Micro Materials
Excellence in Nanomechanics

The nano-impact technique in the framework of high strain rate micromechanical testing of polymer composites

Introduction

Fibre-reinforced polymer composites are increasingly common in the aerospace industry. The latest commercial aircraft programmes from Boeing (the 787) and Airbus (the A350 XWB) are a proof of this. Around 50% of their structural weight comprises composite structures (see Figure 1). However, there are still areas where composite materials have not yet reached their full potential, as is in parts subjected to impacts and high-speed events. In addition, the interest in applying polymer composites in parts subjected to impacts goes beyond the aerospace sector, as the same loading scenarios are seen in automotive, wind energy, space and biomedical applications.

The lack of knowledge about the dynamic behaviour of composites, the complex failure mechanisms of these materials and the challenges associated with the available high strain rate testing methodologies are among the reasons that hinder the application of fibre-reinforced polymer composites in structures subjected to impacts. To this end, an EU-funded project (DYNACOMP) [1,2], was set up to increase the understanding about the dynamic behaviour of composite materials. The main objective of the project was to establish a simulation tool based on multiscale modelling in the framework of the Finite Element Method to predict the mechanical behaviour of composites as a function of strain rate. One of the unique features of the proposed simulation tool is the use of novel micromechanical testing techniques for calibration of the composite constituents: fibre, matrix and fibre-matrix interface.

Multiscale modelling has been proposed as a new approach for prediction of mechanical behaviour in composites. It takes advantage of the hierarchical structure of composites, where there are three clearly differentiated levels of detail (as seen in Figure 2): structure level (m scale), laminate level (mm scale) and ply level (μm scale). Physically-based models that capture the real failure mechanisms of the material are incorporated at each scale of analysis [3]. Information is transferred from lower to upper scales by homogenization into constitutive models. At the microscale, computational micromechanics captures the level of detail of the composite microstructure, including the fibres, matrix and fibre-matrix interface.

There are two approaches: Representative Volume Elements (RVEs), which are used for modelling damage initiation; and Embedded Cell models for simulating fracture propagation. These micromechanical models are partly calibrated with data from micromechanical test techniques, which are based on nanoindentation. This way, the in-situ properties of the matrix and fibre-matrix interface, which may be influenced by the processing conditions of the composite, can be captured. Furthermore, these tests are much faster and require less material than the traditional macroscale testing campaigns.

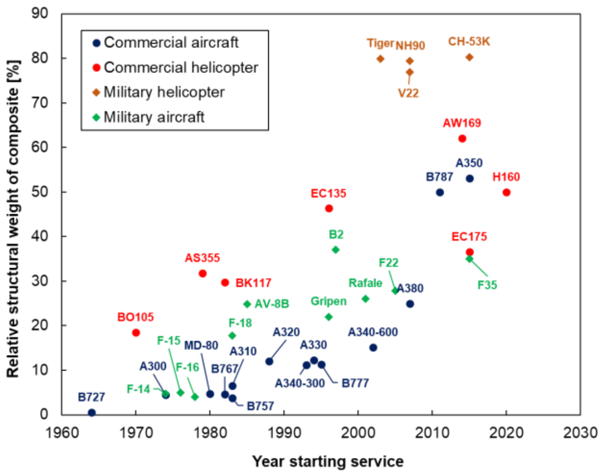

Figure 1 - Use of polymer composites in the aerospace industry.

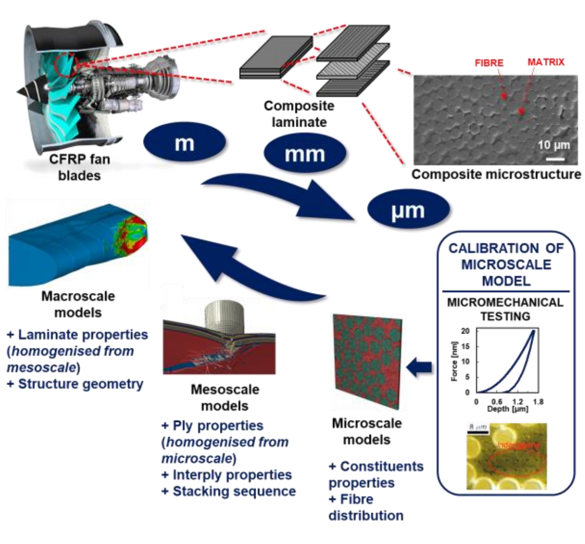

Figure 2 - Bottom-up multiscale modelling of composites.

Use of a load cell for nano-impact test

One of the main outcomes of the DYNACOMP project was the incorporation of additional instrumentation in the NanoTest system to enable force-displacement measurement in the nano-impact test [4]. Figure 3 shows the schematics of the pendulum-based load head and the location of the force sensor. The sensor is in direct contact with the sample back surface so that the total applied force in the material surface can be measured. In the NanoTest, force is actuated in the magnet-coil couple, while two capacitor plates in line with the indenter are used for displacement measurement. The load head rotates around a frictionless pivot and uses a limit stop to have a fixed position in space. The nano-impact test is carried out with the assistance of a solenoid located at the base of the instrument. Before impact, the solenoid switches on at the same time as a constant current is applied in the force actuator (the impulse force). The sample surface is positioned at a predefined distance from the indenter (the impulse distance). Then, the solenoid voltage is switched off and the pendulum swings towards the material surface driven by the acceleration given by the impulse force.

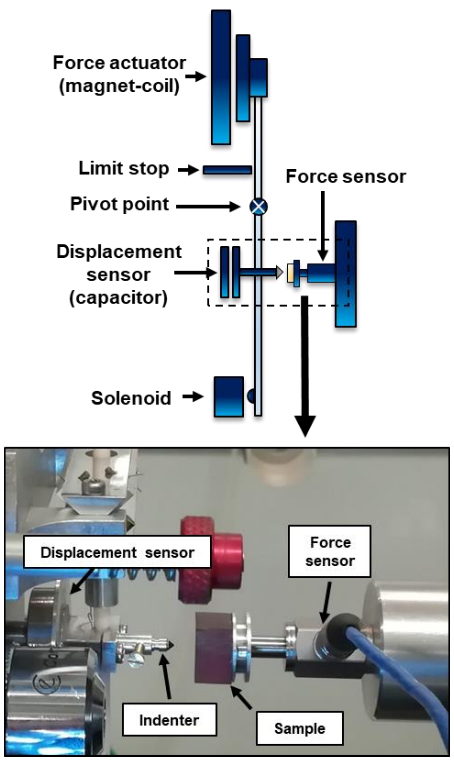

Figure 3 – The NanoTest pendulum-based nanoindenter including force sensor.

Force measurement under impact conditions is necessary because of the significant inertia contribution (product of the load head effective mass and acceleration), which differs to quasistatic indentation where the applied load is equal to the actuated one. Figure 4 shows how the applied force in the material is much higher than the actuated impulse force (5 mN in this case). An estimation of the applied force can be made by assuming a linearized damped harmonic motion to describe the instrument dynamics:

$$P = F_{imp} - m\ddot{h} - \rho\dot{h} - kh$$

F_{imp} is the actuated impulse force and m, ρ and k are the instrument effective mass, damping and spring stiffness, respectively. However, this estimation is not of sufficient quality to provide a force-displacement curve from which to extract quantitative information. This is because of the high noise level in the displacement signal, which is then amplified in its time derivatives ($\dot{h}$ and $\ddot{h}$). In particular, noise in the acceleration signal is the most critical because inertia adds the most important contribution to the total applied force. In addition, the raw estimated force signal needs to be smoothed to reduce noise, so there is an added uncertainty coming from the choice of filter type and parameters. Figure 4 also shows an example of how the estimated force signal looks, even after smoothing procedure.

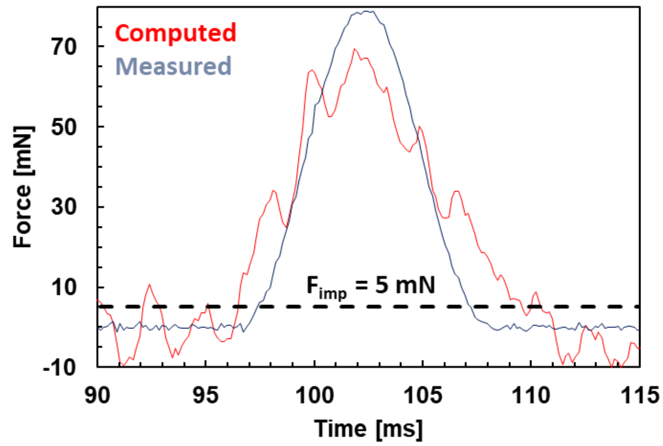

Figure 4 - Computed and measured force signal for a nano-impact in an epoxy resin using an impulse force of 5 mN.

The load cell incorporated to the instrument is a miniature piezoelectric sensor ICP-209C11 from PCB Piezotronics (USA). The sensor incorporates an in-built MOSFET amplifier to convert the measured high impedance charge into a readable low impedance signal. The signal goes through a PCB 482C05 signal conditioner before reaching the acquisition card inside the NanoTest controller. This sensor was selected because of its ability to measure loads in the mN range and its high frequency response. The instrument compliance after adding the force sensor was 0.345 nm/mN, which does not deviate much from the original compliance without sensor, 0.27 nm/mN.

Advantages of using a load cell for nano-impact testing

- It provides the full load-displacement curve during a nano-impact test, which cannot be otherwise determined. The applied force under impact conditions has an important inertia contribution, much higher than the actuated impulse force.
- The ISO-14577 standard for hardness and elastic modulus based on the analysis of the unloading curve can be applied.
- Methods for zero-point correction recommended in ISO-14577 can be applied.
- It opens the door to quantitative testing beyond nanoindentation: microcantilever test, micropillar compression, fibre push-in/push-out test, etc.

Characterization of resin matrix through combined nanoindentation and nano-impact testing

The NanoTest including the force sensor and equipped with a Berkovich indenter was used for testing the epoxy matrix, Hexcel 8552, of a composite system, IM7/8552, used for validation of the multiscale modelling methodology. Constant strain rate nanoindentation tests, using exponential loading schemes, were carried out to cover the low strain rate regime: 0.005, 0.05 and 0.5 s^{-1}. At high strain rates, three impact conditions were applied, using impulse forces of 5, 10 and 20 mN, and an impulse distance of 20 µm in all cases. 5 repetitions per load condition were performed in the nanoindentation cases, and 10 in the nano-impact cases. Figure 5 presents the force-displacement curves of the combined nanoindentation and nano-impact tests. The epoxy exhibits significant elastic behaviour, shown by its large recovery after unloading. Regarding its plastic behaviour, the resin shows a marked strain rate sensitivity, which is revealed in the decreasing depth at constant load with the increase in strain rate. The nano-impact curves at three different impact conditions do not differ significantly, as expected given the slight increase in strain rate with impact velocity shown by the strain rate plots.

A FEM-based inverse analysis was used to extract constitutive parameters that can be directly input in the micromechanical model of a composite ply, details of which can be found in [5]. Figure 7 presents the estimations given by the inverse analysis and they are compared with macroscale compression results on neat resin coupons of the same material. The estimations show significant deviations, which increase with strain rate. This is thought to be caused by the restrictive hypotheses of the inverse analysis. One of them being that viscoelasticity is not considered.

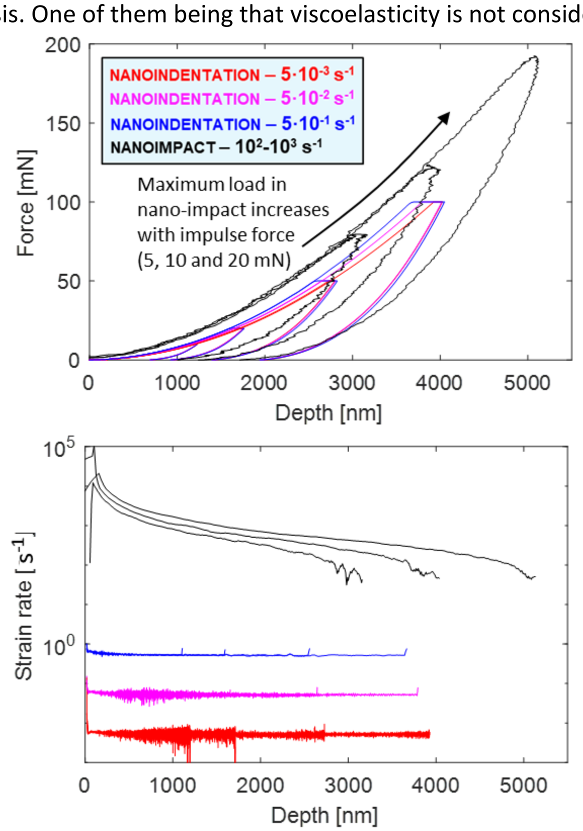

Figure 5 - Force-depth and strain rate-depth curves of the Hexcel 8552 epoxy resin for the combined nanoindentation and nano-impact testing.

Characterization of resin matrix through micropillar compression

Micropillar compression tests were carried out to characterize the constitutive behaviour of the 8552 epoxy matrix. Pillars were carved with a Focused Ion Beam (FIB) of a Helios NanoLab DualBeam 600i of Thermo Fisher Scientific (USA) using annular milling configuration. The procedure resulted in pillars of about 7 µm diameter, which avoids size effects [6], and aspect ratio about 3. They were tested with a 10 µm flat punch. Figure 6 presents the stress-strain curves, which exhibit a similar shape than the macroscale compression curves [5,6]. They also show the significant strain rate sensitivity of the flow stress of the epoxy resin, which is also shown in Figure 7. Unlike the predictions of the inverse analysis fed by nanoindentation results, the flow stress values and their dependency with strain rate correlate well with the macroscale compression results.

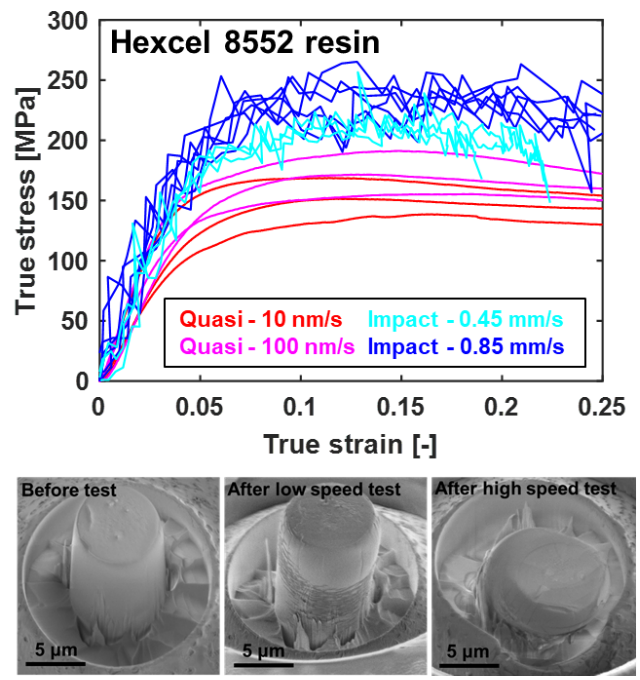

Figure 6 - Stress-strain curves of the micropillar compression test over a wide range of strain rates and observations of the pillars before and after testing. Adapted from [6].

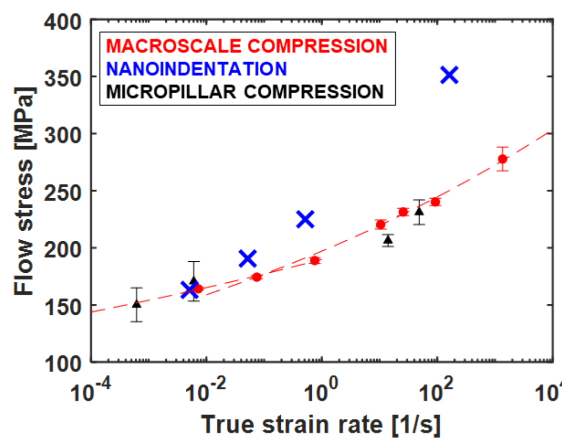

Figure 7 - Comparison of macroscale compression results on Hexcel 8552 neat resin coupons with micropillar compression results and nanoindentation results after applying the inverse analysis.

Micromechanical modelling of fibre-reinforced composites calibrated by micromechanical testing data

The micromechanical testing results of the 8552 epoxy matrix (flow stress as a function of strain rate), together with results from characterization of the fibre-matrix interface through push-in tests (shear strength) [5], were input in the materials models of a Representative Volume Element (RVE) of a composite ply microstructure. Other material parameters were obtained from macroscale testing and literature. The microstructure of the RVE was created with a random-generation algorithm [5] and Periodic Boundary Conditions (PBCs) were imposed to ensure displacement continuity of the nodes in the RVE boundaries and to apply the loading conditions. The RVEs were meshed and analysed in the commercial FEA software Abaqus.

Figure 8 presents a comparison of simulation and macroscale testing results for an in-plane shear loading configuration, which is a deformation mode of the composite ply in which the matrix and fibre-matrix interface play an important role, hence strain rate sensitivity is expected. Two sets of simulations were included: a set with interface properties given by the fibre push-in test in which no strain rate sensitivity was included; and a set with a perfect interface (no damage). While at low strain rates the simulation results of the model with the experimentally-calibrated interface agree reasonably with the experiments, at high strain rates the simulation results with the perfect interface correlate better. These results suggest a change in the failure initiation mechanism of the composite ply with strain rate. At low strain rates, failure initiation is controlled by the combined effect of matrix plasticity and fibre-matrix interface debonding. However, at high strain rates failure initiation is entirely dominated by matrix plasticity. Therefore, these results also suggest that the mechanical behaviour of the fibre-matrix interface is actually rate dependent. Not only that, it seems that the strength of the interface increases with strain rate at a higher rate than the matrix flow stress.

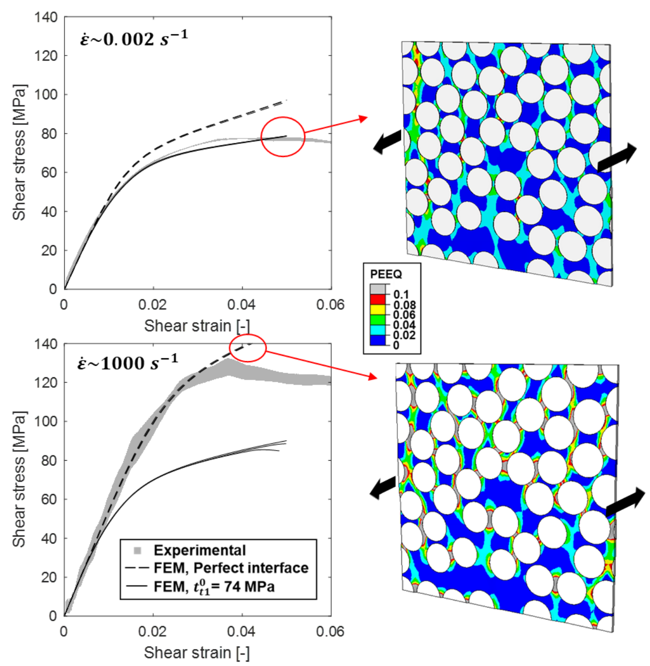

Figure 8 - Comparison of simulation and macroscale experimental results for the in-plane shear response of an IM7/8552 composite ply.

Further proof of this phenomenon would come from testing the fibre-matrix interface over a wide range of strain rates. However, this could not be yet achieved.

Conclusions

- Instrumentation of the NanoTest system with a force sensor to assist in the nano-impact test is possible and necessary in case a full force-displacement curve needs to be obtained.
- The rate-dependent mechanical behaviour of an epoxy resin, Hexcel 8552, could be captured with combined nanoindentation and nano-impact tests. A clear increase in hardness with strain rate was obtained.
- The extraction of material parameters that can go into a constitutive model of the resin was done through a FEM-based inverse analysis. The predictions at low strain rates agree well with macroscale validation data. The estimated flow stress at high strain rates deviates significantly. This is most likely caused by the restrictive hypotheses of the inverse analysis. To this end, micropillar compression tests were proposed as an alternative technique for matrix characterization and they delivered successful results.
- The data from micromechanical testing was used as input for an RVE model of the composite ply that delivered simulations results that compare well with experiments.

Acknowledgements and References

The research leading to these results received funding from the European Union's Horizon 2020 research and innovation programme under the Marie Sklodowska-Curie grant agreement № 722096, DYNACOMP project.

[1] https://cordis.europa.eu/project/id/722096
[2] https://www.youtube.com/watch?v=9OgGmHvqWAM
[3] J. Llorca et al, Advanced Materials 23 (2011) 5130-5147.
[4] M. Rueda- Ruiz et al, Materials & Design 192 (2020) 108715
[5] M. Rueda-Ruiz, PhD Thesis, Universidad Politécnica de Madrid (https://doi.org/10.20868/UPM.thesis.67638).
[6] M. Rueda- Ruiz et al, Extreme Mech. Lett. 40 (2020) 100905.

Micro Materials Ltd

At the forefront of nanomechanics since 1988:

- First commercial high-temperature nanoindentation stage
- First commercial nano-impact stage
- First commercial liquid cell
- First commercial instrument for high-vacuum, high-temperature nanomechanics

Contact us

Micro Materials Ltd
Willow House, Yale Business Village,
Ellice Way, Wrexham, LL13 7YL, UK

Tel: +44(0) 1978 261615
E-mail: info@micromaterials.co.uk
www.micromaterials.co.uk

Dr Mario Rueda-Ruiz
mario.rueda@imdea.org
Dr Jon Molina-Aldareguia
jon.molina@imdea.org

IMDEA Materials Institute
C/Eric Kandel 2, Getafe, Spain