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Conch-shell-inspired porcelain ceramic tile/Kevlar fabric composites with excellent combination of strength, toughness and shock resistance

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Abstract

For various engineering applications in public, ceramics with exceptional strength, toughness and shock resistance are imminently required, but traditional ceramics struggle to meet these demands due to their fragility. Inspired by the shape of natural conch shells, this work offers a simple hot press approach to fabricate layered porcelain ceramic tile/Kevlar fabric composites, yielding excellent mechanical properties and the capacity for non-catastrophic failure. It was concluded that composites' excellent mechanical performance is attributed to the resin/fabric content. The composite with the resin/fabric content of 45.5 vol.% had an outstanding combination of high strength (310.5 ± 6.2 MPa) and excellent fracture toughness (6.83 ± 0.09 MPa·m$^{1/2}$), whereas the sample with 36.4 vol.% resin/fabric content had the maximal impact energy of 2.04 ± 0.09 J, which is much better than those of traditional ceramics. The strong resistance to fracture is a result of the proper interfacial bonding and the presence of elastic component, which enables toughening mechanisms such as crack deflection, fibre pull-out and interfacial debonding. This finding provides useful guidance for replacing low-performance ceramics in engineering applications with cost-effective ceramic composites.

Keywords: *layered ceramics, Kevlar composites, strength, toughness, shock resistance, toughening mechanisms*

I. Introduction

Porcelain ceramic tiles are an essential building material commonly used in various commercial applications, including exterior wall and furniture veneers. This is due to their attractive appearance, remarkable chemical stability and exceptional durability [1,2]. These cost-effective ceramics are predicted to have great application prospects, but they are limited by sudden and catastrophic fracture behaviours while loading caused by their inherent brittleness. As reported by Yuan *et al.* [3], there were nearly 92 million tons of non-degradable porcelain ceramic tile wastes in 2022, mainly due to the brittle fracture. Most of the ceramic wastes are just abandoned or buried, generating many serious environmental disasters and threatening the survival of mankind [4]. Hence, ceramic manufacturers face the pressing challenges of enhancing the bending strength, fracture toughness and shock resistance characteristics of conventional ceramic tiles in a cost-effective manner.

Some organisms in nature have unique biological mechanical structures, which is of great reference value for designing and preparing high-performance ceramic materials [5]. Among them, conch-shell-inspired ceramic composites are considered alternative designs for structural ceramics and they are effective in overcoming the inherent brittleness of ceramic materials [6]. In general, these composites are commonly fabricated by using a variety of methods such as freeze-casting, co-

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extruding and additive manufacturing [7–9]. For example, Li *et al.* [10] found that the shell-inspired structure of Al_2O_3 ceramics exhibits excellent toughness, but the complex processing limits its application area. Sun *et al.* [11] reported that the shock resistance property of shell-inspired ZrO_2/cPMMA composite prepared through additive manufacturing is even equivalent to that of commercial aluminium alloys. However, the above techniques are now commonly used only in fabrication of many advanced ceramics, such as layered carbides and nitrides, etc. [12–14]. The high costs and long processing periods make these techniques difficult to be practical in actual manufacturing. Statistically, only a few studies have reported on the general properties of multilayered traditional ceramics, such as strength and toughness, and the fracture process and shock resistance of the ceramic composites. In addition, the effects of elastic content on their mechanical abilities have rarely been systematically explored.

Inspired by these considerations, the conch-shell-inspired ceramic composite was simply prepared by a straightforward and low-cost hot press process. In addition, the interface bonding state, sectional microstructure, mechanical performances and related mechanism were respectively studied. We hope this conch-shell-inspired ceramic composite with hierarchical design offers crucial guidance for replacing low-performance ceramic materials in engineering applications with cost-effective ceramic composites.

II. Experimental

2.1. Raw materials

Porcelain ceramic tiles with different thicknesses, from 0.5 to 5.5 mm were used as a major phase in composite samples (for additional details, refer to our previous study [15]). Plain-weave Kevlar fabric (G6, Bolton new material Technology Co. LTD, China) was used as reinforcing phase and according to manufacturer had the following features: 0.15–0.16 mm thickness, 280 g/m^2 grammage, 2.70–2.75% elongation, 3350–3750 MPa tensile strength and 85–90 GPa elasticity modulus. Epoxy resin, produced from epoxy resin precursor Part-A and curing agent Part-B (both from Macklin Biochemical Technology Co. LTD, China), had 2.2–2.6% elongation, 0.51–0.92 Pa·s viscosity, 18–20 kJ/m^2 impact strength, 120–125 MPa bending strength, 25–30 MPa tensile strength and 115–125 N/m peel strength (according to data given by the manufacturer). In addition, the following raw materials were also used: acetone solution (C_3H_6O, 99.5%, Macklin Biochemical Technology Co, LTD, China), ethanol (C_2H_6O, 95%, Aladdin Reagent Co. LTD, China), sulphuric acid solution (H_2SO_4, 4.9 mol/l, Aladdin Reagent Co. LTD, China) and abrasive paper (W10, Beijing Abrasives sales Co. LTD, China).

2.2. Fabrication of conch-shell-inspired composites

Preparation process of conch-shell-inspired ceramic composites is schematically illustrated in Fig. 1a and the obtained final heterostructures in Fig. 1b. Firstly, the surface of porcelain ceramic tile was polished and then the tile was immersed in ethanol to clean excess debris. After that, the obtained ceramic tiles were dried at 80 °C for 5 min and treated with 4.9 mol/l sulfuric acid solution for 25 min. This process can facilitate the interface bonding between ceramic tile and epoxy resin. Next, the epoxy resin precursors Part-A, curing agent Part-B and the acetone solution were weighted in a container, in accordance with the mass ratio of 10 : 3 : 1. This mixture was continuously stirred to maintain optimal fluidity in oven at about 22 °C. After that, as shown

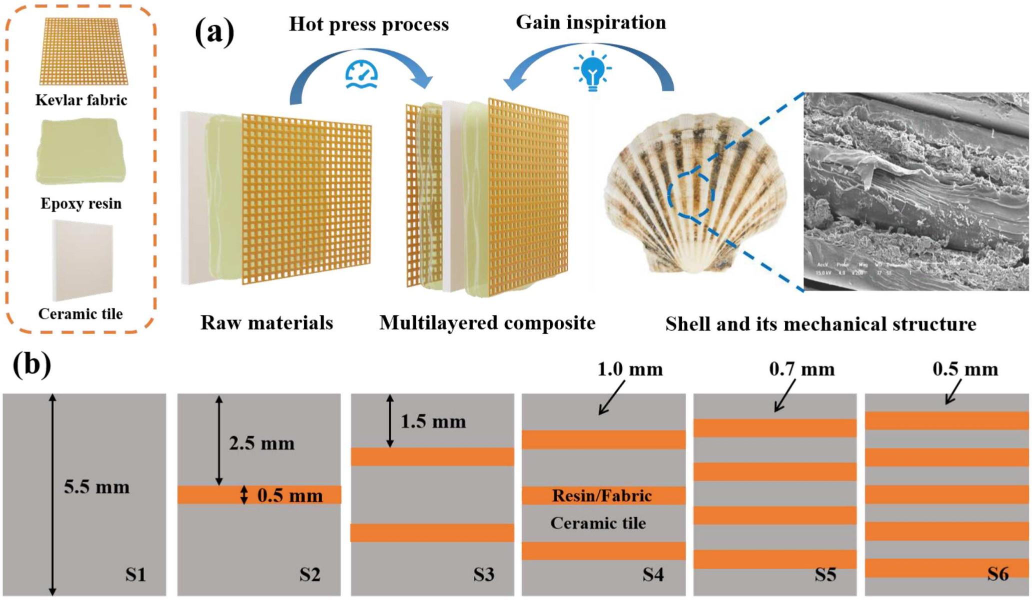

Figure 1. Schematic presentation of structure design (a) and preparation process for conch-shell-inspired ceramic composites (b)

Table 1. Crucial parameters of obtained ceramic composites and contrastive porcelain ceramic tiles

Samples	Layer number		Layer thickness			The volume fraction of
	Ceramics	Resin/fabric	Ceramics	Resin/fabric	Composite	resin/fabric layer [vol.%]
S1	1	-	5.5	-	-	-
S2	2	1	2.5	0.5	5.5	9.1
S3	3	2	1.5	0.5	5.5	18.2
S4	4	3	1.0	0.5	5.5	27.3
S5	5	4	0.7	0.5	5.5	36.4
S6	6	5	0.5	0.5	5.5	45.5

in Fig. 1a, the obtained thin ceramic tile, Kevlar fabric and epoxy resin precursor were combined to form an integrated structure. This prefabricated sample was then subjected to a hot press machine, under uniaxial pressure of 25 MPa at 45 °C for 10 min to produce the conch-shell-inspired composite. The composites with diverse mechanical structures, such as bilayer, three-tier, etc. (refer to Table 1 for details) were obtained by repeating the above process. These multilayered composites were trimmed with emery paper before mechanical testing.

2.3. Characterization

The fracture microstructures of the conch-shell-inspired composites were analysed utilizing digital microscope (VHX-7000, Japan) at magnifications of 20–200×. The sample dimensions were measured with electronic vernier caliper (16ER Mahr, Germany). The bending strength values were determined using a static universal mechanical testing machine (AllroundLine Z150, ZwickRoell, Germany) in accordance with the international testing standard ASTM C1161-13 (loading rate and span were 0.5 mm/min and 30 mm, respectively). The fracture toughness of the samples was measured using a single-edge notch beam method with 30 mm span and 0.5 mm/min loading speed. For accurate fracture toughness measurements, the notch depth to sample height ratio was fixed at 0.35–0.55. The Ramberg-Osgood equation [16] was utilized to obtain stress-strain curves for loaded samples. The fracture work was determined by dividing the area under the stress-strain curves of notched bars by the cross-sectional area of the specimen. To evaluate impact resistance performances, an ISO-125421 compliant falling sphere impact tester with a 500 g mass impact sphere was employed. The top surface dimension of impacted sample was 300 × 300 mm and the loaded one was 40 × 40 mm. Each measurement was tested using five samples and the reported results are the averages to mitigate errors.

III. Results and discussion

3.1. Microstructure

As reported by Zhong *et al.* [17], the mechanical characteristics of the composite are significantly influenced by the interfacial bonding condition between various components. The cross-section microstructure of the ceramics/resin/fabric interface was analysed (Fig. 2) to characterize the interface bonding states of the as-prepared multilayered composites. It can be observed from Figs. 2a,b that there is a typical conch-shell structure with strong interfacial bonding between the ceramics/resin/fabric interface, attributing to chemical polymerization of epoxy resin at a molecular level and with the presence of sulphuric acid as a hydroxylating agent to promote chemical bonding between organic and inorganic components [11,18]. From the magnified images (Figs. 2c,d), it can be seen that not only the fabric/resin interface edges but also the resin/ceramics are quite clear. This indicates that the physical and chemical compatibilities of the two phases at the interface are excellent. According to the Pinho hypothesis [19], this phenomenon also plays a significant role in the transmission of stress and aids in improving the quality of toughening and shock resistance. Since the obtained composites are very similar to the hierarchical structure of natural conch shells, these bio-inspired composites with the multilayered structure are expected to exhibit excellent mechanical properties.

3.2. Mechanical properties

To explore the mechanical failure processes of these biologically inspired composites, the flexural stress-strain curves and fracture work of the bio-inspired multilayered ceramic composites were measured (Fig. 3). It is found that the composites, whose resin/fabric content exceed 18.2 vol.% exhibit typical non-brittle failure (Fig. 3), caused by the layered features and toughening effects from resin/fabric [20]. On the other hand, not only the ceramic tile, but also the composite with only 9.1 vol.% resin/fabric content shows significant brittle failure behaviour. However, the causes of brittle fracture for these samples are entirely different and it can be explained as follows. The brittle fracture of the ceramic tile is attributed to the fact that slip systems have difficulties in slipping and deforming while carrying loads, due to the high bonding energy of the covalent bonds [17,21]. The layered composite exhibits brittle fracture, because internal stress comes from the deformations of ceramics and resin/fabric layer, and it exceeds the admissible shear strength value. Guo *et al.* [22] summarized this phenomenon as a threshold value of ceramic composites, which is related to elastic content and crucial for mechanical performances. In addition, the conch-shell-inspired composites S3, S4, S5

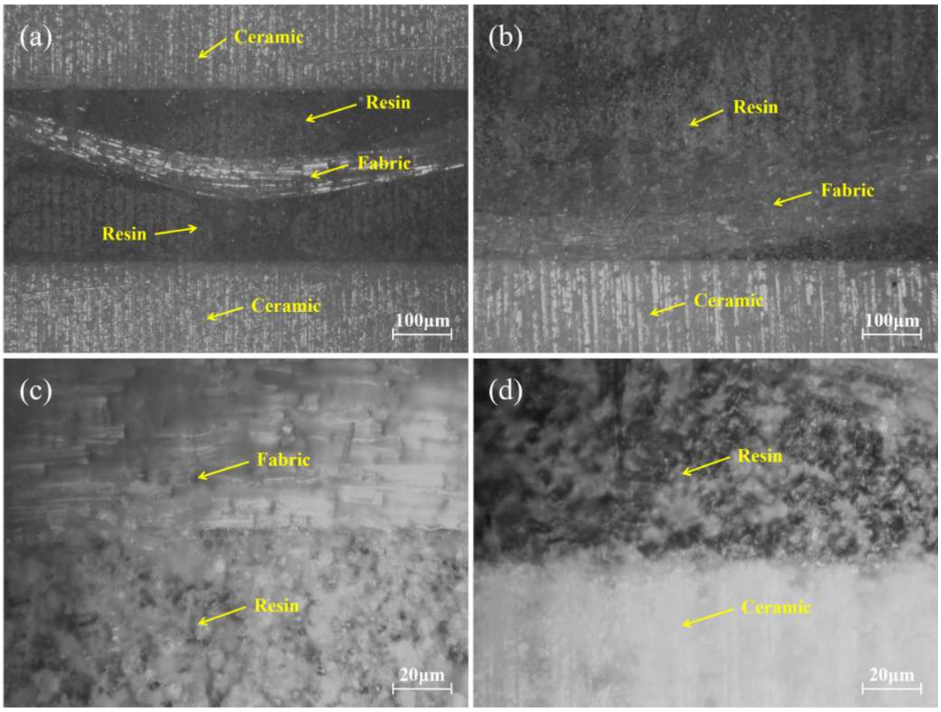

Figure 2. Interface microstructure of conch-shell-inspired composites: a,b) the ceramics/resin/fabric interface, c) magnified image of the fabric/resin interface and d) magnified image of the ceramics/resin interface

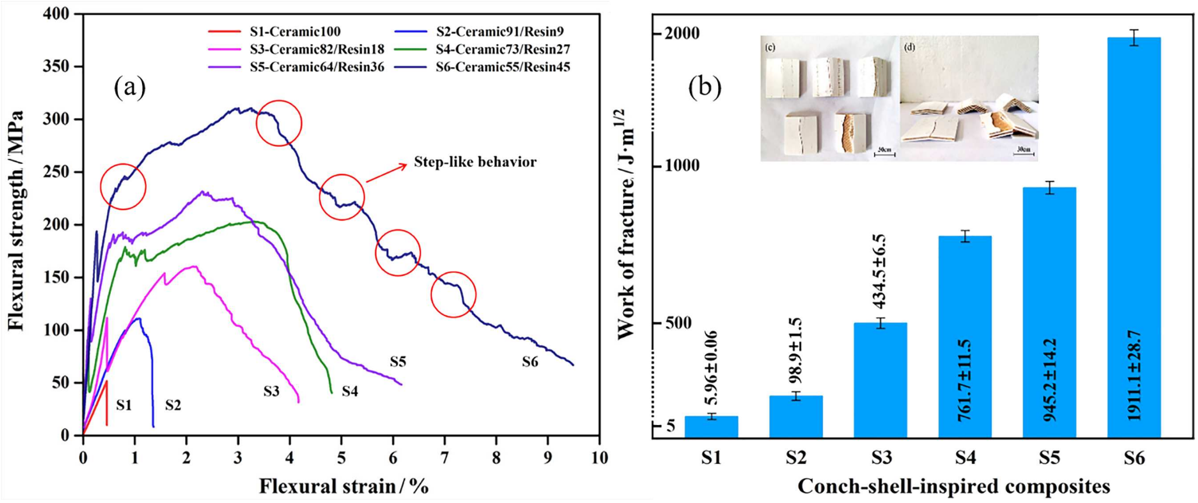

Figure 3. Flexural strength and fracture work of conch-shell-inspired composites; a) flexural stress-strain curves and b) work of fracture histogram

and S6 appear to yield representative step-like fracture behaviours, which are commonly observed in reported ceramics/metal and ceramics/polymer layered ceramic composites. The positive thing is the fact that the step-like fractures have been shown to be crucial in averting the immediate catastrophic collapse of materials in actual engineering [10,23]. Interestingly, the fracture work values for composites are improved with the increase of resin/fabric content and number of layers (Fig. 3b). The performance of the sample S6 with 45.5 vol.% resin/fabric content is about 1911.1 ± 28.7 J·m$^{1/2}$, which is nearly 19.3 times higher than that for the sample S2 (98.9 ± 1.5 J·m$^{1/2}$). Moreover, both values are much higher than the fracture work of 5.96 ± 0.06 J·m$^{1/2}$ for the sample S1. The improvement in the fracture work may correspond to the significant amounts of resin deformation and fibre pull-out, as there are relevant yield ranges in Fig. 3a.

To assess the usage of ceramics and their composites, testing for bending strength and fracture toughness is crucial [24]. Figure 4 provides a comparison of bending strength and fracture toughness values of the conch-shell-inspired composites with different resin/fabric volume fractions. It is interesting to note that these properties are strongly related to the presence of both resin and fabric. The overall mechanical performances of the conch-shell-inspired composites are much better than those of the traditional ceramic tile (Fig. 4). Specifically,

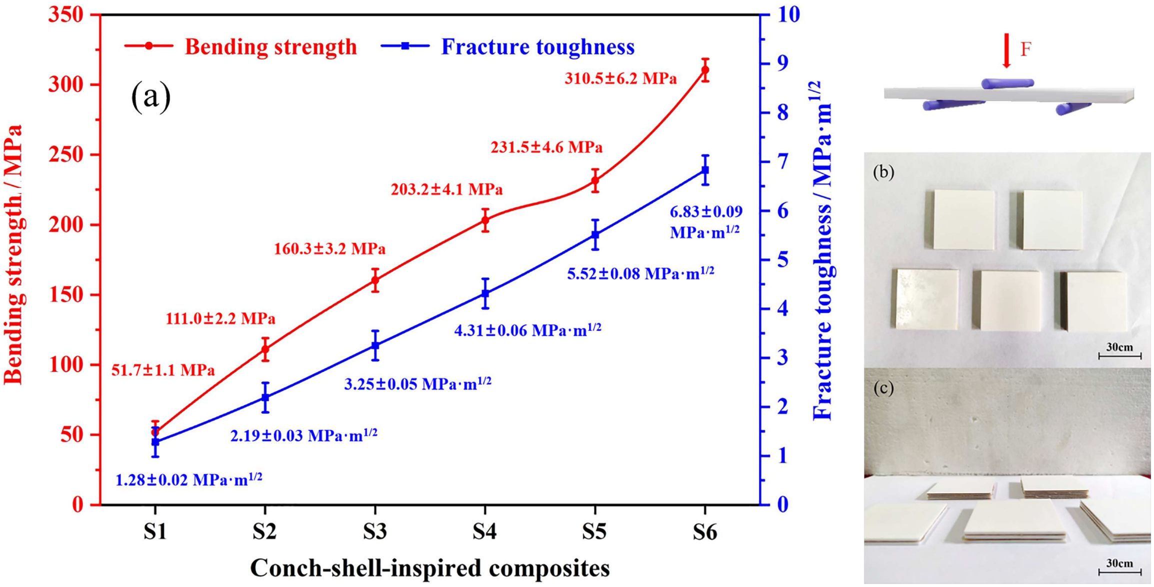

Figure 4. Change of bending strength and fracture toughness with resin/fabric content (a) and photos of as-prepared and tested composites (b,c)

the bending strength value is significantly enhanced by 179.8% from 111.0 ± 2.2 to 310.5 ± 6.2 MPa, as the volume fraction of resin/fabric layer increases from 9.1 to 45.5 vol.%. This is accompanied with the increase of fracture toughness value of 211.9% from 2.19 ± 0.03 to 6.83 ± 0.09 MPa·m$^{1/2}$. These behaviours can be interpreted with results presented by Liu *et al.* [25], who have reported that the component with high elastic modulus can be considered crucial slip system. While carrying external loads, this component can transfer internal stresses from brittle tiles to high-strength fibres in time, helping to prevent catastrophic failures. But due to the deformation of the ceramic and resin/fabric layers, the generated internal shear force reaches a threshold value, resulting in interface failure (Fig. 3). Hence, it could be deduced from the aforementioned mechanical performances that higher resin/fabric content in the conch-shell-inspired composite will enhance its strength and fracture toughness.

Shock resistance is vital as it can measure the mechanical stability of the ceramic composites when subjected to high-strength impact damages. The shock resistance values of the conch-shell-inspired ceramic composites (Fig. 5) confirm that they have better durability and higher shock resistance than the pure ceramics (the sample S1). Interestingly, the trend is not the same as that of the flexural strength and fracture work (Fig. 3). Thus the sample S5 with 36.4 vol.% resin/fabric content has the maximal impact energy of 2.04 ± 0.09 J and the sample S6 with maximal resin/fabric content of 45.5 vol.% has 13.7% lower impact energy, i.e. 1.76 ± 0.09 J. The possible reason is the fact that the impacted ceramic layer in the sample S6 exhibits a lower strength than that in the S5 composites, resulting in more stress transmitted into the underlying layers. This failure occurs as the impacted surface is visibly damaged, but the manifestation of strength and toughness is related to how the composite behaves until the complete failure occurs [26]. As reported by Sarvestani *et al.* [27], it is difficult for a resin/fabric layer to stimulate effective toughening mechanisms, such as fibre pull-out, while suffering instantaneous and huge impacts. However, the deformation of resin can absorb a small proportion of impact energy and it is different from the cases of steady loads. In this case, not only the resin/fabric content, but also the impacted ceramics is essential to the shock resistance capabilities of the bio-inspired ceramic composites, providing valuable reference proposals for the ceramic composite designs.

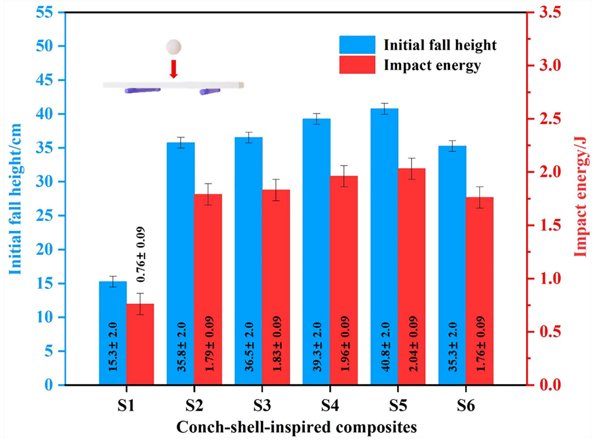

Figure 5. Representative shock resistance properties of conch-shell-inspired composites

In order to better visualize the content effects on representative mechanical properties, Fig. 6 compares the mechanical property map of the obtained ceramic composites (S2 to S6) with the traditional porcelain thin ceramic tile (S1). The performances of the composites are much better than those of the ceramic tile, which is attributed to the fact that the resin/fabric can behave like ductile phase between layers, so that they be-

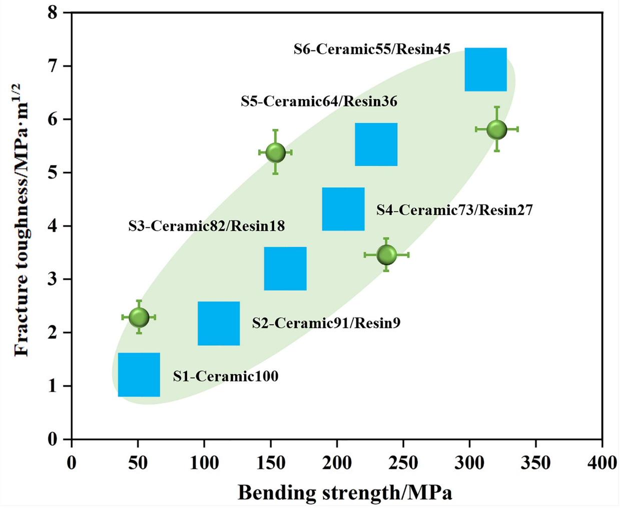

Figure 6. Mechanical property map of conch-shell-inspired ceramic composites as a function of resin/fabric contents (green spheres are error bars)

come stronger and tougher as the resin/fabric content increases, in agreement with the related analysis in Figs. 3 and 4. Hence, multiple performance improvements in the ceramic composites highlight the significance of hierarchical structure for strengthening and toughening, indicating that the bio-inspired composites have bright prospects to replace the traditional brittle ceramic materials in engineering applications.

3.3. Toughening mechanisms

The fracture microstructures of the conch-shell-inspired ceramic composites are displayed in Fig. 7. As shown in Figs. 7b,c, typical brittle fracture occurs in both layers of the S2 composite, related to the flexural stress-strain curves in Fig. 3. However, the resin/fabric layer is just partially destroyed by the stress, which proves that the resin/fabric strengthening and toughening effect is weak, and the brittle fracture mode dominates at low resin/fabric content [28]. As the elastic content increases from 9.1 to 18.2 vol.%, two completely

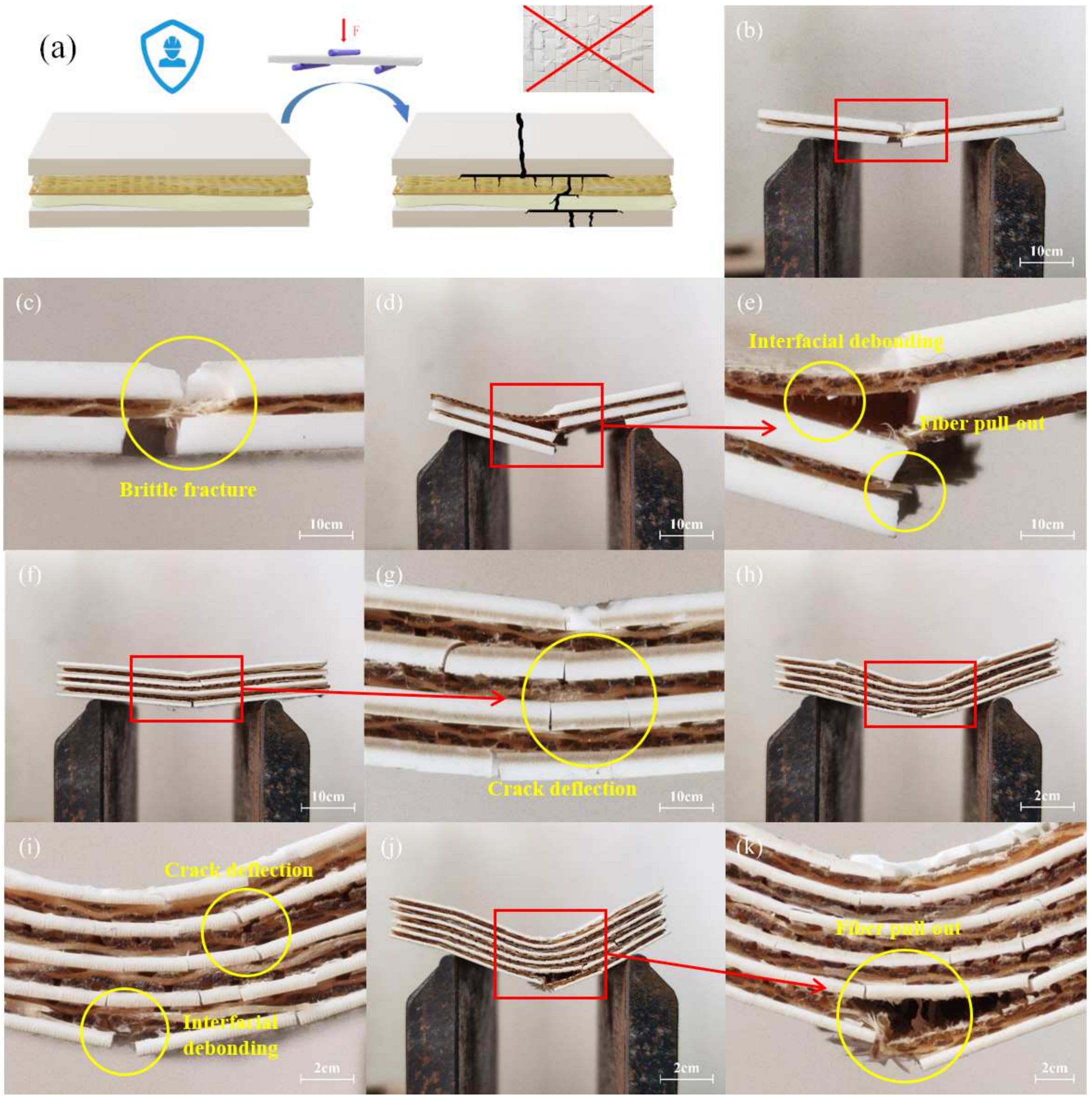

Figure 7. Schematic presentation of mechanical properties testing (a) and fracture microstructures of conch-shell-inspired composites with different resin/fabric contents: (b,c) 9.1 vol.%, (d,e) 18.2 vol.%, (f,g) 27.3 vol.%, (h,i) 36.4 vol.% and (j,k) 45.5 vol.%

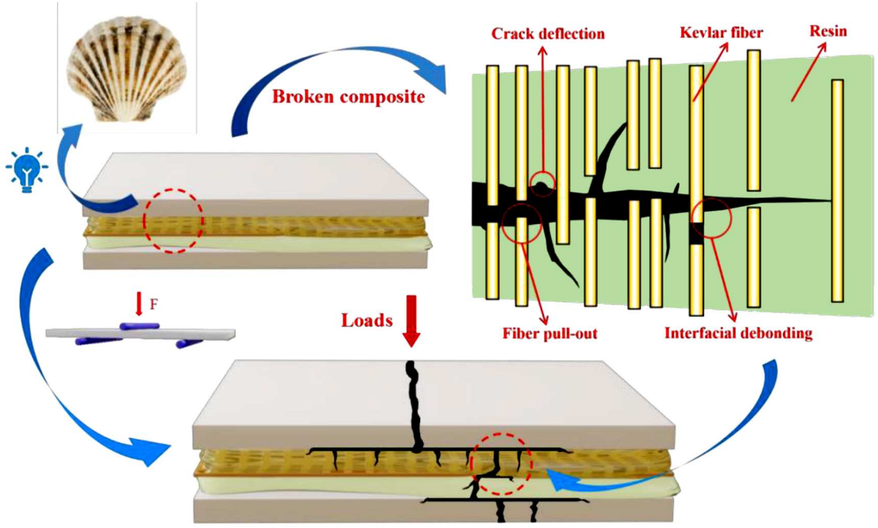

Figure 8. Schematic presentation of toughening mechanisms in conch-shell-inspired porcelain ceramic tile/Kevlar fabric composite [33]

different fracture behaviours, i.e. interfacial debonding and fibre pull-out, are observed in Figs. 7d,e. The cross-section morphology still has many brittle fracture characteristics, but the corresponding curve of the S3 composite exhibits pseudoplastic fracture (Fig. 3). Referring to the research from Diaz *et al.* [29], the interfacial debonding behaviour is commonly associated with the brittle fracture in composites, and the final fracture morphology is the result of competitive effects between brittle and elastic component while carrying loads. Therefore, we can infer that it is the interfacial debonding instead of fibre pull-out that is dominant in the fracture and it yields brittle fracture when the resin/fabric content is 18.2 vol.%. In the sample S4 (Figs. 6f,g) the crack is generated in the ceramic layer and deflected as it comes to the resin/fabric layer. Instead of continuing along the original course, the break tends to expand to the ceramics/resin contact, because inter-layer failure requires less energy than brittle fracture to form a new unit area [30]. Such "crack-tip-shielding" action is thought to be a successful method to blunt the fracture tip, and it could activate toughness mechanisms to prevent cracks transformation from a steady state to an unstable state [31,32]. Interestingly, the crack deflection behaviour is more pronounced in Figs. 6h,i,j,k. The interfacial debonding and fibre pull-out phenomena all occur in the ceramic composites as the resin/fabric contents are 36.4 and 45.5 vol.%, respectively. This can correspond to the suitable interface bonding state due to the presence of hydroxylating agent. The mechanisms shown in Fig. 8 indicate that outstanding mechanical performances of this bio-inspired ceramic composite are related to several factors. Specifically, those factors include the robustness and resilience of the shell-like mechanical structure, as well as the appropriate resin/fabric content. Furthermore, crack deflection, interfacial debonding and fibre pull-out are critical mechanical mechanisms, which help to prevent the composite from brittle fracture by consuming more energy per unit length.

IV. Conclusions

In summary, a surface hydroxylation method was followed by a straightforward and low-cost hot press procedure to effectively fabricate the conch-shell-inspired layered porcelain ceramic tile/Kevlar fabric composites. Due to proper interfacial bonding state between layers and the presence of elastic component, which is crucial for slip systems to transfer internal stresses while carrying external loads, the obtained ceramic composite exhibits outstanding mechanical performance and non-catastrophic failure behaviour. The key conclusions are summarized as follows:

1. The major mechanical properties of the ceramic composites, such as the strength and toughness, are strongly correlated with the resin/fabric content. But the shock resistance properties are affected by the resin/fabric content and the performance of the impacted layer.
2. An outstanding combination of high strength of 310.5 ± 6.2 MPa and excellent fracture toughness of 6.83 ± 0.09 MPa·m$^{1/2}$ is obtained as the resin/fabric content reaches to 45.5 vol.%. However, the sample with 36.4 vol.% resin/fabric content has the maximal impact energy of 2.04 ± 0.09 J, which is much better than those of traditional porcelain thin ceramics.
3. Various strengthening and toughening mechanisms, such as crack deflection, interfacial debonding, and fibre pull-out, are proven to be associated with outstanding mechanical performance and non-catastrophic failure behaviour. This bio-inspired ceramic composite with hierarchical design is supposed to offer guidance for replacing low-performance ceramics in engineering applications with cost-effective ceramic composites.

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