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Quasi-static Characters for UHMWPE Composites after Hygrothermal Aging

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Abstract. This paper studied the Moisture absorption behaviors, DMTA temperature spectrum, Infra-red spectrogram, Quasi-static characters of the dry and hygrothermal UHMWPE composites. The material was UHMWPE fiber reinforced polyurethane matrix composite manufactured by hot press method, which was firstly kept in a heating oven, and then put in a beaker of distilled 70 °C water bath for 24 days and 60 days. The results show that there were two plateaus for the moisture absorption curve of the UHMWPE composites. The coupled effects of moisture and temperature reduce the composite glass transition temperature(T_g) by matrix plasticization and degrades the fiber/matrix interface. The storage modulus and loss modulus of dry and 24 days hygrothermal were gradually decreased in the initiatory calefaction, which drop sharply in T_g . The tensile strength was lossing in the beginning, and comeback to the initial at last in the hygrothermal environment. The quasi-static compressive strength is reduced by about 8% after 24 days treatment. Hygrothermal treatment has no effects on the quasi-static characters.

1. Introduction

Due to the need of protection and the lightweight consideration, the fiber reinforced composite material is gradually applied to the ship protection structures to resist the penetration of high-speed fragments. UHMWPE composite has excellent impact resistance, low density and stable chemical performance, and is one of the ideal materials for ship protection structures. Under the hygrothermal environment, the protective UHMWPE composite may be affected by environmental loads such as water, temperature and salt, resulting in the degradation of its performance. Hygrothermal environment may lead to the plasticization of composite matrix and degrade the fiber/matrix interface, resulting in a decrease of the quasi-static mechanical properties, especially the matrix-dominant mechanical properties. However, researchers have come to different conclusions about the influence of moisture on the static characters of composites. Wollesenbet's study shows that the dynamic compressive peak stress of the graphite/epoxy composite was firstly increased and then decreased with the increase of moisture absorption[1]. However, HAQUE[2] and SNWOSU[3] found that moisture absorption will reduce the dynamic compressive peak stress of composites. The effects of hygrothermal treatment on the compressive behavior of composites needs further research[4-8]. This paper studied the effects of hygrothermal treatment on quasi-static characters of UHMWPE composites. Quasi-static characters conducted by a MTS machine, and DMTA test, and infra-red spectrogram experiment. The moisture absorption and its effects on the composite compressive properties were studied in detail.

2. Experimental work



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2.1. Material description

The material studied in this paper was UHMWPE fiber reinforced polyurethane matrix composite manufactured by hot press method. UHMWPE fibers, coated with polyurethane, were stacked by 0/90° and then kept at pressure of 15MPa, temperature below 120 °C for approximately 30 minutes in a hot press machine to fabricate the plates measuring 300×300mm with a thickness of 4 or 10mm. The single ply of the plates had a thickness of about 0.13mm and the mass fraction of the polyurethane matrix was ~15%. Figure 1 shows the surface of the as-manufactured UHMWPE composite, proving a good compatibility of the UHMWPE fibres and the PU matrix. The composite panels were cut into quasi-static compressive specimens of 10 × 10 × 10mm cubes, and dynamic compressive specimens of $\Phi \times h = 12 \times 4$ mm cylinders by water-jet method. For the dynamic compressive specimens, diameter Φ was the in-plane direction while height h was the through thickness direction. The basic material description is listed in Table1.

Table 1. Material description of the [0/90]_n UHMWPE/polyurethane composites

Composite Density g/cm ³	Resin Fraction	Mass	Ply Thickness / mm	T_g / °C	Tensile Strength / MPa	Tension Modulus / GPa	Short-beam Shear Strength / MPa
0.95	0.1-0.15		0.13 ±0.02	118	541	28.5	6.65

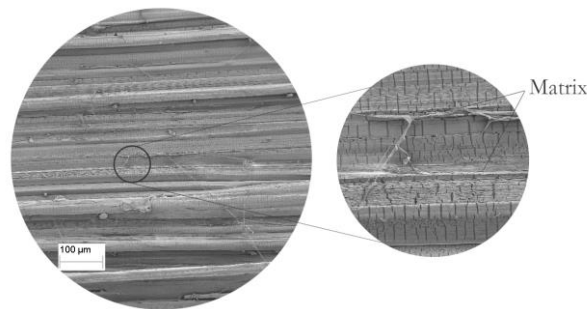


Figure 1. SEM images of the UHMWPE composite surface

2.2. Specimens of Hygrothermal Aging

Specimens were firstly kept in a heating oven to reach the engineering dry condition until the daily mass loss less than 0.02%. Heating temperature was set at 50 °C which is below the composite glass transition temperature $T_g = 118$ °C. Subsequently, the dry specimens were put in a beaker of distilled water and the beaker was kept in a 70 °C water bath for 24 days. Five specimens were numbered and taken out regularly for weighing using an OHAUS analytical balance. Hot, wet specimens were cooled for a short time to reach the room temperature and surface water was removed before weighing. Moisture absorption rate of the composite under hygrothermal environment is calculated according to formula (1)

$$W_t = \frac{M_t - M_0}{M_0} \times 100 \quad (1)$$

where, W_t , M_t and M_0 represent the moisture absorption rate, weight of the hygrothermal treated specimens and weight of the dry specimens, respectively. Average moisture absorption rate of the numbered five specimens was regarded as the moisture absorption rate of the UHMWPE composite.

2.3. DMTA Tests

The DMTA tests of dry specimens and hygrothermal specimens with 15 days, 30days, and 60days were measured by METTLER TOLEDO 1/236 machine. The specimens size were 40×4×2mm with long, wide, and thickness. Every experiment is done for 3 specimens. The method of experiment was three point bending test of frequency 1Hz, loading 5N, temperature 30-140 °C, speed of heating 3K/min.

2.4. Infra-red spectrogram and SEM Tests

Infra-red spectrogram test were done by Nicolet IS5 spectrum machine. The specimens consist of dry, hygrothermal 30 days, and hygrothermal 60 days. The method of test was ATR, which scan scope was $400\text{--}4000\text{cm}^{-1}$. The SEM test of fracture area for drawing specimens were done by Zeiss field emitter electron microscope SUPRATM 55.

2.5. Quasi-static Experiments

The drawing and interceded cutting tests were conducted. The specimens consist of dry, hygrothermal 4 days, 8 days, 15 days, 30 days and 60 days. A test method of drawing was chosen from reference [8]. The specimens were dumbbell shape, which was $240 \times 4\text{mm}$, and the middle wide was 4mm. The specimens were connected with tongs by M4 bolt, which solved the specimens delaminating by traditional methods. The schematic of the tension specimen and test system were showed in Figure 2. A test method of cutting test was choosing from JC/T 773-2010, which was short beam test method. The specimens were $30 \times 8 \times 4\text{mm}$. The span of loading was 20mm. The test machine was MTS E45, whose loading speed was 2mm/min. Each test case was carrier out for 4 specimens.

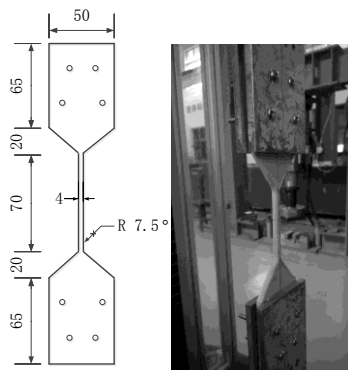


Figure 2. Schematic of the tensile specimen and test system

3. Results and discussions

3.1. Moisture absorption

Moisture absorption rate of the hygrothermal treated UHMWPE material is shown in 0. The moisture absorption rate rises proportionally to the square root of the time initially and then reaches two plateaus of 1.92% and 2.47% on day 6 and day 12, respectively. The moisture absorption process may be explained as follows. Moisture firstly diffused into the composite matrix and occupied the inner voids, reaching the first plateau. The inner voids and cracks expanded under the long time treatment of moisture and high temperature, leading to more moisture absorption and then reaching the second plateau.

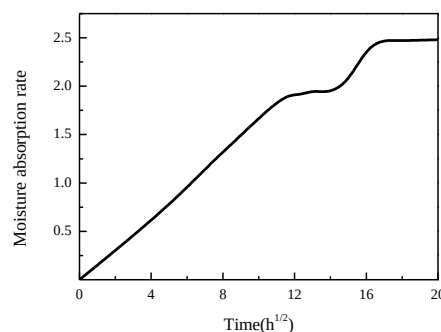


Figure 3. Moisture absorption curve of the hygrothermal treated specimens

The moisture absorption property of UHMWPE/PU was showed in Figure 4, which was hygrothermal in $70 \pm 3\text{ }^{\circ}\text{C}$, $95 \pm 5\%$ R.H. The moisture absorption rate was faster in the initiatory phase. The case was

the water molecule attaching the sample surface, and pervade in PU. The pervasion process of water in PU was slowness. The absorption rate gradually turn slowly, and mitigation ultimately. UHMWPE/PU moisture absorption rate was low, because the UHMWPE fiber was non-polarity material, which absorption rate blow 0.01%[4], and the content of PU was lesser, which showed that the influence was little to whole composite sample.

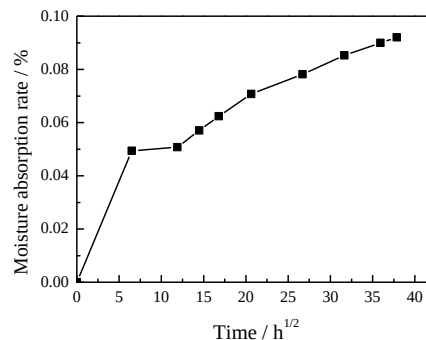


Figure 4. Moisture absorption rate curve of the UHMWPE/PU composite

3.2. DMTA

The DMTA temperature spectrum of the dry UHMWPE/PU composite showed in Figure 5. The storage modulus (E') and loss modulus (E'') were gradually decreased in the initiatory calefaction, which fastly reduce in T_g . The loss factor ($\tan \delta$) gradually hoist, and form a pinnacle in T_g , which suddenly surge after T_g . If the first pinnacle of the composite was the T_g , the T_g of dry sample was 120.5 °C.

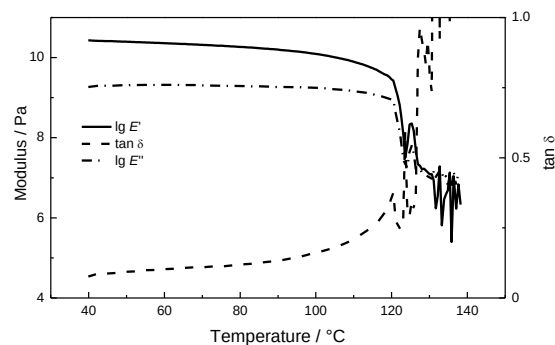
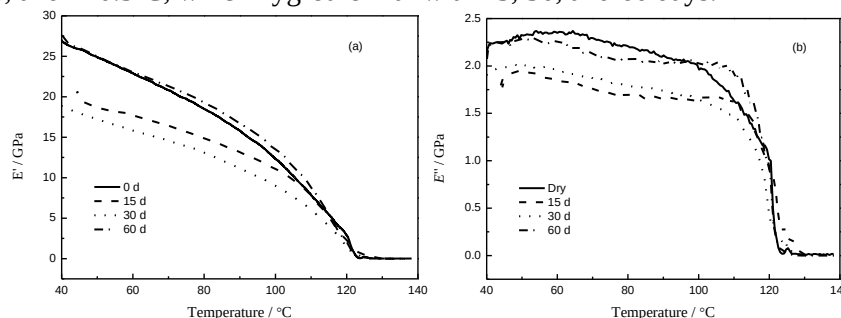


Figure 5. DMTA temperature spectrum of the dry UHMWPE/PU composite

The storage modulus, and loss modulus, and $\tan \delta$ curves of the UHMWPE/PU composite with dry and in different hygrothermal times sample were showed in Figure 6. The modulus didn't deal with logarithm, which showed the change of storage modulus with different hygrothermal time. The storage modulus firstly reduced along with the increasing of hygrothermal time, then rising between the temperture 40-100 °C. The storage modulus curves were superposition of dry and hygrothermal with 60 days. The trend of loss modulus is similar to the storage modulus. The T_g of UHMWPE/PU were 121.4 °C, 120.4 °C, and 120.3 °C, which hygrothermal with 15, 30, and 60 days.



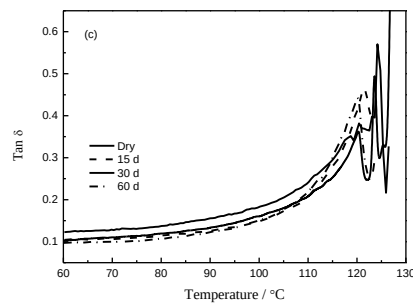


Figure 6. (a) Storage modulus, (b) loss modulus and (c) $\tan \delta$ curves of the UHMWPE/PU composite in hydrothermal environment with time

The DMTA tests results of the dry and 24 days hydrothermal treated UHMWPE composites are shown in 0. Hydrothermal treatment seems to decrease the storage modulus (E') and to reduce the glass transition temperature T_g from 118 °C to about 115 °C (the first peak of $\tan \Delta$). When the temperature exceeds T_g , the storage modulus and the loss modulus drop sharply, leading to the irregular change of the $\tan \Delta$. The decrease of T_g may indicate the plasticization of the specimen matrix because plasticization usually degrades the glass transition temperature [7-8]. Plasticization also leads to lower buckling stress of composite materials and this is consistent with the quasi-static compression results described later.

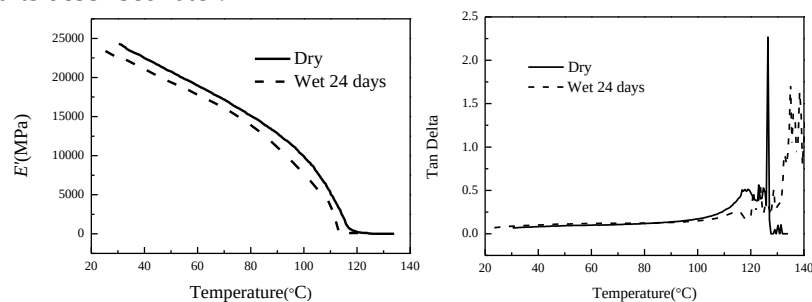


Figure 7. DMTA tests results of the dry 24 days wet specimens

3.3. Infra-red spectrogram

The Infra-red spectrograms of UHMWPE/PU composite at different aging time were showed in Figure 8. There were not new diagnostic peak in IR spectra after hydrothermal environment. The location and relatively size of peak were the same with the dry samples. These showed that there were not new matter engender between the process of hydrothermal.

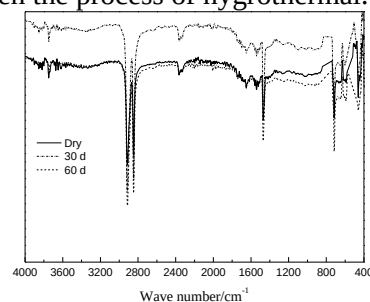


Figure 8. Comparison of IR spectra of UHMWPE/PU composite at different aging time

3.4. Quasi-static characters

The SEM photographs of the fractured the UHMWPE / PU composite was showed in Figure 9. The UHMWPE fiber extend to rupture after neck shrink, which produced the nicer extend property. The PU attaching in the face of the fiber was little, because of the min content of PU, and non- polarity of fiber.

The variation curves of tensile strength and short-beam strength of UHMWPE/PU composite in hygrothermal environment with time were showed in Figure 10. The initial tensile strength was 541.5 MPa. The tensile strength was losing in the beginning, and comeback to the initial at last in the hygrothermal environment. The reason maybe the hygrothermal environment reinforce the tensile strength. The short-beam strength was decreased following the hygrothermal time. The reserve rate of short-beam strength was 69.7% after 60 days hygrothermal.

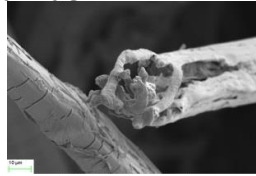


Figure 9. SEM photographs of the fractured the UHMWPE / PU composite

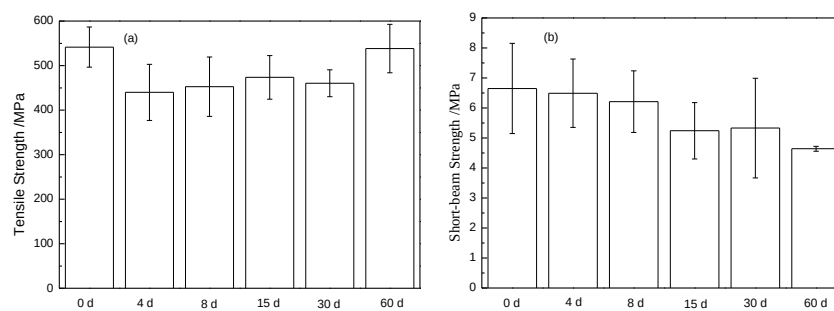


Figure 10. Variation curves of (a) tensile strength and (b) short-beam strength of UHMWPE/PU composite in hygrothermal environment with time

The out-of-plane quasi-static compressions at strain rate 0.001/s and 0.01/s were conducted on the virgin dry samples and 24 days hygrothermal treated samples. Hygrothermal treatment decreased the compressive strength by about 8%, as depicted in Figure 11 (a). The typical compressive stress-strain curves at strain rate 0.001/s are shown in Figure 11 (b). Stress had initially increased nonlinearly with strain before 0.15 and then increased linearly until a catastrophic failure occurred with a very loud burst sound and fractured plies ejecting from the edges of the samples. Fibres extruding out transverse to the local fibre direction can be seen over the side faces of the samples, as shown in Figure 12.

Ravichandran have studied the out-of-plane compression behaviour of Dyneema UHMWPE composites. The existence of shear-lag zone was proved at the periphery of specimens. Lower matrix shear strength, and void-like defects resulting from missing groups of fibres, could degrade the out-of-plane compressive strength^[6]. The degradation effects of the hygrothermal treatment on compressive strength in our tests thus can be explained as follows. Water absorption usually causes plasticization of the composite matrix, inconsistent swell of matrix and fibres, and expansion of the inner voids. The lower matrix quasi-static shear strength caused by plasticization thus decreases the compressive strength. Fibre/matrix interface degradation and inner voids expansion caused water absorption have the same effects as lower the matrix shear strength.

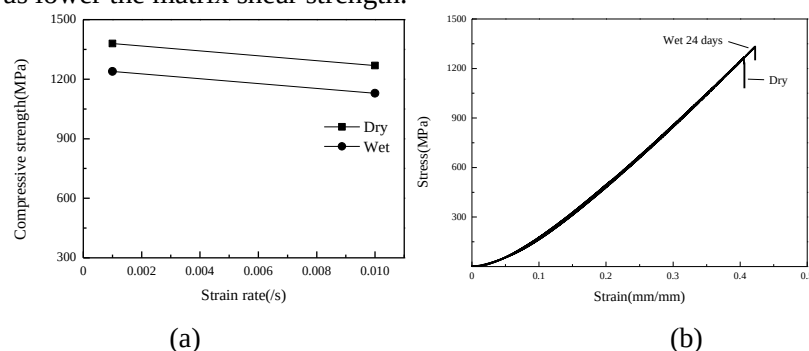


Figure 11. (a) Quasi-static strength and (b) stress-strain curves of dry/wet composite

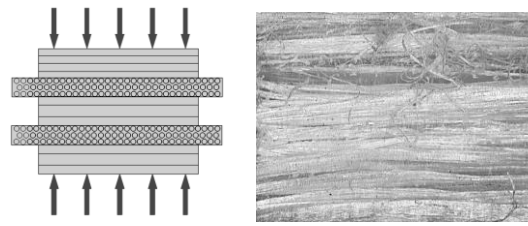


Figure 12. Optical micrograph of the quasi-static compression specimen

4. Conclusions

In this paper, we investigated the quasi-static characters of hygrothermal treatment UHMWPE/PU composites. The conclusion can be summarized as follows:

- (1) There were two plateaus for the moisture absorption curve of the UHMWPE composites. The coupled effects of moisture and temperature reduces the composite T_g by matrix plasticization and degrades the fiber/matrix interface.
- (2) The storage modulus and loss modulus of dry and 24 days hygrothermal were gradually decreased in the initiatory calefaction, which drop sharply in T_g .
- (3) There were not new diagnostic peak in IR spectra after hygrothermal enviroment.
- (4) The quasi-static compressive strength is decreased by about 8% after 24 days treatment.

5. References

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