Comparison of different surface treatments of carbon fibres used as reinforcements in epoxy composites: Interfacial strength measurements by in-situ tensile tests in a scanning electron microscope

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Abstract

The interface plays a very important role in the overall performance of polymer composites. A strong enough interfacial adhesion is generally desired to get an efficient load transfer from the matrix to the reinforcement. In this work, Carbon fibres (CFs) with different surface treatments were studied here. To improve the interactions between CF and the surrounding polymer matrix, two major routes were used: the grafting of carbon nanotubes (CNTs) on their surface and the oxidation of CNT-CF hybrids. In total, four kinds of CFs - raw CFs, desized CFs, carbon nanotubes grafted CF (CNT-CFs) and oxidized CNT-CFs - were used to prepare epoxy composites.

This paper aims to investigate the multiscale (nano-, micro-scale) behaviour of the composites based on a fine investigation using the most modern techniques, to understand the interfaces and to quantify them. The study itself was firstly conducted on a microscopic scale on the interfacial strength between CFs and the epoxy matrix, with tensile tests were carried out in-situ in the chamber of a double-column FIB-SEM microscope (scanning electron microscope coupled to a focused ion beam). The ion beam was used to mill a thin bond-shaped tensile specimen of composite containing both an epoxy and a CF part. The tensile stress field was applied using the nanomanipulator and the test was observed both via the ionic and the electronic columns, giving two different angles of view, to estimate the strain field, hence the interfacial strength when the failure is observed. The fracture strains of raw CF/epoxy, desized CF/epoxy, CNT-CF/epoxy and oxidized CNT-CF/epoxy were clearly observed and the axial interfacial strength was also derived. The interfacial strength decreased from the initial value of 53 MPa for the raw CF/epoxy to 48 MPa for the desized CF/epoxy. However, by grafting CNTs on the CF surface, the interfacial strength of CNT-CF/epoxy reached 55 MPa and was further increased to 58 MPa after the oxidation treatment.

Then, to understand the influence of interface modification, energy dispersion X-ray analysis (EDX) was carried out under scanning transmission electron microscopy (STEM). Based on the EDX mapping, it can be found that oxygen aggregated at the interfaces of raw CF/epoxy and oxidized CNT-CF/epoxy, which indicates that the chemical bonding plays a crucial role in the enhancement of interfacial interactions. This knowledge can be used to guide the modification of other types of reinforcement to fabricate composites with excellent mechanical properties.

Acknowledgement

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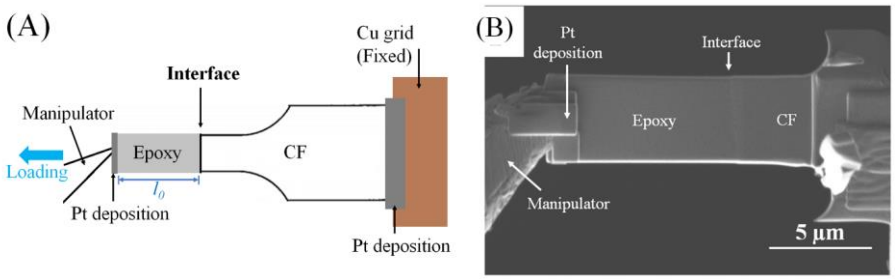

Figure 1 Sketch of a tensile specimen to measure the interfacial strength between a carbon fibre (CF) and an epoxy matrix; (B) SEM image of a final tensile specimen prepared by FIB-SEM.

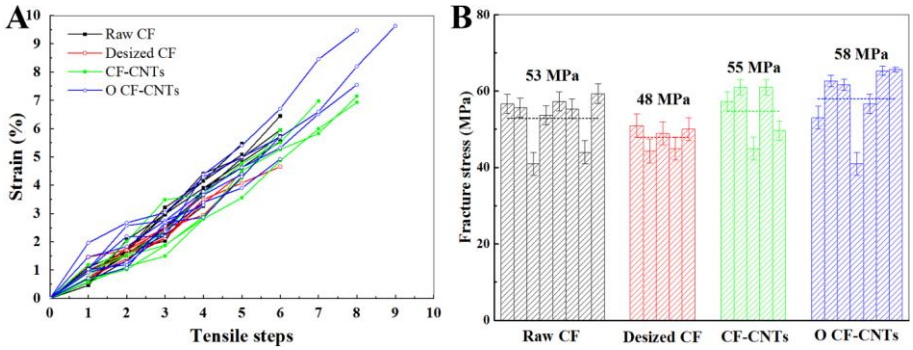

Figure 2 (A) Displacement-tensile step plot of stretching samples extracted from ionic images. The black, red, green and blue lines are the results from the raw carbon fibre (CF)/epoxy, desized CF/epoxy, carbon nanotubes (CNT)-CF/epoxy and oxidized CNT-CF/epoxy, respectively. (B) Fracture strengths of the raw CF/epoxy, desized CF/epoxy, CNT-CF/epoxy and oxidized CNT-CF/epoxy.

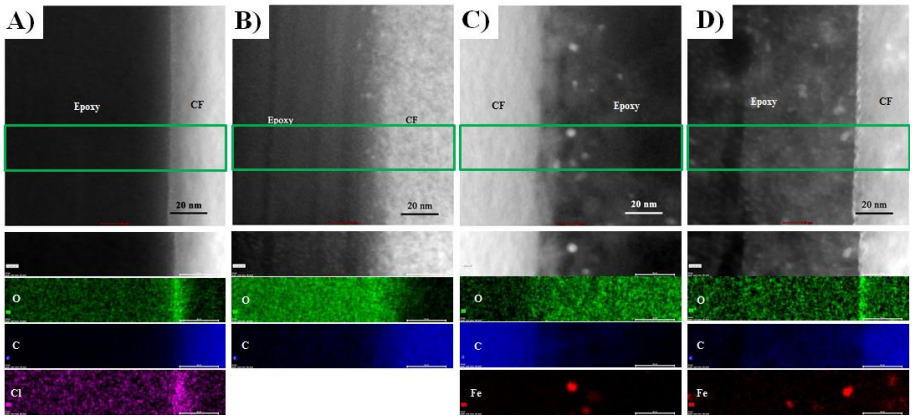

Figure 3 High magnification STEM Z-contrast (HAADF) image showing a carbon fibre (CF) perpendicular to the epoxy foil prepared by FIB: Interfaces of (A) Raw CF/epoxy, (B) Desized CF/epoxy, (C) Carbon nanotube (CNT)-CF/epoxy and (D) Modified CNT-CF/epoxy, respectively. The regions marked out by the green rectangle were analyzed by EDX, the elements were marked out by different colors, green for oxygen, blue for carbon, pink for chlorine and red for iron, respectively.