Introduction

Flexible protective composites have gradually become a research hotspot in the field of protective materials due to their light-weight, strong flexibility, and good protective effect. Most composite materials used for impact resistance are prepared from fibers or fabrics with high toughness and tensile strength. Ultra-high molecular weight polyethylene (UHMWPE) has excellent mechanical properties including, low density, light weight, good toughness, high tensile strength, low moisture absorption, chemical resistance, and strong wear resistance. These mechanical properties have made UHMWPE to be an ideal reinforcing component. It hence widely used in manufacturing of composite materials. However, UHMWPE also has many shortcomings, such as low surface energy and poor heat resistance. These lead to poor interfacial adhesion and hinder the development of high-performance UHMWPE composites. Therefore, it is necessary to modify UHMWPE surface to optimize the fiber adhesion. Plasma surface modification is a commonly used method to increase interfacial adhesion of the composites.

Abstract

This study explored the influence of low temperature glow discharged argon (Ar) plasma on interfacial performance and impact resistance of ultra-high molecular weight polyethylene (UHMWPE) inter-ply hybrid composites. The composites were composed of UHMWPE and meta-aramid plain woven laminates with shear thickening fluid (STF). Water contact angle and drop-weight resistance of the composites with various Ar plasma treatment parameters were tested to investigate the interfacial performance and impact properties of the composites. The tested treatment parameters of this study included treating time, treating power, and gas flow rate. It was found that the best interfacial adhesion of UHMWPE and the impact resistance of the composites was realized at the plasma treatment power of 100 W, treatment time of 150 s, and gas flow rate of 4 sccm. In the follow-up research, this study conducted ballistic test to further explore the bulletproof effect and application prospect of this material.

Keywords

UHMWPE, shear thickening fluid, argon plasma, inter-ply hybrid composites

Date received: 2 March 2021; accepted: 17 August 2021
Plasma technology has the characteristics of surface modification control, fast processing speed, high efficiency, low energy consumption, and hence conserves energy. The technology also protects environment because it has no waste and pollution. It is a dry and clean method. The common plasma methods used for fiber interface treatment include plasma etching, plasma activation, functionalization, plasma induced grafting, and plasma polymerization for polymer surface modification. All kinds of gases can be used to produce plasma. The gas ions produced collide with the exposed sample to modify surfaces. Micro-pits are formed on the fiber surface because of plasma etching and hence, increases the surface roughness. The main factors induced by plasma to increase surface free energy and adhesion are the surface roughness, crystallinity, and surface activation. The influence of plasma treatment on textiles depends on several process parameters of plasma, such as working gas type, treating time, power, pressure, and gas flow rate.

The surface energy and roughness of the textile samples are increased when treated with hydrogen plasma and when treated with argon plasma. According to Magureanu et al. and Slepička et al. the surface adhesion of the samples was improved and their catalytic activity was significantly enhanced. Spyrides et al. found that argon and oxygen plasma were the best choices for surface activation when the main objective is to increase the wettability of polyethylene (PE). Furthermore, the argon plasma was expected to generate surface roughness to physically remove surface material through atomic collisions. After the plasma is applied to the surface of the fabric, the original chemical bonds on the surface of the fabric are broken. The free radicals in the plasma can form a cross-linked structure with the broken bonds and this greatly activates the surface activity.

To prepare flexible protective materials, combination of STF and high-performance fiber fabric has been widely used in China and other countries. Shear thickening fluid is a fluid that shows non-Newtonian behavior. It is a suspension that is usually composed of solid particles (such as silica particles) and dispersion medium (such as water, ethylene glycol, and polyethylene glycol) and shows shear thickening phenomenon. UHMWPE were impregnated in the STF solution and then dried. The SiO₂ particles are uniformly adhered to the surface of the fabric, thereby enhancing the impact resistance of the fabric. Shear thickening fluid impregnation process provides substantial energy absorption enhancements at lower mass densities, maintaining or enhancing the impact resistance, stab resistance and cutting performance of the structure, and even has the potential for ballistic protection.

In this study, UHMWPE was modified by glow discharge low temperature argon plasma with different time, gas flow rate, and power. To obtain UHMWPE/STF flexible composite the UHMWPE surface composite meta-aramid laminates were later then immersed in STF. The surface modification effect of STF/UHMWPE flexible composites was evaluated using SEM scanning electron microscope test and water contact angle test. This is done by dropping the liquid on a solid surface, collect the image, and observe. The contact angle of the liquid to the solid is measured and analyze the wettability of the liquid to the solid is analyzed. The difference of impact resistance was tested through drop-weight impact test. This obtains the load-displacement curve by using the drop hammer with the specified mass and size to impact the sample with the specified height from the specified position. The optimal process was obtained by response surface analysis and the difference of its appearance was observed.

### Materials and methods

#### Materials

Ultra-high molecular weight polyethylene plain fabrics and meta-aramid plain fabric were purchased from Guangdong Tevlon New Materials Co. Ltd. and Dongguan Sanmau Special Weaving Technology Co., Ltd., respectively. The specifications of the high-performance fiber reinforcements were as shown in Table 1.

#### Preparation of Ar plasma modified UHMWPE/STF inter-ply hybrid composites

In order to remove the impurities and oil on the surface of the high-performance fiber reinforcements, the samples were immersed in acetone solution with concentration levels of 50%, 75%, and 100% by the one or two times via ultrasonic cleaning machine (Ningbo Scientz Biotecnology Co., Ltd.)

The UHMWPE plain fabrics were treated with Ar discharge plasma through a glow discharge cold plasma treatment system (HPT-300, Henniker Scientific, UK). This was to improve the chemical modification and surface morphology. According to the change of water contact angle under different treatment conditions, the parameters value with obvious change were selected. The samples Ar discharged plasma modified UHMWPE were fabricated by various treating time, treating power and gas flow rate with three factors and three levels. The specifications of sample design are shown in Table 2.

Polyethylene glycol (PEG) and the silicon dioxide (SiO₂) were utilized as dispersion medium and dispersed phase particle, respectively. Silicon dioxide particles with a diameter of 650 nm were added into PEG with the mass fraction of 45% and were stirred by mechanical agitation. The mixture was then treated using ultrasonic cleaning.
machine for 60 min to obtain STF suspension with uniform distributed silica nanoparticles.

Shear thickening fluid suspension was diluted with absolute ethyl alcohol at the rate of 1:4 v/v. Every UHMWPE and meta-aramid sheet were impregnated in the STF for 60 min by using ultrasonic device. They were then dried in vacuum drying oven at 45°C for 60 min to evaporate the absolute ethyl alcohol and obtain the final UHMWPE/STF inter-ply hybrid composite. The preparation process of UHMWPE/STF inter-ply hybrid composites was as presented in Figure 1.

Testing

Water contact angle test was carried out using JC200D1 contact angle measuring device (Shanghai Zhongchen Digital Technic apparatus Co., Ltd). The angle measurements ranged from 0° to 180° with a resolution of 0.01°. Deionized water was used as the wetting liquid. Five specimens were tested for each sample.

The morphology of reinforcements and composites under different treatment conditions were observed using the HITACHI TM-3000 scanning electron microscopy.

Drop-weight impact test was conducted using the universal material testing machine according to the standard of ASTM D7136/D7137M-12. The impactor head had a circular shape and a weight of 2 kg. The impact energy was set at 10 J. The composites were trimmed into a dimension of $160 \times 110 \text{mm}^2$. Three specimens were tested for each sample.

Results and discussion

Effect of acetone cleaning

The UHMWPE reinforcement were cleaned with once time cleaning of all concentration acetone solution (Figure 2). Results shown in Figure 2(a) and (b) indicates that the UHMWPE reinforcement still retained impurities and oil at low concentration acetone solution of 0% and 50%. Figure 2(c) and (d) shows that the sample was completely cleaned by acetone solution at high concentration of 75% and 100%.

Results shown in Figure 3 indicate that the UHMWPE reinforcement were completely cleaned with double time cleaning of all concentration acetone solution. UHMWPE reinforcement still retained impurities and oil which was not cleaned with acetone (Figure 3(a)). The UHMWPE reinforcement was cleaned with double time cleaning of low concentration acetone solution at 50% concentration (Figure 3(b)). Results shown in Figure 3(c) and (d) indicate that the sample was completely cleaned by double time acetone solution at high concentration of 75% and 100%. It was found that an increase in acetone concentration and cleaning times caused a decrease in the content of oil and impurities on the surface of the fabric (Figure 2(c)). For high efficiency, the UHMWPE reinforcement with a single time cleaning using acetone solution at concentration of 75% was selected for the current study.

Effect of Ar plasma treatment on morphology

It has been found that plasma treatment can improve the roughness and surface adhesion of samples. After argon plasma treated UHMWPE was impregnated with STF, the adhesion amount of SiO$_2$ increased significantly as indicated by SEM photographs.

Results shown in Figure 4 illustrates that the SEM photographs of the UHMWPE reinforcement and composites. The surface without plasma treatment of the fabric was very smooth (Figure 4(a)). The UHMWPE reinforcement sample with 150 W–60 s–4 sccm plasma treatment (Figure 4(b)) exhibited a rougher surface than the UHMWPE reinforcement without plasma treatment (Figure 4(a)).

| Table 1. Specifications of high-performance fiber reinforcements. |
|---|---|---|---|---|---|
| Reinforcement types | Fineness of yarn (filaments/strand) | Areal density (g/m$^2$) | Warp density (ends/inch) | Weft density (picks/inch) |
| UHMWPE | 400 | 130 | 33 | 33 |
| Meta-aramid | 1000 | 200 | 21 | 21 |

| Table 2. Specifications of Ar discharged plasma modified UHMWPE. |
|---|---|---|
| Sample code | Treating time (s) | Treating power (W) | Gas flow rate (sccm) |
| 1 | 120 | 75 | 4 |
| 2 | 120 | 100 | 2 |
| 3 | 120 | 100 | 4 |
| 4 | 120 | 100 | 6 |
| 5 | 120 | 125 | 4 |
| 6 | 120 | 125 | 6 |
| 7 | 150 | 75 | 2 |
| 8 | 150 | 75 | 6 |
| 9 | 150 | 100 | 4 |
| 10 | 150 | 100 | 6 |
| 11 | 150 | 125 | 2 |
| 12 | 150 | 125 | 6 |
| 13 | 180 | 75 | 4 |
| 14 | 180 | 75 | 6 |
| 15 | 180 | 100 | 2 |
| 16 | 180 | 100 | 6 |
| 17 | 180 | 125 | 4 |
In terms of compounding with STF without plasma treatment (Figure 4(c)), the composites with 150 W–60 s–4 sccm plasma treatment Figure 4(d)) were attached more SiO₂ particles, which was because plasma treatment improved the surface roughness of the UHMWPE reinforcement. Furthermore, the interface bonding between reinforcement and STF were also significantly increased. This shows that plasma can change the surface binding property of the fabric and the surface roughness of the fabric can be improved by plasma treatment. Therefore, the interfacial bonding between the fabric and the STF can be improved remarkably which beneficial to the composite of fabric and STF.

Effect of Ar plasma treatment on water contact angle

Argon plasma treatment is the best choice to improve the wettability of fabric.¹¹,¹² With the change of the treatment parameters, water contact angle varies the optimal treatment parameters will be selected.
It was found that the treating power, treating time, and gas flow rate have an impact on the low temperature glow discharged plasma and interfacial performance (Figure 5). The results of this study found that an increase in treating power led to a gradual decrease in the water contact angle (Figure 5(a)). When the power was at 50 W, the water contact angle was 110°, which means that it was hydrophobic. On the other hand, when the power was at 125 W, the water contact angle was 80°, exhibiting a critical state of hydrophobic and hydrophilic. It took 3 min for complete soaking to occur. However, when the power was increased to 175 W, it took 4 s for complete soaking to occur.
Results shown in Figure 5(b) and (c) indicate that water contact angle decreased significantly with an increase in gas flow rate and treating time. When the gas flow rate and treating time was set to 3 sccm and 40 s, respectively, the water contact angle changed gently and was stabilized at 80°. It was evident that the steepness of the image was (a) > (b) > (c), when only one factor is changed. This indicates that the treating power has the greatest effect on the contact angle, followed by the treating time and finally the gas flow rate (Figure 5). Meanwhile, the results of this study show that the treating power has the greatest influence on the low temperature glow discharged plasma on interfacial performance.

**Effect of Ar plasma treatment on resistance impact**

A typical load-displacement curve of impact procedure of composite was as presented in Figure 6. It was found that the fabric is affected by the external force of the drop hammer. Further, the impact force increases with the decrease in the distance between the drop hammer and the fabric. It was found the force increases instantaneously as a result of short duration of the impact. This kind of fabric is a very strong and cannot be hit with a hole, but only a dent. Due to the high concentration of stress points, yarn drawing occurs easily near the stress points and this results into uneven stress around the fabric. Section A of this study showed a static friction stage and the main energy absorption mechanism in this stage was the composites deformation caused by the instantaneous impact. On the other hand, section B was a dynamic friction stage and the main energy absorption mechanism in this stage was sliding friction, resulting in uneven stress and fluctuation.

The influence of plasma treatment on textiles depends on several process parameters of plasma such as treating time, power, pressure, and gas flow rate. The most important influencing factors were found based on the impact strength of composite materials under the different parameters.

The results shown in Figure 7(a) indicates that the gas flow rate is high under the premise of constant time and power. This was shown by the black curve at the top followed by the red curve. Moreover, the results show that the longer the treatment time, the better the impact resistance. This was indicated by black curve of 180 s treatment being higher than the blue curve of 120 treatment. Therefore, it was evident that the impact force of the UHMWPE reinforcement increases rapidly with an increase in gas flow rate and treating time (Figure 7(a)). Similarly, the results in Figure 7(b) and (c) showed that the impact force increases with an increase in gas flow rate and power as well as treatment time and power, respectively. The change of the curve showed that power had a greater impact on the composites compared with the impacts of treating time and gas flow rate.

Based on the secondary regression method, the relationship among the maximum impact force of the UHMWPE reinforcement (Y) and the treating time ($x_1$), the treating power ($x_2$) and the gas flow rate ($x_3$) was as follows:

**Figure 5.** Water contact angle of STF inter-ply hybrid composites with various (a) power, (b) treating time, and (c) gas flow rate.

**Figure 6.** Load-displacement curve of UHMWPE/STF with 100 W–120 s–2 sccm plasma treatment.
The prediction response was considered significant at $p = 0.0031 < 0.01$ it indicates. The $R^2$ was equalled to 0.9876, which indicates that the equation significantly fitted the model. The results also revealed that the order of influencing degree of the three factors was: treating power > treating time > gas flow rate.

The results presented in Figure 8 shows that the interactive effects of two independent process parameters had the maximum impact force of the UHMWPE reinforcement. The third parameter in each figure is the medium level. For example, the third factor of the gas flow rate of 4 sccm in Figure 8(a) and (b). The results shown in Figure 8(a) and (b) indicates that the maximum impact force of the UHMWPE reinforcement with the increase of treating power and treating time increases first but decreases later in the experiment period.

The contour density is increases more with the treating power than with treating time. This indicates that the influence of processing power on the maximum impact force of the UHMWPE reinforcement is greater than that of the treating time. However, Figure 8(c) and (d) shows that the maximum impact force of the UHMWPE reinforcement initially increased with the increase of gas flow rate and treating time, and then tends to level off.

On the other hand, the contour density is increases more with the treating time than with the gas flow rate. This indicates that treating time has more influence on the maximum impact force of the UHMWPE reinforcement than the gas flow rate.

Further results presented in Figure 8(e) and (f) shows that the maximum impact force of the UHMWPE reinforcement initially increased with the increase of treating power and gas flow rate and then decreases. The contour density is increases more with the treating power than with the gas flow rate. This indicated that the influence of the treatment power on the maximum impact force of the UHMWPE reinforcement is greater than that of the gas flow rate. The results presented in Figure 8 illustrates that the response surface map of the maximum impact force is steep and have ellipse of the contour maps. This demonstrates that the interactions between treating power, treating time, treating power, and gas flow rate are significant. However, Figure 8(c) and (d) shows that the curves of the contour map of the maximum impact force were close to the circle. This indicates that the interaction between the treating time and gas flow rate is not significant. Furthermore, this was significant for the maximum impact force of the UHMWPE reinforcement.

The process parameters for the optimal treatment effect of the plasma were 101.79 W, 160.79 s, and 4.24 sccm for the treating power, the treating time, and gas flow rate respectively. However, taking operability of the process into account, these parameters were adjusted to the treating power of 100 W, the treating time of 150 s, and a gas flow rate of 4 sccm.

**Conclusions**

This study revealed that the surface of UHMWPE with Ar plasma treatment was mildly damaged. The treatment improved surface roughness of the UHMWPE reinforcement. The UHMWPE with Ar plasma treatment was compounded with shear thickening liquid to prepare UHMWPE/STF flexible composite.

Although the interfacial performance of UHMWPE fabric is poor, its surface is changed after treatment with Ar plasma. The results of this study showed that the liquid on the surface of UHMWPE could completely infiltrate the fabric when the power is at 100 W. However, when the power is increased to 225 W, the change is not obvious and the influence of the power reaches a moderate state.

$$Y = a_0 + a_1 x_1 + a_2 x_2 + a_3 x_3 + a_{12} x_1 x_2 + a_{13} x_1 x_3 + a_{23} x_2 x_3 + a_{11} x_1^2 + a_{22} x_2^2 + a_{33} x_3^2$$

(1)
The impact force of UHMWPE/STF composites with various Ar plasma treating parameters, including treating time, treating power, and gas flow rate, was also studied. The results showed that an increase in a variable led to an increase in the impact force of UHMWPE/STF composites. The most important factor influencing the impact force was the power, and then the treating time and gas flow rate in order. The optimal effect was realized at the treating time of 150 s, treating power of 100 W, and a gas flow rate of 4 sccm.

Plasma technology has good modification effect and good environmental performance. It has been widely used in the surface treatment of micro/nanostructured materials, including surface activation, functionalization, etching, and polymerization, which improves energy absorption mechanisms of the composite materials. Currently, the continuous treatment of atmospheric pressure plasma technology and the development of large area plasma jet device. These have gradually promoted the application of plasma technology in industrial production, which has a good application prospect. Therefore, our study recommends the following three research direction for future studies: First, we recommend the preparation of plasma treated UHMWPE fiber fabric and STF composite material based to the optimal parameters obtained in this study and conduct ballistic tests. Secondly, to perform further

**Figure 8.** Interaction between the Ar plasma treating parameters (X) and impact force (Y), (a, b) interaction between treating power \((x_1)\) and treating time \((x_2)\) impact force \((Y)\), (c, d) interaction between gas flow rate \((x_3)\) and treating time \((x_2)\) impact force \((Y)\), and (e, f) interaction between treating power \((x_1)\) and gas flow rate \((x_3)\) impact force \((Y)\).
exploration of the effects of nanoparticles diameter, PEG molecular weight, STF concentration, and other factors on shear thickening. Lastly, the impact of different fabric structures, layering sequences, and layering angles on performance of bulletproof should be explored.

Declaration of conflicting interests
The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

Funding
The author(s) disclosed receipt of the following financial support for the research, authorship, and/or publication of this article: The authors would like to thank the foundations for financially supporting this research: Natural Science Foundation of Hebei Province (E2019208424), Youth Talents Plan of Hebei Province, Project of College Students Innovation and Entrepreneurship Training Plan (2020197), Special Project for Cultivating Students Scientific and Technological Innovation Ability (2021H011402).

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