performance, limited the use of these types of composite systems. As a result, the present state of the art-toughened resins that were developed for the interlayer consisted of the same or similar base resin but were toughened using preformed thermoplastic or rubber particles (Fig. 1.12) (Ref 20, 28 to 30). Particles used in these applications have ranged in size from 1 to 100 μm but usually have averaged between 20 and 50 μm , so that the particles remain in the composite interlayer and do not penetrate the fiber tows (Ref 20). The most common method of interlayer toughening is through adding preformed particle-modified resin on the surface of the prepreg, which, when consolidated and cured, results in an interlayer-toughened composite. However, this same method of toughening has recently been extended to infusion-type processing, wherein the fabric plies are sprayed or coated with a tackifier containing preformed particles (Ref 31). When the resin is infused, the preformed particles that were placed on the fabric surfaces are “locked in” and produce an interlayer-modified composite.

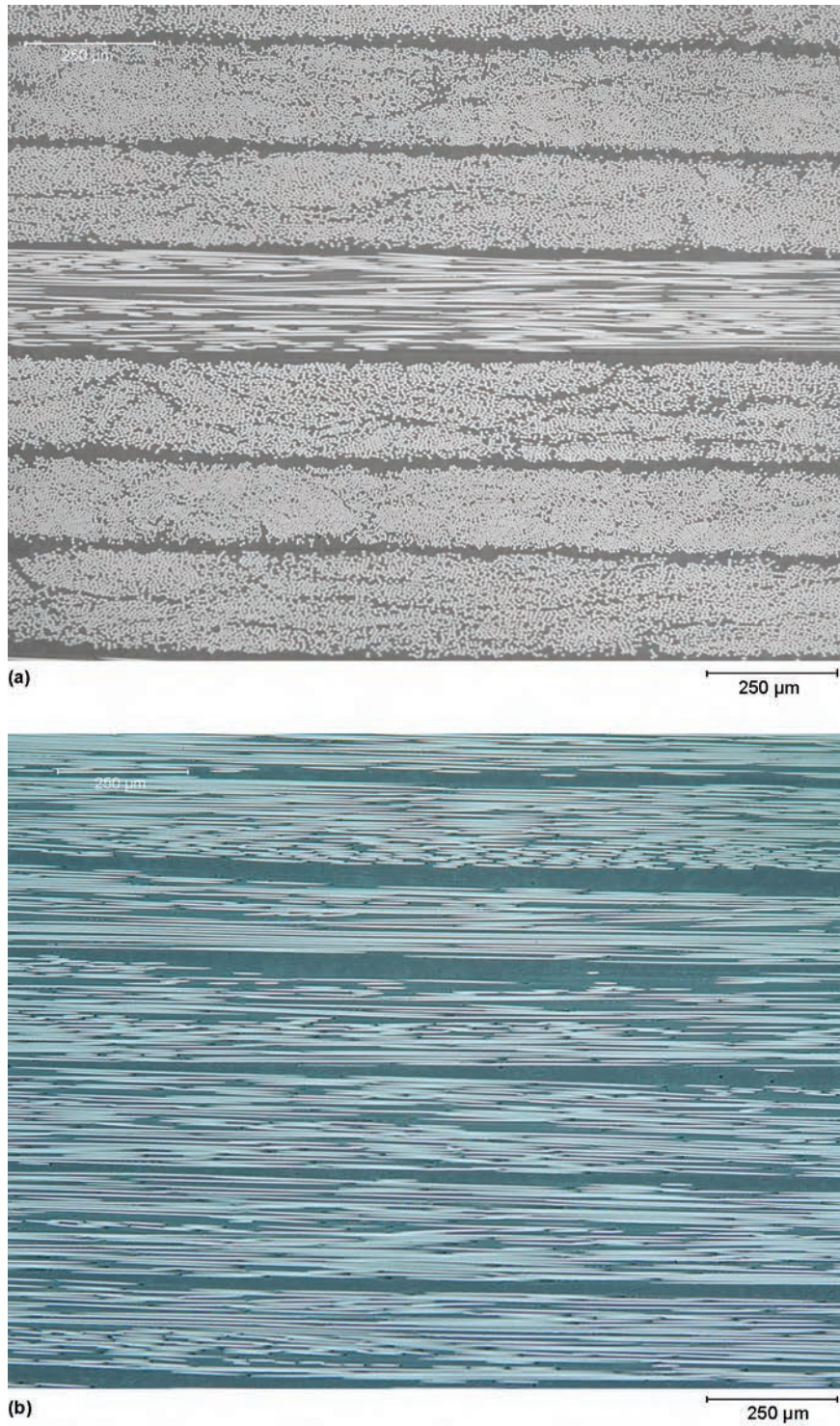


Fig. 1.11 Cross sections of interlayer-modified composite materials. (a) Cross section showing a middle ply at 90° . Bright-field illumination, $10\times$ objective. (b) Cross section taken parallel to the fiber direction. Bright-field illumination, $10\times$ objective

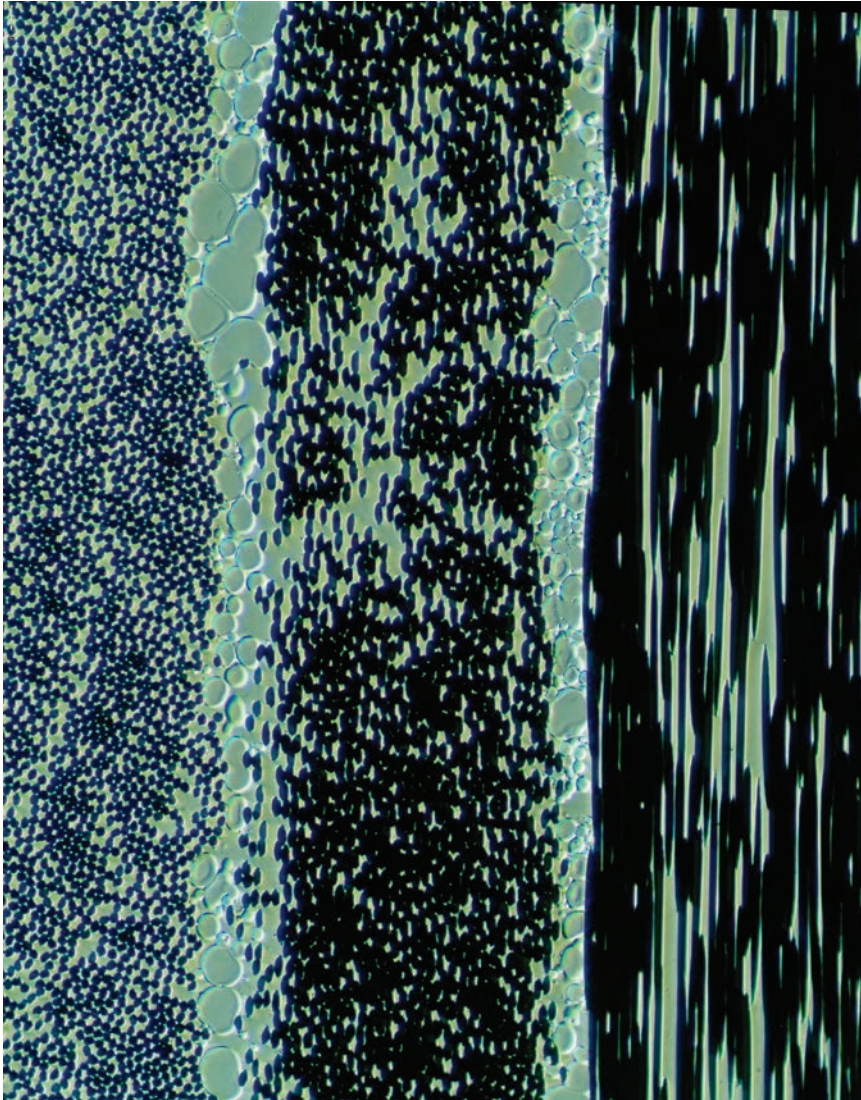


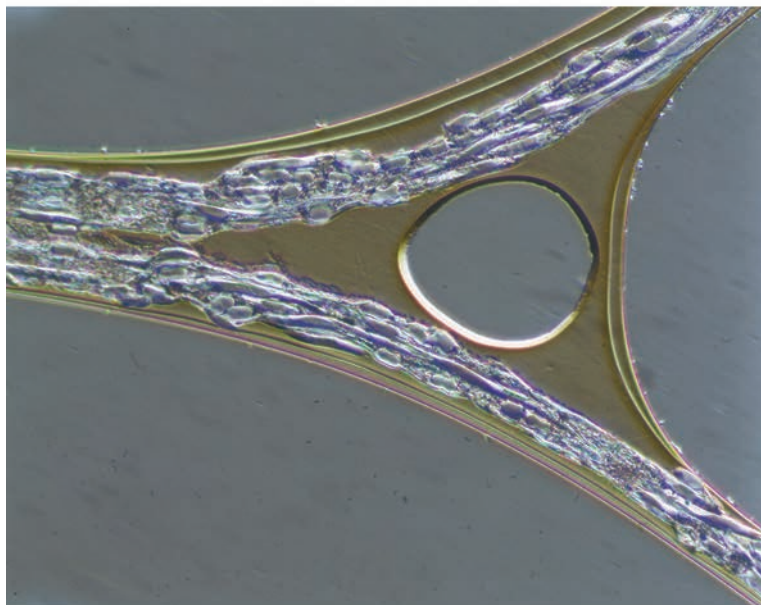
Fig. 1.12 Ultrathin section of a particle-modified interlayer-toughened composite material. Transmitted-light Hoffman modulation contrast, 20x objective

Honeycomb/Foam Structure Composite Materials

To further reduce the already lightweight composite materials and increase stiffness, structures have been developed whereby the composite layers are separated by a lightweight material, making a sandwich structure. Typical materials used for the sandwich materials are honeycomb core or foam materials. Honeycomb core materials are usually based on Nomex (E.I. du Pont de Nemours and Company), glass fiber, aluminum, or carbon, depending on the application (Fig. 1.13a, b). The high strength-



(a)



(b)

Fig. 1.13 Cross sections of honeycomb node areas showing the number of phenolic resin dip coats. (a) Transmitted light, 100× objective. (b) Void in the node area. Transmitted-light phase contrast, 100× objective

to-weight ratios of these structures are especially attractive to the airplane industry, where stiff, lightweight parts are required. In the airplane industry, composite skin honeycomb structures are used for such parts as radomes, fairings, nacelles, internal flooring, storage compartments, and elevators (Ref 32, 33). In addition to aerospace applications, many other composite structures are developed using honeycomb materials, which are found in boats, sporting goods, and infrastructure applications. Thermosetting resins are usually used as the prepreg matrices for composite honeycomb structures because of the necessity of the appropriate tack-and-drape characteristics. The fiber reinforcement usually consists of unidirectional and/or woven fabric developed from glass, Kevlar (E.I. du Pont de Nemours and Company), or carbon fibers. Prepregs with different reinforcing fibers may be used together to make hybrid parts that take advantage of the different mechanical properties supplied by different fibers. If the prepreg material does not satisfy the skin-to-core adhesive characteristics, a film adhesive must be used in combination with the prepreg, adding weight to the overall structure (Fig. 1.14).

Although not generally considered as high performance as honeycomb materials, foam (Fig. 1.15a, b) and wood cores (Fig. 1.16) have also found extensive use in the composites industry for making sandwich structures. These materials have the distinct advantage of not having large open cells and therefore can be used effectively with infusion methods or wet lay-up processing methods. The majority of foam- and balsa-cored composites are found in the marine, sporting goods, and infrastructure industries.

Optical Microscopy of Composite Materials

Optical microscopy is a valuable tool in materials investigations related to problem solving, failure analysis, advanced materials development, and quality control. Microscopy has been used for many decades to provide insight into the micro- and macrostructure of fiber-reinforced composites.

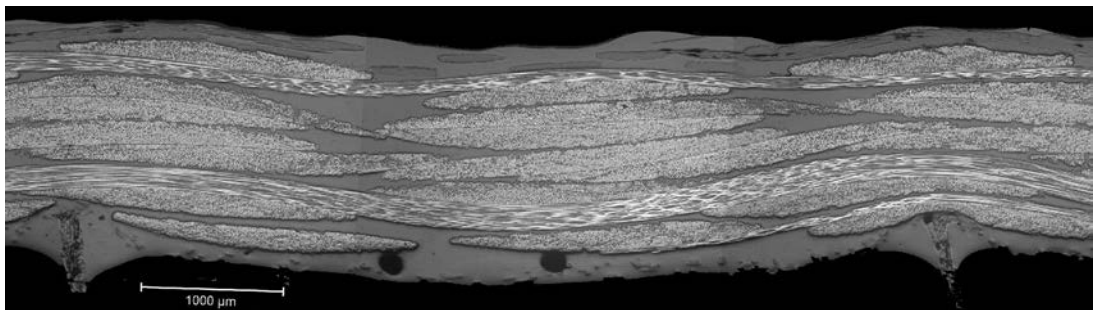
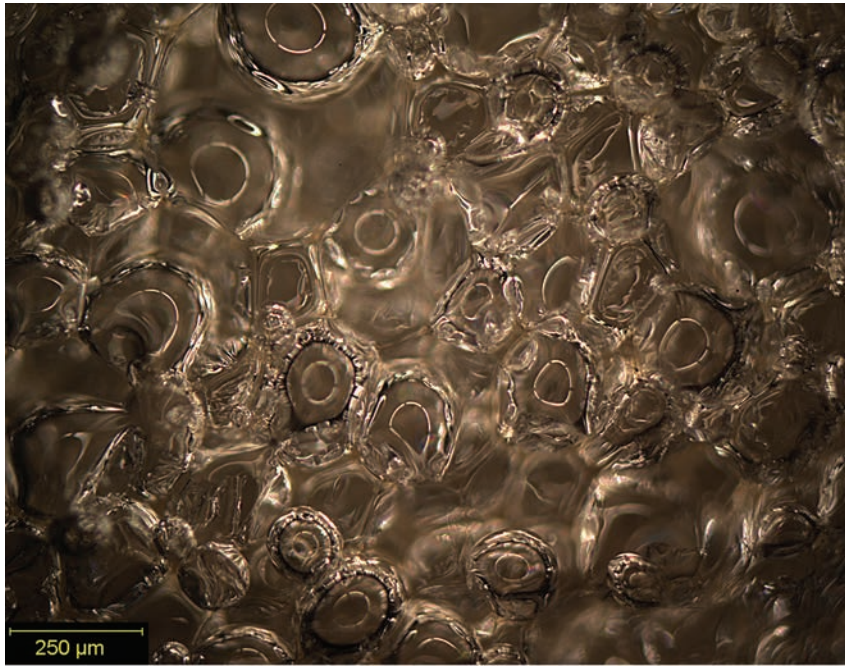
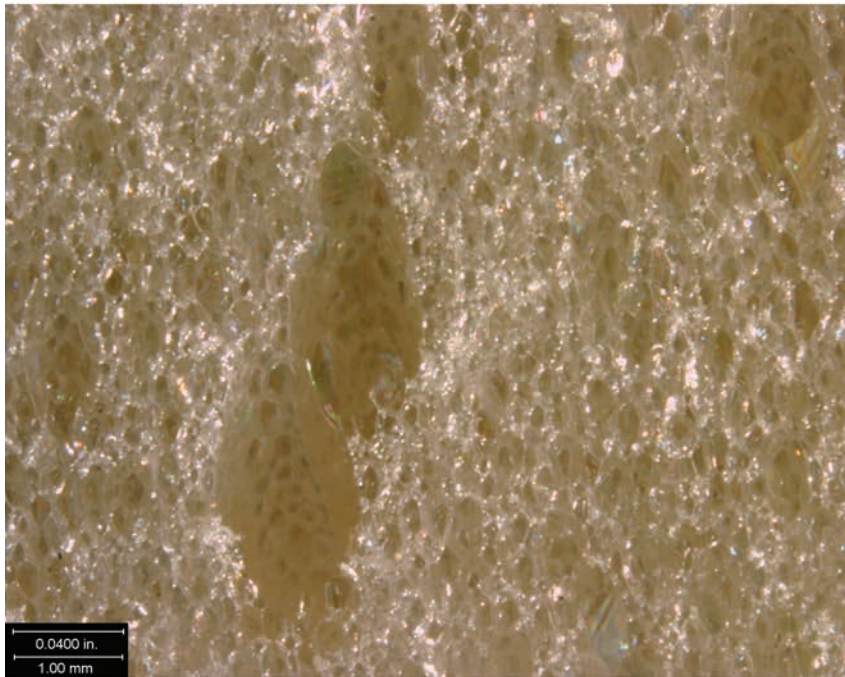


Fig. 1.14 Cross section of a carbon fiber prepreg skin-film adhesive co-cured honeycomb composite showing two fillet regions. A few voids are shown in the adhesive areas. Bright-field illumination montage, 5× objective



(a)



(b)

Fig. 1.15 Unprepared cross sections of structural foams. (a) Dark-field illumination, 10× objective. (b) Bright-field illumination, 65 mm macro-photograph



Fig. 1.16 Ultrathin section showing the microstructure of spruce wood.
Transmitted-light phase contrast, 40× objective

The most widespread use of optical microscopy for composites is determining void content, ply counts, and fiber orientations. While this makes up the majority of analysis, the investigation of failure mechanisms and microstructural analysis is also common. Furthermore, insight into fiber morphology, matrix modifiers, fillers, and the effect of processing parameters used for manufacturing composite materials are also elucidated using optical microscopy techniques.

For the majority of cases, reflected-light microscopy provides most of the necessary information one would desire. In some cases, however, etchants, stains, or dyes may be required for further clarification of the morphology or crack identification. If reflected techniques do not yield the required information, transmitted-light optical microscopy can provide insights into the microstructures of these materials that would otherwise remain hidden when using standard bulk metallographic preparation techniques and reflected illumination. Because many thermoset materials are inert to metallographic etchants, often the sample is best observed with transmitted polarized light and various contrast media to enhance the differences in refractive index of discrete phases in the composite.

Although an array of different types of composite materials is in use today, the utilization of both thermosetting- and thermoplastic (polymer)-

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matrix fiber-reinforced composites continues to dominate the field in terms of both volume and applications. This is due to easy processing, a wide range of materials and properties, and a much lower cost than other composite materials, such as metal-matrix composites and ceramic-matrix composites. While not to discount the limited use of other types of composites, all of these heterogeneous and anisotropic materials have unique properties and characteristics, lending their use in specific applications.

Over the years, many methods for polishing composite materials have been used, most with varying success and quality. This continues today and is frustrating when there is no standard to reference. Throughout this book, the easiest, most cost-effective, and reproducible techniques the authors have found for sample preparation, polishing, and analysis are described. The most common types of polymeric-matrix fiber-reinforced composite materials are the primary focus of the book, but also included is how to prepare more exotic composite materials and hybrids. Most of the materials that are described in this book are continuous thermosetting-matrix fiber-reinforced composites, but all techniques that are explained herein are applicable to short, discontinuous fiber composites and thermoplastic-matrix materials.

Chapter 1: Introduction—Composite Materials and Optical Microscopy

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Chapter 10: Toughening Methods for Thermoset-Matrix Composites

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Chapter 11: Impact Response of Composites

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Chapter 12: Matrix Microstructural Analysis

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Chapter 13: Honeycomb-Cored Sandwich Structure Composites

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Chapter 14: Surface Degradation of Composites

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Chapter 15: The Effects of Lightning Strikes on Polymeric Composites

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