Comparison and Characterization of Dyneema™ HB-210 and HB-212 for Accelerated UV Aging

Jonmichael A. Weaver, David A. Miller

Abstract—Ultra High Molecular Weight Polyethylene (UHMWPE) presents several distinct advantages as a material with a high strength to weight ratio, durability, and neutron stability. Understanding the change in the mechanical performance of UHMWPE due to environmental exposure is key to safety for future applications. Dyneema® HB-210, a 15 µm diameter UHMWPE multi-filament fiber laid up in a polyurethane matrix in [0/ 90]₂, with a thickness of 0.17 mm is compared to the same fiber and orientation system, HB-212, with a rubber-based matrix under UV aging conditions. UV aging tests according to ASTM-G154 were performed on both HB-210 and HB-212 to interrogate the change in mechanical properties, as measured through dynamic mechanical analysis and imaged using a scanning electron microscope. These results showed a decrease in both the storage modulus and loss modulus of the aged material compared to the unaged, even though the tan δ slightly increased. Material degradation occurred at a higher rate in Dyneema® HB-212 compared to HB-210. The HB-210 was characterized for the effects of 100 hours of UV aging via dynamic mechanical analysis. Scanning electron microscope images were taken of the HB-210 and HB-212 to identify the primary damage mechanisms in the matrix. Embrittlement and matrix spall were the products of prolonged UV exposure and erosion, resulting in decreased mechanical properties.

Keywords—Composite materials, material characterization, UV aging, UHMWPE.

I. INTRODUCTION

UHMWPE, sold by DSM as Dyneema®, is used in a broad range of applications. The unique material properties rank this multifilament fiber as a high-performance material. The base monomer ethylene, $-CH_2-$, is structured into long aligned molecular chains through a gel-spinning process as seen in Fig. 1, that creates a fiber with a high tensile strength while maintaining a lightweight hydrophobic structure approximately 15 µm in diameter. The ultra high molecular weight classification comes from the long length of the polymer chains, a distinguishing feature from other forms of thermoplastic polyethylene.

This fiber has a variety of uses due to the unique mechanical properties of high tensile strength while remaining lightweight, non-conductive, and hydrophobic [2]. The filaments are further processed into a composite material with a polyurethane matrix laid up in a unidirectional-cross-ply [0/90]₂ to create Dyneema® HB-210. Dyneema® HB-212 has the exact same fiber and structure as HB-210 but is consolidated with a rubber-based matrix. This composite can provide the same ballistic protection as Kevlar® while reducing weight and increasing the

resistance to damage from humidity [3]. Characterizing Dyneema® HB-210 and HB-212 composites for various environmental profiles, particularly UV, temperature cycling, and humidity stability, is necessary for user safety and accurately predicting the service interval or lifespan of the material in extreme environments.
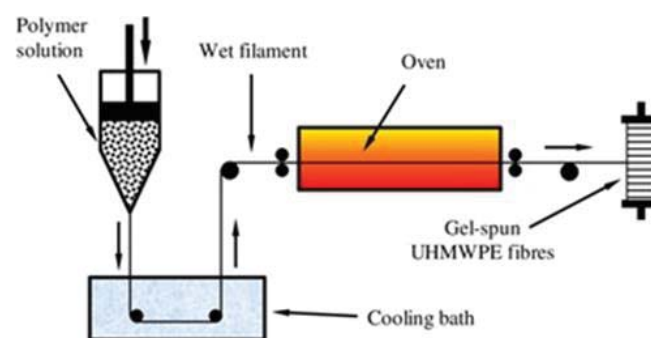

Fig. 1 Gel Spinning Process for Manufacturing UHMWPE Fibers [1]

Ideally, photodegradation of UHMWPE fibers is essentially null, meaning that the material is resistant to damage from sunlight. Pure UHMWPE does not absorb wavelengths longer than 190 nm [4]. UV radiation has three frequency band classifications: UV-A: 315 nm to 400 nm, UV-B: 280 nm to 315 nm, and UV-C: 100 nm to 280 nm [5]. As the wavelength decreases, the energy and subsequent potential for damage increases, making UV-C the most likely to damage materials. However, UV-C is filtered by the earth's atmosphere, whereas UV-A, the lowest energy of the UV spectra accounts for nearly all the UV radiation reaching the earth's surface. UV's non-ionizing radiation has the energy potential to penetrate materials, such as skin, or alter the mechanical properties of UHMWPE through defects, accelerating the aging process of other factors such as oxidation [4], [6], [7].

The non-ionizing UV photons have a minor potential to increase the crystallinity as proven through a morphological analysis [5]. Covalent bonds are the dominant force in polymers and can be damaged by UV through a photolysis, photo dissociation processes. Chain scission occurs on the molecular scale as a photon with an energy above the threshold to break bonds collides with the polymer backbone. These long molecular chains are severed, reducing the molecular weight, and altering the mechanical properties [8]. Additionally, the process of photolysis can release contaminates in the form of

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free radicals or byproducts into the composite system.

Manufacturing processes are not capable of producing pure UHMWPE filaments void of defects; the impurities containing hydroperoxides and other contaminates may shift the UV absorbance of the UHMWPE by over 200 nm [7]. Through UV exposure, the impurities can result in the formation of highly reactive radicals, which seek to join nearby bonding sites at the end of a polymer chain, potentially generating a cascading reaction that can result in both chain scissioning and cross-linking throughout the polymer structure. Free radicals can also bond with oxygen or water molecules, increasing the rate at which the effects of oxidation or hydrolysis take place in polyethylene [8].

In summary, UV degradation promotes electrons to move to higher energy states that ultimately result in the breaking of their respective bonding mechanisms and activating impurities in the material that accelerate other types of aging such as oxidation and hydrolysis. These changes in the physical structure of the material alter both the microstructure and potentially the chemical makeup, which impacts the mechanical properties.

II. METHODS

A. UV Aging

The UV aging was conducted according to ASTM-G154,

with a slight variation to account for equipment limitations. ASTM-G154, Standard Practice for Operating Fluorescent Ultraviolet (UV) Lamp Apparatus for Exposure of Nonmetallic Materials, details various aging profiles for UV degradation of materials. Fig. 2 describes the various cycle parameters of ASTM-G154 UV aging standards. Cycle 4 was selected which uses UVA-340 bulbs to irradiate the samples. The samples were loaded into a Q-Labs QUV Accelerated Weathering Tester, in which the UV bulbs were calibrated using an irradiance meter provided by Q-labs. These calibration tests discovered that the max irradiance the bulbs could maintain was $1.40 \frac{W}{m^2 nm}$ at an approximate wavelength of 340 nm, which is below the typical irradiance standard of $1.55 \frac{W}{m^2 nm}$ set by ASTM G-154. Therefore, the QUV tester was programmed to turn on the lamps at $1.20 \frac{W}{m^2 nm}$ for 8 hours with a back panel temperature of 70 °C cycled with 4 hours of the UV bulbs off while holding the back panel temperature at 50 °C. This cycle is repeated until samples were removed after 100 hours, 200 hours, 400 hours, and 800 hours of UV exposure, to be evaluated in the Dynamic Mechanical Analysis (DMA) and the Scanning Electron Microscope (SEM). Prior to initiating the test, the Q-Labs QUV's water tray and irrigation lines were all cleaned before refilling the water tray with unfiltered tap water.

Cycle	Lamp	Typical Irradiance	Approximate Wavelength	Exposure Cycle	Original Reference and Application, Where Known
1	UVA-340	0.89 W/(m² • nm)	340 nm	8 h UV at 60 (±3) °C Black Panel Temperature; 4 h Condensation at 50 (±3) °C Black Panel Temperature	D4329 cycle A for general Plastics; D4587 Cycle 4 for general metal coatings; C1442 for sealants
2	UVB-313	0.71 W/(m² • nm)	310 nm	4 h UV at 60 (±3) °C Black Panel Temperature; 4 h Condensation at 50 (±3) °C Black Panel Temperature	Unknown
3	UVB-313	0.49 W/(m² • nm)	310 nm	8 h UV at 70 (±3) °C Black Panel Temperature; 4 h Condensation at 50 (±3) °C Black Panel Temperature	SAE J2020
4	UVA-340	1.55 W/(m² • nm)	340 nm	8 h UV at 70 (±3) °C Black Panel Temperature; 4 h Condensation at 50 (±3) °C Black Panel Temperature	Unknown
5	UVB-313	0.62 W/(m² • nm)	310 nm	20 h UV at 80 (±3) °C Black Panel Temperature; 4 h Condensation at 50 (±3) °C Black Panel Temperature	Unknown
6	UVA-340	1.55 W/(m² • nm)	340 nm	8 h UV at 60 (±3) °C Black Panel Temperature; 4 h Condensation at 50 (±3) °C Black Panel Temperature.	Unknown
7	UVA-340	1.55 W/(m² • nm)	340 nm	8 h UV at 60 (±3) °C Black Panel Temperature; 0.25 h water spray (no light), temperature not controlled; 3.75 h condensation at 50 (±3) °C Black Panel Temperature	Unknown
8	UVB-313	28 W/m²	270 to 700 nm	8 h UV at 70 (±3) °C Black Panel Temperature; 4 h Condensation at 50 (±3) °C Black Panel Temperature	Unknown

Fig. 2 ASTM-G154 UV Exposure Times and Temperatures [9]

TABLE I
DYNEEMA® HB-210/HB-212 NUMBER OF TEST SAMPLES FOR UV AGING

Type	HB-210	HB-212
Unaged Solid	1	1
Unaged Laser Perforated	1	1
100 Hour UV Solid	2	2
100 Hour UV Laser Perforated	4	4
200 Hour UV Solid	2	2
200 Hour UV Laser Perforated	4	4
400 Hour UV Solid	1	1
400 Hour UV Laser Perforated	1	1
800 Hour UV Solid	2	2
800 Hour UV Laser Perforated	4	4

Table I contains the number of HB-210 and HB-212 samples in the various precut and solid forms with corresponding UV aging times.

B. Optical Interrogation

After each sample was removed from the Q-labs QUV tester, they were prepared for imaging using the field emission SEM. The samples were mounted onto the imaging tray using carbon tape and were coated with iridium.

Two coupons from each HB-210 and HB-212 aging length were selected at random for imaging using the SEM, with the exception of the unaged samples where only one was imaged.

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The two UV aged samples for each UV aged period were used to compare the side of the Dyneema® facing the UV bulbs and the backside in contact with the aluminum mounting fixture. All HB-210 and HB-212 samples were imaged under the SEM at 3 kV.

C. Dynamic Mechanical Analysis

A TA Instruments – DMA Q800 characterized the dynamic mechanical response of the UHMWPE under a temperature ramp. The samples were loaded, with consistent orientation, into a film tension clamp as shown in Fig. 3. The exposed 90° ply was placed towards the front of the machine with the 0° towards the back.

Fig. 3 DMA Film Tension Clamp with a Dyneema® HB-210 Sample Mounted

A preload of 0.1 N was initiated before oscillating at 1 Hz with a set amplitude of 100 µm, with a temperature heating rate of 2 °C per minute from 40 °C to 150 °C, and a 5-minute soak at 40 °C. Each test was conducted three times with each sample group of environmentally aged or control samples. These testing parameters were chosen based on previous tests of a Dyneema® yarn with a DMA [7]. As the QUV aging fixtures were too large to hold individual DMA samples, larger rectangles were scored with a laser engraver into the DMA coupon size before placing into the QUV tester, with a few unscored rectangles for larger surface exposure.

III. RESULTS AND DISCUSSION

A. Optical Analysis

The unaged samples were imaged to show a baseline for reference on how the material ages with UV exposure. In Fig. 4, comparing baseline (a) HB-210 and (b) HB-212, both samples have a full matrix intact and do not show any spalling, pitting, or matrix cracking.

As shown in Fig. 5, after 100 hours in the UV chamber the HB-210 (a) does not appear to show matrix degradation, while the rubber-based matrix in HB-212 (b) begins to show signs of spall. In the center of the images, a section of the top layer of

the ply is removed and the matrix in the HB-212 (b) is damaged and begins to degrade compared to the HB-210 (a) sample where the fibers are exposed while the matrix seems to be intact. This partial delamination occurred when mounting the HB-212 sample into the SEM.
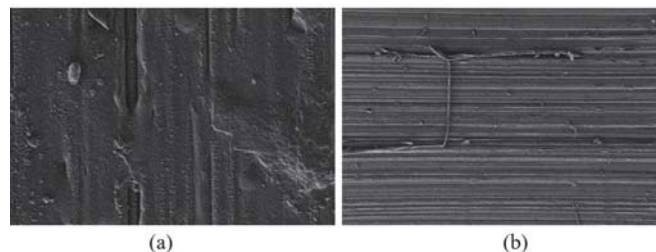

Fig. 4 Unaged single-ply Dyneema® HB-210 (a) and HB-212 (b) before UV exposure

The unaged HB-210 appears to have a rough texture in Fig. 5, while the 100-hour UV sample appears to have a smooth texture on the matrix in Fig. 6, this could have occurred from the temperature cycling causing the matrix to flow, along with some erosion from the dew/humidity in the ASTM-G154 test.
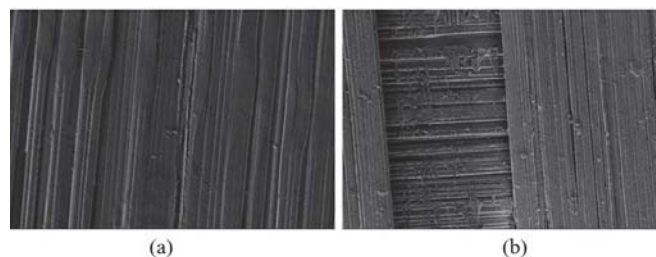

Fig. 5 SEM image of 100 Hour UV Exposure on Dyneema® HB-210 (a) and HB-212 (b)

As the aging of the samples increased to 200 hours of UV exposure, in Fig. 6 (b) the HB-212 begins exhibiting pitting and matrix loss. Most of the matrix has eroded away except for the small, pitted portion in the center of Fig. 6 (b) of the HB-212 sample. The HB-210 sample in Fig. 6 (a) appears to have most of the matrix intact and appears largely unaffected compared to the unaged sample.
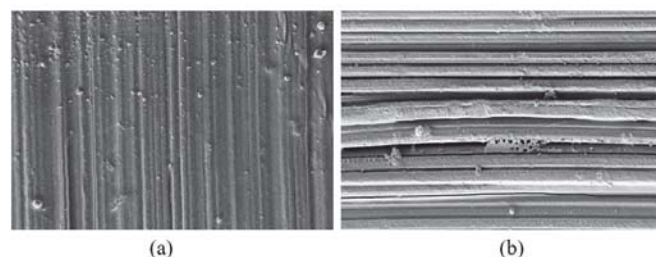

Fig. 6 SEM image of 200 Hour UV Exposure on Dyneema® HB-210 (a) and HB-212 (b)

The difference between the front side and backside of the coupons aged in the QUV machine is shown in Fig. 7, where the matrix of the HB-212 appears to erode faster on the UV

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exposed side. Erosion may have occurred from the humidity and condensation that was present as part of the testing parameters and accelerated the matrix degradation on Fig. 7 (a) compared to Fig. 7 (b). Fig. 7 (b) shows a small amount of matrix torn away as the backside of the coupon had some areas that adhered to the mounting fixture The HB-210 did not have a detectable difference between the exposed and backside faces.
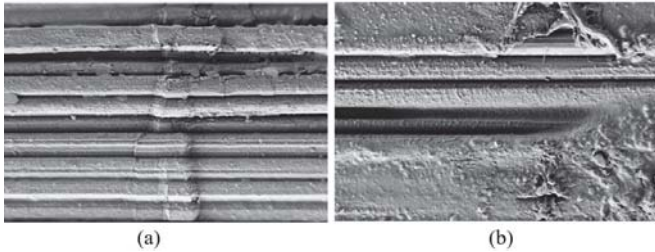
Fig. 7 HB-212 UV exposed face (a) vs backside face (b) for 200 Hour Exposure

After 800 hours with the UV bulbs on the HB-212 matrix had completely dissolved, the fibers became brittle. This sample was difficult to mount as the fibers had nothing holding them together. This made imaging difficult as the fibers did not easily adhere in a planar manner on the SEM sample holders. The fibers are lightweight and the slightest bit of wind or breath will disturb the fibers and cause them to fly off the sample holder. The HB-212 samples were not able to be measured in the DMA after 800 hours of aging. The HB-212 fibers became brittle compared to the unaged sample and would break during handling and removal from the QUV machine.

In Fig. 8 (a) the HB-210 sample after 800 hours of UV exposure shows pitting and matrix degradation similar to the 200-hour aging of the HB-212 sample in Fig. 6 (b), revealing that the polyurethane matrix is more resilient to UV damage compared to the rubber-based matrix but not completely immune to UV damage.
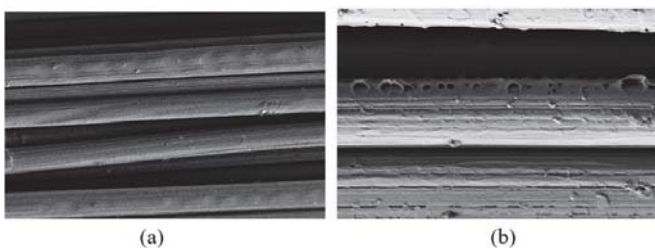

Fig. 8 Comparison of 800 Hour UV Exposure on Dyneema® HB-210 (a) and HB-212 (b)

The heat affected zone of HB-210 in Figs. 9 (a) and (b) is approximately 350 µm in width. This melted zone ends up impacting the DMA results through noise and grip slippage, rendering the DMA tests on laser cut HB-210 and HB-212 indiscernible.

Fig. 10 depicts the unaged HB-210 (a), and the unaged HB-212 (b) respectively as a reference for optical changes as the material ages.
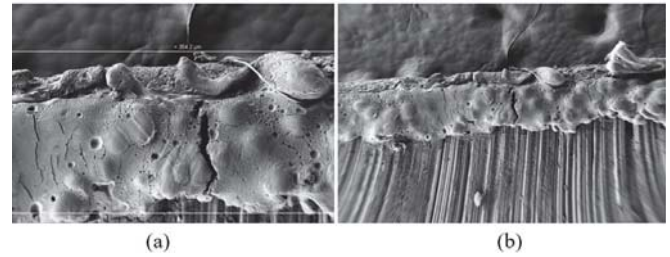

Fig. 9 Heat Affected Zone on Laser cut HB-210 210 Enlarged (a) and standard view (b) of the HB-210 Melted Edge
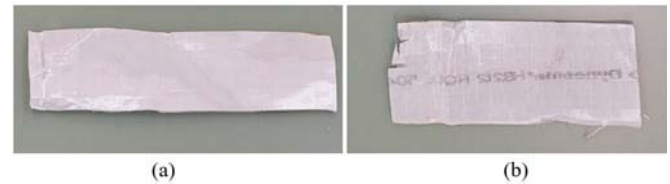

Fig. 10 Reference Image for Unaged HB-210 (a) the Unaged HB-212 (b)

In Fig. 11 (a) the HB-210 after 100 hours of UV exposure remains unchanged in appearance; the DMA coupons had already been removed before photographing resulting in the rectangular holes shown in Fig. 11 (a). Fig. 11 (b) shows the HB-212. After 100 hours of UV aging, the material appearance of HB-212 has changed, with a vertical line in the second column of DMA coupon perforations showing the edge of the window where the UV shines into the coupon. The HB-212 appeared to lighten and become opaquer with UV aging as seen to the right of the vertical delineation.

Fig. 11 100 Hour UV aged HB-210 (a) and HB-212 (b)

After 200 hours of UV exposure the HB-210 had no visible change, as seen in Fig. 12 (a). However, the material became stiffer and less malleable compared to the unaged HB-210. In the HB-212, Fig. 12 (b), the effect of UV became more apparent than the 100-hour sample. The window of UV exposure is more pronounced, with the left edge of the window bisecting the left most column of DMA samples, and the right edge bisecting the third from the right edge column. The DMA samples in the second column from the right were removed before aging due to damage from the laser engraver which was used to perforate the DMA shaped coupons from the larger sample. Discoloration appears most intensely in the center of the sample shown in Fig. 12 (b) in the center 3x3 perforated coupons after 200 hours of UV exposure. This darkening of the normally white composite was accompanied by embrittlement to the point of hold shape under its own weight in a cantilevered position, where the

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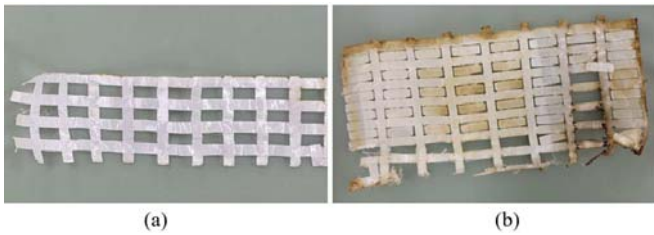unaged coupon appears limp and droops under the material weight in the same position.

Fig. 12 200 Hour UV aged HB-210 (a) and HB-212 (b)

At 800 hours of UV aging, the HB-210 sample in Fig. 13 (a) began to change opacity in the exposure window, but not to the same degree as the HB-212 in Fig. 12 (b). Fig. 13 (b) depicts the HB-212 after 800 hours of UV exposure, the yellowing disappeared as the matrix eroded away and the fibers debonded. The 800-hour HB-212 sample could not be handled without damaging the composite as the fibers were weak and brittle without the presence of the matrix.

Fig. 13 800 Hour UV aged HB-210 (a) and HB-212 (b)

B. Dynamic Mechanical Analysis

HB-210 and HB-212 were evaluated using a DMA to characterize the change in viscoelastic response over temperature and UV aging cycling.

It was discovered that the heat-affected zone from the laser on the edges of each DMA coupon induced noise into the data making the results invalid and inconclusive. Additionally, many of the tests done on the laser cut coupons slipped out of the DMA grips before the test had concluded the temperature ramp. Cutting the HB-210 with a blade by hand allowed for accurate DMA data to be collected and analyzed, mitigating the effects of the laser cut samples. Only the unaged and 100-hour UV tests on the HB-210 have been completed and processed through the DMA characterization for this publication.

Fig. 14 compares the tan δ of unaged and 100-hour UV-exposed HB-210. The overall tan δ increased with the UV aging by an average of 0.15 over the temperature range of the DMA experiment, possibly from molecular weight changes or alterations in the matrix properties. This is consistent with what is seen in tests of UV aged Dyneema® yarn, where the irradiated samples increase in tan δ [7]. The relative stability of the tan δ parameter throughout the DMA test suggests that as the temperature increases both the storage and loss modulus decrease at a constant rate, likely due to the cross-ply orientation of the composite.

With the increase in tan δ during UV aging, the HB-210 slightly increases the ratio of the tensile loss modulus compared to the tensile storage modulus. However, this does not indicate that UV aging increases the material dampening potential, only the ratio between the two moduli that make up the complex modulus. The complex modulus decreases with UV exposure, shown in Fig. 15 which compares the storage and loss modulus of the unaged to the 100-hour UV aged HB-210. Both storage and loss modulus of the unaged sample are greater than that of the aged sample for temperatures less than 125 °C. At 125 °C, the unaged and 100-hour UV loss modulus reach the same temperature and remain nearly identical for the duration of the test up to 150 °C. The unaged storage modulus remained higher than the UV aged storage modulus for the entire experiment.

The decrease in both the loss and storage modulus for the UV aging sample could have occurred from chain scissioning and cross-linking due to photolysis. Additionally, these scission processes can accelerate the effects of hydrolysis and oxidation at the defect and scission sites, further decreasing the storage moduli. The decrease in both storage modulus and loss modulus of the HB-210 after UV aging reduces the potential for this material to dampen and dissipate energy, which will result in diminished utility as a high-performance ballistics protection system.
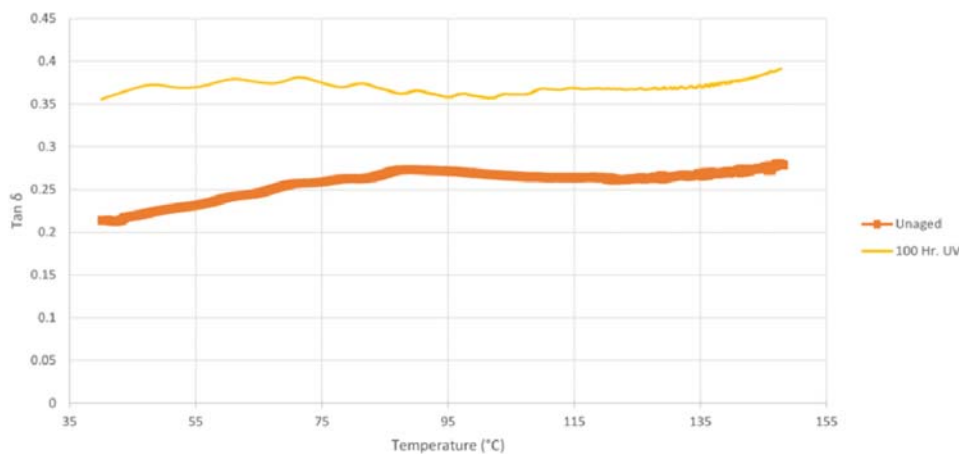

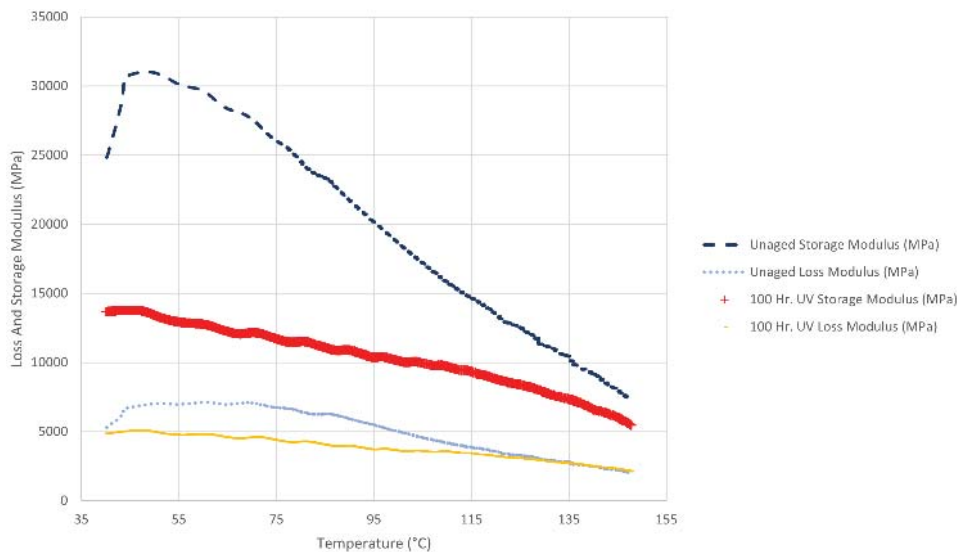Fig. 14 Tan Delta vs Temperature of HB-210 for Unaged and 100 Hour UV

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Fig. 15 HB-210 Storage and Loss Modulus vs Temperature for Both Unaged and 100 Hour UV

IV. CONCLUSION

The UV aging of a composite Dyneema® HB-210 and HB-210 made from UHMWPE fibers produces a quantifiable change in mechanical properties. After a combined UV, humidity, and temperature cycling experiment, the unidirectional cross-ply composite was evaluated using optical and DMA techniques.

Although pure UHMWPE theoretically does not absorb UV-A wavelengths, UV exposure creates a change in material properties. The UHMWPE composites Dyneema® HB-210 and HB-212 responded differently to the UV aging process suggesting that the matrix is the variable that is most susceptible to UV damage and degradation.

After 100 hours, the HB-212 became visibly opaquer with the SEM revealing spall had begun to occur. Under the SEM, the 100-hour HB-210 UV aged sample appeared smooth when compared to the rougher surface of the unaged HB-210. With further UV aging, the matrix continued to erode and spall leaving voids for damage through oxidation and hydrolysis. After 800 hours of UV exposure, the HB-212 would easily fragment, suggesting that nearly all of the matrix was eroded away. The 800-hour UV aged HB-210 under the SEM appeared similar to the 200-hour UV aged HB-212 as matrix pitting and erosion were detected.

Yellowing of HB-212 became visible after 200 hours of UV aging, while the HB-210 did not change in physical appearance. After 200 hours, HB-212 appeared dark yellow and brittle in the exposure window. The delineation between the UV aged coupons and the UV shielded coupons was apparent. HB-210 appeared less susceptible to the UV damage mechanisms compared to the HB-212 in both the 100-hour and 200-hour UV aging tests.

After 800 hours, the HB-212 lost virtually all the matrix; the fibers would not hold together as a composite anymore, and removing them from the QUV aging fixture was impossible without damage. Mounting the sample inside the SEM proved difficult but moderately successful. However, these samples cannot be mounted for DMA analysis as the fibers are too light and move around with the slightest wind or touch. The appearance of the HB-212 lightened, suggesting all the yellowed and browned matrix eroded away.

The DMA characterization concluded that the storage modulus and loss modulus decreased with UV exposure for HB-210, while the tan δ slightly increased, suggesting a change in the molecular weight of the polymer or in the matrix material properties. The edge effects of the melting of the fibers from laser cutting dominated the DMA tests and influenced grip slippage. DMA test with samples cut with a razor blade proved successful.

Further interrogation is needed to fully characterize the aging effects of UV, humidity and temperature cycling on Dyneema® HB-210 and HB-212. The HB-212 samples in DMA testing failed due to grip slippage without any valid DMA data to report. The DMA test temperature range will also be increased to investigate the behavior in the negative Celsius values. Additionally, isolating the effects of the temperature cycling, UV, and the presence of humidity individually on the mechanical characteristics will be explored.

All in all, the effect of UV aging on both Dyneema® HB-210 and HB-212 unidirectional cross-ply composite materials is measurable for a modified ASTM-G154 cycle 4 test. Both the loss modulus and storage modulus decrease, reducing the capacity for this material to function as a ballistics protection composite and decreasing the service. The HB-212 degraded more rapidly under the same environmental exposure compared to the HB-210.

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