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PREVENTION OF ENVIRONMENTALLY INDUCED DEGRADATION OF CARBON/EPOXY COMPOSITE MATERIAL VIA IMPLEMENTATION OF A POLYMER BASED COATING SYSTEM

by

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A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in the Department of Mechanical, Materials, and Aerospace Engineering in the College of Engineering and Computer Science at the University of Central Florida Orlando, Florida

Fall Term 2008
ABSTRACT

As the use of fiber reinforced plastics increases in such industries as aerospace, wind energy, and sporting goods, factors effecting long-term durability, such as environmental exposure, are of increasing interest. The primary objectives of this study were to examine the effects of extensive environmental exposure (i.e., UV radiation and moisture) on carbon/epoxy composite laminate structures, and to determine the relative effectiveness of polymer-based coatings at mitigating degradation incurred due to such exposure. Carbon/epoxy composite specimens, both coated and uncoated, were subjected to accelerated weathering in which prolonged outdoor exposure was simulated by controlling the radiation wavelength (in the UV region), temperature, and humidity. Mechanical test data obtained for the uncoated specimens indicated a reduction in strength of approximately 6% after 750 hours of environmental exposure. This reduction resulted from the erosion of the epoxy matrix in addition to the formation of matrix microcracks. Test data revealed that no further degradation occurred with increased exposure duration. The protective coatings evaluated were all epoxy based and included two different surfacing films and a chromate containing paint primer. The surfacing films were applied during initial cure of the carbon/epoxy composite laminate, and the chromate containing epoxy based paint primer was applied subsequent to curing the carbon/epoxy composite laminate. Although the chromate primer performed well
initially, degradation of the underlying substrate was detected with extended exposure durations. In contrast, the surfacing films provided superior protection against environmentally induced degradation. Similar degradation attributes were identified in the surfacing film as observed in the uncoated composite, but the degradation was either confined within the surfacing film layer or only penetrated the very near surface of the carbon/epoxy substrate. This limited degradation results in a minimal reduction in mechanical strength.
I would like to dedicate this thesis to my family and to my wife Charlene. Without all of your love and support my education would not have been possible.
ACKNOWLEDGEMENTS

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# TABLE OF CONTENTS

LIST OF FIGURES ................................................................. viii  
LIST OF TABLES ................................................................. x  
LIST OF ACRONYMS/ABBREVIATIONS ................................ xi  
1.0 INTRODUCTION ................................................................... 1  
2.0 LITERATURE REVIEW .......................................................... 4  
   2.1 Chemistry of Epoxy Polymers .............................................. 4  
   2.2 Environmental Degradation of Carbon/Epoxy Composites .... 13  
      2.2.1 Degradation Due to Moisture Exposure ...................... 14  
      2.2.2 Degradation Due to Ultraviolet (UV) Radiation Exposure 16 
      2.2.3 Synergistic Effects of Moisture and UV Radiation ........ 18  
   2.3 Mechanisms of Degradation Induced by Exposure to Ultraviolet Radiation .................................................. 20  
      2.3.1 Chemical Reaction Mechanisms .................................. 20  
      2.3.2 Degradation of the Epoxy Matrix as a Function of Depth 28  
3.0 EXPERIMENTAL DETAILS ..................................................... 30  
   3.1 Testing Methodology .......................................................... 30  
   3.2 Test Panel Fabrication ...................................................... 31  
   3.3 Pre-Exposure Testing ....................................................... 35  
   3.4 Accelerated Weathering Exposure Testing ......................... 37  
   3.5 Post Exposure Testing ..................................................... 39  
      3.5.1 Visual Micro-inspection ............................................. 39  
      3.5.2 Mechanical Testing .................................................. 39  
4.0 RESULTS ............................................................................... 42  
   4.1 Weight as a Function of Environmental Exposure Duration .... 42  
   4.2 Visual Inspection of Specimens Subjected to Accelerated Environmental Exposure ............................................. 43  
   4.3 Mechanical Test Results .................................................. 54  
5.0 DISCUSSION ......................................................................... 61  
   5.1 Environmentally Induced Degradation in Carbon/Epoxy Composite Material .................................................... 61  
   5.2 Prevention of Degradation via Implementation of Polymer Based Coatings .............................................................. 63  
6.0 SUMMARY AND RECOMMENDATIONS ................................. 67  
7.0 APPENDIX A: RAW TEST DATA ......................................... 69  
8.0 APPENDIX B: SUPPORTING DOCUMENTATION .................. 80  
9.0 REFERENCES ..................................................................... 111
LIST OF FIGURES

Figure 1  Representation of the Epoxy (a) and the Glycidyl (b) Groups  \cite{3} .......... 6
Figure 2  Common Epoxy Synthesis Reaction  \cite{3} ......................................................... 7
Figure 3 (a) Tri-functional Epoxy; (b) Tetra-functional Epoxy  \cite{3} .............................. 9
Figure 4 Epoxy Curing Reaction with Amine Curing Agent  \cite{3} ....................................... 12
Figure 5 Epoxy Crosslinking Mechanism  \cite{3} ................................................................. 12
Figure 6 Proposed Mechanism for Photo-Oxidation of TGDDM/DDS Epoxy
Polymer Scheme 1  \cite{10} .................................................................................................. 24
Figure 7 Proposed Mechanism for Photo-Oxidation of TGDDM/DDS Epoxy
Polymer Scheme 2  \cite{10} .................................................................................................. 25
Figure 8 Proposed Mechanism for Photo-oxidation of TGDDM/DDS Epoxy
Polymer Scheme 3  \cite{10} .................................................................................................. 26
Figure 9 Proposed Mechanism for Photo-oxidation of TGDDM/DDS Epoxy
Polymer Scheme 4  \cite{10} .................................................................................................. 27
Figure 10 Carbon/Epoxy Composite Test Panel Cure Cycle ................................. 33
Figure 11 Control Test Panels: No Environmental Exposure ................................. 34
Figure 12 Fiber Orientation for ASTM 3518 Test Specimen  \cite{11} ....................... 36
Figure 13 In-Plane Shear Test Specimen Dimensions .............................................. 41
Figure 14 Percentage Weight Loss as a Function of Accelerated Environmental
  Exposure Duration ........................................................................................................... 43
Figure 15 Bare Composite (A) No Exposure (B) 1500 Hrs Exposure ..................... 44
Figure 16 Chromate Primer Coated Composite (A) No Exposure (B) 1500 Hrs
  Exposure ......................................................................................................................... 44
Figure 17 Surfacing Film A (A) No Exposure (B) 1500 Hrs Exposure ..................... 45
Figure 18 Surfacing Film B (A) No Exposure (B) 1500 Hrs Exposure ..................... 45
Figure 19 Secondary Electron SEM Images (1000x) of Bare Carbon/Epoxy
  Composite (A) No Exposure (B) 750 Hrs Exposure (C) 1000 Hrs Exposure
  (D) 1500 Hrs Exposure .................................................................................................. 48
Figure 20 Secondary Electron SEM Image (25000x) of Bare Carbon/Epoxy
  Composite after 1500 Hrs of Environmental Exposure ............................................ 49
Figure 21 Secondary Electron SEM Images (5000x) of Bare Carbon/Epoxy
  Composite (A) No Exposure (B) 750 Hrs Exposure (C) 1000 Hrs Exposure
  (D) 1500 Hrs Exposure .................................................................................................. 50
Figure 22 Secondary Electron SEM Images (50x) of Bare Carbon/Epoxy
  Composite (A) No Exposure (B) 750 Hrs Exposure ..................................................... 50
Figure 23 Secondary Electron SEM Image (1000x) of Chromate Primer Coated
  Carbon/Epoxy Composite (A) No Exposure (B) 1500 Hrs Exposure ............. 51
Figure 24 Secondary Electron SEM Image (1000x) of Carbon/Epoxy Composite
  Coated With Surfacing Film A (A) No Exposure (B) 1500 Hrs Exposure .... 51
Figure 25 Secondary Electron SEM Image (1000x) of Carbon/Epoxy Composite
  Coated With Surfacing Film B (A) No Exposure (B) 1500 Hrs Exposure .... 52
Figure 26  Secondary Electron SEM Image (50x) of Carbon/Epoxy Composite Coated with Surfacing Film A – 750 Hrs Exposure .............................. 52
Figure 27  Cross Section Images (50x) of Carbon/Epoxy Specimens coated with (A) Surfacing Film A and (B) Surfacing Film B ................................................................. 53
Figure 28  Ultimate Load vs. Exposure Time (A) Bare Composite (B) Chromate Primer Coated Composite (C) Surfacing Film A (D) Surfacing Film B ........ 59
Figure 29  Ultimate Load as a Function of Coating Configuration and Exposure Duration .................................................................................................................. 60
Figure 30  Ultimate Load as a Function of Coating Thickness and Exposure Duration .................................................................................................................. 60
LIST OF TABLES

Table 1  Detailed Test Panel Fabrication Matrix ......................................................... 34
Table 2  Detailed Accelerated Weathering Test Matrix .............................................. 38
Table 3  Mechanical Strength Values No Exposure .................................................. 57
Table 4  Mechanical Strength Values 750 Hr Exposure ........................................... 57
Table 5  Mechanical Strength Data 1000 Hr Exposure ............................................. 58
Table 6  Mechanical Strength Data 1500 Hr Exposure ............................................. 58
### LIST OF ACRONYMS/ABBREVIATIONS

<table>
<thead>
<tr>
<th>Acronym</th>
<th>Description</th>
</tr>
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<tbody>
<tr>
<td>ASTM</td>
<td>American Society for Testing and Materials</td>
</tr>
<tr>
<td>DDS</td>
<td>4, 4’-diaminodiphenyl sulfone</td>
</tr>
<tr>
<td>DGEBPA</td>
<td>Diglycidyl Ether of Bisphenol A</td>
</tr>
<tr>
<td>EDX/EDAX</td>
<td>Energy Dispersive X-ray Analysis</td>
</tr>
<tr>
<td>FT-IR Spectroscopy</td>
<td>Fourier Transform Infrared Spectroscopy</td>
</tr>
<tr>
<td>PA FT-IR</td>
<td>Photo Acoustic Fourier Transform Infrared Spectroscopy</td>
</tr>
<tr>
<td>Prepreg</td>
<td>Woven or unidirectional carbon fiber pre-impregnated with matrix resin</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscope</td>
</tr>
<tr>
<td>TGDDM</td>
<td>Tetraglycidyl-4,4’-diaminodiphenylmethane</td>
</tr>
<tr>
<td>UV</td>
<td>Ultraviolet</td>
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1.0 INTRODUCTION

In the most basic sense, a composite material is simply a mixture of two or more distinct solid constituents that are, in theory, mechanically separable and, when combined, produce a material with superior properties to the individual constituents alone. Typically, the composite material consists of a binder or matrix that surrounds and holds reinforcements in place. The separate characteristics of the matrix and reinforcements contribute synergistically to the overall properties of the composite material \[2,3,9\]. This definition includes a wide assortment of materials including steel reinforced concrete, particle filled plastics, ceramic mixtures, and some alloys \[3\]. This study focuses on a class of composites known as fiber reinforced plastics. More specifically, materials composed of an epoxy polymer matrix reinforced with carbon fibers.

The key advantage for using composite materials for structural applications is the weight reduction realized due to the high strength-to-weight and stiffness-to-weight ratios \[1\]. For example, in aerospace applications, weight savings on the order of 25% are generally considered to be achievable using current composite materials in place of metals \[2\].

In composite materials, all of the properties arise, to some extent, from the interaction between the matrix and reinforcement \[3\]. However, each constituent contributes different attributes to the overall composite material performance. The principal role of the reinforcement is to provide mechanical properties such as
strength and stiffness and to carry the load imposed on the composite structure. In structural applications, 70% to 90% of the load is carried by the reinforcements \[^{[3,9]}\]. The primary purpose of the matrix is to bind the reinforcement (fiber) together, transfer loads to and between the fibers, and to protect the fibers from self-abrasion and externally induced scratches. The matrix also protects the fibers from environmental degradation, which can lead to embrittlement and premature failure \[^{[1]}\].

The use of fiber-reinforced plastics has steadily increased in markets such as aerospace, wind energy, and sporting goods. In the past 15 years, the market demand for glass-reinforced plastics has grown by 50% and the market demand for carbon fiber composite products has increased by 250% \[^{[4]}\]. As the use of these fiber reinforced plastic composite materials increases, factors effecting long-term stability and durability, such as environmental exposure, may become a significant concern in the industries where these materials are utilized. Previous research has determined that exposure to environmental factors such as Ultraviolet (UV) radiation, moisture, and temperature results in a reduction in matrix dominated properties, resulting in a decrease in the overall performance of the composite material \[^{[1,7,8]}\].

The primary objectives of this study were to examine the effects of prolonged environmental exposure (specifically UV radiation and moisture) on carbon/epoxy composites and to investigate the effectiveness of various polymer based coatings at preventing composite substrate degradation. In order to
simulate extensive outdoor environmental exposure, carbon/epoxy composite panels, each with a different coating, were subjected to accelerated environmental weathering. After exposure, the panels underwent visual micro-inspection and mechanical testing to determine their load carrying capability. These results were compared with unexposed control specimens to determine the extent of degradation and the performance of the protective coatings.
2.0 LITERATURE REVIEW

2.1 Chemistry of Epoxy Polymers

In composite structures designed for low temperature applications (less than 200°F) the most widely used polymer matrix materials are epoxies. Generally considered the workhorse of the composites industry, epoxies provide outstanding chemical resistance (i.e. fluids and solvents), excellent adhesion strength to fibers, and superior dimensional stability. Epoxies are also favored due to their low cure shrinkage, long shelf life, and lack of void forming volatiles [1].

In order to obtain a better understanding of the specific degradation mechanisms induced by exposure to UV radiation, a basic knowledge of epoxy chemistry must first be attained. Epoxy resins belong in a class of polymers known as thermosets. Thermosetting resins, which are usually liquids at room temperature, are characterized by the ability to form bonds between the molecules of individual chains, also known as crosslinks. This is made possible by certain molecules on the polymer chains that can be activated to form reaction sites. The formation of these crosslinks restricts the movements of the polymer chains, resulting in increased stiffness, strength, and temperature resistance. Once the crosslinks are formed during cure, these materials cannot be melted and will degrade when exposed to extreme temperatures. The other major class
of polymer resins are known as thermoplastics. Contrary to the thermosets, these resin systems, usually solids at room temperature, do not form crosslink bonds and, therefore, can be melted and reformed. Thermoplastics provide improved toughness over thermosets [3].

Epoxies contain a high percentage of aromatic molecules, which are characterized by the presence of variations of the aromatic functional group. In the most basic form, the aromatic group is a cyclic hydrocarbon consisting of six carbon atoms each including one hydrogen atom. When no other polymer groups are attached, it is referred to as benzene. The aromatic group imparts strength and stiffness to the polymer chain and also increases temperature resistance. On the other hand, aliphatic compounds are characterized by their complete lack of aromatic content and an increased presence of straight chain polymers. Aliphatic polymers typically exhibit improved flexibility, toughness, and resistance to weathering [3].

The basic structure of an uncured epoxy resin consists of two parts: a three-member ring epoxy group (epoxy ring) also known as an oxirane group and the rest of the polymer chain. The epoxy group is considered an “active site” because it is the location where crosslinking occurs. The epoxy group also gives rise to many of the characteristic properties observed with epoxies. In the complete epoxy resin structure, the epoxy ring will be attached to another organic group in the polymer chain, either directly or via an intermediate carbon atom, known as a bridge. In the latter case, the epoxy group is then referred to as a
glycidyl (reference Figure 1) [3].

Figure 1
Representation of the Epoxy (a) and the Glycidyl (b) Groups [3]
The most common method of producing uncured epoxy resin is via the condensation polymerization reaction of bisphenol A with epichlorohydrin. This reaction bonds the glycidyl groups to both ends of the aromatic bisphenol A compound (Reference to Figure 2). The epoxy nomenclature is derived from the various components contained within the polymer resin. For example, the epoxy polymer created per the reaction in Figure 2 is referred to as DGEBA, or “DiGlycidyl Ether of BisPhenol A”. This is in reference to the two glycidyl groups (di-glycidyl) attached to the bisphenol A polymer via an ether linkage (R-O-R’) [3].

![Common Epoxy Synthesis Reaction](image)

The other major component of the epoxy resin is the remaining polymer chain. In figure 2, this is represented by the bisphenol A portion of the DGEBA molecule. The particular molecule chosen for this portion of the resin and the
number of repeat units (represented by “n”) can have a significant effect on the final resin characteristics, and, ultimately, the final properties of the cured polymer. For instance, increasing the number of bisphenol A units included in the resin molecule depicted in figure 2, will result in an increased resin viscosity and heat distortion temperature. Besides changing the number of polymer units, different types of polymers can be utilized to increase the functionality of the epoxy resin. Figure 3 illustrates examples of tri-functional and tetra-functional epoxy resins created by utilizing different aromatic polymer linkages. These resins have an increased number of active sites available for crosslinking reactions. Upon cure, the ability for increased crosslinking will lead to a material with higher strength, stiffness, and temperature resistance [3].
Figure 3
(a) Tri-functional Epoxy; (b) Tetra-functional Epoxy"}^{[3]}
The methods used for synthesis of epoxy resins are quite different from those utilized to crosslink and cure them. The crosslink reaction in epoxy resins is based upon the opening of the epoxy ring by a reactive group on the end of another molecule known as the curing agent or hardener. A typical curing agent consists of a polymer molecule with an amine based reactive group (NH$_2$) at each end. The presence of the amine reactive groups on either end allows the curing agent to react with two epoxy groups on two different molecules, thus linking them together [3].

The ring-opening reaction is initiated when the reactive portion of the curing agent comes into close proximity with the epoxy ring. The nitrogen atom has a slightly negative charge and seeks the slightly positive charge of the carbon atom in the epoxy ring. The end carbon of the epoxy ring, which is the terminal carbon of the chain, is usually the more accessible of the two epoxy-ring carbons. It is therefore the atom that usually reacts with the nitrogen. The nitrogen forms a bond with the carbon, breaks open the epoxy ring, and loses a hydrogen atom in the process. This hydrogen atom, which is slightly positive, will then bond to the oxygen that was initially part of the epoxy ring.
This hydroxyl group (OH\textsuperscript{−}) is capable of reacting with other epoxy rings in the crosslinking reaction, which can establish a chain reaction referred to as homopolymerization. In this instance the curing agent can be viewed as an initiator. However, the curing agent and homopolymerization reactions will generally occur together to complete crosslinking during cure. Figures 4 and 5 illustrate the ring-opening reaction mechanism and the epoxy crosslinking mechanism, respectively \footnote{[3]}. 
Figure 4 Epoxy Curing Reaction with Amine Curing Agent [3]

Figure 5 Epoxy Crosslinking Mechanism [3]
2.2 Environmental Degradation of Carbon/Epoxy Composites

As mentioned in the introduction, there are several inherent advantages to using composite materials for structural applications (i.e., high strength and stiffness to weight ratios). Despite these benefits, there are concerns regarding the overall long-term durability of these materials, especially as related to their capacity for sustained performance under harsh and changing environmental conditions \[5\]. Composite structures must be designed to withstand the great diversity of environments encountered in a variety of operations. For instance, in aerospace applications, environmental effects, including combinations of heat, cold, moisture, lightening strikes, UV radiation, fluids, and fuels, can reduce mechanical properties to varying degrees, depending on the composite system and the particular design application \[1\].

Although the most important contribution to the material strength is that of the fiber, the overall performance of the composite structure also depends greatly on the properties of matrix in addition to the quality of the fiber-matrix bond. The matrix, in addition to binding the fibers together and protecting them from environmental effects, serves to transfer applied structural loads to the fibers. The fiber-matrix interface governs these load transfer characteristics and contributes to the overall damage tolerance of the structure \[1,5\].
The composite matrix is generally the component most vulnerable to environmental attack, with UV light and moisture being two of the primary environmental factors contributing to material degradation. In general, matrix degradation induced by environmental exposure is manifested as matrix cracking and erosion that leads to a reduction in matrix dominated properties. Consequently, matrix-dominated properties are of particular concern with regard to environmental exposure of carbon/epoxy composites. \(^1,2,7\).

Previous research determined that a carbon/epoxy laminate exposed to 500 hours of UV exposure, would see a reduction in transverse tensile strength of 9\% and a laminate exposed to 500 hours of moisture via condensation would see a reduction of 20\%. When laminates are exposed to both UV radiation and condensation, either sequentially or in a cyclical manner, the combined effects can produce even greater degradation \(^5\). The synergistic effects of UV radiation and condensation are discussed further in subsequent sections.

### 2.2.1 Degradation Due to Moisture Exposure

The absorption of moisture by the epoxy matrix as a result of environmental exposure can have detrimental effects on the overall mechanical properties of the carbon/epoxy composite structure. The moisture diffuses into the matrix, which leads to dilatation expansion and also chemical changes such as plasticization and hydrolysis \(^1,7\).
In degradation by moisture ingress, the controlling factor is the diffusion constant of water vapor. As water is a very polar molecule, the diffusion mechanism involves hydrogen bonding with polar sites in the polymer molecule. Epoxy resins are the most polar of the normal resins as they contain hydroxyl groups, ether groups, and C-N bonds. Thus, water permeability is highest for epoxy resins. This can result in both reversible and irreversible damage to the epoxy matrix. Plasticization is usually reversible upon desorption of moisture, while hydrolysis of chemical bonds results in permanent irreversible damage. Moisture desorption gradients may induce microcracking as the surface desorbs and shrinks, putting the surface in tension. If the residual tension stress at the surface is beyond the strength of the matrix, cracks occur. Additionally, moisture wicking along the fiber-matrix interface can degrade the fiber-matrix bond, resulting in loss of microstructural integrity \(^{[1,2,5]}\).

All of these factors manifest in a decrease in matrix-dominated properties such as compressive strength, interlaminar shear strength, fatigue, and impact tolerance. Although the carbon fibers do not absorb moisture and their physical properties remain unaffected, the deterioration of the matrix alone is sufficient to cause a decrease in performance and overall reliability \(^{[1,5]}\).
2.2.2 Degradation Due to Ultraviolet (UV) Radiation Exposure

In addition to degradation due to moisture absorption, the epoxy matrix in carbon/epoxy composite structures is also susceptible to attack by incident light. The most important interaction of light with the polymer matrix is from the UV component of light. The UV components of solar radiation incident on the earth surface are in the 290-400 nm band. The energy of these UV photons is comparable to the dissociation energies of polymer covalent bonds, which are typically 290-460 kJ/mole. Therefore, the interactions between this UV light and the electrons are strong, often resulting in excitation of the electrons and a resultant breaking of the bond. Hence, UV light can degrade polymers [3,5].

The nature of the atoms in polymer matrix has some effect on the tendency of the electrons to become excited by the UV light and degrade. Generally, aromatic polymers are more easily degraded by UV light then are aliphatic polymers. All resins containing aromatic groups can absorb sufficient UV radiation to cause bond dissociation. Of the typical resins used in composite structures, phenolics are most sensitive, followed by epoxy resins. The high aromatic content common to most high-performance epoxies makes them particularly susceptible to UV radiation induced degradation [2,3].
The UV photons absorbed by polymers result in photo-oxidative reactions that alter the chemical structure resulting in material deterioration. The chemical reactions typically cause molecular chain scission and/or chain cross-linking. Chain scission lowers the molecular weight of the polymer, giving rise to reduced heat and strength resistance. Chain cross-linking leads to excessive brittleness and can result in microcracking. Previous research discovered that exposure of a carbon/epoxy laminate to UV radiation for as little as 500 hrs results in the formation of microcracks, which lead to a reduction in matrix-dominated properties. This was likely caused by embrittlement of the polymer matrix due to increased crosslinking resulting from photo-oxidation reactions induced by UV exposure. More detailed discussion of UV radiation degradation mechanisms is included in subsequent sections [5].

Some polymers, including epoxies, exhibit a color change when exposed to UV radiation. In addition to inducing chain scission and increasing crosslink density, photo-oxidative reactions can also result in the production of chromophoric chemical species. Chromophores are simply molecules that transmit and absorb light. These chromophores, may impart discoloration to the polymer, if they absorb visible wavelengths. Furthermore, an autocatalytic degradation process may be established if chromophores produced also absorb UV radiation [5].
2.2.3 **Synergistic Effects of Moisture and UV Radiation**

While the previous sections have focused on the individual degradation effects due to UV radiation and moisture exposure, these environmental factors can act in conjunction to further enhance the degradation of the carbon/epoxy composite structure [5].

Exposure to UV radiation results in the formation of a thin surface layer of chemically modified epoxy. Subsequent water condensation leaches away soluble UV degradation products, which exposes a fresh layer that can once again be attacked by UV radiation. In this manner, a repetitive process is established that leads to significant erosion of the epoxy matrix. Furthermore, it is also conceivable that the presence of absorbed water molecules in the epoxy matrix can enhance the photo-oxidation reactions due to increased availability of \( \text{OH}^- \) and \( \text{H}^+ \) ions. This would increase the chain scission and crosslinking reactions occurring on the surface of the epoxy polymer, thus increasing the brittleness of the matrix. These synergistic mechanisms result in more extensive microcracking and loss of fiber confinement due to matrix erosion, ultimately leading to a more significant reduction in the overall mechanical properties of the composite structure [5,7].

In research conducted by Kumar et al., carbon/epoxy laminates exposed to cyclic exposure of both UV radiation and moisture condensation totaling 1000 hrs resulted in extensive matrix erosion, void formation, and fiber-matrix interface...
debonding. The epoxy rich layer on the specimen surface was completely removed and the underlying carbon fibers were exposed. Furthermore, examination of the transverse tensile strength indicated a reduction of 29% as compared with unexposed specimens [5].
2.3 *Mechanisms of Degradation Induced by Exposure to Ultraviolet Radiation*

2.3.1 *Chemical Reaction Mechanisms*

Research performed by Kim et al.\(^6\) examined the degradation due to exposure to UV radiation and moisture. The study utilized an aluminum substrate coated with a bisphenol A based epoxy polymer with a nominal film thickness of 30 \(\mu\)m. These specimens were subjected to accelerated weathering, consisting of cyclic exposures to UV radiation @ 340 nm and water vapor condensation (4 hours each). The exposed specimens were examined using a combination of photo acoustic (PA) Fourier transformed infrared (FT-IR) spectroscopy, FT-IR microscopy, and Raman chemical imaging. Varying the modulation frequencies utilized with PA FT-IR facilitated the determination of molecular level information as a function of depth \(^6\).

Examination of unexposed specimens at depths ranging from 5-24 \(\mu\)m, using the PA FT-IR, detected an increase in the band intensities at 3399 cm\(^{-1}\), which indicates an increase in -OH (hydroxyl group) content with increasing depth. Alternatively, the spectra detected a decrease in the bands at 1250 cm\(^{-1}\) and 1509 cm\(^{-1}\) with increasing depth. These bands are attributed to oxirane ring stretching vibrations of bisphenol A epoxy polymer and N-H deformations of polyamine crosslinker, respectively. Both of these functional groups are reaction
sites responsible for crosslinking reactions of epoxy polymers. This indicates that the ring opening reactions of oxirane groups of bisphenol A epoxy polymer occur further away from the surface, thus resulting in the increase in –OH group content. However, the intensity of the band attributed to C=C stretching vibrations of bisphenol A (1607 cm\(^{-1}\)) does not change as a function of depth, indicating that bisphenol A epoxy polymer is uniformly distributed throughout the film thickness \cite{6}.

PA FT-IR spectroscopy performed on the surface of specimens exposed to various durations (0, 5, 9, and 13 weeks) detected an increase the 3399 cm\(^{-1}\) band, indicating that UV exposure in the presence of water condensation results in the formation of hydroxyl groups on the surface. An exposure time of 5 weeks also detected a decrease in intensity of the 1250 cm\(^{-1}\) and 1509 cm\(^{-1}\) bands. This indicates that UV exposure further promotes crosslinking reactions on the surface. No further decrease in these band intensities was detected with subsequent exposures past 5 weeks. However, the formation of a new band at 1660 cm\(^{-1}\) indicates that carbonyl amide formation is taking place on the surface. This band increases in intensity with continued exposure. These observations indicate that crosslinking reactions are responsible for degradation for exposures up to 5 weeks. After that time, formation of amides dominates the degradation process. FT-IR microscopy and Raman chemical imaging where utilized to examine specific aspects of the degraded surface, comparing areas with and without observed microcracking. The spectra generated detected an increase in band
intensity at 1660 cm\(^{-1}\) and a decrease in band intensity at 1296 cm\(^{-1}\) (C-N vibrations) band in the area with microcracking. These bands are attributed to higher amine content, indicating that the formation of amides, via chain scission, has a greater contribution to epoxy degradation. Spectra from the microcracked area also detected an increase in the 1250 cm\(^{-1}\) and 1509 cm\(^{-1}\), indicating a diminished extent of crosslinking was present\(^6\).

Similar exposure studies support these conclusions. Kumar et al.\(^5\) demonstrated that carbon/epoxy laminates subjected to 500 hrs of UV radiation exposure displayed similar spectra when analyzed with FT-IR. Specifically, reductions in the peaks at 1250 cm\(^{-1}\) and 1509 cm\(^{-1}\) suggesting increased crosslink density on the surface of the epoxy. A reduction in the peak at 1296 cm\(^{-1}\) was also observed, attributed to C-N stretching vibrations due to amide formation. This indicated the presence of chain scission reactions\(^5\).

Both of these studies indicated that crosslinking and chain scission mechanisms operate in a competing manner during the degradation process. Increased crosslinking dominates in the early stages of degradation, after which carbonyl amide formation by chain scission takes over. Both of these mechanisms then result in increased microcracking and surface deterioration, ultimately reducing the mechanical strength of the composite structure\(^5\).

Musto et al.\(^10\) proposed several degradation mechanisms based on FT-IR analysis conducted on tetragnaloyl-4,4'-diaminodiphenylmethane (TGDDM) epoxy resin cured with aromatic hardener 4,4'-diaminodiphenyl sulfone (DDS),
subsequent to exposure to UV radiation and humidity. They concluded that photo-oxidative degradation of TGDDM/DDS could potentially involve several different mechanisms, which ultimately bring about chain-scission, leading to the formation of amide and carbonyl groups. Figures 6 through 9 illustrate proposed degradation Schemes 1 through 4, respectively. Scheme 1 involves scission of the carbon-nitrogen bond following hydrogen abstraction on the methylene group, ultimately resulting in the formation of an aldehyde (carbonyl group). Scheme 2 begins with hydrogen abstraction of the CH-OH bond followed by a similar chain scission reaction at the carbon-nitrogen bond, resulting in the formation of a ketone (carbonyl group). Scheme 3 begins with the oxygen attack of structure VII depicted in scheme 2. Chain scission at the carbon-carbon bond produces a carboxylic acid (carbonyl group) and, via the elimination of H₂O from structure XI, an amide linkage. However, the principal route for amide formation is proposed in scheme 4, with chain scission occurring at the carbon-carbon bond, rather than the carbon-nitrogen bond, producing amide molecules which may propagate the photo-oxidative sequence \(^{[10]}\).
Figure 6
Proposed Mechanism for Photo-Oxidation of TGDDM/DDS Epoxy Polymer
Scheme 1 \cite{10}
Scheme 2

Figure 7
Proposed Mechanism for Photo-Oxidation of TGDDM/DDS Epoxy Polymer
Scheme 2 \cite{10}
Figure 8
Proposed Mechanism for Photo-oxidation of TGDDM/DDS Epoxy Polymer
Scheme 3 [10]
Figure 9
Proposed Mechanism for Photo-oxidation of TGDDM/DDS Epoxy Polymer
Scheme 4
\[^{10}\]
2.3.2 Degradation of the Epoxy Matrix as a Function of Depth

In addition to studying the degradation aspects of an epoxy polymer film exposed to both UV radiation and moisture, Kim et al. [6] also examined the molecular level degradation as a function of depth. As mentioned previously, this study involved an aluminum substrate coated with a bisphenol A based epoxy polymer with a nominal film thickness of 30 μm. These specimens were subjected to accelerated weathering, consisting of cyclic exposures to UV radiation @ 340 nm and water vapor condensation (4 hours each). The exposed specimens were analyzed using step-scan photo acoustic (PA) Fourier transformed infrared (FT-IR) spectroscopy. Varying the modulation frequencies facilitated the determination of molecular level information as a function of depth [6].

The first portion of the study determined that increased crosslinking reactions were initially responsible for degradation. However, with increased exposure time the predominate degradation mechanism was the formation of carbonyl amides. The other portion of the study examined specimens exposed for a 5 weeks, utilizing the PA FT-IR, at depths of 5, 9, 18 and 24 μm. To determine the depth of degradation from the exposed surface, the specimens were examined from the substrate side. Examination of the spectra indicated that
the bands at 1250 and 1509 cm$^{-1}$ increased in intensity as the detection depth approached the exposure surface. These bands are attributed to oxirane ring stretching vibrations of bisphenol A and N-H deformations, respectively. Increased intensity of these bands indicates a lower incidence of crosslinking. Therefore, it can be concluded that a lesser extent of crosslinking reactions occur near the exposure surface. Furthermore, in comparing the spectra at increasing depths from the substrate side, the onset of primary amine formation begins at 24 μm, as evidenced by the first appearance of the band at 1660 cm$^{-1}$. This indicates (based on the nominal coating thickness of 30 μm) that degradation occurs up to a depth of approximately 6 μm from the exposure surface. Additional evaluation indicates that this holds true, even with increased exposure time $[6]$. 
3.0 EXPERIMENTAL DETAILS

3.1 Testing Methodology

As stated in the introduction, the primary focus of this research was to study the effects of environmental weathering (specifically UV radiation and humidity) on carbon/epoxy composite material. Additionally, the effectiveness of various polymer-based coatings in mitigating degradation was also examined. This was accomplished by subjecting carbon/epoxy composite panels, each with a different coating configuration, to accelerated weathering exposure. By controlling the radiation wavelength, temperature, and humidity, extended environmental exposure can be simulated in a relatively short time frame. For example, a 750-hour exposure in an accelerated weathering chamber simulates approximately 6 months of actual exposure in an extreme environment (F. Lopez, personal communication, April 14, 2008). Each composite coating configuration was subjected to several different durations of accelerated weathering. Visual inspection and mechanical testing performed on the exposed specimens were compared to unexposed control panels to determine the extent of degradation.
3.2 Test Panel Fabrication

Previous research indicates that degradation of carbon/epoxy composites due to UV radiation is localized near the surface\(^6\). To increase the probability of detecting degradation this study utilized a thin (4 plies) carbon/epoxy laminate construction. Test panels were constructed utilizing the following materials:

**Carbon/Epoxy Prepreg:**
Standard modulus Carbon fiber woven into a plain weave fabric impregnated with an uncured epoxy resin (designated as 3K-70-PW). The nominal cured ply thickness is 0.008” and the nominal resin content is 36%. Plies of the pre-impregnated carbon fabric are applied to a flat aluminum tool, successively one on top of another and then cured under elevated temperature and pressure to create a composite part.

**Surfacing Film “A”:**
Light weight surfacing film consisting of an epoxy based polymer adhesive supported by a non-woven polyester scrim (carrier). Surfacing film is incorporated during the lay-up and cure of carbon/epoxy prepreg plies. **Nominal Coating Thickness is 0.004”**

**Surfacing Film “B”:**
Heavy weight surfacing film consisting of an epoxy based polymer adhesive supported by a non-woven polyester scrim (carrier). Surfacing film is incorporated during the lay-up and cure of carbon/epoxy prepreg plies. **Nominal Coating Thickness is 0.005”**.

**Chromate Containing Epoxy Paint Primer:** A coating usually applied to components to improve adhesion of subsequent coatings. It is also commonly used to protect substrates against corrosion and environmental degradation. In this case, the epoxy primer was applied to a bare carbon/epoxy test panel after it had been cured. **Nominal Coating Thickness is 0.001”**.
Each test panel was fabricated using four plies of prepreg fabric placed in a [45/-45]s stacking sequence. All composite test panels were cured under elevated temperature and pressure in the same autoclave cycle. The cure cycle consisted of an intermediate hold at 150°F ± 10 ºF for 60 ± 10 minutes followed by a hold at the cure temperature of 350°F ± 10 ºF for 120 ± 10 minutes. The nominal ramp rate used to achieve these temperatures was 4 ºF/min. The maximum autoclave pressure was 100 psi, applied during the ramp up to the cure temperature. The tests panels were cooled to 140°F ± 10 ºF at 4 ºF/min prior to removal from the autoclave (Reference Figure 10). The surfacing film coatings were incorporated in the fabrication of the laminate test panels by laying them on the aluminum tool surface prior to adding the carbon prepreg plies. The surfacing film is then cured along with the carbon/epoxy prepreg layers. The chromate primer coating was added to the carbon/epoxy test panels after they had been cured (Reference Figure 11).
Figure 10
Carbon/Epoxy Composite Test Panel Cure Cycle

Four sets of test panels were constructed with the composite/coating configurations listed below. In addition to the test panels used for visual and mechanical evaluation, smaller specimens were fabricated and used to monitor weight loss over the duration of the UV/moisture exposure. Reference Table 1 for a more detailed test panel fabrication matrix.

1. **Bare Composite (BC):** Carbon/epoxy composite panel with no coating

2. **Chromate Primer (CP):** Carbon/epoxy composite panel coated with a chromate containing epoxy paint primer.

3. **Surfacing Film A:** Carbon/epoxy composite panel coated with light weight surfacing film.

4. **Surfacing Film B:** Carbon/epoxy composite panel coated with heavy weight surfacing film
Table 1
Detailed Test Panel Fabrication Matrix

<table>
<thead>
<tr>
<th>Composite Configuration</th>
<th>Test Panel I.D.</th>
<th>Test Panel Dimensions(1)</th>
<th>Composite Configuration</th>
<th>Test Panel I.D.</th>
<th>Test Panel Dimensions(1)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>8” x 10”</td>
<td></td>
<td></td>
<td>8” x 10”</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2” x 3”</td>
<td></td>
<td></td>
<td>2” x 3”</td>
</tr>
<tr>
<td>Bare Composite</td>
<td></td>
<td></td>
<td>Surfing Film A</td>
<td></td>
<td></td>
</tr>
<tr>
<td>BC-C</td>
<td>●</td>
<td></td>
<td>SFA-C</td>
<td>●</td>
<td></td>
</tr>
<tr>
<td>BC-750</td>
<td>●</td>
<td></td>
<td>SFA-750</td>
<td>●</td>
<td></td>
</tr>
<tr>
<td>BC-1000</td>
<td>●</td>
<td></td>
<td>SFA-1000</td>
<td>●</td>
<td></td>
</tr>
<tr>
<td>BC-1500</td>
<td>●</td>
<td></td>
<td>SFA-1500</td>
<td>●</td>
<td></td>
</tr>
<tr>
<td>BC-1500-WG</td>
<td>●</td>
<td></td>
<td>SFA-1500-WG</td>
<td>●</td>
<td></td>
</tr>
<tr>
<td>Chromate Primer</td>
<td></td>
<td></td>
<td>Surfing Film B</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CP-C</td>
<td>●</td>
<td></td>
<td>SFBC</td>
<td>●</td>
<td></td>
</tr>
<tr>
<td>CP-750</td>
<td>●</td>
<td></td>
<td>SFB-750</td>
<td>●</td>
<td></td>
</tr>
<tr>
<td>CP-1000</td>
<td>●</td>
<td></td>
<td>SFB-1000</td>
<td>●</td>
<td></td>
</tr>
<tr>
<td>CP-1500</td>
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<td></td>
<td>SFB-1500</td>
<td>●</td>
<td></td>
</tr>
<tr>
<td>CP-1500-WG</td>
<td>●</td>
<td></td>
<td>SFB-1500-WG</td>
<td>●</td>
<td></td>
</tr>
</tbody>
</table>

(1) Tolerance of ±0.25”

Figure 11
Control Test Panels:
No Environmental Exposure
3.3 Pre-Exposure Testing

Prior to subjecting the test panels to accelerated weathering, a set of control specimens, representing the composite/coating configurations described in the previous section, were mechanically tested to establish a baseline strength value. This testing was performed immediately after test panel fabrication to preclude any effects due to incidental environmental exposure. In composite laminate structures, tensile strength is considered a fiber-dominated property, while other properties, such as shear and compression, are matrix-dominated properties. UV radiation preferentially affects the polymer matrix, resulting in microcracking and matrix erosion. This decreases the load carrying capability of the matrix, reducing the overall strength of the composite laminate structure. In order to detect the effects of degradation due to environmental exposure, a standard mechanical test method (ASTM D 3518 \(^{[11]}\)) was chosen to evaluate the matrix integrity. ASTM D 3518 performs a standard tensile test (ASTM D 3039 \(^{[12]}\)) on a composite laminate comprised of layers with the fibers oriented at 45° (Reference Figure 12). When the test specimen of this configuration is loaded in tension, the orientation of the fibers creates a maximum shear stress, which is matrix-dominated property. Therefore, this test should identify any degradation in the matrix due to environmental exposure. Due to the inherently variable nature of mechanical properties in composite materials, eight specimens per composite coating configuration were tested to provide statistically significant data.
Figure 12
Fiber Orientation for ASTM 3518 Test Specimen

x (Loading Direction)

z and y represent the Specimen or Reference Axes, while 1 and 2 represent the Material or Local Axes.
3.4 Accelerated Weathering Exposure Testing

Each of the composite configurations described in the previous section were subjected to accelerated weathering exposure. Weathering was performed using an Atlas, Ci4000 Xenon Weatherometer which controls radiation wavelength (in the UV range), temperature, and humidity to simulate extended exposures to outdoor environmental conditions. The humidity in the chamber is created and maintained from a pressurized mixture of air and water, which creates a fine, moist fog that enters the test chamber through the floor vents. Test panels were oriented to ensure that only the coated surface was exposed. Each configuration was exposed for three different time durations: 750, 1000, and 1500 hours. Previous research indicated that the onset of degradation to UV exposure could occur in as little as 500 hrs \cite{5}. These exposure times were selected in an attempt to bound any degradation incurred by the composite material. Throughout the entire 1500-hour exposure, representative panels from each coating configuration were monitored for weight gain/loss at 72-hour intervals. Refer to Table 2 for a detailed test matrix.
The testing was conducted per standard test method, ASTM G 155\textsuperscript{[13]}, with the following parameters:

**Test Method:** ASTM G 155, Cycle 1

**Apparatus Type:** Xenon Arc Lamp

**Optical Filters:** Daylight

**Spectral Irradiance:** 0.55 W/m\^2 x nm (@ 340nm)

**Temperature:** 140 +/- 10° F

**Relative Humidity:** 50 +/- 5% RH

<table>
<thead>
<tr>
<th>Composite Configuration</th>
<th>Test Panel I.D.</th>
<th>Exposure Time (hr)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>0 (Control)</td>
</tr>
<tr>
<td>Bare Composite</td>
<td>BC-C</td>
<td>✓</td>
</tr>
<tr>
<td></td>
<td>BC-750</td>
<td>✓</td>
</tr>
<tr>
<td></td>
<td>BC-1000</td>
<td>✓</td>
</tr>
<tr>
<td></td>
<td>BC-1500</td>
<td>✓</td>
</tr>
<tr>
<td></td>
<td>BC-1500-WG</td>
<td>✓</td>
</tr>
<tr>
<td>Chromate Primer</td>
<td>CP-C</td>
<td>✓</td>
</tr>
<tr>
<td></td>
<td>CP-750</td>
<td>✓</td>
</tr>
<tr>
<td></td>
<td>CP-1000</td>
<td>✓</td>
</tr>
<tr>
<td></td>
<td>CP-1500</td>
<td>✓</td>
</tr>
<tr>
<td></td>
<td>CP-1500-WG</td>
<td>✓</td>
</tr>
<tr>
<td>Surfacing Film A</td>
<td>SFA-C</td>
<td>✓</td>
</tr>
<tr>
<td></td>
<td>SFA-750</td>
<td>✓</td>
</tr>
<tr>
<td></td>
<td>SFA-1000</td>
<td>✓</td>
</tr>
<tr>
<td></td>
<td>SFA-1500</td>
<td>✓</td>
</tr>
<tr>
<td></td>
<td>SFA-1500-WG</td>
<td>✓</td>
</tr>
<tr>
<td>Surfacing Film B</td>
<td>SFB-C</td>
<td>✓</td>
</tr>
<tr>
<td></td>
<td>SFB-750</td>
<td>✓</td>
</tr>
<tr>
<td></td>
<td>SFB-1000</td>
<td>✓</td>
</tr>
<tr>
<td></td>
<td>SFB-1500</td>
<td>✓</td>
</tr>
<tr>
<td></td>
<td>SFB-1500-WG</td>
<td>✓</td>
</tr>
</tbody>
</table>
3.5 Post Exposure Testing

3.5.1 Visual Micro-inspection

Upon completion of the accelerated exposure testing, the panels were examined visually for signs of degradation (i.e., matrix micro-cracking, polymer coating discoloration, etc.). The exposed specimens were compared to the baseline control specimens to determine the extent of degradation as a function of coating configuration and exposure time. Higher magnification images of the specimens (both exposed and unexposed) were obtained using a Hitachi S-4800 Scanning Electron Microscope (SEM) equipped with a field emission electron gun and an EDAX Energy Dispersive X-Ray (EDX) spectrometer. To increase the surface conductivity each specimen was sputter coated with Iridium using an Emitech K575X Peltier cooled coating sputter machine. Secondary electron images were generated using an electron beam with an acceleration voltage of 15keV, with magnification ranging from 50x to 25000x.

3.5.2 Mechanical Testing

Upon completion of the accelerated environmental exposure, each panel was subjected to mechanical testing per standard test method ASTM D 3518\textsuperscript{[11]}. As mentioned previously, this test creates a maximum shear stress in the specimen, which should identify degradation in the composite matrix due to the accelerated weathering. Subsequent to exposure, 0.25” was machined from the
perimeter of each test panel to eliminate any degradation incurred on the edges of the panels. For each coating configuration and exposure duration, eight individual specimens were tested. The edges of each specimen were lightly polished using 400 to 600-grit silicone carbide sand paper to remove any microstructural damage induced during machining. All mechanical testing was performed on a universal load machine utilizing a specimen grip length of 2.75” and a constant head speed of 0.05 in/min (2mm/min). No bonded tabs were required due to the relatively low failure strength expected. However, emery cloth was used to aid in gripping of the specimens during loading. During the test, load vs. cross head speed was monitored. Previous research \cite{5} has shown little effect on the elastic modulus of carbon/epoxy laminates as a result of environmental exposure. Therefore, strain measurements were not acquired. The mechanical test values were compared with the baseline strength values of unexposed specimens to determine the degradation effects of environmental exposure. Due to the difficulty of quantifying the contribution of the coating to the overall composite strength, this study utilized the ultimate load to evaluate the performance of each composite configuration as a function of exposure duration. The contribution of the coating to the shear strength made it difficult to Each test panel was machined into tensile specimens measuring 7.0” in length and 1.0” in width (Reference Figure 13). Due to the inherently variable nature of mechanical properties in composite materials, eight specimens were tested to produce a statistically significant value for strength. The strength values obtained for the
exposed test panels were compared to control panels to determine the extent of degradation.

Figure 13
In-Plane Shear Test Specimen Dimensions
4.0 RESULTS

4.1 Weight as a Function of Environmental Exposure Duration

In conjunction with the environmental exposure testing, small specimens representing each composite coating configuration (Bare, Chromate Primer Coated, Surfacing Film A, and Surfacing Film B) were monitored at 72 hour intervals to determine the weight gain or loss due to environmental exposure. As anticipated, the exposure resulted in a weight loss for each coating configuration indicating that material was being removed from the exposure surface. Furthermore, the decrease in weight continued up to the end of the 1500-hour test duration. It appears that weight loss would have continued had the exposure duration been extended. This coincides with the data presented by Kumar et al. \[5\] and reinforces the synergistic nature of UV and moisture exposure induced degradation of carbon/epoxy composites. The percentage weight loss vs. exposure time for each coating configuration is presented in Figure 14. The panels coated with Surfacing Film B exhibited the greatest amount of cumulative weight loss (-0.3%) while the weight of the panels coated with the chromate primer (-0.14%) was least affected by the environmental exposure.
Figure 14  
Percentage Weight Loss as a Function of Accelerated Environmental Exposure Duration

4.2 Visual Inspection of Specimens Subjected to Accelerated Environmental Exposure

Initial visual inspection of the exposed test panels did not reveal any obvious signs of degradation (i.e. cracking). However, all of the panels exhibited varying degrees of discoloration. The bare composite panels revealed a slight yellow tint post exposure while the chromate primer coated panels exhibited a chalky appearance with a slight dark discoloration. Furthermore, the panels coated with surfacing acquired a brownish tint, which became more pronounced with increasing exposure duration. Refer to Figures 15 through 18 for comparison of each coating configuration before and after environmental exposure.
Figure 15
Bare Composite
(A) No Exposure (B) 1500 Hrs Exposure

Figure 16
Chromate Primer Coated Composite
(A) No Exposure (B) 1500 Hrs Exposure
Figure 17
Surfacing Film A
(A) No Exposure (B) 1500 Hrs Exposure

Figure 18
Surfacing Film B
(A) No Exposure (B) 1500 Hrs Exposure
Figures 19 through 22 show SEM images of the Bare Carbon/Epoxy composite specimens before and after environmental exposure. Figure 19 indicates the formation of matrix microcracking, beginning with as low as 750 hours of exposure and increasing in severity with extended exposure duration. Figure 20 provides the same images at higher magnification (5000x). These images provide better detail of the change in surface topography due to epoxy matrix erosion. As noted in the previous figure, the extent of erosion is more pronounced with increasing exposure duration. Finally, Figure 22 depicts images focused on a bundle of carbon fiber tows. In comparing the images obtained before and after environmental exposure, it is clear that degradation in the form of matrix erosion has resulted increased surface visibility of the carbon fibers.

Images of the Carbon/Epoxy specimens coated with Chromate Primer are show in Figure 23. Comparison of the baseline specimen image (A) with the one obtained after 1500 hours of exposure (B) does reveals minimal signs of degradation. After exposure, the coating appears less continuous with a slightly more rough surface texture.

Figures 24 through 27 depict images obtained for the Carbon/Epoxy specimens coated with Surfacing Film. Examination of Figures 24 and 25 indicates the presence of the microcracking and matrix erosion that increases in severity with exposure duration. This is similar to the observations made from examination of the images generated of the Bare Carbon/Epoxy panels. Another interesting observation can be noted from examination of Figure 26, which
depicts Surfacing Film A after an exposure duration of 750 hours. The image shows several small fibers emanating from the coating surface. These fibers are likely from the non-woven polyester mat carrier used to support the epoxy resin during surfacing film manufacture. This was also observed in specimens coated with Surfacing Film B. Furthermore, cross section images of specimens coated with Surfacing Film A and B (Figure 27) indicate the fibers are only present after an exposure duration of 750 hours. The fact that these fibers are not present in the images of the surfacing film specimens taken at later exposure durations suggest they are degraded or removed during the exposure process.
Figure 19
Secondary Electron SEM Images (1000x) of Bare Carbon/Epoxy Composite
(A) No Exposure (B) 750 Hrs Exposure (C) 1000 Hrs Exposure (D) 1500 Hrs Exposure
Secondary Electron SEM Image (25000x) of Bare Carbon/Epoxy Composite after 1500 Hrs of Environmental Exposure
Figure 21
Secondary Electron SEM Images (5000x) of Bare Carbon/Epoxy Composite
(A) No Exposure (B) 750 Hrs Exposure (C) 1000 Hrs Exposure (D) 1500 Hrs Exposure

Figure 22
Secondary Electron SEM Images (50x) of Bare Carbon/Epoxy Composite
(A) No Exposure (B) 750 Hrs Exposure
Figure 23
Secondary Electron SEM Image (1000x) of Chromate Primer Coated Carbon/Epoxy Composite
(A) No Exposure (B) 1500 Hrs Exposure

Figure 24
Secondary Electron SEM Image (1000x) of Carbon/Epoxy Composite Coated With Surfacing Film A
(A) No Exposure (B) 1500 Hrs Exposure
Figure 25
Secondary Electron SEM Image (1000x) of Carbon/Epoxy Composite Coated With Surfacing Film B
(A) No Exposure (B) 1500 Hrs Exposure

Figure 26
Secondary Electron SEM Image (50x) of Carbon/Epoxy Composite Coated with Surfacing Film A – 750 Hrs Exposure
Figure 27
Cross Section Images (50x) of Carbon/Epoxy Specimens coated with (A) Surfacing Film A and (B) Surfacing Film B
4.3 Mechanical Test Results

Upon completion of the accelerated environmental exposure, each panel was subjected to mechanical testing per standard test method ASTM D 3518. There were no abnormal failures noted during coupon testing (i.e. coupon failed in the specimen grip area). The mechanical test values were compared with the baseline strength values of unexposed specimens to determine the degradation effects of environmental exposure.

The results of the mechanical testing are presented in Tables 3 through 6 as well as Figures 28-30. For the bare composite panels, a decrease in load carrying capability of approximately 6% (as compared with the baseline value) was observed after 750 hours of exposure. Examining the mechanical strength values for bare specimens exposed to 1000 and 1500-hour duration shows they are statistically equivalent (difference is within one standard deviation) to the values obtained after 750 hours of exposure. This indicates that no further degradation takes place in the bare carbon/epoxy composite panels after 750 hours of exposure.

Examining the data for the composite panels coated with chromate primer, there was no statistically significant decrease in load carrying capability (as compared with the baseline value) observed after 750 hours of exposure. The mechanical strength values obtained for panels subjected to 1000 and 1500 hour duration revealed a decrease of approximately 4% and 8%, respectively. This
indicated, as expected, an increased level of degradation with increasing exposure duration.

For the composite panels coated with Surfacing Film A, a decrease in load carrying capability of approximately 5% (as compared with the baseline value) was observed after 750 hours of exposure and a decrease of 12% (as compared with the baseline value) was observed after 1000 hours. Examining the mechanical strength values for specimens exposed to a 1500-hour duration shows they are statistically equivalent (difference is within one standard deviation) to the values obtained after 1000 hours of exposure. This indicates that no further degradation takes place in the carbon/epoxy composite panels coated with the Surfacing Film A after 1000 hours of exposure.

For the composite panels coated with Surfacing Film B, a decrease in load carrying capability of approximately 4% was observed after 750 hours (as compared with the baseline value). Examining the mechanical strength values for specimens exposed to 1000 and 1500-hour duration shows they are statistically equivalent (difference is within one standard deviation) to the values obtained after 750 hours of exposure. This indicates that no further degradation takes place in the carbon/epoxy composite panels coated with Surfacing Film B after 750 hours of exposure.

Figure 30 depicts the strength (ultimate load) of the carbon/epoxy composite material as a function of coating thickness and environmental exposure duration. Examination of this figure indicates that in most cases the
strength increases with increasing coating thickness. Although this trend is
generally upheld regardless of the exposure duration, a deviation is observed at
a coating thickness of 4 mils (corresponding to panels coated with Surfacing Film
A) for exposure times exceeding 750 hrs.
Table 3
Mechanical Strength Values No Exposure

<table>
<thead>
<tr>
<th>Composite Configuration</th>
<th>BC</th>
<th>CP</th>
<th>SFA</th>
<th>SFB</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean Max Load (lbf)</td>
<td>1100</td>
<td>1148</td>
<td>1212</td>
<td>1264</td>
</tr>
<tr>
<td>Standard Deviation</td>
<td>32</td>
<td>44</td>
<td>32</td>
<td>19</td>
</tr>
<tr>
<td>Coefficient of Variation (%)</td>
<td>2.9</td>
<td>3.8</td>
<td>2.7</td>
<td>1.5</td>
</tr>
</tbody>
</table>

Table 4
Mechanical Strength Values 750 Hr Exposure

<table>
<thead>
<tr>
<th>Composite Configuration</th>
<th>BC</th>
<th>CP</th>
<th>SFA</th>
<th>SFB</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean Max Load (lbf)</td>
<td>1045</td>
<td>1140</td>
<td>1157</td>
<td>1214</td>
</tr>
<tr>
<td>Standard Deviation</td>
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<tr>
<td>Coefficient of Variation (%)</td>
<td>2.3</td>
<td>2.4</td>
<td>2.3</td>
<td>2.0</td>
</tr>
<tr>
<td>Decrease in Load 1/</td>
<td>64</td>
<td>8</td>
<td>54</td>
<td>50</td>
</tr>
<tr>
<td>Percentage Decrease in Load</td>
<td>5.8</td>
<td>0.7</td>
<td>4.5</td>
<td>3.9</td>
</tr>
</tbody>
</table>

1/ Decrease is measured with respect to baseline value
### Table 5 Mechanical Strength Data 1000 Hr Exposure

<table>
<thead>
<tr>
<th>Composite Configuration Mean Max Load (lbf)</th>
<th>BC</th>
<th>CP</th>
<th>SFA</th>
<th>SFB</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standard Deviation</td>
<td>11</td>
<td>18</td>
<td>33</td>
<td>37</td>
</tr>
<tr>
<td>Coefficient of Variation (%)</td>
<td>1.0</td>
<td>1.6</td>
<td>3.1</td>
<td>3.1</td>
</tr>
<tr>
<td>Decrease in Load 1/</td>
<td>52</td>
<td>49</td>
<td>140</td>
<td>76</td>
</tr>
<tr>
<td>Percentage Decrease In Load</td>
<td>4.7</td>
<td>4.3</td>
<td>11.6</td>
<td>6.0</td>
</tr>
</tbody>
</table>

1/ Decrease is measured with respect to baseline value

### Table 6 Mechanical Strength Data 1500 Hr Exposure

<table>
<thead>
<tr>
<th>Composite Configuration Mean Max Load (lbf)</th>
<th>BC</th>
<th>CP</th>
<th>SFA</th>
<th>SFB</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standard Deviation</td>
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<td>35</td>
<td>24</td>
</tr>
<tr>
<td>Coefficient of Variation (%)</td>
<td>1.7</td>
<td>2.7</td>
<td>3.2</td>
<td>2.0</td>
</tr>
<tr>
<td>Decrease in Load 1/</td>
<td>59</td>
<td>88</td>
<td>135</td>
<td>57</td>
</tr>
<tr>
<td>Percentage Decrease In Load</td>
<td>5.3</td>
<td>7.7</td>
<td>11.2</td>
<td>4.5</td>
</tr>
</tbody>
</table>

1/ Decrease is measured with respect to baseline value
Figure 28 Ultimate Load vs. Exposure Time
(A) Bare Composite (B) Chromate Primer Coated Composite
(C) Surfacing Film A (D) Surfacing Film B
Figure 29
Ultimate Load as a Function of Coating Configuration and Exposure Duration

Figure 30
Ultimate Load as a Function of Coating Thickness and Exposure Duration
5.0 DISCUSSION

5.1 Environmentally Induced Degradation in Carbon/Epoxy Composite Material

Based on the mechanical results obtained from bare, uncoated panels subsequent to accelerated weathering, it is evident that exposure to UV radiation and moisture, in the presence of elevated temperature, results in degradation of the carbon/epoxy laminate structure. The mechanical test results as detailed in the previous section indicated a reduction in strength of approximately 6% (in comparison with bare unexposed specimens) after 750 hrs of exposure. This conclusion is supported by research performed by Kumar et al.\textsuperscript{[5]}, although the degree of mechanical degradation observed in this study was not as severe. This is likely due to the differences in laminate construction and test methods chosen to identify the extent of degradation.

The observed reduction in strength is primarily attributed to the formation of microcracks in the epoxy matrix in addition to extensive matrix erosion, both of which are caused by the synergistic effects of the simultaneous exposure to both UV radiation and moisture. Microcracks are a direct result of chain scission and crosslinking reactions initiated on the exposure surface by radiation from UV photons. Furthermore, the presence of microcracking can lead to increased moisture ingress, affecting the fiber-matrix bond integrity. All of these factors, when combined, result in a diminished capacity of the epoxy matrix to transfer applied loads to the fibers, ultimately reducing the overall strength of the
composite laminate.

The presence of extensive matrix erosion can be verified by examining the high magnification (SEM) images of the bare carbon/epoxy specimens before and after environmental exposure (Figures 19 through 22). The mechanism for matrix erosion, as suggested by Kumar et al., involves the creation of a thin surface layer of chemically modified epoxy, which is induced by UV radiation. Subsequent water condensation leaches away soluble degradation products, which exposes a fresh layer that can once again be attacked by UV radiation [5]. This mechanism is supported by the weight monitoring data obtained during this study, which demonstrated that the weight of the bare composite specimen continued to decline right up until the end of the exposure duration, thus indicating that material is being removed from the surface.

Several reaction mechanisms have been proposed by Kim et al., Kumar et al, and Musto et al [5,6,10] to explain the degradation in carbon/epoxy composites as a result of exposure to UV radiation and moisture. Exposure to UV radiation results in the formation of microcracks with the dominant mechanism being amide formation due to chain scission of the carbon-nitrogen bonds. Furthermore, combined exposure to both UV and moisture leads to extensive matrix erosion by removing the initial degradation reaction products, thus exposing a fresh surface for UV attack. While the chemical reactions that lead to degradation were not investigated in this study, visual and microscopic observations along with mechanical test data correspond well to previous
research carried out by Kim, Kumar and Musto [5,6,10]. Specifically, exposure to UV and moisture resulted in the formation of microcracks and the presence of matrix erosion, which lead to a decrease in mechanical strength.

5.2 Prevention of Degradation via Implementation of Polymer Based Coatings

This study also evaluated three different polymer-based coating systems at preventing degradation of the underlying carbon/epoxy substrate when subjected to environmental exposure. All of the systems were based on epoxy polymers. Two systems (Surfacing Film A and B) were applied during the initial cure of the carbon/epoxy laminate. The other system (chromate containing epoxy paint primer) was applied to the test panel after the laminate had been cured. As seen in Figure 30, all of the coating applications provided an increase in mechanical strength over the bare, uncoated laminates, with the more substantial improvement noted in the panels coated with surfacing film. This increase can generally be attributed to the presence of the additional cured epoxy resin layer on the surface of the specimens. The difference in strength between the chromate primer coated specimens and those coated with surfacing film can be attributed to the inclusion of polyester fibers in the form of a non-woven mat carrier. This mat is incorporated into the epoxy surfacing film during fabrication as a means of resin support. The combination of this nonwoven fiber carrier and epoxy resin can be viewed as a separate composite system that, when bonded to the carbon/epoxy substrate, provides additionally strength.
Review of the mechanical test data obtained from chromate primer coated panels indicated that the coating performed well initially, but progressively degraded with increased exposure duration. Specimens exposed for 750 hrs exhibited a statistically insignificant decline in strength in comparison to unexposed specimens with the same coating configuration. On the other hand, specimens exposed for longer durations exhibited a more significant reduction in strength (4% for specimens exposed to 1000 hours and 8% for specimens exposed to 1500 hours). This was as expected, as one would anticipate a greater reduction in strength with increasing exposure duration.

Of the two epoxy based surfacing films evaluated, Surfacing Film B performed the best in terms of degradation prevention. Examination of the mechanical test results revealed a small initial drop in strength after 750 hrs (4%) with no additional reduction in strength for subsequent exposure durations. Surfacing Film A exhibited a similar reduction in strength initially (5%), however, a sharp decline (12%) was observed after 1000 hrs of exposure. This sudden decline may be explained by the observations noted in Figure 26 and 27, which depict an SEM image taken of the Surfacing Film A specimen after 750 hrs of exposure and cross section images of specimens coated with Surfacing Film A and B after each exposure duration, respectively. These images reveal the presence of small fibers emanating from the coating surface for the both Surfacing Film A and B after 750 hours. These fibers are likely from the nonwoven polyester mat contained within the epoxy surfacing film. Upon
exposure to UV radiation in combination with moisture, erosion of the epoxy polymer film may have resulted in exposing the underlying polyester fibers. These fibers were not detected during the visual examination of specimens exposed to increased durations (refer to Figure 27), indicating they were degraded. Without these fibers in place, a reduction in strength would be expected. The fundamental difference in Surfacing Film A and B is the weight of the epoxy resin (i.e. the thickness) applied to the polyester mat. This would explain why the “heavier” weight Surfacing Film B did not experience a similar decline in strength after 1000 hrs exposure. The increased thickness of Surfacing Film B prevented the underlying polyester fibers from degrading to the extent observed in Surfacing Film A. It should also be noted that for the sharp decline in strength observed after 1000 hours of exposure for the Surfacing Film A coating, the strength values obtained fell only slightly below the minimum strength values obtained for the bare uncoated carbon/epoxy specimens. This indicates that the degradation may have penetrated through the surfacing film coating and into the underlying substrate. This does not coincide with the results obtained from research conducted by Kim et al, which determined that degradation was confined to a depth of approximately 6 μm from the exposure surface [6].

Visual inspection of the exposed surfacing film under magnification using SEM (Figures 24 and 25) reveals similar degradation characteristics exhibited in the bare carbon/epoxy composite specimens. These images indicate the formation of microcracks and, additionally, the change in surface morphology
signifies some level of matrix erosion. Despite the existence of these epoxy
degradation markers, the mechanical strength data demonstrates that the
underlying carbon/epoxy substrate is only marginally affected in the specimens
coated with Surfacing Film A and not affected at all in those coated with
Surfacing Film B.
6.0 SUMMARY AND RECOMMENDATIONS

This study aspired to investigate the degradation of carbon/epoxy composite materials induced by environmental exposure. Additionally, epoxy based coatings were examined to determine their effectiveness at preventing degradation to the underlying carbon/epoxy laminate. By exposing uncoated composite panels to accelerated weathering, and subsequently performing visual and mechanical tests, it was determined that degradation does occur. Combined exposure to UV radiation moisture results in matrix microcracking and erosion, which reduces the overall strength of carbon/epoxy composite. It was also determined that although applying a chromate containing epoxy based paint primer provides protection initially, the underlying carbon/epoxy substrate is eventually degraded with extended exposure duration. Alternatively, application of an epoxy based polymer film to the exposure surface during the initial laminate cure can prevent environmentally induced degradation from affecting the underlying carbon/epoxy substrate over extended exposure durations. Although similar degradation attributes were identified in the surfacing film as observed in the uncoated composite, it is likely that this degradation was either confined within the surfacing film layer (SF B) or only penetrated the very near surface of the carbon/epoxy substrate (SF A), as it did not result in a substantial reduction in mechanical strength.
The unexpectedly low mechanical strength results obtained for the unexposed bare composite specimens warrant further investigation. Research conducted by Kumar et al. \cite{5} indicated a reduction of approximately 29% in transverse tensile strength of carbon/epoxy composite material exposed to UV radiation and condensation. Although the disparity in values obtained in previous research and this study could be attributed to differences in laminate construction or test methods additional testing is recommended. Such testing should utilize various laminate configurations (i.e. thickness, fiber orientation) constructed from different materials forms (i.e., woven fabric, unidirectional tape) to evaluate the damage tolerance and durability of the composite specimen after environmental exposure. For example, carbon\epoxy composite panels subjected to environmental exposure could be tested for compressive residual strength per ASTM D7136/D7137 \cite{15,16}. Another approach could use ultrasonic non-destructive evaluation to monitor the damage (delamination initiated by an impact) growth characteristics as a function of environmental exposure duration. These tests may identify additional failure mechanisms, not identified in this study, that indicate environmentally induced degradation.
7.0 APPENDIX A: RAW TEST DATA
# Weight Monitoring Data

## Weight Loss (g) vs. Exposure Time

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<th>Exposure Time</th>
<th>Specimen I.D.</th>
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<th>CP-1500-WG</th>
<th>SF3-1500-WG</th>
<th>SF4-1500-WG</th>
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<th>CP-1500-WG</th>
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<th>SF4-1500-WG</th>
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<th>Cross Section Area (in²)</th>
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Mean Peak Load (lbf) 1057
Standard Deviation 11
Coefficient of Variation (%) 1.0
Decrease in Mean Max Load from Baseline (lbf) 52
Percentage Decrease in Mean Max Load from Baseline 4.7

### Test Panel I.D. - BC-1500
Panel Description - Bare Composite, 1500 Hour Exposure

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<th>Coupon I.D.</th>
<th>Width (in)</th>
<th>Thickness (in)</th>
<th>Cross Section Area (in²)</th>
<th>Peak Tensile Load (lbf)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BC-1500-1</td>
<td>1.000</td>
<td>0.031</td>
<td>0.0310</td>
<td>1056</td>
</tr>
<tr>
<td>BC-1500-2</td>
<td>0.997</td>
<td>0.031</td>
<td>0.0309</td>
<td>1046</td>
</tr>
<tr>
<td>BC-1500-3</td>
<td>0.997</td>
<td>0.030</td>
<td>0.0299</td>
<td>1025</td>
</tr>
<tr>
<td>BC-1500-4</td>
<td>0.998</td>
<td>0.031</td>
<td>0.0309</td>
<td>1063</td>
</tr>
<tr>
<td>BC-1500-5</td>
<td>0.999</td>
<td>0.031</td>
<td>0.0310</td>
<td>1042</td>
</tr>
<tr>
<td>BC-1500-6</td>
<td>1.000</td>
<td>0.031</td>
<td>0.0310</td>
<td>1083</td>
</tr>
<tr>
<td>BC-1500-7</td>
<td>0.995</td>
<td>0.031</td>
<td>0.0308</td>
<td>1040</td>
</tr>
<tr>
<td>BC-1500-8</td>
<td>0.998</td>
<td>0.031</td>
<td>0.0309</td>
<td>1045</td>
</tr>
</tbody>
</table>

Mean Peak Load (lbf) 1050
Standard Deviation 17
Coefficient of Variation (%) 1.7
Decrease in Mean Max Load from Baseline (lbf) 59
Percentage Decrease in Mean Max Load from Baseline 5.3
### Chromate Primer Coating Mechanical Test Data

#### Test Panel I.D. - CP-C
<table>
<thead>
<tr>
<th>Panel Description - Chromated Primer Coated Composite, No Exposure</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Coupon I.D.</strong></td>
</tr>
<tr>
<td>-----------------</td>
</tr>
<tr>
<td>CP-C-1</td>
</tr>
<tr>
<td>CP-C-2</td>
</tr>
<tr>
<td>CP-C-3</td>
</tr>
<tr>
<td>CP-C-4</td>
</tr>
<tr>
<td>CP-C-5</td>
</tr>
<tr>
<td>CP-C-6</td>
</tr>
<tr>
<td>CP-C-7</td>
</tr>
<tr>
<td>CP-C-8</td>
</tr>
</tbody>
</table>

**Mean Peak Load (lbf)**: 1148
**Standard Deviation**: 44
**Coefficient of Variation (%)**: 3.8

#### Test Panel I.D. - CP-750
<table>
<thead>
<tr>
<th>Panel Description - Chromated Primer Coated Composite, 750 Hour Exposure</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Coupon I.D.</strong></td>
</tr>
<tr>
<td>-----------------</td>
</tr>
<tr>
<td>CP-750-1</td>
</tr>
<tr>
<td>CP-750-2</td>
</tr>
<tr>
<td>CP-750-3</td>
</tr>
<tr>
<td>CP-750-4</td>
</tr>
<tr>
<td>CP-750-5</td>
</tr>
<tr>
<td>CP-750-6</td>
</tr>
<tr>
<td>CP-750-7</td>
</tr>
<tr>
<td>CP-750-8</td>
</tr>
</tbody>
</table>

**Mean Peak Load (lbf)**: 1140
**Standard Deviation**: 27
**Coefficient of Variation (%)**: 2.4
**Decrease in Mean Max Load from Baseline (lbf)**: 8
**Percentage Decrease in Mean Max Load from Baseline**: 0.7
### Test Panel I.D. - CP-1000
**Panel Description - Chromated Primer Coated Composite, 1000 Hour Exposure**

<table>
<thead>
<tr>
<th>Coupon I.D.</th>
<th>Width (in)</th>
<th>Thickness (in)</th>
<th>Cross Section Area (in²)</th>
<th>Peak Tensile Load (lbf)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CP-1000-1</td>
<td>0.997</td>
<td>0.032</td>
<td>0.0319</td>
<td>1108</td>
</tr>
<tr>
<td>CP-1000-2</td>
<td>0.996</td>
<td>0.033</td>
<td>0.0329</td>
<td>1103</td>
</tr>
<tr>
<td>CP-1000-3</td>
<td>0.996</td>
<td>0.034</td>
<td>0.0338</td>
<td>1086</td>
</tr>
<tr>
<td>CP-1000-4</td>
<td>0.996</td>
<td>0.033</td>
<td>0.0329</td>
<td>1121</td>
</tr>
<tr>
<td>CP-1000-5</td>
<td>0.999</td>
<td>0.033</td>
<td>0.0330</td>
<td>1067</td>
</tr>
<tr>
<td>CP-1000-6</td>
<td>0.996</td>
<td>0.032</td>
<td>0.0319</td>
<td>1119</td>
</tr>
<tr>
<td>CP-1000-7</td>
<td>0.993</td>
<td>0.033</td>
<td>0.0328</td>
<td>1099</td>
</tr>
<tr>
<td>CP-1000-8</td>
<td>1.000</td>
<td>0.033</td>
<td>0.0330</td>
<td>1089</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Mean Peak Load (lbf)</th>
<th>1099</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standard Deviation</td>
<td>18</td>
</tr>
<tr>
<td>Coefficient of Variation (%)</td>
<td>1.6</td>
</tr>
<tr>
<td>Decrease in Mean Max Load from Baseline (lbf)</td>
<td>49</td>
</tr>
<tr>
<td>Percentage Decrease in Mean Max Load from Baseline</td>
<td>4.3</td>
</tr>
</tbody>
</table>

### Test Panel I.D. - CP-1500
**Panel Description - Chromated Primer Coated Composite, 1500 Hour Exposure**

<table>
<thead>
<tr>
<th>Coupon I.D.</th>
<th>Width (in)</th>
<th>Thickness (in)</th>
<th>Cross Section Area (in²)</th>
<th>Peak Tensile Load (lbf)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CP-1500-1</td>
<td>1.003</td>
<td>0.032</td>
<td>0.0321</td>
<td>1091</td>
</tr>
<tr>
<td>CP-1500-2</td>
<td>1.003</td>
<td>0.033</td>
<td>0.0331</td>
<td>1051</td>
</tr>
<tr>
<td>CP-1500-3</td>
<td>1.005</td>
<td>0.033</td>
<td>0.0332</td>
<td>1085</td>
</tr>
<tr>
<td>CP-1500-4</td>
<td>1.005</td>
<td>0.032</td>
<td>0.0322</td>
<td>1077</td>
</tr>
<tr>
<td>CP-1500-5</td>
<td>1.002</td>
<td>0.032</td>
<td>0.0321</td>
<td>1035</td>
</tr>
<tr>
<td>CP-1500-6</td>
<td>1.003</td>
<td>0.033</td>
<td>0.0331</td>
<td>1086</td>
</tr>
<tr>
<td>CP-1500-7</td>
<td>1.000</td>
<td>0.033</td>
<td>0.0330</td>
<td>1016</td>
</tr>
<tr>
<td>CP-1500-8</td>
<td>1.002</td>
<td>0.032</td>
<td>0.0321</td>
<td>1039</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Mean Peak Load (lbf)</th>
<th>1060</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standard Deviation</td>
<td>28</td>
</tr>
<tr>
<td>Coefficient of Variation (%)</td>
<td>2.7</td>
</tr>
<tr>
<td>Decrease in Mean Max Load from Baseline (lbf)</td>
<td>88</td>
</tr>
<tr>
<td>Percentage Decrease in Mean Max Load from Baseline</td>
<td>7.7</td>
</tr>
</tbody>
</table>
## Surfacing Film A Mechanical Test Data

### Test Panel I.D. - SFA-C
Panel Description - 0.035 psf Surfacing Film, No Exposure

<table>
<thead>
<tr>
<th>Coupon I.D.</th>
<th>Width (in)</th>
<th>Thickness (in)</th>
<th>Cross Section Area (in²)</th>
<th>Peak Tensile Load (lbf)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SF3-C-1</td>
<td>0.999</td>
<td>0.037</td>
<td>0.0364</td>
<td>1241</td>
</tr>
<tr>
<td>SF3-C-2</td>
<td>1.004</td>
<td>0.036</td>
<td>0.0361</td>
<td>1155</td>
</tr>
<tr>
<td>SF3-C-3</td>
<td>1.000</td>
<td>0.036</td>
<td>0.0360</td>
<td>1225</td>
</tr>
<tr>
<td>SF3-C-4</td>
<td>1.000</td>
<td>0.036</td>
<td>0.0355</td>
<td>1227</td>
</tr>
<tr>
<td>SF3-C-5</td>
<td>1.001</td>
<td>0.036</td>
<td>0.0360</td>
<td>1239</td>
</tr>
<tr>
<td>SF3-C-6</td>
<td>1.001</td>
<td>0.036</td>
<td>0.0355</td>
<td>1195</td>
</tr>
<tr>
<td>SF3-C-7</td>
<td>1.002</td>
<td>0.036</td>
<td>0.0356</td>
<td>1177</td>
</tr>
<tr>
<td>SF3-C-8</td>
<td>1.003</td>
<td>0.035</td>
<td>0.0351</td>
<td>1235</td>
</tr>
</tbody>
</table>

| Mean Peak Load (lbf) | 1212 |
| Standard Deviation   | 32   |
| Coefficient of Variation (%) | 2.7 |

### Test Panel I.D. - SFA-750
Panel Description - 0.035 psf Surfacing Film, 750 Hour Exposure

<table>
<thead>
<tr>
<th>Coupon I.D.</th>
<th>Width (in)</th>
<th>Thickness (in)</th>
<th>Cross Section Area (in²)</th>
<th>Peak Tensile Load (lbf)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SF3-750-1</td>
<td>0.999</td>
<td>0.034</td>
<td>0.0340</td>
<td>1169</td>
</tr>
<tr>
<td>SF3-750-2</td>
<td>1.000</td>
<td>0.034</td>
<td>0.0340</td>
<td>1156</td>
</tr>
<tr>
<td>SF3-750-3</td>
<td>0.995</td>
<td>0.034</td>
<td>0.0338</td>
<td>1197</td>
</tr>
<tr>
<td>SF3-750-4</td>
<td>1.002</td>
<td>0.034</td>
<td>0.0341</td>
<td>1122</td>
</tr>
<tr>
<td>SF3-750-5</td>
<td>0.997</td>
<td>0.034</td>
<td>0.0339</td>
<td>1168</td>
</tr>
<tr>
<td>SF3-750-6</td>
<td>0.997</td>
<td>0.034</td>
<td>0.0339</td>
<td>1166</td>
</tr>
<tr>
<td>SF3-750-7</td>
<td>0.996</td>
<td>0.035</td>
<td>0.0349</td>
<td>1114</td>
</tr>
<tr>
<td>SF3-750-8</td>
<td>0.998</td>
<td>0.034</td>
<td>0.0339</td>
<td>1167</td>
</tr>
</tbody>
</table>

| Mean Peak Load (lbf) | 1157 |
| Standard Deviation   | 27   |
| Coefficient of Variation (%) | 2.3 |
| Decrease in Mean Max Load from Baseline (lbf) | 54 |
| Percentage Decrease in Mean Max Load from Baseline | 4.5 |
### Test Panel I.D. - SFA-1000
Panel Description - 0.035 psf Surfacing Film, 1000 Hour Exposure

<table>
<thead>
<tr>
<th>Coupon I.D.</th>
<th>Width (in)</th>
<th>Thickness (in)</th>
<th>Cross Section Area (in²)</th>
<th>Peak Tensile Load (lbf)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SF3-1000-1</td>
<td>0.996</td>
<td>0.035</td>
<td>0.0349</td>
<td>1020</td>
</tr>
<tr>
<td>SF3-1000-2</td>
<td>0.999</td>
<td>0.035</td>
<td>0.0349</td>
<td>1070</td>
</tr>
<tr>
<td>SF3-1000-3</td>
<td>1.002</td>
<td>0.035</td>
<td>0.0351</td>
<td>1110</td>
</tr>
<tr>
<td>SF3-1000-4</td>
<td>1.001</td>
<td>0.034</td>
<td>0.0340</td>
<td>1119</td>
</tr>
<tr>
<td>SF3-1000-5</td>
<td>1.000</td>
<td>0.035</td>
<td>0.0350</td>
<td>1100</td>
</tr>
<tr>
<td>SF3-1000-6</td>
<td>1.000</td>
<td>0.035</td>
<td>0.0350</td>
<td>1063</td>
</tr>
<tr>
<td>SF3-1000-7</td>
<td>1.000</td>
<td>0.035</td>
<td>0.0350</td>
<td>1038</td>
</tr>
<tr>
<td>SF3-1000-8</td>
<td>0.998</td>
<td>0.035</td>
<td>0.0349</td>
<td>1051</td>
</tr>
</tbody>
</table>

Mean Peak Load (lbf) 1071
Standard Deviation 33
Coefficient of Variation (%) 3.1
Decrease in Mean Max Load from Baseline (lbf) 140
Percentage Decrease in Mean Max Load from 11.6

### Test Panel I.D. - SFA-1500
Panel Description - 0.035 psf Surfacing Film, 1500 Hour Exposure

<table>
<thead>
<tr>
<th>Coupon I.D.</th>
<th>Width (in)</th>
<th>Thickness (in)</th>
<th>Cross Section Area (in²)</th>
<th>Peak Tensile Load (lbf)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SF3-1500-1</td>
<td>0.997</td>
<td>0.035</td>
<td>0.0349</td>
<td>1003</td>
</tr>
<tr>
<td>SF3-1500-2</td>
<td>0.996</td>
<td>0.034</td>
<td>0.0339</td>
<td>1100</td>
</tr>
<tr>
<td>SF3-1500-3</td>
<td>0.996</td>
<td>0.035</td>
<td>0.0349</td>
<td>1069</td>
</tr>
<tr>
<td>SF3-1500-4</td>
<td>0.995</td>
<td>0.036</td>
<td>0.0353</td>
<td>1072</td>
</tr>
<tr>
<td>SF3-1500-5</td>
<td>1.003</td>
<td>0.035</td>
<td>0.0351</td>
<td>1059</td>
</tr>
<tr>
<td>SF3-1500-6</td>
<td>0.996</td>
<td>0.034</td>
<td>0.0339</td>
<td>1099</td>
</tr>
<tr>
<td>SF3-1500-7</td>
<td>0.998</td>
<td>0.036</td>
<td>0.0359</td>
<td>1103</td>
</tr>
<tr>
<td>SF3-1500-8</td>
<td>1.004</td>
<td>0.035</td>
<td>0.0351</td>
<td>1107</td>
</tr>
</tbody>
</table>

Mean Peak Load (lbf) 1077
Standard Deviation 35
Coefficient of Variation (%) 3.2
Decrease in Mean Max Load from Baseline (lbf) 135
Percentage Decrease in Mean Max Load from 11.2
# Surfacing Film B Mechanical Test Data

## Test Panel I.D. - SFB-C
Panel Description - 0.045 psf Surfacing Film, No Exposure

<table>
<thead>
<tr>
<th>Coupon I.D.</th>
<th>Width (in)</th>
<th>Thickness (in)</th>
<th>Cross Section Area (in(^2))</th>
<th>Peak Tensile Load (lbf)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SF4-C-1</td>
<td>1.001</td>
<td>0.037</td>
<td>0.0370</td>
<td>1280</td>
</tr>
<tr>
<td>SF4-C-2</td>
<td>0.998</td>
<td>0.038</td>
<td>0.0374</td>
<td>1261</td>
</tr>
<tr>
<td>SF4-C-3</td>
<td>1.000</td>
<td>0.038</td>
<td>0.0375</td>
<td>1298</td>
</tr>
<tr>
<td>SF4-C-4</td>
<td>1.003</td>
<td>0.038</td>
<td>0.0376</td>
<td>1248</td>
</tr>
<tr>
<td>SF4-C-5</td>
<td>1.001</td>
<td>0.038</td>
<td>0.0375</td>
<td>1257</td>
</tr>
<tr>
<td>SF4-C-6</td>
<td>1.001</td>
<td>0.038</td>
<td>0.0375</td>
<td>1269</td>
</tr>
<tr>
<td>SF4-C-7</td>
<td>1.002</td>
<td>0.037</td>
<td>0.0371</td>
<td>1235</td>
</tr>
<tr>
<td>SF4-C-8</td>
<td>1.003</td>
<td>0.038</td>
<td>0.0376</td>
<td>1260</td>
</tr>
</tbody>
</table>

| Mean Peak Load (lbf) | 1264 |
| Standard Deviation   | 19   |
| Coefficient of Variation (%) | 1.5 |

## Test Panel I.D. - SFB-750
Panel Description - 0.045 psf Surfacing Film, 750 Hour Exposure

<table>
<thead>
<tr>
<th>Coupon I.D.</th>
<th>Width (in)</th>
<th>Thickness (in)</th>
<th>Cross Section Area (in(^2))</th>
<th>Peak Tensile Load (lbf)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SF4-750-1</td>
<td>1</td>
<td>0.036</td>
<td>0.036</td>
<td>1218</td>
</tr>
<tr>
<td>SF4-750-2</td>
<td>0.998</td>
<td>0.0377</td>
<td>0.0376246</td>
<td>1174</td>
</tr>
<tr>
<td>SF4-750-3</td>
<td>0.999</td>
<td>0.0365</td>
<td>0.0364635</td>
<td>1224</td>
</tr>
<tr>
<td>SF4-750-4</td>
<td>0.998</td>
<td>0.0364</td>
<td>0.0363272</td>
<td>1224</td>
</tr>
<tr>
<td>SF4-750-5</td>
<td>0.9985</td>
<td>0.0365</td>
<td>0.03644525</td>
<td>1253</td>
</tr>
<tr>
<td>SF4-750-6</td>
<td>0.995</td>
<td>0.0365</td>
<td>0.0363175</td>
<td>1223</td>
</tr>
<tr>
<td>SF4-750-7</td>
<td>0.997</td>
<td>0.0358</td>
<td>0.0356926</td>
<td>1185</td>
</tr>
<tr>
<td>SF4-750-8</td>
<td>1.0025</td>
<td>0.036</td>
<td>0.03609</td>
<td>1211</td>
</tr>
</tbody>
</table>

<p>| Mean Peak Load (lbf) | 1214 |
| Standard Deviation   | 25   |
| Coefficient of Variation (%) | 2.0 |
| Decrease in Mean Max Load from Baseline (lbf) | 50 |
| Percentage Decrease in Mean Max Load from Baseline | 3.9 |</p>
<table>
<thead>
<tr>
<th>Coupon I.D.</th>
<th>Width (in)</th>
<th>Thickness (in)</th>
<th>Cross Section Area (in²)</th>
<th>Peak Tensile Load (lbf)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SF4-1000-1</td>
<td>0.997</td>
<td>0.036</td>
<td>0.0359</td>
<td>1242</td>
</tr>
<tr>
<td>SF4-1000-2</td>
<td>1.012</td>
<td>0.037</td>
<td>0.0374</td>
<td>1184</td>
</tr>
<tr>
<td>SF4-1000-3</td>
<td>1.000</td>
<td>0.037</td>
<td>0.0370</td>
<td>1227</td>
</tr>
<tr>
<td>SF4-1000-4</td>
<td>1.000</td>
<td>0.037</td>
<td>0.0370</td>
<td>1149</td>
</tr>
<tr>
<td>SF4-1000-5</td>
<td>0.998</td>
<td>0.037</td>
<td>0.0369</td>
<td>1210</td>
</tr>
<tr>
<td>SF4-1000-6</td>
<td>0.999</td>
<td>0.038</td>
<td>0.0380</td>
<td>1135</td>
</tr>
<tr>
<td>SF4-1000-7</td>
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<td>0.0370</td>
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<tr>
<td>SF4-1000-8</td>
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<td>0.036</td>
<td>0.0360</td>
<td>1170</td>
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Mean Peak Load (lbf): 1187
Standard Deviation: 37
Coefficient of Variation (%): 3.1
Decrease in Mean Max Load from Baseline (lbf): 76
Percentage Decrease in Mean Max Load from: 6.0

---

<table>
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<tr>
<th>Coupon I.D.</th>
<th>Width (in)</th>
<th>Thickness (in)</th>
<th>Cross Section Area (in²)</th>
<th>Peak Tensile Load (lbf)</th>
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</table>

Mean Peak Load (lbf): 1206
Standard Deviation: 24
Coefficient of Variation (%): 2.0
Decrease in Mean Max Load from Baseline (lbf): 57
Percentage Decrease in Mean Max Load from: 4.5
APPENDIX B: SUPPORTING DOCUMENTATION
Designation: D 3518/D 3518M – 94 (Reapproved 2007)

Standard Test Method for
In-Plane Shear Response of Polymer Matrix Composite Materials by Tensile Test of a ±45° Laminate

1. Scope

1.1 This test method determines the in-plane shear response of polymer matrix composite materials reinforced by high-modulus fibers. The composite material form is limited to a continuous-fiber-reinforced composite ±45° laminate capable of being tension tested in the laminate x direction.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1.3 The values stated in either SI units or inch-pound units are to be regarded separately as standard. Within the text the inch-pound units are shown in brackets. The values stated in each system are not exact equivalents; therefore, each system must be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

2. Terminology

2.1 Definitions—Terminology D 3878 defines terms relating to high-modulus fibers and their composites. Terminology D 883 defines terms relating to plastics. Terminology E 256 and Practice E 177 define terms relating to statistics. In the event of a conflict between terms, Terminology D 3878 shall have precedence over the other standards.

2.2 Definitions of Terms Specific to This Standard:

2.2.1 ±45° laminate—in laminated composites, a balanced, symmetric lay-up composed only of +45° plies and −45° plies. (See also ply orientation.)

2.2.2 balanced, adj.—in laminated composite, having, for every off-axis ply oriented at +θ, another ply oriented at −θ that is of the same material system and form.

2.2.3 lamina, m—pl. laminae, in laminated composites, a single, thin, uniform layer that is the basic building block of a laminate. (Syn. ply)

2.2.4 material coordinate system, m—in laminated composites, a 123 Cartesian coordinate system describing the principle material coordinate system for a laminated material, where the
l-axis is aligned with the ply principal axis, as illustrated in Fig. 1. (See also ply orientation, ply principal axis, and principal material coordinate system.)

3.2.5 Nominal value, n—a value, existing in name only, assigned to a measurable property for the purpose of convenient designation. Tolerances may be applied to a nominal value to define an acceptable range for the property.

3.2.6 Off-axis, adj.—in laminated composites, having a ply orientation that is neither 0 nor 90°.

3.2.7 Ply, n—in laminated composites, synonym for lamina.

3.2.8 Ply orientation, n—in laminated composites, the angle between a reference direction and the ply principal axis. The angle is expressed in degrees, greater than—90° but less than or equal to 90°, and is shown as a positive quantity when taken from the reference direction to the ply principal axis, following the right-hand rule.

3.2.8.1 Discussion—The reference direction is usually related to a primary load-carrying direction.

3.2.9 Ply principal axis, n—in laminated composites, the coordinate axis in the plane of each lamina that defines the ply orientation. (See also ply orientation and material coordinate system.)

3.2.9.1 Discussion—The ply principal axis will, in general, be different for each ply of a laminate. The angle that this axis makes relative to a reference axis is given by the ply orientation. The convention is to align the ply principal axis with the direction of maximum stiffness (for example, the fiber direction of unidirectional tape or the warp direction of fabric-reinforced material).

3.2.10 Principal material coordinate system, n—a coordinate system having axes that are normal to planes of symmetry within the material. (See also material coordinate system.)

3.2.10.1 Discussion—Common usage; at least for Cartesian coordinate systems (for example, 123 or xy2), aligns the first axis of the principal material coordinate system with the direction of highest property value; for elastic properties, the axis of greatest elastic modulus is aligned with the 1 or x axes.

3.2.11 Symmetric, adj.—in laminated composites, when the constituents, material form, and orientation for the plies located on one side of the laminate midplane are the mirror image of the plies on the other side of the midplane.

3.2.12 Transition region, n—a strain region of a stress-strain or strain-strain curve over which a significant change in the slope of the curve occurs within a small strain range.

3.2.12.1 Discussion—Many filamentary composite materials exhibit a nonlinear stress-strain response during loading, such as seen in plots of either longitudinal stress versus longitudinal strain or transverse strain versus longitudinal strain. In certain cases, the nonlinear response may be conveniently approximated by a linear fit. There are varying physical reasons for the existence of a transition region. Common examples include matrix cracking under tensile loading and ply delamination.

3.3 Symbols:

3.3.1 A—cross-sectional area of a coupon.

3.3.2 \( \sigma \)—coefficient of variation statistic of a sample population for a given property (in percent).

3.3.3 \( F_{12} \) (offset)—the value of the \( F_{12} \) shear stress at the intersection of the shear chord modulus of elasticity and the stress stress curve, when the modulus is offset along the shear strain axis from the origin by the reported strain offset value.

3.3.4 \( G_{12} \)—in-plane shear modulus of elasticity.

3.3.4.1 Discussion—Indices 1 and 2 indicate the fiber direction and transverse to the fiber direction in the plane of the ply, respectively, as illustrated in Fig. 2.

3.3.5 n—number of coupons per sample population.

3.3.6 P—load carried by test coupon.

3.3.7 \( P_{0} \)—the load carried by test coupon that is the lesser of the (1) maximum load before failure or (2) load at 5% shear strain.

3.3.8 \( \sigma_n \)—standard deviation statistic of a sample population for a given property.

3.3.9 \( \bar{X} \)—mean (or average) of a sample population for a given property.

3.3.10 \( \sigma \)—general symbol for strain, whether normal strain or shear strain.

3.3.11 \( e \)—indicated normal strain from strain transducer or extensometer.

3.3.12 \( F_{12} \) shear stress on the plane perpendicular to the l-axis that axes parallel to the 2-axis.
3.3.14 $\tau_{12}^f$—the calculated value of the $\tau_{12}$ shear stress taken at the lesser of (1) maximum shear stress before failure or (2) shear stress at 5% shear strain.

3.3.15 $\sigma_{12}$—shear strain on the plane perpendicular to the 1-axis that acts parallel to the 2-axis.

3.3.16 $\sigma_{12}^f$—the value of the $\sigma_{12}$ shear strain at the maximum shear stress before failure, or 5%, whichever is less.

4. Summary of Test Method

4.1 A uniaxial tension test of a $\pm45^\circ$ laminate is performed in accordance with Test Method D 3039, although with specific restrictions on stacking sequence and thickness. Use of this test for evaluation of in-plane shear properties was originally proposed by Petit and was later improved by Rosen. Using expressions derived from laminael plate theory, the in-plane shear stress in the material coordinate system is directly calculated from the applied axial load, and the related shear stress is determined from longitudinal and transverse normal strain data obtained by transducers. This data is used to create an in-plane shear stress-shear strain curve.

5. Significance and Use

5.1 This test method is designed to produce in-plane shear property data for material specifications, research and development, quality assurance, and structural design and analysis. Factors that influence the shear response and should therefore be reported include the following: material, methods of material preparation and lay-up, specimen stacking sequence and overall thickness, specimen preparation, specimen conditioning, environment of testing, specimen alignment and gripping, speed of testing, time at temperature, load content, and volume percent reinforcement. Properties that may be derived from this test method include the following:

5.1.1 In-plane shear stress versus shear strain response.
5.1.2 In-plane shear modulus of elasticity.
5.1.3 Off-axis shear properties.
5.1.4 Maximum in-plane shear stress for a $\pm45^\circ$ laminate, and
5.1.5 Maximum in-plane shear strain for a $\pm45^\circ$ laminate.

6. Interferences

6.1 Impurity of Stress Field—The material in the gauge section of this specimen is not in a state of pure in-plane shear stress, as an in-plane normal stress component is present throughout the gauge section and a complex stress field is present close to the free edges of the specimen. Although this test method is designed to provide reliable initial material response and can establish shear stress-shear strain response values in the nonlinear region, the calculated shear stress values at failure do not represent true material strength values and should only be used with caution. Despite attempts to minimize these effects, the shear stress at failure obtained from this test method, even for otherwise identical materials that differ only in cured ply thickness or fabric area weight, may have differing failure modes and may not be able to be statistically pooled. The technical basis for the further discussion below is taken from the work by D. L. Keller et al.

6.2 Interferences—Of particular concern in the in-plane stress component normal to the fiber direction. This component of stress is present in all plies and throughout the gauge section of the specimen. The effect of this stress on a given ply is minimized by the fiber reinforcement of the neighboring plies. Since the ply constraint is reduced with increasing ply thickness, the thickness of the individual plies is an important parameter that influences both the shear stress-shear strain response and the ultimate failure load of this specimen. Moreover, the surface plies of a given specimen, being constrained by only one neighboring ply (as opposed to interior plies, which are constrained by a ply on each side), represent the weakest link in a $\pm45^\circ$ specimen. During the tensile loading of this test coupon, the first ply failures consist primarily of normal stress (or mixed mode) failures, rather than pure shear failures. Because of this, the actual material shear strength cannot be obtained from this test. Except for the case of materials capable of sustaining large axial test coupon strains (greater than about 5%), the shear stress at failure is believed to underestimate the actual material shear strength.

6.3 Interference of Load—As a result of the failure processes discussed above, the shear stress-shear strain response at higher stress levels depends upon the total number of plies. As the total number of plies in the specimen configuration is increased, the relative contribution of the two weak surface plies to the total load-carrying capacity is decreased. After the surface plies of the laminate fail, their portion of the load is redistributed to the remainder of the intact plies. The higher the total number of plies, the greater the chance that the remaining plies will be able to carry the load without immediate ultimate failure of the coupon. However, with each successive ply matrix failure the number of remaining intact plies diminishes, to the point where the applied load can no longer be carried. Because of this pattern, higher ply count specimens tend to achieve higher failure loads. To minimize these effects, this test method requires the use of a homogenous stacking sequence and requires a fixed number of plies, for which the only repeating plies are the two required for symmetry on opposite sides of the laminate midplane.

6.4 Effects of Large Deformation—Note that extreme fiber scissoring can occur in this specimen for the cases of ductile matrixes, weak fiber/matrix interfaces, thick specimens with a large number of repeated plies, or a combination of the above. Keller et al suggest that a general rule of thumb for this specimen is that a fiber rotation of $\pi$ takes place for every 2%
of axial strain (or every 3.5% shear strain for commonly tested materials). Such fiber scissoring, if left unbounded, would lead to an unacceptable violation of the assumptions in this test method of a nominal ±45° laminate. This is the principal rationale for terminating this test at a large strain level, even if load is still increasing on the specimen. This test method terminates data reporting at 5% calculated shear strain; this limit fiber eccentricity to about 1.5", is approximately the limit of foil strain gage technology (if used), and it is also well beyond the strain levels required for common engineering practice. Further details of the effects of stacking sequence, specimen geometry, and, in particular, specimen and ply thickness, are presented in the reference by Kelso, et al.

6.1.4 Effects of Edge Stresses—Even though interlaminar stresses reach a maximum value near the free edges of this laminate, the effect of interlaminar stresses on the failure process of ±45° laminates is insignificant when compared to the effect of the normal stress component transverse to the fiber direction in the plane of the specimen. Therefore, the effect of specimen width is much less important than stacking sequence and specimen thickness effects.

6.1.5 Effects of Axial Stress Nonuniformity—Both the shear stress and the shear modulus calculations depend upon the uniformity of the applied axial stress. Since the average applied load is used to calculate the shear stress, this will not necessarily correspond to the stress in the vicinity of the measured shear stress, unless the axial stress is uniform throughout the volume of the stressed material. Therefore, the greater the degree of material inhomogeneity, such as with coarsely woven fabrics or materials with significant axis-rich regions, the greater the potential for inaccuracies in the measured response.

6.2 Other—Additional sources of potential scatter in testing of composite materials are described in Test Method D 3039.

7. Apparatus

7.1 Apparatus shall be in accordance with Test Method D 3039. However, this test method requires that load-normal strain data be measured at both the longitudinal and transverse directions of the coupon.

8. Sampling and Test Specimens

8.1 Sampling—Sampling shall be in accordance with Test Method D 3039.

8.2 Geometry—The coupon geometry shall be in accordance with Test Method D 3039, as modified by the following:

8.2.1 The stacking sequence shall be [45/-45/], where 4 ≤ n ≤ 6 for unidirectional tape (16, 20, or 24 plies) and 2 ≤ n ≤ 4 for woven fabric 68, 12, or 16 plies). The recommended coupon width is 25 mm (1.0 in.), and the recommended coupon length range is 200 to 300 mm (8 to 12 in.) inclusive.

Note 2—Tapes, which are optional for the Test Method D 3039 D 309M test coupon, are normally not required for a successful conduct of this Practice.

8.3 Specimen Preparation—Specimen preparation shall be in accordance with Test Method D 3039/D 309M.

9. Calibration

9.1 Calibration shall be in accordance with Test Method D 3039/D 309M.

10. Conditioning

10.1 Conditioning shall be in accordance with Test Method D 3039/D 309M.

11. Procedure

11.1 Perform a tension test on the ±45° laminate coupon in accordance with Test Method D 3039/D 309M, with normal strain instrumentation in both longitudinal and transverse directions and continuous or nearly continuous load-normal strain data recording. If ultimate failure does not occur within 5% shear strain, the data shall be truncated to the 5% shear strain mark (see 6.1.3 for the explanation). When the data is truncated, for the purpose of calculation and reporting, this 5% shear strain point shall be considered the maximum shear stress. Any truncation of data shall be noted in the report.

12. Calculation

12.1 Maximum Shear Stress/Shear Strain—Calculate the maximum in-plane shear stress for the ±45° laminate using Eq 1 and report the results to three significant figures. The shear modulus is to be calculated, determine the shear stress at each required data point using Eq 2.

\[ \tau_s^{\text{max}} = \frac{P}{A} \]  

\[ \tau_s = \frac{P_1}{A} \]

where:

- \( \tau_s^{\text{max}} \) = maximum in-plane shear stress, MPa [psi];
- \( P \) = maximum load at or below 5% shear strain, N [lb];
- \( P_1 \) = load at 1-ft data point, N [lb]; and
- \( A \) = cross-sectional area in accordance with Test Method D 3039/D 309M, mm² [in.²].

12.2 Shear Stress/Maximum Shear Strain—If shear moduli or maximum shear strain is to be calculated, determine the shear strain at each required data point using Eq 3. The maximum shear strain is determined from Eq 4. Report the results to three significant figures.

\[ \gamma_{12} = \tau_s - \tau_s^{\text{max}} \]  

\[ \tau_{12}^{\text{max}} = \min \{\tau_{12}^{\text{max}} \text{ at maximum shear stress}\} \]

where:

- \( \gamma_{12} \) = shear strain at i-th data point, µ;
- \( \tau_s \) = longitudinal normal strain at i-th data point, µ;
- \( \tau_s^{\text{max}} \) = lateral normal strain at i-th data point, µ; and
- \( \tau_{12}^{\text{max}} \) = maximum shear strain, µ.

12.3 Shear Modulus of Elasticity—Calculate the shear modulus of elasticity using Eq 5, applied over a 4000 ± 200 µ shear strain range, starting with the lower strain point in the range of 1500 to 2500 µ, inclusive. Report the
chord shear modulus of elasticity to three significant figures. Also report the shear strain range used in the calculation. A graphical example of chord shear modulus is shown in Fig. 3.

Note 3—The shear strain range of 2000 to 6000 µε for shear modulus determination was selected, based on the shear response of a ±45° tensile coupon with a Poisson ratio near 0.3 to correspond approximately to the normal strain range of 1000 to 3000 µε used to report the tensile chord modulus of elasticity in Test Method D 3039/D 3039M.

12.3.1.1 A different strain range must be used for materials that fail or exhibit a transition region (a significant change in the slope of the stress-strain curve) before 6000 µε. In such cases, the upper strain range value for the sample population shall be determined after testing; defined as 50% of the average value of the upper limit of the essentially linear region, rounded down to the nearest 500 µε. Any presence of a transition region shall be reported, along with the strain range used.

\[
\frac{C_{12}^{\text{chord}}}{\Delta_{12}} = \frac{\Delta_{12}}{\Delta_{12}^{\text{chord}}}
\]

(5)

where:

- \(C_{12}^{\text{chord}}\) = shear chord modulus of elasticity, GPa [ksi],
- \(\Delta_{12}\) = difference in applied shear stress between the two shear strain points, MPa [ksi], and
- \(\Delta_{12}^{\text{chord}}\) = difference between the two shear strain points (nominally 0.04).

12.3.2 Shear Modulus of Elasticity (Other Definitions)—Other definitions of elastic moduli may be evaluated and reported at the user's discretion. If such data is generated and reported, also the definition used, the shear strain range used, and the results to three significant figures. Test Method E 111 provides additional guidance in the determination of modulus of elasticity.

Note 4—An example of another modulus definition is the secondary chord modulus of elasticity for materials that exhibit essentially bilinear stress-strain behavior.

12.4 Offset Shear Strength—If desired, an offset shear strength may be determined from the shear stress versus shear strain curve. Translate the shear chord modulus of elasticity line along the strain axis from the origin by a fixed strain value and extend this line until it intersects the stress-strain curve. Determine the shear stress that corresponds to the intersection point and report this value as the offset shear strength, along with the value of the offset strain, as in:

\[
F_{\text{os}} (0.2 \% \text{ offset}) = 70 \text{ MPa}
\]

(6)

A graphical example of offset shear strength is shown in Fig. 3.

Note 5—In the absence of evidence suggesting the use of a more appropriate value, an offset strain value of 0.2% is recommended.

12.5 Statistics—For each series of tests calculate the average value, standard deviation, and coefficient of variation (in percent) for each property determined:

\[
\bar{x} = \frac{\sum x_i}{n}
\]

(7)

\[
s_{x,1} = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}}
\]

(8)

\[
CV = 100 \times \frac{s_{x,1}}{\bar{x}}
\]

(9)

where:

- \(\bar{x}\) = sample mean (average),
- \(s_{x,1}\) = sample standard deviation,
- \(CV\) = sample coefficient of variation, in percent,
- \(n\) = number of specimens, and
- \(x_i\) = measured or derived property.

13. Report

13.1 The data reported with this test method include mechanical testing data, material identification data, fiber filler, and core material identification data and shall be in accordance with Guides E 1434, E 1309, and E 1471, respectively. Each data item discussed is identified as belonging to one of the following categories: (VT) required for reporting of valid test
result, (VM) required for valid material traceability, (RT) recommended for maximum test method traceability, (RM) recommended for maximum material traceability, or (O) for optional data items. At a minimum, the report shall include all (VT) category items from Guide E 1434.

3.1.1.1 Field A1, Test Method—The response shall be either "D3518 – 94" or "D3518M – 94," as appropriate.

3.1.1.2 Field A5, Type of Test—The response shall be "in-plane shear."

3.1.1.3 Field E2, Specimen Orientation—The response shall be "0.0."

3.1.1.4 Block E, Transducer Block—Used twice; once for each transducer.

3.1.1.5 Block F, Specimen Geometry Block—F6 (reinforcement volume) may be actual values, or it may be the average value for a sample F7 (overall length) and F8 (gage length) may be actual values, or they may be the nominal or average value for the sample. F9 (area) is the actual area in accordance with Test Method D 3039/D 3039M. F10 must also satisfy Test Method D 3039/D 3039M.

11.1.1.6 H2/K58, Progressive Damage Parameter—The response shall be "0.2% offset strength."

11.2 In addition to the data reported in accordance with Test Method D 3039/D 3039M, the report shall include the following information:

11.2.1 Theresolution level or date of issue of this test method.

11.2.2 Shear strain range used for chord shear modulus determination.

11.2.3 If another definition of shear modulus of elasticity is used in addition to chord shear modulus, describe the method used, the resulting correlation coefficient (if applicable), and the shear strain range used for the evaluation.

11.2.4 Individual values of shear modulus of elasticity, and the average, standard deviation, and coefficient of variation (in percent) values for the population.

11.2.5 Individual values of offset shear strength with the value of the offset strain, along with the average, standard deviation, and coefficient of variation (in percent) for the population. Note any test in which the failure load was less than the maximum load before failure.

11.2.6 Individual maximum shear stress, and the average, standard deviation, and coefficient of variation (in percent) for the population. Note any test that was truncated to 5% shear strain.

14. Precision and Bias

14.1 Precision—The data required for the development of a precision statement is not available for this test method.

14.2 Bias—Bias cannot be determined for this test method as no acceptable reference standard exists.

15. Keywords

15.1 composite materials; shear modulus; shear properties; shear strength

APPENDIX

(Nonmandatory Information)

XI. SIGNIFICANT POINTS OF MAJOR REVISIONS TO THIS TEST METHOD

XI.1 1991 Revision:

XI.1.1 Updated the format to conform to 1989 Form and Style for ASTM Standards.

XI.1.2 Changed the title and clarified the scope.

XI.1.3 Transformed the document from an inch-pound standard to a dual-units standard.

XI.1.4 Updated the Terminology section.

XI.1.5 Added a new Interferences section.

XI.1.6 Added a new Calibration section.

XI.2 1994 Revision:

XI.2.1 Updated the format to conform to current practices of Committee D 30.
X1.2.2. Relaxed the scope to allow usage of this test method on material forms reinforced by woven fabrics.

X1.2.3. Extensively updated the Interferences section to discuss difficulty with this test method in obtaining reliable values for ultimate shear strength, and generally replaced the modifier ultimate with the term maximum throughout the text, as appropriate.

X1.2.4. Added to the coupon geometry, limitations on the stacking sequence of the test specimen laminate, and provided recommended values for coupon length and width.

X1.2.5. Indirectly incurred several procedural changes, through changes to Test Method D 3039/D 3039M, including the approach to standard conditioning described by Test Method D 5229/D 5229M.

X1.2.6. Added the determinations and reporting of an offset shear strength.

X1.2.7. Shear modulus reporting now includes, at a minimum, a strain-range-based chord shear modulus and also requires documentation of the strain range used for any other modulus definitions reported.

X1.2.8. Shear stress/shear strain data has been truncated at 5% shear strain.

X1.2.9. Ultimate shear strength has been removed from this practice, replaced by reporting of maximum shear stress, determined as the maximum shear stress not exceeding 5% shear strain.

X1.2.10. New illustrations have been included.

X1.2.11. Data reporting now follows Guide E 1434.
Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials

1. Scope

1.1 This test method determines the in-plane tensile properties of polymer matrix composite materials reinforced by high-modulus fibers. The composite material forms are limited to continuous fiber or discontinuous fiber-reinforced composites in which the laminate is balanced and symmetric with respect to the test direction.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. Within the text, the inch-pound units are shown in brackets. The values stated in each system are not exact equivalents; therefore, each system must be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

D 792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement
D 883 Terminology Relating to Plastics
D 2584 Test Method for Ignition Loss of Cured Reinforced Resins
D 2734 Test Methods for Void Content of Reinforced Plastics
D 3171 Test Methods for Constituent Content of Composite Materials
D 3878 Terminology for Composite Materials

D 5229/D 5229M Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials
E 4 Practices for Force Verification of Testing Machines
E 5 Terminology Relating to Methods of Mechanical Testing
E 83 Practice for Verification and Classification of Extensometer Systems
E 11 Specification for Wire Cloth and Sieves for Testing Purposes
E 122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process
E 132 Practice Method for Poisson’s Ratio at Room Temperature
E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E 251 Test Methods for Performance Characteristics of Metallic Bonded Resistance Strain Gages
E 456 Terminology Relating to Quality and Statistics
E 1012 Practice for Verification of Test Frame and Specimen Alignment Under Tensile and Compressive Axial Force Application
E 1237 Guide for Installing Bonded Resistance Strain Gages

3. Terminology

3.1 Definitions—Terminology D 3878 defines terms relating to high-modulus fibers and their composites. Terminology D 883 defines terms relating to plastics. Terminology E 6 defines terms relating to mechanical testing. Terminology E 456 and Practice E 177 define terms relating to statistics. In the event of a conflict between terms, Terminology D 3878 shall have precedence over the other standards.

3.2 Definitions of Terms Specific to This Standard:

Note—If the term represents a physical quantity, its analytical dimensions are stated immediately following the term (or letter symbol) in fundamental dimension form, using the following ASTM standard symbology for fundamental dimensions, shown within square brackets: [M] for mass, [L] for length, [T] for time, [Θ] for thermodynamic temperature, and [N] for nondimensional quantities. Use of these symbols is
restricted to analytical dimensions when used with square brackets, as the symbols may have other definitions when used without the brackets.

3.2.1 nominal value, \( n \) — a value, existing in name only, assigned to a measurable property for the purpose of convenient designation. Tolerances may be applied to a nominal value to define an acceptable range for the property.

3.2.2 transition region, \( \tau \) — a strain region of a stress-strain or strain-strain curve over which a significant change in the slope of the curve occurs within a small strain range.

3.2.3 transition strain, \( \frac{\varepsilon_{\text{transition}}}{\text{max}} \), \( n \) — the strain value at the mid range of the transition region between the two essentially linear portions of a bilinear stress-strain or strain-strain curve.

3.2.3.1 Discussion — Many filamentary composite materials show essentially bilinear behavior during force application, such as seen in plots of either longitudinal stress versus longitudinal strain or transverse strain versus longitudinal strain. There are varying physical reasons for the existence of a transition region. Common examples include: matrix cracking under tensile force application and ply delamination.

3.3 Symbols:

- \( A \) — minimum cross-sectional area of a coupon.
- \( B \) — percent bending for a uniaxial coupon of rectangular cross section about \( y \) axis of the specimen (about the narrow direction).
- \( B \) — percent bending for a uniaxial coupon of rectangular cross section about \( z \) axis of the specimen (about the wide direction).
- \( C V \) — coefficient of variation statistic of a sample population for a given property (in percent).
- \( E \) — modulus of elasticity in the test direction.
- \( F_{\text{ult}} \) — ultimate tensile strength in the test direction.
- \( F_{\text{ult}}^{\text{shear}} \) — ultimate shear strength in the test direction.
- \( h \) — coupon thickness.
- \( L_e \) — extensional gage length.
- \( L_{\text{net}} \) — minimum required bonded tab length.
- \( m \) — number of coupons per sample population.
- \( P \) — force carried by test coupon.
- \( P_{\text{max}} \) — maximum force carried by test coupon at failure.
- \( S \) — standard deviation statistic of a sample population for a given property.
- \( w \) — coupon width.
- \( x \) — test result for an individual coupon from the sample population for a given property.

- \( \delta \) — mean or average (estimate of mean) of a sample population for a given property.
- \( \varepsilon \) — general symbol for strain, whether normal strain or shear strain.
- \( \sigma \) — normal stress.
- \( \nu \) — Poisson's ratio.

4. Summary of Test Method

4.1 A thin flat strip of material having a constant rectangular cross section is mounted in the grips of a mechanical testing machine and monotonically loaded in tension while recording the force. The ultimate strength of the material can be determined from the maximum force carried before failure. If the coupon strain is monitored with strain or displacement transducers then the stress-strain response of the material can be determined, from which the ultimate tensile strain, tensile modulus of elasticity, Poisson's ratio, and transition strain can be derived.

5. Significance and Use

5.1 This test method is designed to produce tensile property data for material specifications, research and development, quality assurance, and structural design and analysis. Factors that influence the tensile response and should therefore be reported include the following: material, methods of material preparation and lay-up, specimen stacking sequence, specimen preparation, specimen conditioning, environment of testing, specimen alignment and gripping, speed of testing, time at temperature, void content, and volume percent reinforcement. Properties, in the test direction, which may be obtained from this test method include the following:

- 5.1.1 Ultimate tensile strength,
- 5.1.2 Ultimate tensile strain,
- 5.1.3 Tensile chord modulus of elasticity,
- 5.1.4 Poisson's ratio, and
- 5.1.5 Transition strain.

6. Interferences

6.1 Material and Specimen Preparation — Poor material fabrication practices, lack of control of fiber alignment, and damage induced by improper coupon machining are known causes of high material data scatter in composites.

6.2 Gripping — A high percentage of grip-induced failures, especially when combined with high material data scatter, is an indicator of specimen gripping problems. Specimen gripping methods are discussed further in 7.2.4, 8.2, and 11.5.

6.3 System Alignment — Excessive bending will cause premature failure, as well as highly inaccurate modulus of elasticity determination. Every effort should be made to eliminate excess bending from the test system. Bending may occur as a result of misaligned grips or from specimens themselves if improperly installed in the grips or out-of-tolerance causes due to poor specimen preparation. If there is any doubt as to the alignment inherent in a given test machine, then the alignment should be checked as discussed in 7.2.5.

6.4 Edge Effects in Angle Ply Laminates — Premature failure and lower stiffnesses are observed as a result of edge softening in laminates containing off-axis plies. Because of this, the strength and modulus for angle ply laminates can be drastically underestimated. For quasi-isotropic laminates containing significant 0° plies, the effect is not as significant.

7. Apparatus

7.1 Micrometers — A micrometer with a 4 to 5 mm [0.16 to 0.20-in] nominal diameter double-ball interface shall be used to measure the thickness of the specimen. A micrometer with a flat anvil interface shall be used to measure the width of the specimen. The accuracy of the instruments shall be suitable.
for reading to within 1% of the sample width and thickness. For typical specimen geometries, an instrument with an accuracy of ±2.5 μm [±0.0001 in.] is adequate for thickness measurement, while an instrument with an accuracy of ±25 μm [±0.001 in.] is adequate for width measurement.

7.2 Testing Machine—The testing machine shall be in conformance with Practices E 4 and shall satisfy the following requirements:

7.2.1 Testing Machine Heads—The testing machine shall provide a stationary head and a moveable head.

7.2.2 Drive Mechanism—The testing machine drive mechanism shall be capable of imparting to the moveable head a controlled velocity with respect to the stationary head. The velocity of the moveable head shall be capable of being maintained as specified in 11.3.

7.2.3 Force Indicator—The testing machine force-sensing device shall be capable of indicating the total force being carried by the test specimen. This device shall be essentially free from inertia lag at the specified rate of testing and shall indicate the force with an accuracy over the force range(s) of interest of within ±1% of the indicated value. The force range(s) of interest may be fairly low for modulus evaluation, much higher for strength evaluation, or both, as required.

7.2.4 Grips—Each head of the testing machine shall carry one grip for holding the test specimen so that the direction of force applied to the specimen coincides with the longitudinal axis of the specimen. The grips shall apply sufficient lateral pressure to prevent slippage between the grip face and the coupon. If tabs are used the grips should be long enough that they overhang the beveled portion of the tab by approximately 10 to 15 mm (0.5 in.). It is highly desirable to use grips that are rotationally self-aligning to minimize bending stresses in the coupon.

7.2.5 System Alignment—Poor system alignment can be a major contributor to premature failures, to elastic property data scatter, or both. Practice E 1012 describes bending evaluation guidelines and describes potential sources of misalignment during tensile testing. In addition to Practice E 1012, the degree of bending in a tensile system can also be evaluated using the following related procedure. Specimen bending is considered separately in 11.6.1.

7.2.5.1 A rectangular alignment coupon, preferably similar in size and stiffness to the test specimen of interest, is instrumented with a minimum of three longitudinal strain gages of similar type, two on the front face across the width and one on the back face of the specimen, as shown in Fig. 1. Any difference in indicated strain between these gages during loading provides a measure of the amount of bending in the thickness plane (B₁) and width plane (B₂) of the coupon. The strain gage location should normally be located in the middle of the coupon gage section (if modulus determination is a concern), near a grip (if premature grip failures are a problem), or any combination of these areas.

7.2.5.2 When evaluating system alignment, it is advisable to perform the alignment check with the same coupon inserted in each of the four possible installation permutations (described relative to the initial position): initial (top-front facing observer), rotated back to front only (top-back facing observer), rotated end for end only (bottom front facing observer), and rotated both front to back and end to end (bottom back facing observer). These four data sets provide an indication of whether the bending is due to the system itself or to tolerance in the alignment check coupon or gaging.

7.2.5.3 The zero strain point may be taken either before gripping or after gripping. The strain response of the alignment coupon is subsequently monitored during the gripping process, the tensile loading process, or both. Eq. 1-3 use these indicated strains to calculate the ratio of the percentage of bending strain to average extensional strain for each bending plane of the alignment coupon and the total percent bending, B₅₀₉₁. Plotting percent bending versus axial average strain is useful in understanding trends in the bending behavior of the system.

7.2.5.4 Problems with failures during gripping would be reason to examine bending strains during the gripping process in the location near the grip. Concern over modulus data scatter would be reason to evaluate bending strains over the modulus evaluation force range for the typical transducer location. Excessive failures near the grips would be reason to evaluate bending strains near the grip at high loading levels. While the maximum allowable amount of system misalignment is material and location dependent, good testing practice is generally able to limit percent bending to a range of 3 to 5% at moderate

![FIG. 1 Gage Locations for System Alignment Check Coupon](image-url)
strain levels (>1000 µ`). A system showing excessive bending for the given application should be reajdusted or modified.

\[
B_y = \frac{\varepsilon_{ax} - \varepsilon_{ay}}{\varepsilon_{ay}} \times 100 \quad (1)
\]

\[
B_x = \frac{4\varepsilon_{ax} - \varepsilon_{ay}}{\varepsilon_{ay}} \times 100 \quad (2)
\]

where:

- \(B_y\) = percent bending about system \(y\) axis (about the narrow plane), as calculated by Eq 1, %;
- \(B_x\) = percent bending about system \(x\) axis (about the wide plane), as calculated by Eq 2, %;
- \(\varepsilon_{ax}, \varepsilon_{ay}\), and \(\varepsilon_{ay}\) = indicated longitudinal strains displayed by Gages 1, 2, and 3, respectively, of Fig. 1, pp.; and
- \(\varepsilon_{ave}\) = \((\varepsilon_{ax} + \varepsilon_{ay})/2 + \varepsilon_{ay})/2 \)

The total bending component is:

\[
\varepsilon_{ave} = |B_x + iB_y| \quad (3)
\]

Note 2—Experimental error may be introduced by sources such as poor system alignment, specimen preparation and strain gage precision and calibration. These sources of error may result in an average calculated strain (\(\varepsilon_{ave}\)) of 6, causing \(B_x\) and \(B_y\) (Eq 1 and Eq 1) to approach infinity as the average calculated strain is the denominator. To minimize the potential for this occurrence during system alignment evaluation, it is recommended that force be applied to the alignment coupon until all three strain gauges measure positive strain of no less than 500 µ` with an \(\varepsilon_{ave}\) of no less than 1000 µ`. If these conditions can not be met, the test configuration should be adjusted prior to performing further system alignment evaluation.

7.3 Strain-Indicating Device—Force-strain data, if required, shall be determined by means of either a strain transducer or an extensometer. Attachment of the strain-indicating device to the coupon shall not cause damage to the specimen surface. If Poisson's ratio is to be determined, the specimen shall be instrumented to measure strain in both longitudinal and lateral directions. The modulus of elasticity is to be determined, the longitudinal strain should be simultaneously measured on opposite faces of the specimen to allow for a correction as a result of any bending of the specimen (see 11.6 for further guidance).

7.3.1 Bonded Resistance Strain Gage Selection—Strain gage selection is a compromise based on the type of material. An active gage length of 6 mm [0.25 in.] is recommended for most materials. Active gage lengths should not be less than 3 mm [0.125 in.]. Gage calibration certification shall comply with Test Methods E 251. When testing woven fabric laminates, gage selection should consider the use of an active gage length that is at least as great as the characteristic repeating unit of the weave. Some guidelines on the use of strain gages on composites follow. A general reference on the subject is Tuttle and Brinson.4

7.3.1.1 Surface preparation of fiber-reinforced composites in accordance with Practice E 1237 can penetrate the matrix material and cause damage to the reinforcing fibers resulting in improper coupon failures. Reinforcing fibers should not be exposed or damaged during the surface preparation process. The strain gage manufacturer should be consulted regarding surface preparation guidelines and recommended bonding agents for composites pending the development of a set of standard practices for strain gage installation surface preparation of fiber-reinforced composite materials.

7.3.1.2 Consideration should be given to the selection of gages having larger resistances to reduce heating effects on low-conductivity materials. Resistances of 350 Ω or higher are preferred. Additional consideration should be given to the use of the minimum possible gage excitation voltage consistent with the desired accuracy (1 to 2 V is recommended) to reduce further the power consumed by the gage. Heating of the coupon by the gage may affect the performance of the material directly, or it may affect the indicated strain as a result of a difference between the gage temperature compensation factor and the coefficient of thermal expansion of the coupon material.

7.3.1.3 Consideration of some form of temperature compensation is recommended, even when testing at standard laboratory atmosphere. Temperature compensation is required when testing in nonambient temperature environments.

7.3.1.4 Consideration should be given to the transverse sensitivity of the selected strain gage. The strain gage manufacturer should be consulted for recommendations on transverse sensitivity corrections and effects on composites. This is particularly important for a transversely mounted gage used to determine Poisson's ratio, as discussed in Note 13.

7.3.2 Extensometers—For most purposes, the extensometer gage length should be in the range of 10 to 50 mm [0.5 to 2.0 in.]. Extensometers shall satisfy, at a minimum, Practice E 83, Class B-1 requirements for the strain range of interest and shall be calibrated over that strain range in accordance with Practice E 83. For extremely stiff materials, or for measurement of transverse strains, the fixed error allowed by Class B-1 extensometers may be significant, in which case Class A extensometers should be considered. The extensometer shall be essentially free of inertia lag at the specified speed of testing, and the weight of the extensometer should not induce bending strains greater than those allowed in 6.3.

Note 4—It is generally less difficult to perform strain calibration on extensometers of longer gage length as less precision in displacement is required of the extensometer calibration device.

7.4 Conditioning Chamber—When conditioning materials at nonlaboratory environments, a temperature/vaporcontrolled environmental conditioning chamber is required that shall be capable of maintaining the required temperature to

----

Within ±3°C [±5°F] and the required relative vapor level to within ±3%, chamber conditions shall be monitored either on an automated continuous basis or on a manual basis at regular intervals.

7.5 Environmental Test Chamber—An environmental test chamber is required for test environments other than ambient testing laboratory conditions. This chamber shall be capable of maintaining the gage section of the test specimen at the required test environment during the mechanical test.

8. Sampling and Test Specimens

8.1 Sampling—Test at least five specimens per test condition unless valid results can be gained through the use of fewer specimens, such as in the case of a designed experiment. For statistically significant data, the procedures outlined in Practice E122 should be consulted. Report the method of sampling.

Note 5—If specimens are to undergo environmental conditioning to equilibrium, and use of such type or geometry that the weight change of the material cannot be properly measured by weighing the specimen itself (such as a tabbed mechanical coupon), then another travel coupon of the same nominal thickness and appropriate size (but without tabs) to determine when equilibrium has been reached for the specimens being conditioned.

8.2 Geometry—Design of mechanical test coupons, especially those using end tabs, remains to a large extent an art rather than a science, with no industry consensus on how to approach the engineering of the gripping interface. Each major composite testing laboratory has developed gripping methods for the specific material systems and environments commonly encountered within that laboratory. Comparison of these methods shows them to differ widely, making it extremely difficult to recommend a universally useful approach or set of approaches. Because of this difficulty, definition of the geometry of the test coupon is broken down into the following three levels, which are discussed further in each appropriate section:

<table>
<thead>
<tr>
<th>Purpose</th>
<th>Degree of Geometry Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>8.2.1 General Requirements</td>
<td>Mandatory Shape and Tolerances</td>
</tr>
<tr>
<td>8.2.2 Specific Recommendations</td>
<td>Nonmandatory Suggested Dimensions</td>
</tr>
<tr>
<td>8.2.3 Detailed Examples</td>
<td>Nonmandatory Typical Practices</td>
</tr>
</tbody>
</table>

8.2.1 General Requirements:

8.2.1.1 Shape, Dimensions, and Tolerances—The complete list of requirements for specimen shape, dimensions, and tolerances is shown in Table 1. Failure modes may occur with reasonable frequency, then there is no reason to change a given gripping method.

8.2.2 Specific Recommendations:

8.2.2.1 Width, Thickness, and Length—Select the specimen width and thickness to promote failure in the gage section and assure that the specimen contains a sufficient number of fibers in the cross section to be statistically representative of the bulk material. The specimen length should normally be substantially longer than the minimum requirement to minimize bending stresses caused by minor grip eccentricities. Keep the gage section as far from the grips as reasonably possible and provide a significant amount of material under stress and therefore produce a more statistically significant result. The minimum requirements for specimen design shown in Table 1 are by themselves insufficient to create a properly dimensioned and tolerated coupon drawing. Therefore, recommendations on other important dimensions are provided for typical material configurations in Table 2. These geometries have been found by a number of testing laboratories to produce acceptable failure modes on a wide variety of material systems, but use of them does not guarantee success for every existing or future material system.

8.2.2.2 Gripping/Use of Tabs—There are many material configurations, such as multidirectional laminates, fabric-based materials, or randomly reinforced sheet-molding compounds, which can be successfully tested without tabs. However, tabs are strongly recommended when testing unidirectional materials (or strongly directionally dominated laminates) to failure in the fiber direction. Tabs may also be required when testing unidirectional materials in the matrix direction to prevent gripping damage.

8.2.2.3 Tab Geometry—Recommendations on important dimensions are provided for typical material configurations in Table 2.

<table>
<thead>
<tr>
<th>Table 2 Tensile Specimen Geometry Recommendations</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Fiber Orientation</td>
<td>Width, mm (in.)</td>
</tr>
<tr>
<td>0° Unidirectional</td>
<td>16 [0.6]</td>
</tr>
<tr>
<td>90° Unidirectional</td>
<td>26 [1.0]</td>
</tr>
<tr>
<td>Balanced and Symmetric</td>
<td>26 [1.0]</td>
</tr>
</tbody>
</table>

* Dimensions in this table and the tolerances of Fig. 2 or Fig. 3 are recommendations only and may be varied as long as the requirements of Table 1 are met.
Table 2. These dimensions have been found by a number of testing laboratories to produce acceptable failure modes on a wide variety of material systems, but use of them does not guarantee success for every existing or future material system. The selection of a tab configuration that can successfully produce a gage section tensile failure is dependent upon the coupon material, coupon ply orientation, and the type of grips being used. When pressure-operated nonwedge grips are used with care, squared-off 90° tabs have been used successfully. Wedge-operated grips have been used most successfully with tabs having low bevel angles (7° to 10°) and a feathered smooth transition into the coupon. For alignment purposes, it is essential that the tabs be of matched thickness.

8.3.2.4 Positioning Tabs—Tabs need not always be bonded to the material under test to be effective in introducing the force into the specimen. Friction tabs, essentially nonbonded tabs held in place by the pressure of the grips, are often used with emery cloth or some other light abrasive between the tab and the coupon, have been successfully used in some applications. In specific cases, lightly serrated wedge grips (see Note 2) have been successfully used with only emery cloth as the interface between the grips and the coupon. However, the abrasive used must be able to withstand significant compressive forces. Some types of emery cloth have been found ineffective in this application because of disintegration of the abrasive.

8.2.2.5 Tab Material—The most consistently used bonded tab material has been continuous E-glass fiber-reinforced polymer matrix materials (woven or unwoven) in a [0/90] laminate configuration. The tab material is commonly applied at 45° to the force direction to provide a soft interface. Other configurations that have reportedly been successfully used have incorporated steel tabs or tabs made of the same material as is being tested.

8.2.2.6 Bonded Tab Length—When using bonded tabs, estimate the minimum suggested tab length for bonded tabs by the following simple equation. As this equation does not account for the peaking stresses that are known to exist at the ends of bonded joints, the tab length calculated by this equation should normally be increased by some factor to reduce the chance of joint failure:

\[
L_{\text{tab}} = \frac{F'_{\text{tab}}}{F''} \frac{t}{h}
\]

where:
- \(L_{\text{tab}}\) = minimum required bonded tab length, mm [in.];
- \(F'_{\text{tab}}\) = ultimate tensile strength of coupon material, MPa [psi];
- \(h\) = coupon thickness, mm [in.]; and
- \(F''\) = ultimate shear strength of adhesive, coupon material, or tab material (whichever is lowest), MPa [psi].

8.2.2.7 Bonded Tab Adhesive—Any high-strength (tough) adhesive system that meets the environmental requirements may be used. When bonding tabs to the material under test, a uniform bondline of minimum thickness is desirable to reduce undesirable stresses in the assembly.

8.2.3 Detailed Examples—The minimum requirements for specimen design discussed in 8.2.1 are by themselves insufficient to create a properly dimensioned and tolerated coupon drawing. Dimensionally tolerated specimen drawings for both tabbed and untabbed forms are shown as examples in Fig. 2 (SI) and Fig. 3 (inch-pound). The tolerances on these drawings are fixed, but satisfy the requirements of Table 1 for all of the recommended configurations of Table 2. For a specific configuration, the tolerances on Fig. 2 and Fig. 3 might be able to be relaxed.

8.3 Specimen Preparation:

8.3.1 Panel Fabrication—Control of fiber alignment is critical. Improper fiber alignment will reduce the measured properties. Erratic fiber alignment will also increase the coefficient of variation. The specimen preparation method shall be reported.

8.3.2 Machining Methods—Specimen preparation is extremely important for this specimen. Mold the specimens individually to avoid edge cutting effects or cut them from plates. If they are cut from plates, take precautions to avoid notches, undercut, edges, or uneven surfaces, or delaminations caused by inappropriate machining methods. Obtain final dimensions by water-lubricated precision sawing, milling, or grinding. The use of diamond tools has been found to be extremely effective for many material systems. Edges should be flat and parallel within the specified tolerances.

8.3.3 Labeling—Label the coupons so that they will be distinct from each other and traceable back to the raw material and in a manner that will both be unaffected by the test and not influence the test.

9. Calibration

9.1 The accuracy of all measuring equipment shall have certified calibrations that are current at the time of use of the equipment.

10. Conditioning

10.1 The recommended pre-test condition is effective moisture equilibrium at a specific relative humidity as established by Test Method D 5229/D 5229M; however, if the test requestor does not explicitly specify a pre-test conditioning environment, no conditioning is required and the test specimen may be tested as prepared.

10.2 The pre-test specimen conditioning process, to include specified environmental exposure levels and resulting moisture content, shall be reported with the test data.

Note 6—The term moisture, as used in Test Method D 5229/D 5229M, includes not only the water of a liquid and its condensate, but the liquid itself in large quantities, as for immersion.

10.3 If no explicit conditioning process is performed, the specimen conditioning process shall be reported as "unconditioned" and the moisture content as "unknown."

11. Procedure

11.1 Parameters To Be Specified Before Test:

11.1.1 The tension specimen sampling method, coupon type and geometry, and conditioning chambers (if required).

11.2 The tensile properties and data reporting format desired.

Note 7—Determine specific material property, accuracy, and data reporting requirements before test for proper selection of instrumentation and data-recording equipment. Estimation of stress and strain levels
to aid in transducer selection, calibration of equipment, and determination of equipment settings.

11.1.3 The environmental conditioning test parameters.

11.1.4 If performed, the sampling method, coupon geometry, and test parameters used to determine density and reinforcement volume.

11.2 General Instructions:

11.2.1 Report any deviations from this test method, whether intentional or inadvertent.

11.2.2 If specific gravity, density, reinforcement volume, or void volume are to be reported, then obtain these samples from the same panels being tension tested. Specific gravity and density may be evaluated by means of Test Methods D 792. Volume percent of the constituents may be evaluated by one of the matrix digestion procedures of Test Method D 3171, or, for certain reinforcement materials such as glass and ceramics, by the matrix burn-off technique of Test Method D 2584. The void content equations of Test Methods D 2734 are applicable to both Test Method D 2584 and the matrix digestion procedures.

11.2.3 Following final specimen machining and any conditioning, but before the tension testing, determine the specimen area as $A = w \times h$, at three places in the gage section, and report the area as the average of these three determinations to the accuracy in 7.1. Record the average area in units of $\text{in}^2$ (

11.3 Speed of Testing—Set the speed of testing to effect a nearly constant strain rate in the gage section. If strain control is not available on the testing machine, this may be approximated by repeated monitoring and adjusting of the rate of force application to maintain a nearly constant strain rate, as measured by strain transducer response versus time. The strain rate should be selected so as to produce failure within 1 to 10 min. If the ultimate strain of the material cannot be reasonably estimated, initial trials should be conducted using standard speeds until the ultimate strain of the material and the compliance of the system are known, and the strain rate can be adjusted. The suggested standard speeds are:

11.3.1 Strain-Controlled Tests—A standard strain rate of 0.01 min$^{-1}$.

11.3.2 Constant Head-Speed Tests—A standard head displacement rate of 2 mm/min [0.05 in./min].
Note 8—Use of a fixed head speed in testing machine systems with a high compliance may result in a strain rate that is much lower than required. Use of wedge grips can cause excessive compliance in the system, especially when using compliant tab materials. In some such cases, actual strain rates 10 to 50 times lower than estimated by head speed have been observed.

11.4 Test Environment—Condition the specimen to the desired moisture profile and, if possible, test under the same conditioning fluid exposure level. However, cases such as elevated temperature testing of a moist specimen place unrealistic requirements on the capabilities of common testing machine environmental chambers. In such cases, the mechanical test environment may need to be modified, for example, by testing at elevated temperature with no fluid exposure control, but with a specified limit on time of failure from withdrawal from the conditioning chamber. Modifications to the test environment shall be recorded. In the case where there is no fluid exposure control, the percentage moisture loss of the specimen prior to test completion may be estimated by placing a conditioned traveler coupon of known weight within the test chamber at the same time as the specimen is placed in the chamber. Upon completion of the test, the traveler coupon is removed from the chamber, weighed, and the percentage weight calculated and reported.

11.4.1 Store the specimen in the conditioned environment until test time, if the testing area environment is different than the conditioning environment.

11.5 Specimen Insertion—Place the specimen in the grips of the testing machine, taking care to align the long axis of the gripped specimen with the test direction. Tighten the grips, recording the pressure used on pressure controllable (hydraulic or pneumatic) grips.

Note 5—The ends of the grip jaws on wedge-type grips should be even with each other following insertion to avoid inducing a bending moment that results in premature failure of the specimen at the grip. When using untabbed specimens, a folded strip of medium grade (80 to 150 grit) emery cloth between the specimen faces and the grip jaws (grit-side toward specimen) provides a nonrip grip on the specimen without jaw serration damage to the surface of the specimen. When using tabbed specimens, insert the coupon so that the grip jaws extend approximately
10 to 15 mm (0.5 in.) past the beginning of the tapered portion of the tab. Coupons having tabs that extend beyond the grips are prone to failure at the tab ends because of excessive interlaminar stresses.

11.6 Transducer Installation—If strain response is to be determined, attach the strain-indication transducer(s) to the specimen, symmetrically about the mid-span, mid-width location. Attach the strain-recording instrumentation to the transducer on the specimen.

11.6.1 When determining modulus of elasticity, it is recommended that at least one specimen per like sample be evaluated with back-to-back axial transducers to evaluate the percent bending, using Eq 5, at the average axial strain checkpoint value (the mid range of the appropriate chord modulus strain range) shown in Table 3. A single transducer can be used if the percent bending is no more than 3%. When bending is greater than 3% averaged strains from back-to-back transducers of like kind are recommended.

\[ B_p = \frac{\varepsilon_f - \varepsilon_i}{\varepsilon_f + \varepsilon_i} \]  
(5)

where:
- \( \varepsilon_f \) = indicated strain from front transducer, \( \mu \varepsilon \); 
- \( \varepsilon_i \) = indicated strain from back transducer, \( \mu \varepsilon \); and
- \( B_p \) = percent bending in specimen.

11.7 Loading—Apply the force to the specimen at the specified rate until failure, while recording data.

11.8 Data Recording—Record force versus crosshead displacement (and force versus strain, if extensometers are utilized) continuously or at frequent regular intervals. For this test method, a sampling rate of 2 to 3 data recordings per second, and a target minimum of 100 data points per test are recommended. If a transition region or initial ply failures are noted, record the force, strain, and mode of damage at such points. Record the method used to determine the initial failure (visual, acoustic emission, etc.). If the specimen is to be failed, record the maximum force, the failure force, and the strain (or transducer displacement) at, or as near as possible to, the moment of rupture.

Note 10—Other valuable data that can be useful in understanding testing anomalies and gripping or specimen slipping problems includes force versus head displacement data and force versus time data.

11.9 Failure Mode—Record the mode and location of failure of the specimen. Choose, if possible, a standard description using the three-part failure mode code that is shown in Fig. 4.

TABLE 3 Specimen Alignment and Chord Modulus Calculation Strain Ranges

<table>
<thead>
<tr>
<th>Chord Modulus Calculation</th>
<th>Longitudinal Strain Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Start Point</td>
<td>End Point</td>
</tr>
<tr>
<td>( \mu \varepsilon )</td>
<td>( \mu \varepsilon )</td>
</tr>
<tr>
<td>( \varepsilon_f )</td>
<td>( \varepsilon_i )</td>
</tr>
<tr>
<td>9000</td>
<td>3000</td>
</tr>
</tbody>
</table>

* 12000 \( \mu \varepsilon \) = 0.99% absolute strain
* This strain range is to be contained in the lower half of the stress-strain curve.

12. Validation

12.1 Values for ultimate properties shall not be calculated for any specimen that breaks at some obvious flaw, unless such flaw constitutes a variable being studied. Retests shall be performed for any specimen on which values are not calculated.

12.2 Grip/Tab Failures—Reexamine the means of force introduction into the material if a significant fraction of failures in a sample population occurred within one specimen width of the tab or grip. Factors considered should include the tab alignment, tab material, tab angle, tab adhesive, grip type, grip pressure, and grip alignment.

13. Calculation

13.1 Tensile Stress/Tensile Strength—Calculate the ultimate tensile strength using Eq 6 and report the results to three significant figures. If the tensile modulus is to be calculated, determine the tensile stress at each required data point using Eq 7.

\[ F = \frac{P_{max}}{A} \]  
(6)

\[ \sigma = \frac{P}{A} \]  
(7)

where:
- \( F \) = ultimate tensile strength, MPa [psi];
- \( P_{max} \) = maximum force before failure, N [lb];
- \( \sigma \) = tensile stress at ith data point, MPa [psi];
- \( P_i \) = force at ith data point, N [lb]; and
- \( A \) = average cross-sectional area from 11.2.3, \( \text{mm}^2 \) [\( \text{in.}^2 \)].

13.2 Tensile Strain/Ultimate Tensile Strain—If tensile modulus or ultimate tensile strain is to be calculated, and material response is being determined by an extensometer, determine the tensile strain from the indicated displacement at each required data point using Eq 8 and report the results to three significant figures.

\[ \varepsilon = \frac{L_d}{L_2} \]  
(8)

where:
- \( \varepsilon \) = tensile strain at ith data point, \( \mu \varepsilon \); 
- \( L_d \) = extensometer displacement at ith data point, \( \text{mm} \) [\( \text{in.} \)]; and
- \( L_2 \) = extensometer gage length, \( \text{mm} \) [\( \text{in.} \)].

13.3 Tensile Modulus of Elasticity:

Note 11—To minimize potential effects of bending it is recommended that the strain data used for modulus of elasticity determination be the average of the indicated strain from each side of the specimen, as discussed in 7.3 and 11.6.

13.3.1 Tensile Chord Modulus of Elasticity—Select the appropriate chord modulus strain range from Table 3. Calculate the tensile chord modulus of elasticity from the stress-strain data using Eq 9. If data is not available at the exact strain range end points (as often occurs with digital data), use the closest available data point. Report the tensile chord modulus of elasticity to three significant figures. Also report the strain range used in the calculation. A graphical example of chord modulus is shown in Fig. 5.

13.3.1.1 The tabulated strain range should only be used for materials that do not exhibit a transition region (a significant
change in the slope of the stress-strain curve) within the given strain range. If a transition region occurs within the recommended strain range, then a more suitable strain range shall be used and reported.

\[ E_{\text{mod}} = \Delta\sigma / \Delta e \]  

(9)

where:

- \( E_{\text{mod}} \) = tensile chord modulus of elasticity, GPa [psi];
- \( \Delta\sigma \) = difference in applied tensile stress between the two strain points of Table 3, MPa [psi]; and
- \( \Delta e \) = difference between the two strain points of Table 3 (nominally 0.002).

13.3.2 Tensile Modulus of Elasticity (Other Definitions)—
Other definitions of elastic modulus may be evaluated and reported at the user’s discretion. If such data is generated and reported, report also the definition used, the strain range used, and the results to three significant figures. Test Method E 111 provides additional guidance in the determination of modulus of elasticity.
Note 12—An example of another modulus definition is the secondary chord modulus of elasticity for materials that exhibit essentially linear stress-strain behavior. An example of secondary chord modulus is shown in Fig. 5.

13.4 Poisson's Ratio:

Note 13—If bonded resistance strain gages are being used, the error produced by the transverse sensitivity effect on the transverse gage will generally be much larger for composites than for metals. An accurate measurement of Poisson’s ratio requires correction for this effect. The strain gage manufacturer should be contacted for information on the use of correction factors for transverse sensitivity.

13.4.1 Poisson’s Ratio By Chord Method—Select the appropriate chord modulus longitudinal strain range from Table 3. Determine (by plotting or otherwise) the transverse strain (measured perpendicular to the applied force), \( \varepsilon_y \), at each of the two longitudinal strains (measured parallel to the applied force), \( \varepsilon_x \), strain range end points. If data is not available at the exact strain range end points (as often occurs with digital data), use the closest available data point. Calculate Poisson’s ratio by Eq 10 and report to three significant figures. Also report the strain range used.

\[

\nu = -\frac{\Delta\varepsilon_y}{\Delta\varepsilon_x}

\]

where:

\( \nu \) = Poisson’s ratio;
\( \Delta\varepsilon_y \) = difference in lateral strain between the two longitudinal strain points of Table 3, m, and
\( \Delta\varepsilon_x \) = difference between the two longitudinal strain points of Table 3 (nominally either 0.001, 0.002, or 0.005).

13.4.2 Tensile Poisson’s Ratio (Other Definitions)—Other definitions of Poisson’s ratio may be evaluated and reported at the user’s discretion. If such data is generated and reported, report also the definition used, the strain range used, and the results to three significant figures. Test Method E 132 provides additional guidance in the determination of Poisson’s ratio.

13.5 Transition Strain—Where applicable, determine the transition strain from either the bilinear longitudinal stress versus longitudinal strain curve or the bilinear transverse strain versus longitudinal strain curve. Create a best linear fit or chord line for each of the two linear regions and extend the lines until they intersect. Determine to three significant digits the longitudinal strain that corresponds to the intersection point and record this value as the transition strain. Report also the method of linear fit (if used) and the strain ranges over which the linear fit or chord lines were determined. A graphical example of transition strain is shown in Fig. 5.

13.6 Statistics—For each series of tests calculate the average value, standard deviation and coefficient of variation (in percent) for each property determined:

\[

\bar{x} = \left( \frac{\sum x_i}{n} \right)\bar{n}

\]

\[

\sigma_x = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}}

\]

\[

CV = 100 \times \frac{\sigma_x}{\bar{x}}

\]

where:

\( \bar{x} \) = sample mean (average);
\( \sigma_x \) = sample standard deviation;
\( CV \) = sample coefficient of variation, in percent;
\( n \) = number of specimens; and
\( x_i \) = measured or derived property.

14. Report

14.1 Report the following information, or references pointing to other documentation containing this information, to the maximum extent applicable (reporting of items beyond the control of a given testing laboratory, such as might occur with material details or panel fabrication parameters, shall be the responsibility of the requestor):

14.1.1 The revision level or date of issue of this test method.
14.1.2 The date(s) and location(s) of the test.
14.1.3 The name(s) of the test operator(s).
14.1.4 Any variations to this test method, anomalies noticed during testing, or equipment problems occurring during testing.
14.1.5 Identification of the material tested including: material specification, material type, material designation, manufacturer, manufacturer’s lot or batch number, source (if not from manufacturer(s), date of certification, expiration of certification, filament diameter, tow or yarn filament count and twist, sizing, form or weave, fiber areal weight, matrix type, prepreg matrix content, and prepreg volatiles content.
14.1.6 Description of the fabrication steps used to prepare the laminate including: fabrication start date, fabrication end date, process specification, cure cycle, consolidation method, and a description of the equipment used.
14.1.7 Ply orientation stacking sequence of the laminate.
14.1.8 If requested, report density, volume percent reinforcement, and void content test methods, specimen sampling method and geometries, test parameters, and test results.
14.1.9 Average ply thickness of the material.
14.1.10 Results of any nondestructive evaluation tests.
14.1.11 Method of preparing the test specimen, including specimen labeling scheme and method, specimen geometry, sampling method, coupon cutting method, identification of tab geometry, tab material, and tab adhesive used.
14.1.12 Calibration dates and methods for all measurement and test equipment.
14.1.13 Type of test machine, grips, jaws, grip pressure, alignment results, and data acquisition sampling rate and equipment type.
14.1.14 Results of system alignment evaluations, if any such were done.
14.1.15 Dimensions of each test specimen.
14.1.16 Conditioning parameters and results, use of travelers and traveler geometry, and the procedure used if other than that specified in the test method.
14.1.17 Relative humidity and temperature of the testing laboratory.
14.1.18 Environment of the test machine environmental chamber (if used) and soak time at environment.
14.1.19 Number of specimens tested.
14.1.20 Speed of testing.
14.1.21 Transducer placement on the specimen and transducer type for each transducer used.
14.1.22 If strain gages were used, the type, resistance, size, gage factor, temperature compensation method, transverse sensitivity, lead-wire resistance, and any correction factors used.

14.1.23 Stress-strain curves and tabulated data of stress versus strain for each specimen.

14.1.24 Percent bending results for each specimen so evaluated.

14.1.25 Individual strengths and average value, standard deviation, and coefficient of variation (in percent) for the population. Note if the failure force was less than the maximum force before failure.

14.1.26 Individual strength at failure and the average value, standard deviation, and coefficient of variation (in percent) for the population.

14.1.27 Strain range used for chord modulus and Poisson’s ratio determination.

14.1.28 If another definition of modulus of elasticity is used in addition to chord modulus, describe the method used, the resulting correlation coefficient (if applicable), and the strain range used for the evaluation.

14.1.29 Individual values of modulus of elasticity, and the average value, standard deviation, and coefficient of variation (in percent) for the population.

14.1.30 If another definition of Poisson’s ratio is used in addition to the chordwise definition, describe the method used, the resulting correlation coefficient (if applicable), and the strain range used for the evaluation.

14.1.31 Individual values of Poisson’s ratio, and the average value, standard deviation, and coefficient of variation (in percent) for the population.

14.1.32 If transition strain is determined, the method of linear fit (if used) and the strain range over which the linear fit or chord lines were determined.

14.1.33 Individual values of transition strain (if applicable), and the average value, standard deviation, and coefficient of variation (in percent) for the population.

14.1.34 Failure mode and location of failure for each specimen.

15. Precision and Bias

15.1 Precision:

15.1.1 The precision and bias of tension test strength and modulus measurements depend on strict adherence to the Test Method D 3039/D 3039M and are influenced by mechanical and material factors, specimen preparation, and measurement errors.

15.1.2 Mechanical factors that can affect the test results include: the physical characteristics of the testing machine (stiffness, damping, and mass), accuracy of force application and displacement/strain measurement, speed of force application, alignment of test specimen with applied force, parallelism of the grips, grip pressure, and type of force control (displacement, strain, or force).

15.1.3 Material factors that can affect test results include: material quality and representativeness, sampling scheme, and specimen preparation (dimensional accuracy, tab material, tab taper, tab adhesive, and so forth).

15.1.4 The mean tensile strength for a strain rate sensitive, glass-epoxy tape composite testing in the fiber direction was found to increase by approximately two standard deviations with decreasing time to failure tested at the limits of the recommended time to failure prescribed in Test Method D 3039/D 3039M. This result suggests that caution must be used when comparing test data obtained for strain rate sensitive composite materials tested in accordance with this standard.

15.1.5 Measurement errors arise from the use of specialized measuring instruments such as load cells, extensometers and strain gages, micrometers, data acquisition devices, and so forth.

15.1.6 Data obtained from specimens that fracture outside the gage area should be used with caution as this data may not be representative of the material. Failure in the grip region indicates the stress concentration at the tab is greater than the natural strength variation of the material in the gage section. A tapered tab, bonded with a ductile low-modulus adhesive has a relatively low-stress concentration and should result in the lowest frequency of grip failures. Low-strength bias increases with the frequency of grip failures by an amount proportional to the stress concentration at the tab.

15.1.7 An interlaboratory test program was conducted where an average of five specimens each, of six different materials and lay-up configurations, were tested by nine different laboratories. Table 4 presents the precision statistics generated from this study as defined in Practice E 691 for tensile strength, modulus, and failure strain. All data except that for Material B (90° lay-up) was normalized with respect to an average thickness. The materials listed in Table 4 are defined as:

<table>
<thead>
<tr>
<th>No.</th>
<th>Material Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>615/S/500-6 6&quot; tape (0°)</td>
</tr>
<tr>
<td>B</td>
<td>615/S/500-6 6&quot; tape (90°)</td>
</tr>
<tr>
<td>C</td>
<td>615/S/500-6 6&quot; tape (90°)</td>
</tr>
<tr>
<td>D</td>
<td>Glass/Carbon fabric (77%)</td>
</tr>
<tr>
<td>E</td>
<td>Glass/Carbon fabric (77%)</td>
</tr>
<tr>
<td>F</td>
<td>Glass/Carbon fabric (64°)</td>
</tr>
<tr>
<td>G</td>
<td>Glass/Carbon fabric (64°)</td>
</tr>
</tbody>
</table>

15.1.8 The averages of the coefficients of variation are in Table 5. The values of $S_{xx}$ and $S_{yy}$ represent the representability and the reproducibility coefficients of variation, respectively. These averages permit a relative comparison of the repeatability (within laboratory precision) and reproducibility (between laboratory precision) of the tension test parameters. Overall, this indicates that the failure strain measurements exhibit the least repeatability and reproducibility of all the parameters measured while modulus was found to provide the highest repeatability and reproducibility of the parameters measured.

15.1.9 The consistency of agreement for repeated tests of the same material is dependent on lay-up configuration, material and specimen preparation techniques, test conditions, and measurements of the tension test parameters.

15.2 Bias—Bias cannot be determined for this test method as no acceptable reference standard exists.

---

16. Keywords

16.1 composite materials; modulus of elasticity; Poisson’s ratio; tensile properties; tensile strength

### TABLE 4 Precision Statistics

<table>
<thead>
<tr>
<th>Material</th>
<th>( x )</th>
<th>( s )</th>
<th>( S_x )</th>
<th>( S_y )</th>
<th>( S_y/S_x )</th>
<th>( S_y/S_y )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strength</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>A</td>
<td>942.69</td>
<td>0.69</td>
<td>10.69</td>
<td>12.75</td>
<td>1.12</td>
<td>3.73</td>
</tr>
<tr>
<td>B</td>
<td>8.62</td>
<td>0.52</td>
<td>0.85</td>
<td>0.92</td>
<td>0.94</td>
<td>10.84</td>
</tr>
<tr>
<td>C</td>
<td>155.57</td>
<td>3.84</td>
<td>10.85</td>
<td>19.85</td>
<td>6.94</td>
<td>9.44</td>
</tr>
<tr>
<td>P</td>
<td>6.18</td>
<td>0.20</td>
<td>1.52</td>
<td>3.46</td>
<td>2.30</td>
<td>5.26</td>
</tr>
<tr>
<td>G</td>
<td>121.52</td>
<td>1.99</td>
<td>3.92</td>
<td>3.92</td>
<td>3.23</td>
<td>3.23</td>
</tr>
<tr>
<td>Modulus, ksi</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>A</td>
<td>23.57</td>
<td>0.95</td>
<td>0.63</td>
<td>0.26</td>
<td>2.99</td>
<td>3.66</td>
</tr>
<tr>
<td>B</td>
<td>1.90</td>
<td>0.05</td>
<td>0.64</td>
<td>0.06</td>
<td>1.12</td>
<td>4.57</td>
</tr>
<tr>
<td>C</td>
<td>12.98</td>
<td>0.20</td>
<td>0.37</td>
<td>0.44</td>
<td>2.54</td>
<td>3.84</td>
</tr>
<tr>
<td>P</td>
<td>3.08</td>
<td>0.08</td>
<td>0.46</td>
<td>0.05</td>
<td>1.01</td>
<td>2.28</td>
</tr>
<tr>
<td>G</td>
<td>9.47</td>
<td>0.16</td>
<td>0.12</td>
<td>0.20</td>
<td>1.29</td>
<td>2.69</td>
</tr>
<tr>
<td>Failure Strain, %</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>A</td>
<td>1.06</td>
<td>0.06</td>
<td>0.07</td>
<td>0.08</td>
<td>4.95</td>
<td>6.15</td>
</tr>
<tr>
<td>B</td>
<td>0.66</td>
<td>0.04</td>
<td>0.09</td>
<td>0.09</td>
<td>12.47</td>
<td>13.62</td>
</tr>
<tr>
<td>C</td>
<td>1.22</td>
<td>0.00</td>
<td>0.06</td>
<td>0.05</td>
<td>5.05</td>
<td>5.27</td>
</tr>
<tr>
<td>P</td>
<td>2.04</td>
<td>0.15</td>
<td>0.17</td>
<td>0.16</td>
<td>3.19</td>
<td>6.03</td>
</tr>
<tr>
<td>G</td>
<td>1.27</td>
<td>0.03</td>
<td>0.05</td>
<td>0.05</td>
<td>3.85</td>
<td>4.13</td>
</tr>
</tbody>
</table>

### TABLE 5 Averages of the Coefficients of Variation

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Average of ( S_x ) %</th>
<th>Average of ( S_y/S_x ) %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strength</td>
<td>5.11</td>
<td>6.02</td>
</tr>
<tr>
<td>Modulus</td>
<td>2.22</td>
<td>2.22</td>
</tr>
<tr>
<td>Failure strain</td>
<td>5.94</td>
<td>7.32</td>
</tr>
</tbody>
</table>

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Designation: G 155 − 05a

Standard Practice for Operating Xenon Arc Light Apparatus for Exposure of Non-Metallic Materials

1 This standard is issued under the fixed designation G 155; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the basic principles and operating procedures for using xenon arc light and water apparatus intended to reproduce the weathering effects that occur when materials are exposed to sunlight (either direct or through window glass) and moisture as rain or dew in actual use. This practice is limited to the procedures for obtaining, measuring, and controlling conditions of exposure. A number of exposure procedures are listed in an appendix; however, this practice does not specify the exposure conditions best suited for the material to be tested.

Note 1—Practice G 151 describes performance criteria for all exposure devices that use laboratory light sources. This practice replaces Practice G 26, which describes very specific designs for devices used for xenon-arc exposures. The apparatus described in Practice G 26 is covered by this practice.

1.2 Test specimens are exposed to filtered xenon arc light under controlled environmental conditions. Different types of xenon arc light sources and different filter combinations are described.

1.3 Specimen preparation and evaluation of the results are covered in ASTM methods or specifications for specific materials. General guidance is given in Practice G 151 and ISO 4892-1. More specific information about methods for determining the change in properties after exposure and reporting these results is described in Practice D 5870.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1.6 This practice is technically similar to the following ISO documents: ISO 4892-2, ISO 11341, ISO 105 B02, ISO 105 B05, ISO 105 B08, and ISO 105 B06.

2. Referenced Documents

2.1 ASTM Standards:

D 3980 Practice for Interlaboratory Testing of Paint and Related Materials

D 5870 Practice for Calculating Property Retention Index of Plastics

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

G 26 Practice for Operating Light-Exposure Apparatus (Xenon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials

G 113 Terminology Relating to Natural and Artificial Weathering Tests for Nonmetallic Materials

G 151 Practice for Exposing Nonmetallic Materials in Accelerated Test Devices That Use Laboratory Light Sources

2.2 CIE Standards:


2.3 International Standards Organization Standards:

ISO 1143, Paint and Varnishes—Artificial Weathering Exposure to Artificial Radiation to Filtered Xenon Arc Radiat

ISO 105 B02, Textiles—Tests for Colorfastness—Part B02 Colorfastness to Artificial Light: Xenon Arc Fading Lamp Test

ISO 105 B04, Textiles—Tests for Colorfastness—Part B04 Colorfastness to Artificial Weathering: Xenon Arc Fading Lamp Test

ISO 105 B05, Textiles—Tests for Colorfastness—Part B05

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Detection and Assessment of Photochromism
ISO 105 B06, Textiles—Tests for Colorfastness—Part B06
Colorfastness to Artificial Light at High Temperatures:
Xenon Arc Fading Lamp Text
ISO 4892-1, Plastics—Methods of Exposure to Laboratory
Light Sources, Part 1, General Guidance
ISO 4892-2, Plastics—Methods of Exposure to Laboratory
Light Sources, Part 2, Xenon-Arc Sources
SAE J1885, Accelerated Exposure of Automotive Interior
Trim Components Using a Controlled Irradiance Water
Cooled Xenon Arc Apparatus
SAE J1940, Accelerated Exposure of Automotive Exterior
Materials Using a Controlled Irradiance Water Cooled
Xenon Arc Apparatus
SAE J3212, Accelerated Exposure of Automotive Interior
Trim Components Using a Controlled Irradiance Xenon-
Arc Apparatus
SAE J3257 Accelerated Exposure of Automotive Exterior
Materials Using a Controlled Irradiance Xenon-Arc
Apparatus.

3. Terminology
3.1 Definitions—The definitions given in Terminology
G 113 are applicable to this practice.
3.2 Definitions of Terms Specific to This Standard:
3.2.1 As used in this practice, the term sunlight is identical
to the terms daylight and solar irradiance, global as they are
defined in Terminology G 113.

4. Summary of Practice
4.1 Specimens are exposed to repetitive cycles of light and
moisture under controlled environmental conditions.
4.1.1 Moisture is usually produced by spraying the test
specimen with demineralized/deionized water or by condensa-
tion of water vapor onto the specimen.
4.2 The exposure condition may be varied by selection of:
4.2.1 Lamp filter(s).
4.2.2 The lamp's irradiance level.
4.2.3 The type of moisture exposure.
4.2.4 The timing of the light and moisture exposure.
4.2.5 The temperature of light exposure.
4.2.6 The temperature of moisture exposure.
4.2.7 The timing of a light/ dark cycle.
4.3 Comparison of results obtained from specimens exposed
in the same model of apparatus should not be made unless
reproducibility has been established among devices for the
material to be tested.
4.4 Comparison of results obtained from specimens exposed
in different models of apparatus should not be made unless
reproducibility has been established among devices for the
material to be tested.

5. Significance and Use
5.1 The use of this apparatus is intended to induce property
changes associated with the end use conditions, including the
effects of sunlight, moisture, and heat. These exposures may
include the means to introduce moisture to the test specimen.
Exposures are not intended to simulate the deterioration caused
by localized weather phenomena, such as atmospheric pollution,
biological attack, and saltwater exposure. Alternatively,
the exposure may simulate the effects of sunlight through
window glass. Typically, these exposures would include moisture
in the form of humidity.

Note 2—Caution: Refer to Practice G 151 for full cautionary guidance
applicable to all laboratory weathering devices.

5.2 Variation in results may be expected when operating
conditions are varied within the accepted limits of this practice.
Therefore, no reference shall be made to results from the use
of this practice unless accompanied by a report detailing the
specific operating conditions in conformance with the Report
Section.

5.2.1 It is recommended that a similar material of known
performance (a control) be exposed simultaneously with the
test specimen to provide a standard for comparative purposes.
It is recommended that at least three replicates of each material
evaluated be exposed in each test to allow for statistical
evaluation of results.

6. Apparatus
6.1 Laboratory Light Source—The light source shall be one
or more quartz-jacketed xenon arc lamps which emit radiation
from below 270 nm in the ultraviolet through the visible
spectrum and into the infrared. In order for xenon arcs to
simulate terrestrial daylight, filters must be used to remove
short wavelength UV radiation. Filters to reduce irradiance at
wavelengths shorter than 310 nm must be used to simulate
daylight filtered through window glass. In addition, filters to
remove infrared radiation may be used to prevent unrealistic
heating of test specimens that can cause thermal degradation
not experienced during outdoor exposures.
6.1.1 The following factors can affect the spectral power
distribution of filtered xenon arc light sources as used in these
apparatus:
6.1.1.1 Differences in the composition and thickness of
filters can have large effects on the amount of short wavelength
UV radiation transmitted.
6.1.1.2 Aging of filters can result in changes in filter
transmission. The aging properties of filters can be influenced
by the composition. Aging of filters can result in a significant
reduction in the short wavelength UV emission of a xenon
burner.
6.1.1.3 Accumulation of deposits or other residue on filters
can effect filter transmission.
6.1.1.4 Aging of the xenon burner itself can result in
changes in lamp output. Changes in lamp output may also be
caused by accumulation of dirt or other residue in or on the
burner envelope.
6.1.2 Follow the device manufacturer’s instructions for
recommended maintenance.

---

* Available from Society of Automotive Engineers (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001.
TABLE 1 Relative Ultraviolet Spectral Power Distribution Specification for Xenon Arc with Daylight Filters

<table>
<thead>
<tr>
<th>Wavelength (n m)</th>
<th>Minimum A1</th>
<th>Measured B</th>
<th>Maximum B</th>
</tr>
</thead>
<tbody>
<tr>
<td>λ &lt; 300</td>
<td>2.4</td>
<td>6.8</td>
<td>18</td>
</tr>
<tr>
<td>300 &lt; λ ≤ 520</td>
<td>9.3</td>
<td>20.8</td>
<td>40.0</td>
</tr>
<tr>
<td>520 &lt; λ ≤ 400</td>
<td>54.2</td>
<td>54.2</td>
<td>57.0</td>
</tr>
</tbody>
</table>

*Data in Table 1 are the incidence in the given bandwidth expressed as a percentage of the total incidence from 350 to 400 n m. The manufacturer is responsibility for determining compliance to Table 1. Appendix A1 states how to determine relative spectral incidence.*

6.1.3 Spectral Irradiance of Xenon Arc with Daylight Filters—Filters are used to filter xenon arc lamp emissions in a simulation of terrestrial sunlight. The spectral power distribution of xenon arcs with new or pre-aged filters shall comply with the requirements specified in Table 1.

6.1.4 Spectral Irradiance of Xenon Arc With Window Glass Filters—Filters are used to filter xenon arc lamp emissions in a simulation of sunlight filtered through window glass. Table 2 shows the relative spectral power distribution limits for xenon arcs filtered with window glass filters. The spectral power distribution of xenon arcs with new or pre-aged filters shall comply with the requirements specified in Table 2.

6.1.5 Spectral Irradiance of Xenon Arc With Extended UV Filters—Filter that transmit more short wavelength UV are sometimes used to accelerate test result. Although this type of filter has been specified in some tests, they transmit significant radiant energy below 300 n m (the typical cut-off wavelength for terrestrial sunlight) and may result in aging processes not occurring outdoors. The spectral irradiance for a xenon arc with extended UV filters shall comply with the requirements of Table 3.

6.1.6 The actual irradiance at the test's specimen plane is a function of the number of xenon burners used, the power applied to each, and the distance between the test specimens and the xenon burner. If appropriate, report the irradiance and the bandwidth in which it was measured.

6.2 Test Chamber—The design of the test chamber may vary, but it should be constructed from corrosion resistant material and, in addition to the radiant source, may provide for means of controlling temperature and relative humidity. When required, provision shall be made for the spraying of water on the test specimen, for the formation of condensate on the exposed face of the specimen or for the immersion of the test specimen in water.

6.2.1 The radiation source(s) shall be located with respect to the specimens such that the irradiance at the specimen face complies with the requirements in Practice G 151.
TABLE 8 Relative Ultraviolet Spectral Power Distribution Specifications for Xenon Arc with Extended UV Filters

<table>
<thead>
<tr>
<th>Spectral Bandwidth</th>
<th>Minimum</th>
<th>Nominal</th>
<th>Minimum</th>
<th>Nominal</th>
<th>Maximum</th>
</tr>
</thead>
<tbody>
<tr>
<td>λ ≤ 300</td>
<td>0.1</td>
<td>0.1</td>
<td>0.1</td>
<td>0.1</td>
<td>0.1</td>
</tr>
<tr>
<td>300 ≤ λ ≤ 600</td>
<td>5.0</td>
<td>5.0</td>
<td>5.0</td>
<td>5.0</td>
<td>5.0</td>
</tr>
<tr>
<td>600 ≤ λ ≤ 1,100</td>
<td>32.4</td>
<td>32.4</td>
<td>32.4</td>
<td>32.4</td>
<td>32.4</td>
</tr>
<tr>
<td>1,100 ≤ λ ≤ 600</td>
<td>52.0</td>
<td>52.0</td>
<td>52.0</td>
<td>52.0</td>
<td>52.0</td>
</tr>
<tr>
<td>600 ≤ λ ≤ 400</td>
<td>62.0</td>
<td>62.0</td>
<td>62.0</td>
<td>62.0</td>
<td>62.0</td>
</tr>
</tbody>
</table>

* Data in Table 8 are the radiant flux in the given bandwidth expressed as a percentage of the total radiant flux from 250 to 400 nm. The manufacturer is responsible for determining conformance to Table 8. (See Appendix A4.)

6.6 Moisture—The test specimen may be exposed to moisture in the form of water spray, condensation, immersion, or high humidity.

6.6.1 Water Spray—The test chamber may be equipped with a means to introduce intermittent water spray onto the front or the back of the test specimens, under specified conditions. The spray shall be uniformly distributed over the specimens. The spray system shall be made from corrosion resistant materials that do not contaminate the water employed.

6.6.1.1 Quality of Water for Sprays and Immersion—Spray water must have a conductivity below 5 µS/cm, contain less than 1 ppm solids, and leave no observable stains or deposits on the specimens. Very low levels of silica in spray water can cause significant deposits on the surface of test specimens. Care should be taken to keep silica levels below 0.1 ppm. In addition to distribution, a combination of deionization and reverse osmosis can effectively produce water of the required quality. The pH of the water used should be reported. See Practice G 151, for detailed water quality instructions.

6.6.2 Relative Humidity—The test chamber may be equipped with a means to measure and control the relative humidity. Such instruments shall be shielded from the lamp radiation.

6.6.3 Water Immersion—The test chamber may be equipped with a means to immerse specimens in water under specified conditions. The immersion system shall be made from corrosion resistant materials that do not contaminate the water employed.

6.7 Specimen Holders—Specimen holders shall be made from corrosion resistant materials that will not affect the test results. Corrosive resistant alloys of aluminum or stainless steel have been found acceptable. Brass, steel, or copper shall not be used in the vicinity of the test specimens.

6.7.1 The specimen holders are typically, but not necessarily, mounted on a revolving cylindrical rack that is rotated around the lamp system at a speed dependent on the type of equipment and that is centered both horizontally and vertically with respect to the exposure area.

6.7.2 Specimen holders may be in the form of an open frame, leaving the back of the specimen exposed, or they may provide the specimen with a solid backing. Any backing used may affect test results and shall be agreed upon in advance between the interested parties.

6.7.3 Specimen holders may rotate on their own axis. When these holders are used, they may be filled with specimens placed back to back. Rotation of the holder on its axis alternately exposes each specimen to direct radiation from the xenon burner.

6.8 Apparatus to Assess Changes in Properties—Use the apparatus required by the ASTM or other standard that describes determination of the property or properties being monitored.

7. Test Specimen

7.1 Refer to Practice G 151.
8. Test Conditions

8.1 Any exposure conditions may be used as long as the exact conditions are detailed in the report. Appendix XI lists some representative exposure conditions. These are not necessarily preferred, and no recommendation is implied. These conditions are provided for reference only.

9. Procedure

9.1 Identify each test specimen by suitable indelible marking, but not on areas to be used in testing.

9.2 Determine which property of the test specimens will be evaluated. Prior to exposing the specimens, quantify the appropriate properties in accordance with recognized international standards. If required (for example, destructive testing), use unexposed file specimens to quantify the property. See Practice D 5870 for detailed guidance.

9.3 Mounting of Test Specimens.—Attach the specimens to the specimen holders in the equipment in such a manner that the specimens are not subject to any applied stress. To assure uniform exposure conditions, fill all of the spaces, using blank panels of corrosion resistant material if necessary.

Note 3.—Evaluation of color and appearance changes of exposed materials must be made based on comparisons to unexposed specimens of the same material which have been stored in the dark, masking or shielding the face of test specimens with an opaque cover for the purpose of showing the effects of exposure on one panel is not recommended. Misleading results may be obtained by this method, since the masked portion of the specimen is still exposed to temperature and humidity that in many cases will affect results.

9.4 Exposure to Test Conditions.—Program the selected test conditions to operate continuously throughout the required number of repetitive cycles. Maintain these conditions throughout the exposure. Interruptions to service the apparatus and to inspect specimens shall be minimized.

9.5 Specimen Repositioning.—Periodic repositioning of the specimens during exposure is not necessary if the irradiance at the positions farthest from the center of the specimen area is at least 90 % of that measured at the center of the exposure area. Irradiance uniformity shall be determined in accordance with Practice G 151.

9.5.1 If irradiance at positions farthest from the center of the exposure area is between 70 and 90 % of that measured at the center, one of the following three techniques shall be used for specimen placement.

9.5.1.1 Periodically reposition specimens during the exposure period to ensure that each receives an equal amount of radiant exposure. The repositioning schedule shall be agreed upon by all interested parties.

9.5.1.2 Place specimens only in the exposure area where the irradiance is at least 90 % of the maximum irradiance.

9.5.1.3 To compensate for test variability, randomly position replicate specimens within the exposure area that meets the irradiance uniformity requirements as defined in section 9.5.1.

9.6 Inspection.—If it is necessary to remove a test specimen for periodic inspection, take care not to handle or disturb the test surface. After inspection, the test specimen shall be returned to the test chamber with its test surface in the same orientation as previously tested.

9.7 Apparatus Maintenance.—The test apparatus requires periodic maintenance to maintain uniform exposure conditions. Perform required maintenance and calibration in accordance with manufacturer’s instructions.

9.8 Expose the test specimens for the specified period of exposure. See Practice G 151 for further guidance.

9.9 At the end of the exposure, quantify the appropriate properties in accordance with recognized international standards and report the results in conformance with Practice G 151.

Note 4.—Periods of exposure and evaluation of test results are addressed in Practice G 151.

10. Report

10.1 The test report shall conform to Practice G 151.

11. Precision and Bias

11.1 Precision

11.1.1 The repeatability and reproducibility of results obtained in exposures conducted according to this practice will vary with the materials being tested, the material property being measured, and the specific test conditions and cycles that are used. In round-robin studies conducted by Subcommittee G03.03, the 60° gloss values of replicate PVC tape specimens exposed in different laboratories using identical test devices and exposure cycles showed significant variability. The variability shown in these round-robin studies restricts the use of “absolute specifications” such as requiring a specific property level after a specific exposure period.

11.1.2 If a standard or specification for general use requires a definite property level after a specific time or radiant exposure in an exposure test conducted according to this practice, the specified property level shall be based on results obtained in a round-robin test that takes into consideration the variability due to the exposure and the test method used to measure the property of interest. The round-robin shall be conducted according to Practice E 691 or Practice D 5870 and shall include a statistically representative sample of all laboratories or organizations who would normally conduct the exposure and property measurement.

11.1.3 If a standard or specification for use between two or three parties requires a definite property level after a specific time or radiant exposure in an exposure test conducted according to this practice, the specified property level shall be based on statistical analysis of results from at least two separate, independent exposures in each laboratory. The design of the experiment used to determine the specification shall take into consideration the variability due to the exposure and the test method used to measure the property of interest.

11.1.4 The round-robin studies cited in 11.1.1 demonstrated that the gloss values for a series of materials could be ranked with a high level of reproducibility between laboratories. When reproducibility in results from an exposure test conducted according to this practice have not been established through round-robin testing, performance requirements for materials shall be specified in terms of comparison (ranked) to a control material. The control specimens shall be exposed simultaneously with the test specimen(s) in the same device. The specific control material used shall be agreed upon by the
concerned parties. Expose replicates of the test specimen and the control specimen so that statistically significant performance differences can be determined.

11.2 Bias—Bias cannot be determined because no acceptable standard weathering reference materials are available.

ANNEX

A1. DETERMINING CONFORMANCE TO RELATIVE SPECTRAL POWER DISTRIBUTION TABLES

(Mandatory Information for Equipment Manufacturers)

A1.1 Conformance to the relative spectral power distribution tables is a design parameter for xenon-arc source with the different filters provided. Manufacturers of equipment claiming conformity to this standard shall determine conformance to the spectral power distribution tables for all lamp/filter combinations provided, and provide information on maintenance procedures to minimize any spectral changes that may occur during normal use.

A1.2 The relative spectral power distribution data for this standard were developed using the rectangular integration technique. Eq A1.1 is used to determine the relative spectral irradiance using rectangular integration. Other integration techniques can be used to evaluate spectral power distribution data, but may give different results. When comparing relative spectral power distribution data to the spectral power distribution requirements of this standard, use the rectangular integration technique.

A1.3 To determine whether a specific lamp for a xenon-arc device meets the requirements of Table 1, Table 2, or Table 3, measure the spectral power distribution from 250 nm to 400 nm. Typically, this is done at 2 nm increments. If the manufacturer’s spectral measurement equipment cannot measure wavelengths as low as 250 nm, the lowest measurement wavelength must be reported. The lowest wavelength measured shall be no greater than 270 nm. For determining conformance to the relative spectral irradiance requirements for a xenon-arc with extended UV filters, measurement from 250 nm to 400 nm is required. The total irradiance in each wavelength bandpass is then summed and divided by the specified total UV irradiance according to Eq A1.1. Use of this equation requires that each spectral interval must be the same (for example, 2 nm) throughout the spectral region used.

\[
I_\Delta = \frac{\sum_{\lambda=\Delta}^{\lambda_{\text{max}}} I_{\lambda}}{\sum_{\lambda=\Delta}^{\lambda_{\text{max}}} I_{\lambda}} \times 100
\]  

(A1.1)

where:

- \( I_{\Delta} \) = relative irradiance in percent,
- \( E \) = irradiance at wavelength \( \lambda \) (irradiance steps must be equal for all bandpasses),
- \( A \) = lower wavelength of wavelength bandpass,
- \( B \) = upper wavelength of wavelength bandpass,
- \( C \) = lower wavelength of total UV bandpass used for calculating relative spectral irradiance (290 nm for daylight filters, 300 nm for window glass filters, or 250 nm for extended UV filters), and
- \( \lambda \) = wavelength at which irradiance was measured.

APPENDIXES

(Nonmandatory Information)

X1. APPARATUS WITH AIR-COOLED XENON ARC LAMPS

X1.1 This test apparatus uses one or more air-cooled xenon arc lamps as the source of radiation. Different type and different size lamps operating in different wattage ranges may be utilized in different sizes and types of apparatus.

12. Keywords

12.1 accelerated; accelerated weathering; durability; exposure; laboratory weathering; light; lightfastness; non-metallic materials; temperature; ultraviolet; weathering; xenon arc
X.2. APPARATUS WITH WATER-COOLED XENON ARC LAMPS

X.2.1 The test apparatus uses a water-cooled xenon arc lamp as the source of radiation. Different size lamps operating in different wattage ranges may be utilized in different sizes and types of apparatuses.

X.2.2 The xenon-arc lamp used consists of a xenon burner tube, an inner filter of glass or quartz, an outer glass filter, and the necessary accessories. To cool the lamp, distilled or deionized water is circulated over the burner tube and then directed out of the lamp between the inner and outer glass filters.

X.3. EXPOSURE CONDITIONS

X.3.1 Any exposure conditions may be used, as long as the exact conditions are detailed in the report. Following are some representative exposure conditions. These are not necessarily preferred and no recommendation is implied. These conditions are provided for reference only (see Table X.3.1).

Note X.3.1—These exposure conditions are brief summaries of the actual exposure procedure. Consult the applicable test method or material specification for detailed operating instructions and procedures. Historical convention has established Cycle I as the commonly used exposure cycle. Other cycles may give a better simulation of the effects of outdoor exposure.

### TABLE X.3.1 Common Exposure Conditions

<table>
<thead>
<tr>
<th>Cycle</th>
<th>Filter</th>
<th>Incidence</th>
<th>Wavelength</th>
<th>Exposure Cycle</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Daylight</td>
<td>0.36 W/m²•m</td>
<td>340 nm</td>
<td>102 min light at 60°C Black Panel Temperature</td>
</tr>
<tr>
<td></td>
<td></td>
<td>18 min light and water spray (all lamp not controlled)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Daylight</td>
<td>0.36 W/m²•m</td>
<td>340 nm</td>
<td>102 min light at 63°C Black Panel Temperature</td>
</tr>
<tr>
<td></td>
<td></td>
<td>18 min light and water spray (all lamp not controlled)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>Daylight</td>
<td>0.36 W/m²•m</td>
<td>340 nm</td>
<td>15.6 h light, 70% RH, at 77°C Black Panel Temperature</td>
</tr>
<tr>
<td></td>
<td></td>
<td>6 h dark at 0.4% RH, at 24°C Uninsulated Black Panel Temperature</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>Window Glass</td>
<td>0.30 W/m²•m</td>
<td>340 nm</td>
<td>166 min light, 55% RH, at 65°C Black Panel Temperature</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.5 h light and water spray (all lamp not controlled)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Window Glass</td>
<td>1.10 W/m²•m</td>
<td>420 nm</td>
<td>102 min light, 56% RH, at 63°C Black Panel Temperature</td>
</tr>
<tr>
<td></td>
<td></td>
<td>18 min light and water spray (all lamp not controlled)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>Window Glass</td>
<td>1.10 W/m²•m</td>
<td>420 nm</td>
<td>3.8 h light, 55% RH, at 63°C Black Panel Temperature</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1 h dark, 56% RH, at 43°C Black Panel Temperature</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>Extended UV</td>
<td>0.56 W/m²•m</td>
<td>340 nm</td>
<td>40 min light, 60% (±5%) RH, at 70°C Black Panel Temperature and 47°C (±2) Chamber Air Temperature</td>
</tr>
<tr>
<td></td>
<td></td>
<td>20 min light and water spray on specimen face</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7A</td>
<td>Daylight</td>
<td>0.56 W/m²•m</td>
<td>360 nm</td>
<td>40 min light, 60% (±5%) RH, at 70°C Black Panel Temperature and 47°C (±2) Chamber Air Temperature</td>
</tr>
<tr>
<td></td>
<td></td>
<td>20 min light and water spray on specimen face</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>Extended UV</td>
<td>0.55 W/m²•m</td>
<td>360 nm</td>
<td>38 h light, 50% (±5%) RH, at 70°C Black Panel Temperature and 47°C (±2) Chamber Air Temperature</td>
</tr>
<tr>
<td></td>
<td></td>
<td>60 min light, 50% (±5%) RH, at 70°C Black Panel Temperature</td>
<td></td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>Daylight</td>
<td>160 W/m² at 390-400 nm</td>
<td>380-400 nm</td>
<td>102 min light at 63°C Black Panel Temperature</td>
</tr>
<tr>
<td></td>
<td></td>
<td>18 min light and water spray (temperature not controlled)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>Window Glass</td>
<td>162 W/m² at 390-400 nm</td>
<td>390-400 nm</td>
<td>106% light, 59% RH at 80°C Black Panel Temperature</td>
</tr>
<tr>
<td>11</td>
<td>Window Glass</td>
<td>1.6 W/m²•m</td>
<td>420 nm</td>
<td>Continuous light at 62°C uninsulated black panel temperature, 59% RH</td>
</tr>
<tr>
<td>12</td>
<td>Daylight</td>
<td>0.30 W/m²•m</td>
<td>340 nm</td>
<td>Continuous light at 62°C uninsulated black panel temperature, 29% RH</td>
</tr>
<tr>
<td></td>
<td></td>
<td>6 h dark at 50% RH, at 35°C dry bulb temperature</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
exposure. Cycle 3 has been used for exterior grade textile materials. Cycle 4 has been used for interior plastics. Cycles 5 and 6 have been commonly used for indoor textile materials. Cycle 7 has been used for automotive exterior materials. Cycle 8 has been used for automotive interior components.

Note X3.2—Cycle 7 corresponds to the test cycles specified in SAE J2412 and SAE J1885. Cycle 8 corresponds to the test cycles specified in SAE J2412 and SAE J1885. Consult the appropriate test procedure for detailed cycle descriptions, operating instructions, and a description of the filters used in this application. The filter system specified in these procedures is characterized in .

Note X3.3—More complex cycles may be programmed in conjunction with dark periods that allow high relative humidity and the formation of condensation at elevated chamber temperatures. Condensation may be produced on the face of the specimen by spraying the rear side of the specimen to cool them below the dew point.

Note X3.4—For special tests, a high operating temperature may be desirable, but this may increase the tendency for thermal degradation to adversely influence the test results.

Note X3.5—Surface temperature of specimens is an essential test quantity. Generally, degradation processes accelerate with increasing temperature. The specimen temperature permissible for the accelerated test depends on the material to be tested and on the aging criterion under consideration.

Note X3.6—The relative humidity of the air as measured in the test chamber is not necessarily equivalent to the relative humidity of the air very close to the specimen surface. This is because test specimens having varying colors and thicknesses may be expected to vary in temperature.

X3.2. Unless otherwise specified, operate the apparatus to maintain the operational fluctuations specified in Table X3.2 for the parameters in Table X3.1. If the actual operating conditions do not agree with the machine settings after the equipment has stabilized, discontinue the test and correct the cause of the disagreement before continuing.

Note X3.7—Set points and operational fluctuations could either be listed independently of each other, or they could be listed in the format: Set point ± operational fluctuations. The set point is the target condition for the sensor used at the operational control point as programmed by the user. Operational fluctuations are deviations from the indicated set point at the control point indicated by the readout of the calibrated control sensor during equilibration operation and do not include measurement uncertainty. At the operational control point, the operational fluctuation can exceed no more than the listed value at equilibrium. When a standard calls for a particular set point, the user programs that exact number. The operational fluctuations specified with the set point do not imply that the user is allowed to program a set point higher or lower than the exact set point specified.

X3.3 For conversion of test cycles from G26 to G155 see Table X3.3.
### TABLE X3.3 Conversion of Test Cycles from G26 to G155

<table>
<thead>
<tr>
<th>G26 Test Cycle Description</th>
<th>Corresponding Test Cycle in G155</th>
</tr>
</thead>
<tbody>
<tr>
<td>G26, Method A — Continuous light with intermittent water spray</td>
<td>Three cycles in G155, Table X3.1 use continuous light and the same water spray times as the conditions described in G26, Method A</td>
</tr>
<tr>
<td>The following test cycle is the only specific condition described</td>
<td></td>
</tr>
<tr>
<td>162 mm light only (uninsulated black panel temperature at 63 ± 3°C)</td>
<td>Cycle 1 uses daylight filters with 340 nm irradiance controlled at 0.35 W/m² (the suggested minimum 340 nm irradiance for daylight filters in G26, Method A)</td>
</tr>
<tr>
<td>16 min light, 1 min water spray</td>
<td>Cycle 5 uses window glass filters with 420 nm irradiance controlled at 1.10 W/m²/m² (the suggested minimum 340 nm irradiance for window glass filters in G26 is 0.7 W/m²/m²)</td>
</tr>
<tr>
<td>The type of filter and relative humidity during this light period are not specified</td>
<td>Cycle 9 uses daylight filters and 340 nm irradiance controlled at 1.35 W/m²/m² from 1000-400 nm</td>
</tr>
</tbody>
</table>

G25—Method B — alternate exposure to light and dark and intermittent exposure to water spray

No specific light-dark-water cycle described

The only conditions during the light period that are described are those of Method A. The length of dark period is not specified, nor are temperature or relative humidity conditions during the dark period.

G26—Method C — continuous exposure to light with no water spray

Uses window glass filters

Uninsulated black panel temperature is 63 ± 3°C, relative humidity is 56 ± 5%

Typical irradiance is 1.1 W/m²/m²

G155, Table X3.1, Cycle 11

G36—Method D — alternate exposure to light and darkness without water spray

No specific periods of light/dark are described

Type of filter and specified irradiance is not specified. Suggested minimum irradiance is 0.36 W/m²/m² at 430 nm with daylight filters or 0.2 W/m²/m² at 430 nm with window glass filters

RH controlled to 56 ± 5% during light period

Dark cycle requires a dry bulb temperature of 25 ± 3°C and 56 ± 5% RH.

G153, Table X3.1 Cycle 12
X4. COMPARISON OF BENCHMARK SOLAR UV SPECTRUM AND CIE 85 TABLE 4 SOLAR SPECTRUM


X4.2 Previous versions of this standard used CIE 85 Table 4 as the benchmark solar spectrum. Table X3.4 compares the basic atmospheric conditions used for the benchmark solar spectrum and CIE 85 Table 4 solar spectrum.

X4.3 Table X3.5 compares irradiance (calculated using rectangular integration) and relative irradiance for the benchmark solar spectrum and CIE 85 Table 4 solar spectrum, in the bandpasses used in this standard.

---

TABLE X3.4 Comparison of Basic Atmospheric Conditions Used for Benchmark Solar Spectrum and CIE 85 Table 4 Solar Spectrum

<table>
<thead>
<tr>
<th>Atmospheric Condition</th>
<th>Benchmark Solar Spectrum</th>
<th>CIE 85 Table 4 Solar Spectrum</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ozone (atm)</td>
<td>0.30</td>
<td>0.34</td>
</tr>
<tr>
<td>Relative humidity</td>
<td>6.47</td>
<td>6.42</td>
</tr>
<tr>
<td>Altitude (ft)</td>
<td>2000</td>
<td>2000</td>
</tr>
<tr>
<td>Azimuth</td>
<td>30° facing Equator</td>
<td>0° (horizontal)</td>
</tr>
<tr>
<td>Relative humidity</td>
<td>1.00</td>
<td>1.00</td>
</tr>
<tr>
<td>Albedo (ground reflectance)</td>
<td>Light Soil wavelength-dependent</td>
<td>Constant at 0.2</td>
</tr>
<tr>
<td>Aerosol extinction</td>
<td>Shuttle &amp; Fenn Rural</td>
<td>Equivalent to Linke turbidity factor of about 0.8</td>
</tr>
<tr>
<td>Aerosol optical thickness at 500 nm</td>
<td>0.05</td>
<td>0.10</td>
</tr>
</tbody>
</table>

---

TABLE X3.5 Irradiance and Relative Irradiance Comparison for Benchmark Solar Spectrum and CIE 85 Table 4 Solar Spectrum

<table>
<thead>
<tr>
<th>Bandpass</th>
<th>Benchmark Solar Spectrum</th>
<th>CIE 85 Table 4 Solar Spectrum</th>
</tr>
</thead>
<tbody>
<tr>
<td>Irradiance [Wm²] in stated bandpass</td>
<td>0.05</td>
<td>0.05</td>
</tr>
<tr>
<td>290 &lt; λ ≤ 320</td>
<td>3,758</td>
<td>3,060</td>
</tr>
<tr>
<td>320 &lt; λ ≤ 365</td>
<td>35,364</td>
<td>25,350</td>
</tr>
<tr>
<td>365 &lt; λ ≤ 400</td>
<td>31,250</td>
<td>20,050</td>
</tr>
<tr>
<td>400 &lt; λ ≤ 400</td>
<td>84,171</td>
<td>75,600</td>
</tr>
<tr>
<td>400 &lt; λ ≤ 600</td>
<td>462,330</td>
<td>473,830</td>
</tr>
<tr>
<td>Percent of 200 to 400 nm irradiance</td>
<td>0.0%</td>
<td>0.0%</td>
</tr>
<tr>
<td>290 &lt; λ ≤ 290</td>
<td>0.0%</td>
<td>0.0%</td>
</tr>
<tr>
<td>290 &lt; λ ≤ 320</td>
<td>0.8%</td>
<td>0.8%</td>
</tr>
<tr>
<td>320 &lt; λ ≤ 365</td>
<td>4.6%</td>
<td>3.8%</td>
</tr>
<tr>
<td>365 &lt; λ ≤ 400</td>
<td>54.5%</td>
<td>56.4%</td>
</tr>
<tr>
<td>Percent of 200 to 300 nm irradiance</td>
<td>0.0%</td>
<td>0.0%</td>
</tr>
<tr>
<td>290 &lt; λ ≤ 365</td>
<td>0.8%</td>
<td>11.0%</td>
</tr>
</tbody>
</table>

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9.0 REFERENCES


