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Review

# A review of key developments and pertinent issues in nanoindentation testing of fibre reinforced plastic microstructures

M. Hardiman [a], T.J. Vaughan [b], C.T. McCarthy [a,*]

[a] The Irish Centre for Composites Research (IComp), Bernal Institute, School of Engineering, University of Limerick, Limerick, Ireland
[b] Biomechanics Research Centre (BMEC), Biomedical Engineering, National University of Ireland, Galway, Ireland

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ABSTRACT

In recent decades, nanoindentation has emerged as a useful experimental technique for characterising the in situ properties of fibrous composite constituents. However, the elastic theory used by the nanoindentation technique assumes that the substrate is a stress-free single-phase homogeneous continuum. Therefore, the application of nanoindentation theory to inhomogeneous composite materials composed of discrete regions with distinct material properties has proven to be problematic in certain scenarios. In this paper, a review of the key developments and pertinent issues reported by authors in relation to the nanoindentation of polymer matrix composites is presented. The effects of sample preparation, neighbouring constituents, residual stress, pile-up, time-dependent deformation and hydrostatic stress on the important nanoindentation parameters and properties are highlighted. The review also details the use of numerical simulations to gain greater insight into the stress and deformation fields produced during the nanoindentation of FRP microstructures, and includes recommendations regarding the standardisation of nanoindentation protocols for composite and polymeric materials.


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* Corresponding author.
*E-mail address:* conor.mccarthy@ul.ie (C.T. McCarthy).

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## 1. Introduction

The use of composite materials in structural applications has increased significantly in recent decades due to their higher specific stiffness and strength, compared to metallic materials. Fibre-reinforced plastics (FRP) are the most common type of composite material, and are used extensively in the automotive, marine and aerospace applications. The increasing demand for composite materials has been mirrored by an increase in demand for predictive analysis tools that are required to gain a greater understanding of their behaviour under various types of loading. Thus, determining accurate reliable failure criteria for fibre-reinforced composite materials is currently a very active research area. Much of the analysis of the composite structures relies on design practices and strength predictions which are based on ply-level analysis and macroscale testing. Macroscopic stress and strain criteria are often used to predict the response of composite materials to various loading scenarios, as opposed to criteria based on the actual physical mechanisms of failure. As a result, a significant amount of research has been carried out in order to gain a greater understanding of the link between the damage at the composite microscale and that experienced by the larger composite structure. Micromechanical finite element models of composite microstructures have been successfully used to predict the macroscopic stress-strain behaviour of composite materials, and could potentially prove to be a useful tool in future composite structural design, by limiting the amount of costly coupon and structural testing required to analyse composite structural elements. However, in order to ensure the accurate simulation of microscale deformation and damage, quantitatively accurate properties of the in situ constituents and interfaces must be determined. In recent years, the field of micromechanical testing has grown as researchers strive to quantify unknown microscale properties. Nanoindentation is a non-destructive testing technique that can be used to determine the properties of materials at the micro and nano scales. These directly measured in situ properties can then be used to establish the effect of the composite manufacturing process on the material quality, as well as allowing direct comparison between different polymer matrix blends and fibre treatments. The technique can also be used to provide, experimentally verified, input properties to micromechanical simulations of the composite deformation and failure process, providing a critical link between the microscale properties and the properties of large composite structural elements, as illustrated in Fig. 1. This review outlines the importance of this testing technique to the broader understanding of the FRP microscale interactions and highlights the work carried out and the issues reported by numerous authors relating to the application of the nanoindentation technique to these materials.

## 2. Fibrous composite micromechanics

While the failure behaviour of most structural materials is largely controlled by a single failure mechanism, the macroscopic deformation and failure behaviour of FRP materials is controlled by a number of local micromechanisms, whose initiation and propagation are dependent on the constituent materials properties, the local fibre distribution, and the direction of the applied load relative to the fibre direction, as illustrated in Fig. 2. Parallel to the fibres, the tensile failure is dominated by the strength of the fibre material, while under compressive loading failure occurs due to fibre buckling due to slight off-axis orientations of the unidirectional reinforcement. Failure due to transverse and shear loading is largely controlled by the matrix and interface properties, with failure occurring due to matrix plasticity and cracking accompanied by fibre-matrix debonding. It is clear that the large number of failure micromechanisms complicates failure predictions for composite structures. As more accurate strength predictions are required, the field of micromechanics has grown in order to gain a greater insight into the microscale damage process, and relate the observed processes to the macroscopic stress-strain response. Recent advances in experimental analysis techniques have allowed for unparalleled observation of composite micromechanical deformation and damage, where modern experimental equipment such as in situ microtest machines allow tensile [1], compression [2] and bending [3] tests to be carried out in the chamber of a Scanning Electron Microscope (SEM). These tests provide a useful qualitative understanding of the microscale composite failure process. However, quantitative insight into the effects of local fibre distribution, constituent properties and interface strength through experimentation has proven difficult. Micromechanical finite element models provide a more quantitative insight into the material's micromechanical behaviour based on the properties of the individual composite constituents. A number of advanced micromechanics damage models have been developed by Llorca, González and coworkers [2,4–7], who carried out numerous studies characterising the behaviour of fibre reinforced composites when subjected to transverse tensile loading [6], transverse compressive loading [2,5] and transverse shear loading [6,8]. Vaughan and McCarthy examined the micromechanical behaviour and effect of thermal

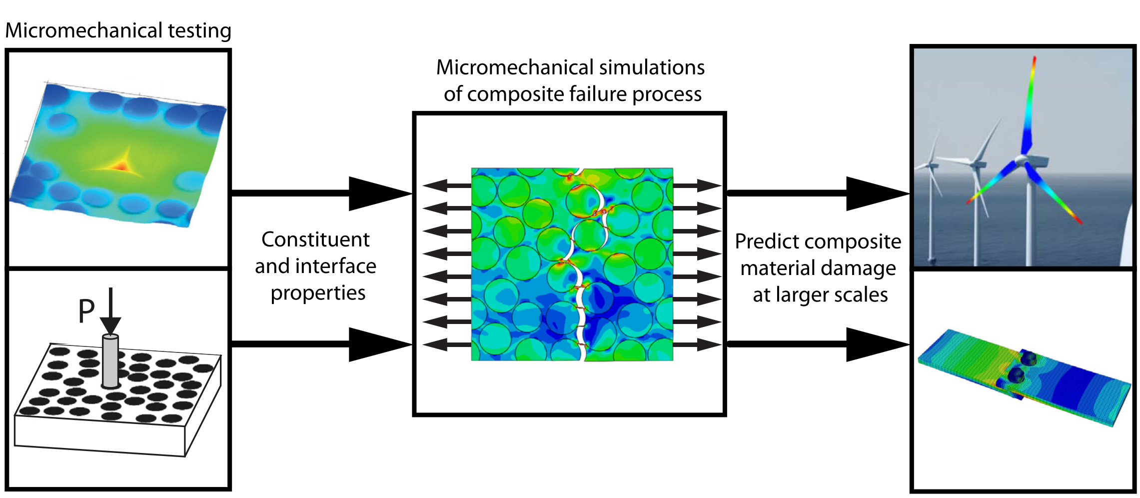


**Fig. 1.** Multiscale testing and modelling of fibre reinforced composite materials [10,110,118,119].