STRAIN SENSING OF COMPLEX SHAPED 3D WOVEN COMPOSITES USING MXENE NANOPARTICLES

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Abstract: This study aims to develop smart 3D woven nano composite by vacuum infusion process, which can sense deformation and damage at the joints of complex shapes like T-profile. The MXene were delaminated by chemical etching and dispersed in deionised water. In next stage, they were sprayed directly on the joint of 3D woven T-profile composite to form a conductive coating. The resultant nano composites were subjected to tensile loading to study the sensitivity of MXene to applied elongation. Results show that the MXene network is sensitive to tensile deformation. It can be concluded from the study that MXene smart strain gauge manufactured during this research work can be used for in-situ deformation monitoring of complex shaped composites while being cost effective and easy to manufacture.

Keywords: 3D woven composite; MXene; mechanical testing; T-profile

1. Introduction

The quality evaluation of fibre reinforced composites in aerospace, civil and mechanical engineering structures is usually accomplished by locating damage via different techniques. These damage detection techniques can be broadly classified into two major categories namely destructive and non-destructive testing (NDT). Bulk manufactured cost-efficient components are often evaluated by destructive testing whereas large complex components, which are not feasible to be damaged are evaluated via NDT. Researchers combine various scientific techniques to develop systems for sensing damage in the composite structures. Some of the NDT methods are ultrasonic testing, radiography, thermographic, acoustic [1], and shearography testing [2] etc. However, these methods fail to evaluate the component during its actual usage. The concept of structural health monitoring (SHM) becomes important in such scenarios where quality of the component needs to be evaluated during its actual usage.

The most feasible method of SHM is by integrating sensor elements during various stages of composite manufacturing. A brief overview of such methods is integrating metallic wires, optical fibres [3], yarn coated with conductive dyes [4] etc., in the composite layup or in the fabric preform during weaving process. However, placing sensor elements during fabric layup introduces defect in the composite structure while integrating the sensor at weaving stage of fabric manufacturing imparts damage to the sensor element and often results in improper functioning of the sensor. To resolve this problem and enhance the sensing efficiency, conductive nanoparticles like MXene [5], carbon nanotube (CNT) [6], graphene [7] etc., are either dispersed in matrix or applied as coating on the composite. The mechanical deformation of composite structure affects the electrical properties of these nanoparticles, which forms the basis of monitoring the structural health of the material.

The MXene nanoparticles are two dimensional (2D) structures of titanium carbide $Ti_3C_2T_z$ synthesized from its MAX phase via various etching and delamination methods. The manufacturing process of MXene allows bonding of functional groups such as -O, -OH and/or-F, denoted as T_z in the formula, to impart wide range of properties. The reported tensile strength of MXene nanoparticles is 570 MPa (940 nm thick film) [8]. Recent studies have successfully modified MXene to achieve high adhesion with epoxy resin [9].

The most common research methodology of effective SHM by MXene nanoparticles is focused only on planar surfaces [10]. However, the practical applications of composites consist of complex shapes. Such shapes are usually in the form of 'T' or 'I'. One of the most extensively used shape is T-profile, which transfers load between mutually perpendicular directions. The conventional method of manufacturing T-joint composites is by laying up fabric plies on 2 opposing 'L' shaped rigid mould [11]. The laminated T-joint composite manufactured in this manner is reported to have less load bearing capacity and prone to delamination [12]. To overcome the structural limitations of laminated composite, the layers of fabric plies are either z pinned/tufted [13] or the T-profile preform is 3D woven directly on weaving machine. Numerous studies have shown that the 3D woven T-profile composites exhibit higher mechanical properties without delamination of constituent layers [14].

Majority of the research on NDT techniques for SHM of laminated and 3D woven composite Tprofile has been done via process such as ultrasonic vibration [15], integrating piezoresistive layers [16] and electromechanical response of CNT [17], etc. However, there is a research gap in deformation and damage sensing of complex shapes by MXene nanoparticles. The aim of this research work was to develop smart 3D woven composite T-profile capable of monitoring its deformation and damage in real time using MXene nanoparticles. T-profile preform was woven on shuttle loom and infused with epoxy resin to manufacture composite. In the next step, the MXene nanoparticles were sprayed at the composite junction region and copper wires were soldered to measure the resistance via multimeter. The electromechanical response of MXene coating was studied while subjecting the sample to tensile loading.

2. Materials and Methods

2.1 Preparation of MXenes

The MXenes were sourced in their Ti_3AlC_2 MAX phase. The etching process was done by adding the MAX phase to hydrochloric acid and lithium fluoride solution. After stirring for 24 h, the multilayer MXene sediment was delaminated using 99 wt.% LiCl. The $Ti_3C_2T_2$ MXene nanoparticles were further centrifuged at 3500 rpm for 10–15 times and rinsed with deionised water until the pH of the supernatant reached 6.5. After measuring the concentration of MXenes in the supernatant to 0.335 mg/mL, it was finally concentrated to 3.3 mg/mL by centrifuge to obtain conductivity in the order of $10^5 \Omega$.

2.2 Weaving of T-profile preform and composite manufacturing

Fibre reinforcement in the shape of T-profile was woven in folded form on an 8 head shaft dobby loom with shuttle insertion. The draft and denting plan as shown in Figure 1a was designed to thread glass rovings of 300 tex. The thread density (warp and weft) measured in final fabric was 8 threads/cm.

Bisphenol F resin hardener system CR-122 was used as matrix and the woven T-profile was used as reinforcement to manufacture composite via resin transfer moulding process in double 'L' shaped metal mould. The wet preform was then subjected to 101.3 kPa vacuum for 3 h. The preform was cured at room temperature for 24 h and post cured in oven at 80 °C for 5 h.

To increase the hydrophilicity of epoxy surface, T-profile composite was subjected to argonoxygen plasma and sprayed with 10 layers of MXene using airbrush with a 0.4 mm diameter nozzle. The composite T-profile was cut into dimensions as shown in Figure 1b.



Figure 1. 3D woven composite T-profile: (a) Weave design and draft; (b) Sample dimensions and resistance measurement scheme

2.3 Tensile testing

The tensile testing of five composite specimens was carried out on Instron ElectroPuls E10000T machine with optical strain sensor and testing speed of 2 mm/min. The specimen dimensions and mounting scheme are as shown in Figures 1b and Figure 2, respectively. The copper wires were soldered on the MXene coating and silver paste was applied to reduce the contact resistance. The resistance measurement was performed with Fluke 287 RMS multimeter by using two-probe method.



Figure 2. Tensile testing of T-profile composite

3. Results and Discussion

3.1 Tensile testing results and discussion

Due to premature failure at the bottom restraints, four out of five samples were successful in demonstrating monotonic relation between deformation and resistance values of the MXene coating. The graphs of $\Delta R/R_0$ against force-displacement for one representative sample is shown in Figure 3. ΔR is change in resistance and R_0 is initial resistance of the MXene coating. A typical graph on T-joint composites can be divided in two distinct zones i.e., initial failure and final failure. Initial failure is usually detected as drop in the tensile load values for the 1st time.



Figure 3. Real time deformation monitoring of composite T-profile during tensile loading

With increase in the tensile load, the stress started to accumulate at the junction region of T-profile. At 2.47 mm the initial failure occurred. Further loading beyond this point lead to crack propagation and ultimate failure of the composite at 6.54 mm in the junction region. The major failure mode observed was yarn/matrix damage.

The resistance values of MXene coating increased linearly in proportion to the applied stress which matches well with the previous studies related to MXene based SHM of composites [5], [10]. The initial failure of composite was detected as disturbance in the resistance value while the deformation due to stress accumulation till failure was detected as steady increase in the resistance of the MXene coating. The main source of change in MXene coating resistance is quantum tunnelling and contact resistance. Quantum tunnelling is the phenomenon where the electrons can transfer between nano flake structures, which has a gap in the order of magnitude 1 nm. Contact resistance is due to the overlapping of MXene over each other, thus providing a continuous path for electrical conductivity. The applied deformation of the composite tends to increase the contact and tunnelling resistance of the MXene coating, thereby leading to sensing of the damage and deformation.

4. Conclusions

The 3D woven composites T-profile were manufactured by resin transfer moulding process and MXene coating was sprayed at the junction region to monitor deformation and damage. The results show that the composites were able to sense the initial failure and deformation at the junction in real time under tensile loading. The graphs of change in resistance versus force-displacement were plotted to study the electromechanical response of the MXene layer. The following conclusions can be derived from the study:

- The concept of spraying MXene nanoparticles at the junction of complex shaped composite is feasible for real time deformation monitoring.
- During tensile testing of the samples, the MXene layer was able to sense the total deformation till failure in response to the applied displacement.

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5. References

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