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**Experimental and computational
micromechanics of fibre-reinforced
polymer composites at high strain rates**

TESIS DOCTORAL

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Ingeniero Aeroespacial

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Departamento de Ciencia de Materiales

Escuela Técnica Superior de Ingenieros de
Camino, Canales y Puertos

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2021

*A mis padres,
Manuel y Gema,
y mis hermanos,
Pablo y Manuel*

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Resumen

Los materiales compuestos de matriz polimérica y refuerzo de fibra son en la actualidad el material preferente en aplicaciones que requieren altas prestaciones estructurales y bajo peso. Sin embargo, hay aplicaciones donde los materiales compuestos no han alcanzado todavía todo su potencial, como por ejemplo en componentes sujetos a cargas de impacto, que es una sollicitación típica en las industrias aeronáutica y de automoción. Esto se debe al escaso conocimiento que hay sobre su comportamiento dinámico y a la falta de métodos de análisis precisos. En este contexto, este trabajo propone el uso de una estrategia de simulación multiescala basada en micromecánica computacional para estudiar el efecto que la velocidad de deformación tiene en la iniciación del daño en una lámina de material compuesto. Los ensayos virtuales basados en modelado multiescala abajo-arriba tienen como objetivo la reducción del coste de las campañas de certificación a través de simulaciones de alta fidelidad que complementan los experimentos en todos los niveles de la pirámide de ensayos (LLorca et al., 2011). Este trabajo es una extensión del estado del arte de este tipo de estrategias de simulación (Canal Casado, 2011; Rodríguez, 2012; Naya Montáns, 2017; Herráez Matesanz, 2018) hacia el rango de las altas velocidades de deformación.

Una de las singularidades de la estrategia de simulación multiescala propuesta es el uso de ensayos micromecánicos para la caracterización *in situ* de la matriz y la intercara, que se realizan mediante nanoindentación instrumentada (Herráez et al., 2017). Por ello, el primer paso en este trabajo ha consistido en el desarrollo de una nueva técnica de ensayo micromecánica para extraer las propiedades de los constituyentes del material compuesto en un rango amplio de velocidades de deformación. Se ha modificado un nanoindentador comercial con la capacidad de realizar ensayos de impacto en la nano-/micro-escala para añadir la capacidad de medir la fuerza durante el ensayo. Esto ha posibilitado la determinación de curvas de fuerza-desplazamiento, que es el resultado principal de un ensayo de nanoindentación, en un

amplio rango de velocidades de indentación. En paralelo, se propone un análisis inverso basado en el método de elementos finitos para transformar la respuesta directa del ensayo combinado de nanoindentación y nanoimpacto en parámetros de material necesarios para la calibración del modelo constitutivo de la matriz. El análisis inverso se ha validado en materiales convencionales (cobre y PMMA) para los que existe un conocimiento amplio sobre el comportamiento mecánico a altas velocidades de deformación. Las predicciones del análisis se corresponden adecuadamente con los resultados esperados. Sin embargo, se encontró que las predicciones en el caso de las matrices de los dos materiales compuestos estudiados (IM7-8552 e IM7-M91) se desvían significativamente de los resultados macroscópicos. Por ello, se ha propuesto una técnica adicional para la caracterización de la matriz, el ensayo de compresión de micropilares. Estos ensayos han proporcionado valores precisos del esfuerzo de fluencia en compresión en un rango amplio de velocidades de deformación (10^{-3} - 10^2 s^{-1}) que están de acuerdo con los resultados obtenidos en la macroescala. Adicionalmente, se ha comprobado que la nueva configuración experimental permite la realización de ensayos de push-in de la intercara fibra-matriz a velocidades de impacto, si bien se ha constatado que es necesario mejorar el sistema de adquisición de datos para obtener resultados libres de ruido experimental.

Los resultados de los ensayos micromecánicos, complementados con ensayos en la macroescala y valores de literatura, se han usado para calibrar los modelos de material de una herramienta de simulación basada en micromecánica computacional que emplea un volumen representativo para representar la lámina del material compuesto. La curva de esfuerzo-deformación simulada de la lámina se ha obtenido para dos modos de deformación dominados por la matriz, compresión transversal y cortadura plana, en un rango amplio de velocidades de deformación y se ha comparado con resultados de ensayos en la macroescala. Las predicciones de las simulaciones están de acuerdo con los experimentos. Además, la comparación ha revelado un cambio en el mecanismo de fallo con la velocidad de deformación. A bajas velocidades, el inicio del daño está controlado por el efecto combinado del fallo de la intercara fibra-matriz y la deformación plástica de la resina. A altas velocidades, la deformación plástica de la resina es la principal causa de iniciación del daño.

Abstract

Fibre-reinforced polymer composites are nowadays the material of choice in applications requiring high structural performance and low weight. However, there are still areas where composite materials have not reached their full potential, such as in components subjected to impacts and high speed events, which are often seen in the aerospace and automotive industry. This is mostly due to the insufficient knowledge about their dynamic behaviour and the lack of accurate analysis methodologies. In view of this, this work proposes the use of a physically-based multiscale simulation strategy based on computational micromechanics to study the effect that the strain rate has on the initiation of failure within the composite ply. Virtual testing based on bottom-up multiscale modelling aims at reducing the cost of certification test campaigns by providing high-fidelity simulations that can complement the experiments at all levels of the test pyramid (LLorca et al., 2011). This work represents an extension of this state of the art simulation strategy (Canal Casado, 2011; Rodríguez, 2012; Naya Montáns, 2017; Herráez Matesanz, 2018) to the high strain rate regime.

One of the singularities of the proposed multiscale simulation strategy is the use of micromechanical testing for the *in situ* mechanical characterization of the matrix and the fibre-matrix interface, which is carried out by instrumented nanoindentation (Herráez et al., 2017). To this end, the first step of this work involved the development of a novel micromechanical testing methodology to extract properties of the composite constituents over a wide range of strain rates. A commercially available nanoindenter with the in-built capability of performing impact tests at the nano-/microscale was modified to add force sensing capability. This enabled the determination of a force-displacement curve, which is the essential output of nanoindentation. In parallel, a FEM-based inverse analysis methodology was devised to transform the direct outputs of the combined nanoindentation and nano-impact tests into material parameters needed to calibrate the rate dependent constitutive model of the matrix. The inverse analysis was validated against materials (Cu and

PMMA) with well known rate dependent mechanical behaviour. The predictions of the analysis correlated well with the literature results. However, the predictions for the matrix material of the two composite systems under study (IM7-8552 and IM7-M91) deviated significantly from test results at the macroscale. As a result, an additional technique for matrix characterization, the micropillar compression test, was proposed. The microscale tests provided accurate values of the compression flow stress over a wide range of strain rates (10^{-3} - 10^2 s^{-1}) that were in good agreement with the results obtained at the macroscale. In addition, it was confirmed that the new experimental setup allows for the implementation of push-in tests for the characterization of the fibre-matrix interface at impact conditions. However, the acquisition system of the setup must be improved to obtain results free of experimental noise.

The inputs from micromechanical testing, complemented by macroscale testing and literature values, were used to calibrate the material models of a simulation tool based on computational micromechanics that uses the representative volume element (RVE) approach to represent the composite ply. The stress-strain response at the ply level was simulated for two matrix-dominated deformation modes, transverse compression and in-plane shear, over a wide range of strain rates and compared with macroscale test results. The numerical predictions were in good agreement with the experiments. Furthermore, the comparison revealed a change in failure mechanism with strain rate. At low strain rates, failure initiated due to the combined effect of fibre-matrix debonding and matrix plasticity. At high strain rates, failure initiation was caused primarily by matrix plasticity.

List of acronyms

AFP	Automatic fibre placement
AITM	Airbus industries test method
ALE	Arbitrary Lagrangian-Eulerian
ASTM	American society for testing and materials
ATL	Automatic tape laying
CDM	Continuum damage mechanics
CFRP	Carbon fibre-reinforced polymer
CNC	Computer numerical control
CZM	Cohesive zone modelling
DIC	Digital image correlation
DMA	Dynamic mechanical analysis
ECAP	Equal channel angular pressing
EN	European norm
FAA	Federal aviation administration
FEM	Finite element method
FEA	Finite element analysis
FIB	Focused ion beam
FRP	Fibre-reinforced polymer
GFRP	Glass fibre-reinforced polymer
IPS	in-plane shear
MEMS	Microelectromechanical systems
MOSFET	Metal-oxide-semiconductor field-effect transistor
NASA	National aeronautics and space administration
NNA	Nearest neighbour algorithm
NU	Northwestern University
PBC	Periodic boundary condition
RFI	Resin film infusion

ROI	Region of interest
RSA	Random sequential algorithm
RTM	Resin transfer moulding
RVE	Representative volume element
SEM	Scanning electron microscope
SHPB	Split-Hopkinson pressure bar
SRS	Strain rate sensitivity
TC	Transverse compression
TTSP	Time temperature superposition principle
UD	Unidirectional
UFG	Ultrafine grain
WWFE	World Wide Failure Exercise
XFEM	Extended finite element method

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Introduction

1

1.1 Composite materials for aerospace structural applications

Composites are hybrid materials formed by the combination of two or more materials that present enhanced properties not provided by its constituents individually (Ashby, 2011). Composite materials have been used since the early stages of humankind. Adobe constructions, for example, use a mix of mud and organic materials like straw. The organic phase works as binder and the mud gives the necessary compression strength. Civil engineering materials have evolved over the years, but composite materials are still the usual choice for maximum performance. Most constructions nowadays employ steel-reinforced concrete. Concrete possesses high compression strength, but tensile strength and ductility are low. The steel reinforcement increases the overall tensile strength of the composite concrete structure.

The aerospace industry has experienced in the last fifty years an increase in the use of composite materials, in particular fibre-reinforced polymer composites. The fibre (in short or continuous form) works as reinforcement while the polymer matrix binds the fibres together, gives shape to the composite structure and transfers the loads to the fibres. Low density ceramic fibres are generally used (carbon, glass, aramid, etc.) and the polymer matrix can be made of thermoplastics or thermosets. Composite materials made of carbon fibre and epoxy resin (thermoset) are the most popular choice for application in aerospace primary structures due to the superior properties of the carbon fibre. Thermosets are preferred over thermoplastics because of their lower viscosity (prior to curing) and lower processing temperatures that make them more versatile for manufacturing. Thermoplastics consist of long entangled molecules that need high temperatures ($> 300\text{ }^{\circ}\text{C}$) to gain mobility so that the

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material can be shaped, while thermosets are formed by a 3D network built by crosslinking of short monomer molecules that react with hardener molecules upon heating ($\sim 180\text{ }^{\circ}\text{C}$) during a process called curing. Epoxy resins are a type of high performance thermosetting polymers that exhibit good compatibility and adhesion to the carbon fibre. The most typical processing route for carbon-fibre reinforced polymer (CFRP) composites used in aerospace primary structures is the lay up of fibre laminates preimpregnated with uncured resin (prepreg) over the appropriate tooling, followed by curing in an autoclave that applies homogeneous temperature and pressure. The prepreg ensures accurate control over the fibre volume fraction. The pressure applied in the autoclave helps eliminating entrapped air, leading to a high quality laminate with a very low porosity content ($< 1\%$). CFRP composites present a set of key advantages that place them as material of interest in airframe construction technology:

- CFRP composites offer higher specific (per unit of density) strength and stiffness compared to the traditional lightweight alloys used in aircraft. The high specific properties come from the low density of the constituents used plus the high strength and stiffness of the carbon fibre. The IM7 fibre, for example, which is a typical fibre used for aerospace structures, has a tensile modulus and strength of 276 and 5.5 GPa, respectively. It is about three times stiffer and ten times stronger than typical high strength aluminium alloys. Nevertheless, in reality the mix of carbon fibre with the polymer matrix reduces by a half the strength and stiffness of the composite ply, and it is further reduced (again about a half) when a laminate is built stacking plies with the reinforcement in different directions. Even so, the composite laminate still presents better specific properties than most lightweight alloys.
- CFRP composites are relatively insensitive to fatigue and do not exhibit corrosion. This reduces the amount of maintenance procedures that the structure needs and increases the lifetime of the components.
- High degree of tailoring and versatility for the design. Composite structures enable the design of smooth aerodynamic surfaces with double curvature that are difficult to realize with metallic structures. In addition, joining techniques like cocuring and cobonding eliminate the amount of fasteners in outer surfaces, further improving the aerodynamic efficiency. These types of joining methods enable manufacturing of large components with low part count, thus

reducing the amount of metallic fasteners (and associated weight) as compared with the traditional riveted stiffened stressed metallic skin.

Nonetheless, CFRP composite structures also present certain disadvantages that have delayed their widespread application until recently. Even today, they still hinder further extension of the use of composite materials:

- Higher cost of composite structures compared to their metallic counterparts is one of the most important factors that has slowed down the implementation of composites, especially in commercial aircrafts. In addition, analysis methods are less reliable because of the lower experience with this type of materials, hence their development cost derived from certification test campaigns is higher. Design and development cost predictions are also more unreliable than in metallic design because of the lack of maturity.
- Susceptibility to environmental degradation due to moisture ingestion. This has a quantifiable effect on the mechanical properties of the material, so the design allowables can account for this effect.
- Sensitivity to concentrated and out-of-plane loads. Microcracks are easily generated when the structure is subjected to hail and debris impact. Impact energy is absorbed by the composite structure through creation of cracks, unlike metallic structures in which impact damage is generally translated into plastic deformation. This susceptibility to impact damage in the form of microcracks rises special concerns about damage that is under the sensitivity limit of the detection devices during inspection. Therefore, in composite structures damage tolerance design is equally important as ultimate strength design.
- Damage by lightning strike. Due to the lower conductivity of composite materials compared to metallic structures, structures need to be protected to avoid damage in the event of a lightning strike, using solutions that most of the time increase the overall weight of the structure.
- Complex recyclability. The 3D crosslinked network structure of epoxy resins is irreversible and cannot be separated from the fibre. The carbon fibre can be recycled upon heating and incineration of the epoxy resin.

Despite these disadvantages, composite materials are nowadays the predominant material in aerospace structures. This is the result of a long development and

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Figure 1.1: Evolution of the use of composites for structural applications in commercial and military aircraft and rotorcraft. Data gathered from several sources (Kassapoglou (2010); Blanco Varela (2017); Linde (2010); Harris et al. (2002); Paul et al. (2002); CompositesWorld (2015)).

maturity process that started around the second half of the 20th century. First airplanes were, in fact, made of composite materials consisting of wood, wire and fabric, like the one used in the first flight of the Wright brothers in 1903. Wood got quickly replaced by steel tubes during the 1910s. In parallel, the first all-metal airplanes started appearing, like the designs of Hugo Junkers. These first metallic designs employed mainly a monocoque construction, but by the 1930s the riveted stiffened stressed aluminium skin became the industry standard. It was in the 1960s when polymer matrix composites were first used in aerospace structures. Sailplanes and the helicopter industry were early adopters of the technology (see Figure 1.1) because of the higher importance of improving performance and reducing weight over cost. Business jets were also early adopters of the technology because flyaway costs in this aircraft sector is higher than in commercial aerospace, hence reductions in maintenance and operation costs are more interesting. In fact, the first FAA-certified all-composite aircraft was a business jet, the Beech Starship (Abbott, 1989; Froes, 1989). The FAA had to issue special conditions for the certification of this first all-composite airframe, and focused on: analysis of damage tolerance, strength after impact, failure of adhesive joints and evaluation of environmental effects. The improvement of performance over cost also drove the implementation of the first

composite components in military aircraft. In the early 1970s several public agencies, like NASA or the United States Air Force, funded programmes to stimulate the use of composites in airframe construction (Tenney et al., 2009). Composites were first applied to secondary structure like fairings and control surfaces (e. g. horizontal stabilizers of the F14 and F15) to minimize risk and gather test and in-service data. Use of composites in military applications was extended in the early 1980s in the F-18 and AV-8B programmes after having gained enough maturity (see Figure 1.1). The maturity obtained from the use of composites in the military and rotorcraft industry led to the implementation of the first composite components in commercial aircraft, again starting first with secondary structure like the horizontal stabilizer of the Boeing B737 or the horizontal and vertical stabilizer in the Airbus A320. However, it took more time for composites to gain acceptance in the commercial aerospace business. The reason for this was the much higher cost of the technology compared to the metallic designs. The driver for innovation in composites went then from improved performance to lowering development and productions costs. This involved work on automation of manufacturing processes, examples of which are the creation of the Automatic Tape Laying (ATL) and Automatic Fibre Placement (AFP) techniques. Also new manufacturing technologies were developed, such as dry textile reinforcements fabricated by automated techniques (knitting, weaving, braiding and stitching) combined with liquid moulding processes like Resin Transfer Moulding (RTM) or Resin Film Infusion (RFI). The first major use of composite materials in commercial aircrafts came in the first decade of the 21st century with the Airbus A380. Composites were used in the central wing box, rear unpressurized fuselage, pressure bulkhead, wing ribs and empennage. Further improvements in automation and reduction of cost led then to a major application of composite materials in the last two programmes of Airbus and Boeing with the Boeing B787 and the Airbus A350, featuring fuselage and wings made of CFRP composites. Similar technologies have been used in both programmes and the main difference is in the fuselage construction. Boeing designed a procedure based on full barrel manufacturing, while Airbus preferred a design based on assembly of fuselage sectors to form the full barrel (Hiken, 2018).

Figure 1.1 shows an increasing trend in the use of composite materials over the years, which has been justified by the reduction of manufacturing costs and increased confidence due to the maturity gained in low risk applications (secondary structure). Further implementations of composites in airframe structures are possible, but needs

Figure 1.2: Representation of the Building Block approach (test pyramid) for certification of aerospace composite structures.

additional optimization of costs and improvement of analysis methods used during development and certification to ensure the integrity of the structure. In fact, certification represents the highest nonrecurring costs in an aircraft programme, followed by tooling, nonrecurring fabrication (prototypes) and testing (Kassapoglou, 2010). Therefore, the optimization of analysis methodologies must seek the reduction of the amount of necessary tests, in addition to the enhancement of reliability and robustness.

The traditional analysis procedure for certification of composite structures in the aerospace industry follows the so called Building Block approach, which is represented by the test pyramid in figure 1.2. The pyramid represents an increasing level of complexity of the test set-ups, going from tests to determine the most basic material properties to full scale and component tests that consider all critical service conditions. The first two levels at the bottom of the pyramid comprise the coupon and element tests. They are generic, i.e., the information provided at these levels can be used in all kinds of structures made of the specific material under study. A

large amount of test articles is required in these levels to provide a statistical basis. At the coupon level, the most basic material properties and material allowables for design are determined. The materials are tested under extreme conditions (high and low temperatures, after environmental aging, etc). The next level is occupied by the element tests. This level comprises different structural solutions, in general common to all structures. For example, bolted joints, cocured and adhesive joints and stiffener tests. The next level of the pyramid corresponds to the details tests and they are specific of the structure being certified. They represent discrete parts of the structure, normally critical for the load bearing capability and integrity of the whole airframe or an entire component. Examples of detail tests are stiffened panels, parts of the structure containing load introduction elements, etc. The last two levels of the pyramid correspond to the sub-component (wingbox, fuselage panels, etc.), component (fuselage section, entire wing, empennage, etc.) and full scale testing. These tests are very expensive and require long preparation times; hence, it is convenient to have as much understanding as possible about the mechanical behaviour of the structure from tests at lower levels of the pyramid (Breuer, 2016). The test campaigns at the top of the pyramid must consider all critical load cases, manufacturing and service defects (like impact damage) below the detectability limit and environmental effects. Composites present the additional challenge, compared to metallic structures, of having several damage mechanisms (see section 1.2) and location dependency derived from the susceptibility to out-of-plane loads, thus increasing the amount of testing and cost of certification (Backman, 2005).

In parallel to the test campaigns defined by the pyramid of figure 1.2, analytical and numerical simulation methods also play an important role in the certification process. In addition, they are essential in the initial sizing of the structure. They are carried out at all levels of the pyramid. The test campaigns at the bottom provide inputs for the simulation tools, while the tests at the upper part provide data to validate the predictions made by the analysis methods. Traditionally, structural analysis methods follow a global-to-local approach. Linear FEM analysis of the entire structure is used to obtain distributions of internal loads, followed by detail FEM analysis of critical parts of the structure. In recent years, nonlinear FEM analysis has been incorporated to achieve more accurate stress assessments and to make better predictions about the failure mechanisms of the structure (Ostergaard et al., 2011).

In the traditional global-to-local or top-down analysis approach for composite

1.1. Composite materials for aerospace structural applications

Figure 1.3: Multiscale modelling in fibre-reinforced polymer composite materials (UltraFan image courtesy of Rolls-Royce and image of Molecular Dynamics cell from Okabe et al. (2016)).

structures, the detailed models of the critical areas of the structure often rely on phenomenological material models that are calibrated with the tests at the bottom of the certification pyramid. They are thus constraint to the materials that have been used for certification. This creates a high inertia against material innovation because the certification of a structure made of a different material would require the repetition of the test pyramid. To this end, in recent years a new bottom-up multiscale simulation strategy has been proposed that takes advantage of the hierarchical structure of FRP composites (Cox and Yang, 2006; LLorca et al., 2011, 2013; Lopes et al., 2016; González et al., 2017). Physically-based models that capture the real failure mechanisms of the material are incorporated at each scale of the analysis. The bottom-up strategy is based on the idea of transfer of information between scales by homogenization into constitutive models, rather than coupling several models with different levels of detail, as it is done in the traditional top-down approach. In the bottom-up strategies, three scales of the composite are considered, as shown in figure 1.3: the ply level (μm scale), the laminate level (mm scale) and

the structure level (m scale).

At the microscale, computational micromechanics models are used to capture the level of detail of the composite microstructure, including fibres, matrix and fibre-matrix interface. Two types of models are considered: representative volume elements (RVEs) and embedded cells (see section 6.1). The RVE approach is used to predict failure initiation of the composite ply in the form of failure envelopes, which are equivalent to the ones obtained by classical failure criteria of composites (see section 1.2). RVEs are restricted to failure initiation as propagation of damage is a local phenomenon. In this case, embedded cell models are more suited to determine parameters related to damage propagation (González and LLorca, 2006, 2007b; Canal et al., 2012). Computational micromechanics models need the input of the material properties of the constituents and information regarding volume fraction and fibre distribution. There are several possibilities for the calibration of material models. Fibres properties are usually determined by inverse analysis using analytical micromechanics and test data from standard laminate tests (see section 2.2). Testing of the fibre-matrix interface is challenging, but several approaches can be found in literature (see section 5.3.1). Matrix properties have been often obtained from macroscale testing of neat resin coupons. However, this data does not contain the change in properties derived from the specific processing conditions used in the consolidation of the composite laminate. As a result, micromechanical testing methodologies have been developed to perform an *in situ* characterization of the matrix mechanical behaviour (Herráez et al., 2017). A third option for determining matrix properties is the use of lower scale modelling, like Molecular Dynamics or Monte Carlo simulations (Li and Strachan, 2015).

In the bottom-up multiscale simulation strategy, the laminate (mm) level is modelled with computational mesomechanics models that consist of a stack of several plies. The plies are modelled as a continuum and the interface between plies is generally represented by cohesive zone modelling (CZM). Interply behaviour is calibrated using standard interlaminar fracture toughness tests (ASTM D5528 and ASTM D7905). For intraply behaviour, failure initiation is defined by the failure envelope obtained from computational micromechanics. Damage progression is usually modelled by continuum damage mechanics (CDM). This modelling tool assumes the existence of a homogeneous distribution of microcracks, which is translated in a degradation of the stiffness of the ply. Damage progression is controlled by a damage tensor that accounts for the different failure modes. The information from

1.2. Failure and damage of UD FRP composites

micromechanical simulations of intraply damage (embedded cell models) can be used to calibrate CDM models. Alternative to the CDM tool, CZM or XFEM can be used to model intraply damage evolution. Finally, the last scale in the bottom-up strategy is the structure level. The laminate is modelled as a continuum using shell elements calibrated with the homogenized information from the simulations at the mesoscale. At this scale, structural failure mechanisms like buckling and large nonlinear deformations are considered.

1.2 Failure and damage of UD FRP composites

The prediction of failure and propagation of damage in fibre-reinforced polymer composite materials is challenging because they are formed by two constituents with remarkably different mechanical behaviour. Fibres are very strong and stiff, but brittle. In addition, certain fibres are very anisotropic, e.g. carbon fibres, thus making the composite ply highly anisotropic too. The polymer matrix is much weaker and less stiff than the fibres. Furthermore, it presents a different failure mechanisms depending on the loading condition. In particular, thermoset polymers loaded in compression present large nonlinear deformation before failure, while they behave brittle in tension (see section 4.2.2). Therefore, the combination of both components in a composite materials leads to complex failure mechanisms that depend on the loading configuration. At the microscale (ply level), there are six distinctive failure modes that result from the application of different loading configurations. They are described in figure 1.4.

Failure under longitudinal tension and longitudinal compression leads to failure mechanisms mostly dominated by the fibres, although Naya et al. (2017b) and Herráez et al. (2020a) found that matrix shear yielding and fibre-matrix debonding also play an important role on the longitudinal compression strength. In both cases, the load is applied parallel to the fibres, but the response is notably different. Failure in longitudinal tension is brittle and the response is nearly elastic up to damage initiation, which is controlled by the tensile strength of the fibres and results in cracks perpendicular to the loading direction. Due to the brittle nature of the fibres, their tensile strength depends significantly on the size and distribution of pre-existing defects along their length. To account for this, Naya Montáns (2017) carried out an experimental statistical analysis of the tensile strength of the fibres using the Weibull distribution and incorporated this in a computational micromechanics model based on a RVE of the composite microstructure, by including a

Figure 1.4: Failure mechanisms in a fibre-reinforced polymer composite ply as a function of the loading configuration.

distribution of cohesive surfaces along the fibres length. Other damage mechanisms like fibre-matrix interface debonding were also included. However, Naya Montáns (2017) observed that interface debonding only had a minor effect on the longitudinal tensile strength of the ply. It triggered upon breakage of the first fibre, inducing a slight nonlinearity in the stress-strain curve before the maximum stress. In the case of longitudinal compression loading, however, the response is slightly nonlinear up to brittle failure. Damage is controlled by the fibre kinking mechanism, which is an instability in the fibres loaded in compression that leads to loss of load bearing capability. Naya et al. (2017b) and Herráez et al. (2020a) assumed that the initial fibre misalignment, a defect from manufacturing, is responsible for triggering fibre kinking. Herráez et al. (2020a) performed a comprehensive study of the fibre kinking mechanism using computational micromechanics. They observed that fibre kinking is triggered when the matrix cannot hold the shear stresses any longer and it is followed by further fibre rotation during the post-peak regime. The matrix is loaded in shear because of the misalignment of the fibres. Herráez et al. (2020a) also found that when the interface is weaker than the matrix, the formation of the kink band is triggered earlier because of interface failure.

Transverse tension and transverse compression loading induce failure mechanisms

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in the ply that are controlled by the mechanical behaviour of the matrix and the fibre-matrix interface. Under transverse tension, damage starts at the fibre-matrix interface in locations of high stress concentration like fibre clusters (Naya et al., 2017a). Thermal residual stresses from the manufacturing process may cause even earlier initiation of damage if the interface is much weaker than the matrix (Vaughan and McCarthy, 2011a). However, the opposite happens if the interface has a similar strength than the matrix. The compressive residual stresses delay the initiation of interface damage. Interface cracks induce then severe plastic deformation in the matrix ligaments that remain in between failed interfaces. Final failure takes place when the stress in the ligaments reaches the matrix tensile strength, leading to a nearly linear stress-strain response up to brittle failure through the development of cracks perpendicular to the applied load. The transverse compression response presents a higher degree of nonlinearity before failure. In this case, the ply fails because of the formation of a shear band of localized intense plastic deformation. Two mechanism contribute to the formation of the shear band: yielding of the polymer matrix and the development of damage in the fibre-matrix interface (González and LLorca, 2007a). If the interface is stronger than the matrix, the shear band forms an angle with the loading direction that is defined by the pressure sensitivity of the matrix. However, if the interface is weaker, plastic deformation of the matrix is triggered earlier by interface debonding and the orientation of the shear band changes (Naya et al., 2017a).

The last two failure mechanism in figure 1.4 are caused by shear loading of the ply. Damage is controlled by the mechanical behaviour of the matrix and fibre-matrix interface. In-plane shear loading leads to a highly nonlinear response before failure. At the microscale, damage initiates by the formation of shear cracks in the fibre-matrix interfaces that lead to the appearance of a band of high plastic deformation parallel to the fibres (Totry et al., 2009, 2010). In the case of transverse shear, the stress-strain response is also highly nonlinear. Failure initiation at the microscale depends on whether the interface is stronger or weaker than the matrix. A weaker interface will lead to interface cracks aligned along a direction that forms about 45° with the shear loading axis (Vaughan and McCarthy, 2011b). If the interface is stronger, then failure is initiated by the formation of bands of intense plastic deformation and interface cracks then develop along these bands.

These variety of failure mechanisms contributes to the complexity of predicting failure in fibre-reinforced polymer composites. In addition, the failure mechanisms

interact with each other. For example, shear stresses decrease the transverse compression strength of the ply. Composite materials usually consist of a stack of plies with fibres in different directions, hence several failure mechanism will often appear at the same time in the laminate upon load application (Canal Casado, 2011). Over the years, researchers have developed failure criteria that take into account the different failure mechanisms and the interaction among them. These models are phenomenological and they are based on the definition of a failure envelope obtained by intersecting several smooth surfaces. However, designers often face the challenge of selecting among the many failure criteria available because of the lack of information regarding their validity and limitations. To this end, researchers all over the world gathered efforts to assess many of the available failure criteria in an initiative called the World Wide Failure Exercise (WWFE). A total of 19 failure criteria were selected and the laboratories from the different partners involved were responsible for comparing them against each other and against experimental data provided for validation (Hinton and Soden, 1998). The Puck and Schürmann (1998) model was one of the failure criteria that ranked higher in the WWFE (Soden et al., 2004).

The Puck and Schürmann (1998) model is a modification of the failure criterion proposed by Hashin (1980), which was the first to consider the different failure mechanisms of unidirectional composite plies. Hashin (1980) considered fibre and matrix dominated failure, and differentiated between tension and compression fracture modes. He introduced their effect in a set of failure surfaces, which are defined by a quadratic interaction between the normal and tangential stresses acting on the fracture plane. However, the model failed to provide the orientation of the fracture plane, which led to a poor representation of the transverse compression and longitudinal compression cases. The modification proposed by Puck and Schürmann (1998) included the calculation of the orientation of the fracture plane. Therefore, the angle of the fracture plane in transverse compression and its variation when combined with shear stresses could be accurately determined. Nevertheless, the Puck and Schürmann (1998) failure criterion presents certain drawbacks, one of them being the dependence on non-physical material parameters that are difficult to determine, especially for novel materials. A new failure criterion was proposed by Davila et al. (2005), named LaRC, that solved this issue by determining the fracture angle using the Mohr-Coulomb failure criterion (see section 4.2.2) to represent the response of the ply to transverse loading.

Another conclusion from the WWFE was that the full assessment of the validity

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of the failure criteria is difficult because of the lack of experimental data for failure under multiaxial stress states. This is because testing under multiaxial loading conditions is complex. In this regard, computational micromechanics presents a competitive advantage because multiaxial stress states can be easily introduced in the models. They can thus provide virtual testing data for validation of the failure criteria. Furthermore, they consider all possible failure mechanisms in the composite microstructure, there is control over the fibre distribution and volume fraction and the properties of the constituents can be easily changed. Therefore, not only they can provide validation data for traditional failure criteria, they can also provide accurate failure envelopes to be used by designers. In fact, there have been a significant amount of studies on this topic. Totry et al. (2008a,b) determined the failure envelope under combined transverse compression and in-plane shear loading, and combined transverse compression and transverse shear loading using computational micromechanics models based on the RVE approach. Canal et al. (2009) also used the RVE approach to obtain the failure envelope under combined transverse tension and transverse shear. They all arrived to the same conclusions. When assuming an interface stronger than the matrix, the predictions provided by computational micromechanics agreed well with the predictions from the Puck and Schürmann (1998) model. However, in cases where the interface is weaker than the matrix, the traditional failure criteria failed to match the prediction from computational micromechanics. Melro et al. (2013b,a) arrived to the same conclusion in their computational micromechanics study of all the ply failure mechanisms. Computational micromechanics has not been used exclusively for the study of matrix dominated failure. Romanowicz (2012) carried out a failure analysis under combined longitudinal tension and transverse compression. They found that the micromechanical model provided a failure envelope closer to the experiments compared to the Puck and Schürmann (1998) model prediction.

1.3 Effect of strain rate in the mechanical properties of UD FRP composites

The study of the dynamic properties of FRP composites has attracted the interest of researchers for many years, driven by the many potential applications that need to fulfil crashworthiness requirements. High strain testing techniques are challenging *per se* (see next section 1.4), and the complexity is increased by the many different failure mechanisms of FRP composites (see section 1.2). Ultimately, the mechanical

behaviour of composites at high strain rates is defined by the dynamic properties of its constituents and the interactions between them.

The dynamic properties of the reinforcement phase depend upon the nature of the material with which they are made of (ceramic or polymeric). Wang and Xia (1998a) studied the mechanical behaviour of kevlar fibre bundles in a range of strain rates from 10^{-4} to 10^3 s^{-1} . They found that the tensile strength increases with strain rate, although not as much as the strength of glass fibres bundles, which they also tested in another study (Wang and Xia, 1998b). The same research group tested SiC fibres and also found an increasing tensile strength with strain rate (Huang et al., 2003). In all three cases, the elastic modulus and strain to failure increased with strain rate too. Russell et al. (2013) studied the mechanical behaviour of ultra high molecular weight polyethylene fibre yarns and found that the strength, modulus and strain to failure were not sensitive to strain rates in the range from 10^{-1} to 10^3 s^{-1} . However, at lower strain rates the yarns exhibited creep, thus the strength and modulus decreased with the decrease in strain rate, and the strain to failure increased. Zhou et al. (2007, 2010) studied the mechanical behaviour of carbon fibre bundles over a wide range of strain rates and found that the material properties did not change with strain rate.

Polymers, used as matrix material in FRP composites, are well known to be rate sensitive materials, as well as temperature and pressure sensitive (Siviour and Jordan, 2016). Rate or time dependency manifests both for reversible deformation (viscoelasticity) and nonreversible deformation (viscoplasticity). This rate dependency is related to the molecular motions (translations and rotations) of the main molecular chain and side groups of the polymer, each of them defined by a characteristic time. If the deformation time is below this characteristic time, then the molecular motions will be restricted. Temperature has the same effect on the molecular motions, i. e., low temperatures will lead to a similar response as increasing the strain rate. This is known as the time-temperature superposition principle, and some authors have used it to convert temperature dependency to strain rate dependency or viceversa (Kendall and Siviour, 2013). The mechanical behaviour of polymers from low to high strain rates has been extensively studied. Siviour and Jordan (2016) have compiled a detailed review with many of the studies in this field. In the case of epoxies, which is often the material of choice for the matrix of high performance structural composites, most of the studies in literature deal with the dynamic properties of epoxy-based composites, but there are not many that anal-

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use the material in neat resin form. Gerlach et al. (2008) tested the RTM6 epoxy under compression and tensile loading over a wide range of strain rates (10^{-3} to 10^4 s^{-1}). They found a significant increase in yield stress and elastic modulus in both loading conditions with the increase in strain rate, and an increase in flow stress with strain rate for the compression case. They also found that the strain to failure decreased with strain rate in the tensile case, while it increased under compression. On the contrary, Jordan et al. (2008) did not find an increase in strain to failure with strain rate under compression loading for Epon 826/DEA epoxy. However, they did observe a significant increase in flow stress. Gilat et al. (2007) also found an increase in yield stress and a decrease in strain to failure with strain rate in their characterization of two epoxy resins in a tensile test.

The dynamic properties of UD FRP composites in the longitudinal direction have been studied both under tension and compression, and the behaviour was found to be remarkably different. In longitudinal tension, deformation and failure are dominated by the fibres, so the dynamic properties depend upon the rate dependency of the fibres used. In the case of CFRPs, Harding and Welsh (1983) and Taniguchi et al. (2007) found that the tensile strength and modulus of CFRP laminates were indeed not dependent on strain rate, in line with the rate insensitivity of carbon fibres reported earlier. In contrast, Gerlach et al. (2013) and Harding and Welsh (1983) tested glass fibre reinforced polymer (GFRP) composites and found a pronounced increase of strength, modulus and strain to failure with the increase in strain rate. This is in line with the findings of Wang and Xia (1998b), who concluded that glass fibre bundles are remarkably strain rate sensitive under tensile loads. In the case of longitudinal compression, fibres, matrix and fibre-matrix interface all play an important role in deformation and failure (see section 1.2). Since polymers are highly rate sensitive, this failure mode is expected to exhibit rate sensitivity too. Ploeckl et al. (2017) and Koerber and Camanho (2011) both tested the IM7-8552 composite system from quasistatic strain rates up to strain rates about 100 s^{-1} . They found that the longitudinal compressive modulus was not strongly affected by strain rate, but they found a significant increase in compressive strength.

In the case of matrix dominated failure modes, previous works have found a significant strain rate dependent response. Taniguchi et al. (2007) studied the T700S-2500 composite system in several configurations (transverse tension and $\pm 45^\circ$ in-plane shear) over a wide range of strain rates. They found an increase in modulus and strength in both configurations when increasing the strain rate, specially in

the shear properties. Furthermore, the strain to failure in the in-plane shear tests decreased with the rise in strain rate. Cui et al. (2016) found similar results in their study of the IM7-8552 composite under in-plane shear stress: an increase in shear strength and decrease in strain to failure with strain rate. Both studies also reported a transition from failure dominated by fibre-matrix debonding at low strain rates to failure controlled by combined fibre-debonding and matrix cracks at high strain rates. Under transverse compression loading, Koerber et al. (2010) reported an increase in modulus and strength with strain rate for the IM7-8552 composite, while the strain to failure remained unchanged. Schaefer et al. (2014) arrived to the same results, also in the IM7-8552 system, by testing at strain rates from 10^{-4} to 10^3 s^{-1} .

The study of the dynamic behaviour of UD FRP composites has not been only focused on analysing the rate dependency of the different failure mechanism individually. Some works have also dealt with the determination of failure envelopes of combined loading. Daniel et al. (2011) tested off-axis specimens of AS4/3501-6 in compression at three strain rates (10^{-4} , 1 and $\sim 200 \text{ s}^{-1}$). Multiaxial loading was obtained by decomposing the applied stress in transverse normal and shear components. They then compared the experimental failure envelope at the different strain rates with several failure criteria, finding a good correlation with the Northwestern University (NU) criterion. In a follow-up article, Schaefer et al. (2014) applied the same methodology to the IM7-8552 composite and also found that the NU theory prediction agreed well with the experimental failure envelopes for all strain rates. Koerber et al. (2010) also studied the IM7-8552 composite under multiaxial loading using end-loaded off-axis compression specimens. The in-plane shear and transverse compression response was obtained by applying a transformation of the applied stress and strain from the global coordinate system to the material coordinate system. In this case, the authors compared the experimental failure envelopes at different strain rates with the predictions from the Puck and Schürmann (1998) criterion and found an excellent agreement. Kuhn et al. (2015) extended the study to the combined in-plane shear and transverse tension behaviour using off-axis tensile specimen. They also found that the prediction from the Puck and Schürmann (1998) model correlated accurately with the experimental failure envelopes.

In line with the current trend towards multiscale modelling of composites and virtual testing, several authors have also approached the study of dynamic properties of composites from a multiscale modelling perspective. Lidgett et al. (2011) set up a

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microscale unit cell with hexagonal packing arrangement and analysed it with FEM. They introduced different constitutive models for the fibre, matrix and fibre-matrix interface. They calibrated the fibre and matrix models with experiments at the macroscale, and the interface model with fibre push-out results from other authors (Tanoglu et al., 2000). Sorini et al. (2020) used a semi-analytical micromechanics model, the Generalized Method of Cells, to carry out predictions of the ply behaviour under several loading configurations. The constitutive models were calibrated with macroscale neat resin tests. Li and Ghosh (2020) opted for the RVE approach to perform a computational micromechanics study at high strain rates. They used two types of RVEs: one with a single fibre (squared arrangement) and the other with a random distribution of fibres. Again, macroscale testing data was used for model calibration.

1.4 Techniques for the mechanical characterization of materials from low to high strain rates and across length scales

The mechanical characterization of materials is an essential discipline of engineering and science that serves several purposes: (1) qualification of a new materials and determination of material allowables for design; (2) assistance during material development programmes for screening of mechanical properties; (3) calibration of material models for numerical analysis of structures; (4) provision of experimental data for validation of numerical simulations; (5) quality control during production; among others. There are many types of mechanical characterization techniques and one way to classify them is based on the mechanical behaviour that aims at assessing: elasticity, plasticity, fracture, fatigue, etc. The working principle of most of these techniques is the same. A specimen of the material with a certain geometry is deformed in a controlled manner by mechanically loading the specimen. There are many types of loading configurations: tension, compression, bending, torsion, etc. Some techniques, the ones for stiffness and strength testing, produce a stress-strain relation that defines the constitutive behaviour of the material; others, like the fracture tests, lead to controlled propagation of cracks from which quantifiable material properties can be extracted. However, there is another set of techniques that are based on a different working principle. These are the indentation or hardness testing techniques. In a hardness test, the material surface is deformed by contact loading

with a tip of a very hard material. In this case, the output is a material metric that can be used to rank or compare several materials, but there has been large research efforts to extract properties equivalent to the ones obtained in tensile or compression testing (see section 4.1).

Material testing is carried out over a wide range of environments and loading scenarios, representative of the real conditions that the material will encounter in service. One of these scenarios is the deformation at high speeds, which is found in most machining and forming processes and, in general, in applications where the material is subjected to impacts, like in the automobile and aerospace industries. Material testing at high strain rates is challenging because it involves the emergence of physical phenomena that do not materialize at low (quasistatic) strain rates. Beyond 10^0 - 10^2 s^{-1} , depending on the size and density of the specimen and test fixtures, inertia effects and the propagation of stress waves along the sample must be considered (Field et al., 2004; Kuhn and Medlin, 2000). When the test time is in the order of magnitude of the time that the stress waves take to travel along the samples, the loading of the specimen will not be uniform, thus the stress-strain relation will not be comparable with those obtained at quasistatic conditions. Regarding inertia, the inertia term (force needed to accelerate the particles of the material) will also increase with increasing speed up to a point, beyond 10^4 s^{-1} , where the inertial stress increases exponentially and induces confinement of the material, causing a transition from a state of uniaxial stress to a state of uniaxial strain (Field et al., 2004). Thus, material testing at high strain rates requires the use of specialized equipment because the conventional testing machines used for testing at quasistatic strain rates cannot reach strain rates beyond 10 - 100 s^{-1} .

Material testing has been traditionally constraint to the millimetre scale, and even to larger scales if component and full scale structural testing are considered. However, the advances in scientific instrumentation and electronics over the second half of the 20th century have led to the miniaturization of the mechanical testing devices, enabling the extension of the material testing field to the nanometre and micrometre scales. At these scales, material testing is carried out using instrumented nanoindenters, which are designed to perform hardness tests while monitoring load and displacement with sub-micronewton and sub-nanometre precision, respectively (Fischer-Cripps, 2011). Nanoindentation testing has become increasingly popular because it allows testing of small volumes and thin films that could not be tested otherwise by macroscopic techniques. This is particularly useful in emerging fields

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like microelectromechanical systems (MEMS) and advanced coatings, and to understand the physics behind the size-dependent deformation of materials. Therefore, nowadays a true multiscale mechanical characterization of materials is possible.

The combination of material testing at multiple length scales with testing at multiple loading rates is specially interesting because one can achieve a certain desired strain rate range either by changing the loading rate or the size of the specimen. For a given strain rate, the needed loading rate decreases by decreasing the specimen length scale, thus potentially avoiding the typical issues of high strain rate macroscale testing, like the inertia contributions and the stress wave equilibrium times (Ramachandramoorthy et al., 2019). Figure 1.5 represents a map including most of the techniques available for material testing, as a function of length and time scales. It clearly shows how the same level of strain rate can be achieved at lower actuating speeds by decreasing the characteristic size of the specimen. For example, the laser-induced particle impact technique reaches strain rates that would only be possible to achieve at the macroscale by testing at speeds over 10 km/s, only realizable with very specialized equipment like multistage gas guns or plasma guns (Veysset et al., 2021).

The lower right part of figure 1.5 is populated by conventional testing techniques, which are performed in universal testing frames. These machines are designed to test materials under various loading conditions (tension, compression, bending, etc.). They have evolved from purely mechanical devices to modern electronically-controlled devices with microprocessors that increase the quality and reliability of the test results. Material testing in these machines is carried out by moving a crosshead or bench at a controlled speed while measuring the applied load with a load cell. There are two types of actuating systems: (1) electro-mechanical actuation in which the crosshead is moved by a screw connected to a motor through a gear box; and (2) servo-hydraulic actuation in which a piston is moved by a hydraulic pump and servohydraulic valves. These devices are limited to the low and intermediate strain rate regimes. Actuation rates in electro-mechanical devices are limited by the speed of the motor and the design of the transmission system, and by the capacity of the hydraulic system (size of the actuator, type of pressure valve, etc.) in the servo-hydraulic machines, with servo-hydraulic frames covering a wider range of displacement rates (Kuhn and Medlin, 2000). They can go from the typical rates of creep testing to the intermediate strain rate regime ($1\text{-}100\text{ s}^{-1}$). However, at the limit of their operation ($100\text{-}1000\text{ mm/s}$), servo-hydraulic frames usually experience

Figure 1.5: Material testing techniques across length and velocity scales. Sources: Kuhn and Medlin (2000); Perogamvros et al. (2016); Siviour (2017); Maudlin et al. (1999); Klopp et al. (1985); Ramesh (2008); Lu et al. (2003); Marshall et al. (1983); Nobre et al. (1997); Shore (1918); Barnoush et al. (2019); Golovin (2021); Sudharshan Phani and Oliver (2017); Guillonneau et al. (2018); Wheeler and Gunner (2013); Hassani et al. (2020)

issues that challenge the determination of accurate material data: (1) oscillatory response in the load signal coming from instrument ringing; (2) likely lack of stress equilibrium if the distance between the actuation system and load measuring device is high; (3) poor control over the displacement rate that leads to the strain rate not being constant; and (4) uncertainties coming from lack of synchronization between the different signals recorded during the test. Regarding the later, Spronk et al. (2018) carried out an in-depth analysis of all the factors to take into account concerning data acquisition at high loading rates, also highlighting the importance of using acquisition equipment with frequency response well above the characteristic test time. They also studied through FEM simulations the effect that the delay in either the stress or the strain signal has in the stress-strain curve and the effect of load cell ringing. Regarding dynamic equilibrium, Hufner and Hill (2017) carried out a detailed analysis on how to ensure dynamic equilibrium in intermediate strain rate testing in hydraulic frames. Although the intermediate strain rate regimes can be covered with hydraulic testing frames, there are also many studies that have

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opted for a drop-weight impact tester. For example, Perogamvros et al. (2016) and Fernie and Warrior (2002) carried out tensile and compression testing at intermediate strain rates in a drop-weight tower using special fixtures to transform the impact loading into tensile or compression loading on the sample.

The high strain rate regime (upper right corner of figure 1.5) is covered by a variety of techniques. The split-Hopkinson pressure bar (SHPB) or Kolsky bar is the most popular among them because it allows testing the material in a range from 10^3 to 10^4 s^{-1} at a state of uniaxial stress (Kuhn and Medlin, 2000). The stress-strain response obtained from the SHPB can be used therefore to extract directly the constitutive behaviour of the material at high strain rates. Nevertheless, the SHPB technique is not exempt from challenges. At these high strain rates, the characteristic test time is in the order of magnitude of the travel time of the stress waves. Therefore, it is necessary to ensure that the sample is in dynamic equilibrium in order to extract a valid stress-strain response. This is especially challenging in polymeric materials, which have low density and elastic modulus, and therefore, present a speed for elastic wave propagation that is significantly lower than for metals or ceramics (Chen et al., 2002). In the SHPB technique, the material is tested by placing a specimen in between two bars, the incident bar and the transmission bar. A strain pulse is introduced at the edge without sample of the incident bar using an impactor bar. All bars are made of the same material and have the same diameter. The strain pulse deforms the material and stress and strain can be obtained from 1D analysis of the stress waves (see section 2.3.1).

Beyond 10^4 s^{-1} there are several techniques and they all have in common that a state of uniaxial stress is no longer reachable due to the inertia effects. One of these techniques is the Taylor impact test. A cylinder of the material under study is launched against a rigid anvil. Initially, this test was intended at estimating the dynamic flow stress of the material by measuring the change in shape. However, this method lacked the accuracy of other techniques, like the Hopkinson bar test for which a full stress-strain relation can be obtained Field et al. (2004). Therefore, it is nowadays more widely used as an experimental technique to validate constitutive models of materials. Another set of techniques are the plate impact and ballistic impact experiments. In plate impact, a plate of the material under study (the flyer plate) is impacted against a stationary plate or anvil (Ramesh, 2008). The flyer plate is normally launched using a gas gun. The plate is often mounted on a sabot which works as a support for the flyer plate while it travels along the gas gun barrel.

The plate is normally launched perpendicular to the motion direction, but there is a variant in which the flyer plate and the target plates are inclined. This is the so called pressure-shear plate impact experiment. A very thin specimen (1-100 μm) is placed on the surface of a disc made of a harder material (Klopp et al., 1985). The disc-sample is launched at a high speed against the surface of a fixed anvil made of the same hard material used for the disc that supports the sample. When impacting, the sample is sandwiched in between the two hard plates. Because of the inclination, a combined shear-pressure (hydrostatic pressure) loading is introduced in the sample. Shear stress and strain can be derived from the measurement of the normal and transverse motions of the rear surface of the fixed anvil (the target plate). This is normally carried out by laser interferometry. This test requires special caution when comparing the stress-strain response with the one at lower speeds because the manufacturing of very thin samples may lead to microstructural changes as compared to the microstructure of a specimen for conventional testing.

The left side of figure 1.5 contains the nano-/micro-scale testing techniques. These techniques are based on instrumented nanoindentation testing (see section 3.1.1). Although, in recent years the range of techniques has been expanded by the combination of nanoindentation testing with nano- and micro-fabrication, which has enabled carrying out uniaxial tension and compression and fracture tests at these low length scales. The micropillar compression test is specially popular nowadays for probing the stress-strain response of materials at the nano-/micro-scale (see section 5.2). The operating principle of instrumented nanoindenters consists of bringing a hard indenter (usually diamond) in contact with the material surface while monitoring load and displacement. Unlike conventional macroscale testing machines, nanoindenters are normally load-controlled. In most of the instruments, load is actuated using an electromagnetic coil (Fischer-Cripps, 2011); although, some instruments employ electrostatic actuation or displacement-controlled piezo-actuators. Some instruments incorporate a load cell to measure the applied load on the sample, but most devices rely on the reading from the coil in combination with a load calibration procedure. In most designs, displacement sensing is accomplished with capacitance transducers. The configuration of most instrumented nanoindenters consists of a loading head in which the actuator and sensors are all aligned vertically with the indenter. However, there are other configurations, like the pendulum design in which displacement sensor, indenter and sample are aligned horizontally at one side of the hinge, and the actuation system is placed at the other side of the

1.4. Techniques for the mechanical characterization of materials from low to high strain rates and across length scales

pendulum hinge (see section 3.2). Instrumented nanoindenters present many advantages compared to conventional testing techniques, such as lower testing and sample preparation times, versatility and high automation. However, they are susceptible to uncertainties derived from other factors, such as thermal drift, surface roughness, zero-depth determination, indenter tip imperfections and instrument compliance, among others (Fischer-Cripps, 2011).

Nano-/micro-scale characterization techniques have also been extended to the study of high strain rate mechanical behaviour. The first attempt to carry out instrumented nanoindentation tests at high strain rates dates back to the late nineties and was based on nano-impact testing. The technique was primarily developed to perform repetitive impacts on a material surface for studying coating wear resistance, toughness and adhesion (Beake et al., 2001, 2002); and subsequently modified to allow high frequency data acquisition to obtain a complete time-evolution of the displacement signal throughout the impact, enabling the study of single-impact events (Tweedie et al., 2004; Constantinides et al., 2008, 2009). More recently, Phani et al. have developed a nanoindentation technique based on rapid step loading for which strain rates in the order of 10^3 s^{-1} could be achieved (Sudharshan Phani and Oliver, 2017). Another high strain rate technique was recently proposed by Guillon et al. (2018) based on a displacement-modulated force-sensing device, for which they also showed that strain rates in the order of 10^3 s^{-1} could be achieved. The authors have shown that the piezo-actuator enables an accurate control over the displacement scheme at high rates, allowing to carry out constant strain rate nanoindentation tests at high strain rate. They have demonstrated the capabilities of this novel nanoindentation device to perform high strain rate micropillar compression, and applied the technique to study the rate dependent deformation behaviour of nanocrystalline nickel. The technique was later expanded to the study of ceramic materials (Ramachandramoorthy et al., 2019) and polymers (Rohbeck et al., 2020).

All these techniques for high strain rate testing at the nano-/micro-scale cover a wide range of strain rates up to 10^3 s^{-1} , which is representative of many loading scenarios that materials experience in service, like typical debris impacts in automobile and aerospace industry, materials for sports, etc. However, there are certain scenarios that require the characterization of the material mechanical response at even higher strain rates. For example, in space exploration and additive manufacturing. A group of researchers have developed a technique, the laser-induced particle impact test, that covers this strain rate range (Hassani et al., 2020; Veyssset et al., 2021).

Strain rates as high as $10^6 - 10^8 \text{ s}^{-1}$ were reached by impacting alumina particles launched by a laser pulse. The authors used a high speed camera to measure the impact and rebound velocity of the particle and optical imaging techniques to measure the indentation mark after the impact. Then, they obtained hardness values from the dissipated energy in the impact and the indentation volume.

There are no techniques in the low speed μm - mm range in figure 1.5. However, in practice this range can be covered by several techniques available both commercially and in academia. Gianola and Eberl (2009) have gathered a detailed review of methods for microscale testing that serve as a bridge between the nano- and macro-scales. For example, microtensile testers have become specially popular because they allow *in situ* imaging inside an optical or electronic microscope while deforming the sample. At high strain rates, several techniques based on indentation have been proposed over the years and they cover a large range of high strain rates from 10^2 to 10^6 s^{-1} (see figure 1.5). The first studies were based on indentations using drop-weight impact (Shore, 1918) (velocity around 1-10 m/s). More recent studies have employed gas guns to launch an indenter towards the material surface (Lu et al., 2003; Marshall et al., 1983) (velocity around 1-50 m/s) and pendulum impact (Nobre et al., 1997) (velocity around 0.1-1 m/s). In all cases, the length scale of the indentation is about 1-100 μm .

1.5 Motivation and objectives of the work

One area where composite materials have not reached their full potential is in low-weight applications where the material is subjected to impacts and high speed events, which are often seen in the aerospace and automobile industry. This is mostly due to the insufficient knowledge about their dynamic behaviour and the lack of accurate analysis methods. To this end, multiscale modelling presents itself as a promising tool to provide reliable and robust predictions of the dynamic behaviour of composites, given its success at quasistatic conditions (LLorca et al., 2011). Multiscale methods have been already proposed for the study of the dynamic behaviour of composites (see section 1.3). However, there has not been any attempt yet to develop an integrated physically-based multiscale simulation strategy that uses *in situ* micromechanical testing to calibrate strain rate dependent material models of the composite constituents. Two research objectives have been established with this idea in mind:

1.6. Structure of the dissertation

- The development of novel micromechanical testing techniques for the mechanical characterization of the composite constituents (fibre, matrix and fibre-matrix interface) over a wide range of strain rates (10^{-3} to 10^3 s^{-1}) at the nano-/micro-scale. The novel technique needs to provide the necessary inputs for the material constitutive models of the composite constituents implemented in the multiscale simulation strategy.
- The creation of a multiscale simulation tool based on computational micromechanics that can predict the mechanical behaviour of the composite ply over a wide range of strain rates (10^{-3} to 10^3 s^{-1}). Furthermore, the tool needs to be validated against macroscale experimental results. Two composite materials systems, one well-known to the industry and the other recently developed and optimized for high strain rate applications, have been selected to assess the validity of the novel computational micromechanics tool.

1.6 Structure of the dissertation

The accomplishment of the previously stated research objectives has involved a step-wise process consisting of several tasks (some of them performed in parallel). Each of these tasks and the associated results are presented in a series of chapters, which were structured as follows:

Chapter 2 presents the two composite materials systems (IM7-8552 and IM7-M91) used for testing the novel micromechanical testing methodologies and the computational micromechanics simulations. In addition, it contains results from several test campaigns performed at the macroscale with two objectives: (1) providing validation data for the computational micromechanics models and the novel micromechanical testing techniques; and (2) completing the set of inputs needed for the models that could not be provided by the micromechanical testing techniques. Based on the review in section 1.3, in-plane shear and transverse compression tests were chosen for validation, because they present a potentially high strain rate sensitivity.

The development of the novel micromechanical testing techniques involved two main challenges. First, the implementation of a suitable instrumentation to perform high strain rate tests at the nano-/micro-scale. Among the techniques presented in the review of section 1.4, the nano-impact test was selected. The test device had to be modified with new instrumentation in order to obtain suitable outputs to

extract material properties, i. e. a force-displacement curve. Furthermore, the new instrumentation was assessed by testing materials with a wide spectrum of mechanical behaviour. This is the main topic covered in chapter 3.

The second step in the development of the novel micromechanical testing methodologies was the creation of an inverse analysis framework to extract the material properties, needed as input in the computational micromechanics simulations, from the indentation tests, which is covered in chapter 4.

Chapter 5 presents the micromechanical characterization of the composite microconstituents using the modified instrument presented in chapter 3. In particular, the results of applying the nano-impact indentation test and the analysis framework proposed in chapter 4 to extract the strain rate dependent behaviour of the matrix materials in the composites under study are presented in this chapter. The outcomes are compared against the results obtained in the macroscale tests presented in chapter 2 and against other potential micromechanical testings techniques, like the micropillar compression test, performed using the same modified instrument. This chapter also contains the results from the characterization of the fibre-matrix interface at different strain rates.

Finally, chapter 6 presents the computational micromechanics approach that was used to predict in-plane ply properties over a wide range of strain rates for the two materials under study (IM7-8552 and IM7-M91). Inputs from chapters 2 and 5 were used to calibrate the material models of the three composite constituents. The simulation results were validated against the macroscale experiments presented in chapter 2.

Finally, chapter 7 compiles the main conclusions and proposed future work, while chapter 8 contains a list of publications and contributions to conferences in which the work carried out has been presented.

Materials and macroscale characterization

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The novel experimental and computational methodologies presented throughout this work were tested on two composite materials systems. The IM7-8552 is a well-known material used extensively in the aerospace industry. There are many studies in literature that focus on this material, thus making it perfect to validate new testing and computational methodologies. The second composite system is the IM7-M91. This is a newer composite material that presents a superior performance in high strain rate and impact applications. Further details about these materials are included in section 2.1. This chapter contains the results of a series of test campaigns that aimed at providing experimental results for validation of the novel micromechanical testing and micromechanical modelling methodologies developed in this work. It also includes results from test campaigns needed to provide inputs for the micromechanical models presented in chapter 6. Particularly, section 2.2 presents the results of a test campaign aimed at estimating the IM7 fibre properties, which are needed for the micromechanical modelling and cannot be obtained from micromechanical testing. Section 2.3 contains the results of the two tests used for validation of the computational micromechanical simulations in chapter 6: (1) the in-plane shear test and (2) the transverse compression test, both performed over a strain rate range of six orders of magnitude: from 10^{-3} to 10^3 s^{-1} . Section 2.3 also contains the results of two test campaigns on neat resin specimens: (1) dynamic mechanical analysis (DMA) testing to characterize the elastic modulus over a wide range of strain rates, and (2) compression tests to validate the micromechanical test methodologies in chapter 5.

2.1 Materials

Two unidirectional carbon fibre reinforced epoxy systems were studied: IM7-8552 and IM7-M91. The same fibre is used in both materials, the HexTow IM7 of Hexcel (USA), an intermediate modulus carbon fibre of high performance with widespread application in the aerospace industry. The Hexcel 8552 resin is an amine-cured epoxy containing polyethersulphone. This is a tough resin that has been used for aerospace applications for many years. The IM7-8552 system is very popular and its properties and performance are well known to the industry; hence it is the perfect material for the development of new test methodologies and validation of simulation tools. The Hexcel M91 resin has a similar formulation to the 8552. It also contains polyethersulphone as toughening agent, but it incorporates additional thermoplastic interleaf particles for extra toughening. The IM7-M91 system is a third generation composite material intended for applications that require high toughness; for example, structures subjected to impact events. Figure 2.1 shows SEM images of the two composite systems and an example of a C-scan analysis after a 30 J impact in panels of both materials carried out in a drop-weight tower. The IM7-M91 clearly exhibits a superior performance.

Samples for mechanical testing were extracted from panels manufactured by autoclave consolidation. The material was supplied by Hexcel in prepreg form with area weight of 268 gsm (resulting in a consolidated ply thickness of 0.25 mm). Panels were prepared by hand lay-up followed by vacuum bagging. Debulking of the panels was carried out every 4 plies in a vacuum press for around 10 minutes. Then, they were cured in an autoclave at 180 °C for 135 minutes, with a heating/cooling rate of 2 °C/min. A pressure of 7 bar was applied during heating, curing and cooling. The nominal fibre volume fraction of both composite systems was 60 %.

Panels of neat resin material were also prepared. The 8552 and M91 epoxies are high viscosity systems, thus they were heated up to 100 °C for 30 minutes and speed-mixed at 2300 rpm for 1 minute before pouring them into open moulds. This is to reduce the amount of entrapped air in the material. The same autoclave curing scheme applied in the composite systems was used.

Figure 2.1: Typical microstructures of IM7-8552 and IM7-M91 composite systems and example C-scan inspections after 30 J impact in 110x110 mm panels of both materials.

2.2 Mechanical tests of composite coupons for estimation of fibre mechanical properties

The direct determination of fibre mechanical properties is challenging because they are not easy to handle (thickness of a few micrometres). Certain properties, like the longitudinal tensile strength and modulus, can be obtained by testing fibre bundles. Micromechanical testing is neither a straightforward option, specially in carbon fibres, which are transversely isotropic. Therefore, the calibration of fibre properties is normally done by testing coupons of the composite and neat resin material, followed by inverse analysis based on analytical micromechanics (Naya Montáns,

2.2. Mechanical tests of composite coupons for estimation of fibre mechanical properties

Figure 2.2: Coupons used for testing the composite and neat resin materials following industry standards.

2017; Rodríguez, 2012). One of the most popular methods is the Chamis rule of mixtures (Chamis, 1984). This model assumes transverse isotropy, thus the fibre and the laminate are defined by five independent elastic constants: the longitudinal elastic modulus, E_1 , transverse elastic modulus, E_2 , longitudinal shear modulus, G_{12} , transverse shear modulus, G_{23} , and the longitudinal Poisson's ratio, ν_{12} . The resin is assumed to be fully isotropic, thus the elastic behaviour is defined by two independent constants: the elastic modulus and Poisson's ratio.

The elastic properties of the composite laminate and the neat resin coupons were obtained by macroscale testing following industry standards. Four tests were required: longitudinal tensile test of composite coupons (ASTM D3039), transverse tensile test of composite coupons (EN2597), in-plane shear test of composite coupons (AITM 1-0002) and tensile test of neat resin coupons (ASTM D638). A schematic of the geometry of the samples is shown in figure 2.2. All composite coupons had the same shape, but the dimensions differed. Table 2.1 compiles the nominal dimensions of the specimens. Tabs were used in all three composite specimens. They were made of a composite panel consisting of four plies ($[\pm 45]_2$) of E-glass and Hexcel 924 epoxy, and bonded with Redux 312/5 epoxy adhesive. The neat resin coupons were made with a dogbone shape to promote failure away from the grips.

Tests were carried out at a low speed, as specified in the standards, of about 0.07 s^{-1} . Figure 2.3 presents the stress-strain curves of the four tests. Only the results for the IM7-8552 system are presented and the properties of the IM7 fibre are estimated from them, but the estimation using the IM7-M91 properties provides similar results.

2. Materials and macroscale characterization

Test	Standard	Laminate	L	W	G	T	W _g	t	tt
Longitudinal tension	ASTM D3039	[0] ₄	250	15	130	60	-	1	1.2
Transverse tension	EN2597	[90] ₈	250	25	130	60	-	2	1.2
In-plane shear	AITM 1-0002	[±45] _{2s}	230	25	130	50	-	2	1.2
Tensile (dogbone)	ASTM D638	-	165	19	50	25	13	4	-

Table 2.1: Dimensions of the test samples for standard characterization of the composite and neat resin materials. Dimensions according to sketches in Figure 2.2 (in mm).

The longitudinal and transverse tensile response of the composite laminate was nearly linear with brittle failure, as expected. The longitudinal tensile properties of the laminate are dominated by the stiff and brittle behaviour of the carbon fibres, while failure in transverse tension is dominated by fibre-matrix interface debonding and brittle tensile failure of the matrix. Most of the transverse tension specimens failed near the tab area where the machine grips were grabbing the specimens, driven by the higher stress concentrations in this area. The standard recommends the careful alignment of the set-up to avoid bending or torsion loading modes that may trigger failure initiation in the tab area. However, even after cautious alignment of the set-up, it was not possible to ensure failure within the gauge (untabbed) length. This is the reason to use a dogbone geometry in the neat resin samples. In this case, only some of the specimens failed in the tab area. The resin tensile behaviour presented a slight nonlinearity, but the specimen still failed in a brittle manner. The in-plane shear test was the one that presented higher nonlinearities, as expected. Failure in in-plane shear involves a complex interaction of different mechanisms: damage is initiated by fibre-matrix interface shear failure and matrix yielding, and propagates towards the interleaf region, where delaminations appear.

Table 2.2 compiles the elastic properties of the composite laminate and neat resin samples obtained in the tests and the estimation of the IM7 fibre using the inverse Chamis rule of mixtures. The obtained laminate properties are close to the values in literature; although, the transverse and shear moduli present certain deviations. These tests are more susceptible to experimental uncertainties because the elastic modulus is much lower than the value in longitudinal direction. Small misalignments in the fibre direction ($\sim 1^\circ$) or in the specimen set-up may have a significant effect in the modulus. In the case of the shear modulus, the value obtained

2.2. Mechanical tests of composite coupons for estimation of fibre mechanical properties

Figure 2.3: Stress-strain curves of the standard mechanical tests of IM7-8552 composite and 8552 neat resin coupons.

2. Materials and macroscale characterization

in the tests for characterization over a wide range of strain rates (see section 2.3.5) has been included in the table for comparison. The transverse shear modulus was derived from literature. The estimated IM7 elastic properties were also compared with literature. (Kaddour and Hinton, 2012) reported a comprehensive review of properties of several commercial fibres and composites.

IM7-8552 properties					
	E_1^l [GPa]	ν_{12}^l	E_2^l [GPa]	G_{12}^l [GPa]	G_{23}^l [GPa]
Test results	167.1 ± 2.6	0.287 ± 0.028	8.6 ± 0.1	6.2 ± 0.1 (5.30 ^a)	3.2^b
Literature range	[150-171.4]	[0.32-0.33]	[9-12]	[4.7-5.6]	[2.3-3.9]
8552 properties					
	E_m [GPa]	ν_m			
Test results	4.2 ± 0.1	0.374 ± 0.010			
IM7 estimated properties					
	E_1^f [GPa]	ν_{12}^f	E_2^f [GPa]	G_{12}^f [GPa]	G_{23}^f [GPa]
Inverse Chamis analysis	275.7	0.23	12.2	55.5 (18.3)	4.7
Kaddour and Hinton (2012)	276	0.2	19	27	7

Table 2.2: Summary of elastic properties obtained for the IM7-8552 composite and 8552 neat resin materials, and estimation of elastic properties of IM7 fibres. Composite lamina characterization compared against literature (Hexcel, a; Camanho et al., 2007; Krueger et al., 2007; Krueger, 2012; Marlett, 2011; Schaefer et al., 2014). (a) The obtained shear modulus is significantly higher compared to literature, so the value obtained in in-plane shear test over wide range of strain rates (see Section 2.3.5) is included for comparison. (b) This value is an average from literature (Krueger et al., 2007; Krueger, 2012; MIL-HDBK-17, 2002).

2.3 Mechanical characterization over a wide range of strain rates

2.3.1 Instrumentation

The mechanical characterization of composite laminates over a wide range of strain rates (from 10^{-3} to 10^3 s^{-1}) required the use of several mechanical test frames. There is not a single mechanical test device that can cover such a wide range of strain rates (see section 1.4). Three types of mechanical testing machines were used in this study.

2.3. Mechanical characterization over a wide range of strain rates

Low strain rate testing was carried out in a screw-driven electromechanical frame. This type of machine covers the very low strain rates range (creep testing) and are limited in their upper limit to speeds of about 1-10 mm/s. The electromechanical frame used was an Instron 5966 equipped with a 10 kN load cell. Mechanical wedge grips were employed for the in-plane shear tests, and special attention was given to the alignment of the two grips to avoid torsion of the sample. The neat resin compression samples were tested using two parallel plates. A calibration test was carried out by bringing the plates together and loading the frame to estimate the instrument compliance.

Higher strain rates were achieved with a servo-hydraulic machine. This type of machines has a larger actuation speed range, covering speeds from 10^{-3} to 10^3 mm/s. However, in the higher speeds range (10^2 - 10^3 mm/s) they present ringing issues due to inertia effects that may affect the accuracy of the results. The servo-hydraulic mechanical tester used was a Gleeble 3800 of Dynamic Systems (USA) equipped with a 250 kN load cell. The set-up for the compression tests consisted of two self-aligning plates, so loads were transferred to the sample by contact. A test with the two plates in contact was performed prior to testing the samples to have an estimate of the frame compliance because strains were computed from bench displacement and sample length. In the in-plane shear tests, custom-built mechanical wedge grips were used to fix the sample.

The highest strain rate regime was covered with a split-Hopkinson pressure bar (SHPB), with which stroke rates in the order of 10^4 mm/s can be achieved. Two SHPB set-ups were used. A compression SHPB set-up was used for the composite transverse compression and neat resin compression samples and a tensile SHPB set-up was used for the in-plane shear specimens. Figure 2.4 shows schematics of both SHPB set-ups. Loads are transmitted by contact in the compression bars, but load transfer in the tensile bars required more preparation. Custom-built grips were made with a notch where the sample was inserted and glued (Loctite EA 9466). These grips were screwed to the incident and transmitted bars. In both set-ups, the bars are made of Inconel and the striker bar is propelled by a gas gun. The incident and transmitted bars are both instrumented with strain gauges in Wheatstone bridge configuration to measure the strain pulses. A sheet of thick paper is placed in the contact between the striker and incident bar for reducing ringing and for pulse shaping.

The analysis of the SHPB experiments followed the 1D wave propagation theory,

Figure 2.4: Split-Hopkinson pressure bar (SHPB) test set-ups used for tensile and compression testing.

which is based on several assumptions: (1) the bars are made of homogeneous and isotropic materials and have an uniform cross-section; (2) the deformation of the bars is linear elastic; (3) the specimen must be in a state of dynamic stress equilibrium; among others (Körber, 2010). This analysis makes use of three strain pulses obtained during the test. The incident wave, ϵ_i , is recorded by the strain gauge in the incident bar. The incident wave reaches the bar end where the sample is located and splits into the reflected wave, ϵ_r , and the transmitted wave, ϵ_t , due to the impedance mismatch between the bar and the sample. The reflected wave travels backwards in the incident bar and it is recorded by the same strain gauge that records the incident wave. The transmitted wave travels through the specimen and the transmitted bar and it is recorded by the strain gauge in the transmitted bar. The first step in the analysis is the shifting of the signals to the same start time, which is done manually in this work. Then, by applying the D'Alembert solution to the 1D wave equation (more details in the work of Körber (2010)), one can derive the following expression for the engineering strain:

$$\epsilon_{eng} = \int_0^t \dot{\epsilon}_{eng} dt = \int_0^t \frac{V_a - V_b}{l} dt = \int_0^t \frac{c_{bar}(\epsilon_i - \epsilon_r - \epsilon_t)}{l} dt \quad (2.1)$$

V_a and V_b are the velocities at both sides of the sample. c_{bar} is the sound speed in the bars ($c_{bar} = \sqrt{E_{bar}/\rho_{bar}}$), being E_{bar} and ρ_{bar} the elastic modulus and density of the bars material, respectively. ϵ_i , ϵ_r and ϵ_t are the incident, reflected and transmitted strain pulses, respectively. l is the specimen length.

2.3. Mechanical characterization over a wide range of strain rates

The engineering stress (or true if the real cross-sectional area is used) is computed from the forces at both sides of the specimen:

$$F_a = A_{bar}E_{bar}(\epsilon_i + \epsilon_r) \quad F_b = A_{bar}E_{bar}\epsilon_t \quad (2.2)$$

The force signal from the side in contact with the transmitted bar (F_b) is often used for the calculation of stress because it is less noisy. This is because the sample damps part of the oscillations (Körber, 2010). Compliance of the SHPB test with the dynamic equilibrium assumption requires that the forces at both sides of the specimen are equal. This was checked and both forces overlap in all tests performed in this work, being the force at the incident side much noisier. There is a first period, corresponding to the elastic part, where both forces did not overlap. Therefore, the elastic modulus results should be treated with care.

	Gleeble (highest speed case)			SHPB		
	Resin compression	IPS	TC	Resin compression	IPS	TC
Travel time of the stress wave (back and forth) [ms]	0.10	0.10	0.11	0.01	0.01	0.02
Number of reflections in elastic region	10	24	45	2	3	2
Number of reflections during entire test	43	48	91	13	5	4

Table 2.3: Estimations to check dynamic equilibrium in the two highest speed cases of the three test set-ups. The travel time is estimated from the sample and fixture length (between the sample and load cell) in the Gleeble case and only from the sample length in the SHPB case.

Furthermore, the dynamic equilibrium assumption was assessed by analysing the characteristic times of stress wave travel and the number of stress wave reflection during the test. Table 2.3 contains estimation of the travel time of the stress wave in each of the test set-ups and for the two highest speed conditions. The estimated time in the highest speed case of the Gleeble takes into account the length of the fixed fixture located between the sample and the load cell plus the sample length. The length considered for the SHPB is the sample length. According to SAE-International, the dynamic equilibrium is reached when 10-15 reflected stress waves pass through the

2. Materials and macroscale characterization

specimen and fixture during the period of elastic deformation. Table 2.3 shows that this condition is fulfilled in the Gleeble tests; although, the resin compression test is just in the limit of the recommended range. The estimations for the SHPB tests are far off the recommended 10-15 reflections. However, many authors claim that dynamic equilibrium in SHPB tests is achieved after 3-4 reflections of the stress waves (Siviour and Jordan, 2016; Gama et al., 2004; Chen et al., 2002). In this case, only the IPS complies with this condition for the elastic regime, and all tests comply if the entire test duration is to be considered. This means that the plastic properties derived from the tests can be trusted, but the elastic modulus results should be treated with care.

Several videoextensometry systems were used to capture video and perform digital image correlation (DIC) measurements. Table 2.4 summarizes the different cameras and optics used in the tests. Images were processed with a commercial DIC algorithm of Correlated Solutions (USA) to extract the displacement and strain fields in the sample surface.

Mechanical test set-up	Camera	Optics
Instron 5966	Stingray F-504 of Allied Vision	Pentax C7528-M f2.8/75mm
Gleeble 3800	Phantom VEO 1310 of Vision Research	AF Zoom-Nikkor 24-85mm f/2.8-4D IF
Tension/compression SHPB	Phantom VEO 710 of Vision Research	Micro-Nikkor 105 mm f/2.8

Table 2.4: Videoextensometry systems used in the experiments.

DMA testing was employed to characterize the elastic modulus of the neat resin samples over a wide range of strain rates. DMA tests were performed in a Q800 of TA Instruments (USA) using the single cantilever beam configuration with both edges clamped. The test was displacement-controlled, with force measurement at the same point of displacement application. Tests were performed inside a temperature-controlled chamber. Temperatures above room temperature were reached using heaters inside the chamber, and temperatures below room temperature using liquid nitrogen.

2.3. Mechanical characterization over a wide range of strain rates

2.3.2 Dynamic Mechanical Analysis on neat resin coupons

A series of isothermal frequency sweeps were carried out to characterize the viscoelastic behaviour of the resins over a wide range of strain rates. Prisms of 17.5 x 10 x 2.8 mm were used for the test. 17.5 mm is the length in between clamps, not the total length of the specimens. Tests were performed under a displacement-controlled scheme by application of an oscillation of 15 μm amplitude. For each isotherm, the sample was oscillated at ten different frequencies (1 to 30 Hz). Isotherms from -80 to 250 °C were tested, with 10 °C steps up to 150 °C and 5 °C for higher temperatures. A standard steel calibration sample was used to determine instrument compliance (0.945 $\mu\text{m}/\text{N}$) and clamp mass (29.7 mg).

The complex elastic modulus was obtained as a result of the data reduction process in the DMA test. The complex modulus (E_{cpx}) can be divided into an elastic part (storage modulus, $E_{st} = E_{cpx}\cos\phi$) and a viscous part (loss modulus, $E_{ls} = E_{cpx}\sin\phi$). The storage and loss modulus are thus defined by the phase delay between the displacement and force signals, ϕ . The equation used for determination of elastic modulus depends on the loading and clamping conditions. For the single cantilever tests, and applying Timoshenko's beam theory, the complex modulus is defined as:

$$E_{cpx} = f_{inst}L\frac{P_0}{w_0}\left[\frac{2(1+\nu)}{\kappa ab} + \frac{L^2}{ab^3}\right] \quad (2.3)$$

P_0 and w_0 are load and displacement amplitudes, respectively. L is the length in between clamps and a and b are the dimensions of the sample cross-section. κ is the Timoshenko's shear coefficient and f_{inst} is a correction factor given by the instrument manufacturer.

The characterization of modulus over a wide range of frequencies was carried out making use of the Time Temperature Superposition Principle (TTSP). This is a method to characterize the viscoelastic behaviour of polymers at very low or very high frequencies from the behaviour at standard frequencies (0.1-100 Hz) at different temperatures. This method can be applied only to thermorheologically simple materials, i.e. materials in which all contributing relaxation mechanisms have the same temperature dependence (Dealy and Plazek, 2009). There is no standard procedure to apply the TTSP. In general, the idea is to pick a reference isotherm (reference temperature) and shift the rest of isotherms to build a master curve.

In this case, a method based on least-squares to minimize the distance between isotherms was used.

DMA testing combined with the application of the TTSP provided a distribution of storage and loss moduli over a wide frequency range. However, the data necessary for simulation needed to be expressed as a function of strain rate. To this end, the DMA frequency was converted into an average strain rate by assuming that half of the sinusoidal displacement scheme had a triangular shape, thus the average strain rate ($\dot{\epsilon}_{avg} = \Delta\epsilon/\Delta t$) can be computed as $4f\epsilon_{max}$, being ϵ_{max} the strain in the specimen at maximum amplitude (Mulliken and Boyce, 2006; Siviour and Jordan, 2016). Furthermore, since the strain in a single cantilever specimen is not constant along the sample, the maximum strain in the specimen was used instead as a representative value. McAninch et al. (2015) also carried out tests with the Q800 tester of TA Instruments and gave an expression (see equation 2.4) to compute the maximum strain in the single cantilever configuration, which was used in this study to estimate ϵ_{max} :

$$\epsilon_{max} = \frac{3 \cdot w_0 \cdot t \cdot f_{inst}}{L^2[1 + (12/5)(1 + \nu)(t/L)^2]} \quad (2.4)$$

L and t are the sample length and thickness, respectively. w_0 is the displacement amplitude and f_{inst} is the correction factor given by the instrument manufacturer.

Upon application of these calculations to estimate the elastic modulus as a function of strain rate from the storage modulus as a function of frequency, the resulting value was about 1 GPa lower than that obtained from the standard dogbone tensile tests (see section 2.2). Previous studies have also found discrepancies and attributed it to experimental factors like the sample geometry, clamp type, loading configuration, oscillation amplitude, among others. McAninch et al. (2015) showed that the elastic modulus is highly dependent on span-to-thickness ratio and recommended a value around 10, which is actually higher than the ratio of the samples tested in this study. Deng et al. (2007) compared the results of DMA testing (as a function of temperature) with three loading configurations (single and double cantilever, and 3-point bending) and found significant discrepancies among the three set-ups and with conventional tensile test data. However, the trend with temperature was similar in all cases. Therefore, they proposed shifting of the DMA data taking the room temperature as reference to make it coincide with the tensile test data. The same approach was adopted in the present study. The value of elastic modulus ob-

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Figure 2.5: Elastic modulus estimated from DMA testing, applying the TTSP to cover a wide range of strain rates, scaled with the results of tensile tests with dogbone specimens.

tained from tensile testing (4.23 GPa at $4.3 \cdot 10^{-4} \text{s}^{-1}$ for the 8552 and 3.79 GPa at $4.9 \cdot 10^{-4} \text{s}^{-1}$ for the M91) was used as reference to shift the estimated elastic modulus distribution obtained with DMA. Figure 2.5 presents the results of the estimations.

2.3.3 Compression tests on neat resin coupons

End-loading 10x5x5 mm prism specimens were employed for the neat resin compression tests. The 2:1 aspect ratio is recommended in the ASTM D695 standard for compressive properties of plastics. It ensures a state of uniaxial stress along the material. Samples were extracted from neat resin panels by CNC machining, paying special attention to the parallelism of the two surfaces that are in contact with the compression plates in the test.

Low speed testing was performed in the electromechanical frame Instron 5966. Intermediate strain rate tests were carried out in the hydraulic frame Gleeble 3800. The highest strain rate case was tested in the compression SHPB set-up. Table 2.5 compiles the most relevant experimental parameters of the tests. In the SHPB tests, a high speed camera was employed to study the failure mechanism of the samples.

True stress-strain curves in the low and intermediate strain rate cases were obtained from the measured load and input displacement. A correction for instrument compliance was applied to the displacement values: $d_{corrected} = d_{initial} - c_{frame}P$, being P the measured load and c_{frame} the estimated instrument compliance ($2.4 \cdot 10^{-5}$

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Test device	Test speed [mm/s]	Acq. Freq. [Hz]
Instron	0.07	10
	0.7	100
	6.7	500
Gleeble	100	5000
	200	10^4
	1000	10^4
SHPB	$\sim 1.6 \cdot 10^4$	10^7

Table 2.5: Experimental parameters of the neat resin compression tests for mechanical characterization over wide range of strain rates. Acq. freq. means acquisition frequency.

mm/N for the Instron and $1.2 \cdot 10^{-5}$ mm/N for the Gleeble). Force and strain in the SHPB tests were obtained from 1D wave analysis (see section 2.3.1). All stress-strain curves were modified to eliminate the toe region at the start of the curve, before the elastic regime. Guidelines from the ASTM D695 were followed. The tangent stiffness (derivative of stress with respect to strain) of the low and intermediate strain rate cases did not exhibit a clear constant region, so the toe was corrected by building the tangent to the strain point where the tangent stiffness is maximum. The tangent stiffness did show a constant value in the SHPB tests in the 1-2 % range of deformation, so the toe was corrected by performing a linear fit in this range.

Figure 2.6 shows the stress-strain curves for the two neat resin materials and all test cases. The two resins exhibit a significant strain rate sensitive behaviour in compression, being the M91 resin slightly softer than the 8552. The M91 resin has a similar formulation than the 8552 (see section 2.1), thus one would expect a similar mechanical performance. However, the M91 resin contains soft thermoplastic particles that overall decrease the flow stress. This could be also the reason for the larger strain to failure obtained in the M91 resin. The failure strain is about 5 to 10% higher. The curves from the SHPB tests present an early drop in stress at about 20% of deformation. This is caused by the end of the strain pulse and not by the failure of the sample. Figure 2.7 shows images from the end of the first three strain pulses in the SHPB test. Failure in the 8552 samples happened near the end of the second strain pulse, i.e. at a deformation of about 35-40%. Failure in the M91 samples took place at a higher deformation, near the end of the third strain

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Figure 2.6: Stress-strain curves of the neat resin compression tests of the 8552 (left) and the M91 (right).

pulse (45-50% deformation).

Overall, the results show no change in the strain to failure with strain rate. The predominant failure mechanism in both materials and all strain rate cases is the growth of a longitudinal crack that leads to a drop in the stress during the post-yield softening. A few specimens in the low speed cases exhibited pronounced hardening, hence leading to a higher strain to failure. However, the stress-strain for such high strains is likely to have lost the uniaxial condition due to barreling of the samples, which is in fact shown in the images of figure 2.7. The occurrence of post-yield hardening is typical in compression tests of polymers and most likely the rest of tested samples would show it if they had not prematurely failed. Interestingly, Naya Montáns (2017) showed that the size of the specimen may have an effect on the post-yield behaviour of the resin. They examined the compressive properties of two type of prismatic specimens, having the same 2:1 aspect ratio, but different size. Most of the specimens of smaller size showed a pronounced hardening before failure, while the 10x5x5 specimens, as in this work, failed in a brittle manner before hardening. Other authors have also shown this type of size-dependent post-yield behaviour. Fard et al. (2012) showed that cubic specimens experienced a strong

Figure 2.7: Failure analysis in the SHPB compression tests of the two resins. Size of the specimen surface shown in the image: 10x5 mm.

hardening, while prismatic specimens failed within the softening region.

The data reduction process was carried out by computing the elastic modulus and compression yield stress, following guidelines from the ASTM D695 standard. In the low and intermediate strain rate cases, the tangent stiffness did not exhibit a constant value, so the elastic modulus was calculated as the maximum of the tangent stiffness. In the SHPB tests, the tangent stiffness was constant in the strain range from 1 to 2 %. The elastic modulus was computed as the average of the tangent stiffness in that range. The compression yield stress was obtained at the point where the tangent stiffness goes to zero (first point of the stress-strain curve where there is an increase in strain without change in stress). This is the definition given in the ASTM D695 standard.

Figure 2.8 presents the two material properties as a function of strain rate. The elastic modulus values at the three lower strain rates do not show a significant effect of the strain rate. The elastic modulus is calculated at the first stages of deformation, a period where the electromechanical instrument could be still accelerating from the rest state. In this case, there would not be a significant difference among the strain rates of the three cases, explaining why the elastic modulus did not change. In the other hand, the elastic modulus for the higher strain rates tested in the hydraulic frame and the SHPB did show an increase with strain rate. The SHPB values should be treated with care because the sample was likely not in equilibrium during the elastic regime (see table 2.3). The elastic modulus values of the highest speed case of the hydraulic frame were excluded because they were notably higher than the rest

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Figure 2.8: Data reduction results of the neat resin compression tests of the 8552 and the M91.

of values. There could be a number of reasons for this. First, table 2.3 shows that the number of reflections of the stress waves in this case was about 10, when the SAE-International standard recommends a minimum of 10-15 reflections to consider the test in dynamic equilibrium. Second, the acquisition frequency used (see table 2.5) may have not been enough as it led only to a few points within the elastic regime. Overall, the elastic modulus values obtained in the compression tests were lower than the values obtained in the standard tensile tests on neat resin coupons presented in section 2.2. The ASTM D695 standard actually recommends specimens with an aspect ratio of at least 4:1 when the focus of the test is on elastic modulus calculation. Therefore, the specimens tested in this work were not optimized for the determination of the elastic modulus, but for the determination of the neat resin flow stress, which was required for the validation of the micromechanical testing techniques developed in this work.

The compression yield stress values exhibit a significant strain rate sensitivity. Furthermore, a different trend with strain rate is observed for the low speed and intermediate-high speed cases. This type of bilinear behaviour has been reported before in epoxy resins (Jordan et al., 2008). It is attributed to a change in the molecular mechanism driving deformation. The mechanical behaviour at high strain rates is dominated by the molecular β -transition, while the behaviour at low strain rates is dominated by the α -transition, which is also responsible for the glass transition process upon heating of the material.

2.3.4 Transverse compression tests on composite coupons

End-loading 20x10x5 mm prism samples were used in the transverse compression tests. They were extracted from 5 mm thick laminates with $[90]_{20}$ lay-up. The samples were prepared by CNC machining, ensuring that the two 10x5 mm surfaces for load introduction are parallel and smooth to ensure the proper loading of the specimen. The tests were carried out only in the IM7-8552 composite system. The behaviour in transverse compression is dominated by the matrix. Since the M91 and 8552 resin formulations are very similar (see section 2.1), a similar mechanical response in transverse compression is expected. The main difference between both systems is in the interleaf region, where the thermoplastic particles are located in the IM7-M91 composite.

Low and medium speed tests were carried out in the servo-hydraulic system Gleeble 3800 and the highest speed case was carried out in the compression SHPB set-up. Table 2.6 summarizes the experimental conditions of each of the test cases. Low and high speed video acquisition was employed to perform DIC and to analyse the initiation and propagation of damage in the specimen. A mirror was used to record images of the front and the side of the specimen at the same time.

Mechanical				Video		DIC
Test device	Test speed [mm/s]	Acq. Freq. [Hz]	ROI size [pixel]	fps	Exp. Time [μ s]	Subset/step size [pixel]
Gleeble	0.2	200	180x90	100	50	21/5
	20	$2 \cdot 10^4$	180x90	10^4	50	21/5
	1000	$5 \cdot 10^4$	180x90	$6 \cdot 10^4$	3	21/5
SHPB	$\sim 1.5 \cdot 10^4$	10^7	120x60	$2.4 \cdot 10^5$	3	13/3

Table 2.6: Experimental parameters of the TC tests for mechanical characterization over wide range of strain rates. Acq. freq. means acquisition frequency and Exp. Time is exposure time.

Unlike the neat resin compression tests, engineering and not true stress-strain curves were determined in the composite transverse compression test because the deformation did not exceed 5%. The stress-strain curves are presented in figure 2.9-a. In the tests using the hydraulic frame, stress and strains were calculated using

2.3. Mechanical characterization over a wide range of strain rates

Figure 2.9: (a) Stress-strain curves of the TC tests over a wide range of strain rates of the IM7-8552 composite system. (b) Original stress-strain curves obtained in the SHPB tests showing high degree of oscillation and smooth curves after filtering.

the measured load and input displacement, respectively. A correction for instrument compliance was applied to the displacement values: $d_{corrected} = d_{initial} - c_{frame}P$, being P the measured load and c_{frame} the estimated instrument compliance ($1.2 \cdot 10^{-5} \text{ mm/N}$). Force and strain in the SHPB tests were obtained from 1D wave analysis (see section 2.3.1). In addition, the stress-strain curves of the SHPB tests required signal treatment to smooth the high degree of oscillation observed in the raw data (see figure 2.9-b). This oscillation is an indication of the specimen not being in dynamic equilibrium during the test (Körber, 2010), which is not surprising given the estimations in table 2.3. The number of reflections during the entire test is around 4, which is just above the 3-reflections rule of thumb recommended in literature. The neat resin compression tests also present certain oscillatory response during the elastic part of the deformation (see figure 2.6), in line with the estimations of table 2.3. However, the oscillations damped out during the plastic regime because there were more than 10 reflections of stress waves, unlike the composite transverse compression test. All stress-strain curves were corrected to eliminate the toe region at the start of the curve, following the recommendations of the ASTM D695 standard. The toe was corrected by extending the linear fit in the strain range where the tangent stiffness is constant (around 1-1.5% of deformation) to the point of zero load.

The stress-strain curves of figure 2.9-a exhibit a clear dependency of the trans-

Figure 2.10: Representative images of the specimen side (20x5 mm) depicting the evolution of fracture angle in TC tests over a wide range of strain rates.

verse compression strength with the strain rate. There seems to be a higher degree of nonlinear deformation for the low speed tests, while the high speed tests exhibit a near linear elastic behaviour up to failure. This same trend has been observed by Koerber et al. (2010) and Schaefer et al. (2014), who also tested the IM7-8552 composite in transverse compression configuration. The results show that the strain to failure is not significantly affected by the change in strain rate, which is in line too with the observations by other previous works (Körber, 2010; Hsiao and Daniel, 1998).

The failure mechanism did not change with strain rate. All transverse compression samples failed by the propagation of cracks parallel to the fibres. The fracture planes are inclined with respect to the loading direction (see figure 2.10), as expected in transverse compression specimens (González and LLorca, 2007a). This type of failure is the result of the development of two mechanisms at the microscale: localization of plastic strain along a shear band in the resin matrix and nucleation of interface cracks in the fibre-matrix interface. The prevalence of one mechanism over the other will depend on the interface-matrix relative strength. If the interface is significantly stronger than the matrix, then failure will take place by the development of a plastic shear band in the matrix, which forms an angle, α , with the plane perpendicular to the loading direction. This angle is related to the pressure sensitivity of the resin, and it is typically 50-60° for epoxies (González and LLorca, 2007a). Weak interfaces lead to early nucleation of damage by interface decohesion even when the matrix is still experiencing elastic deformation. The interface cracks promote the formation of plastic shear bands, which then link with other interface cracks, leading to a failure by the coalescence of interface cracks. In this case, the fracture angle is not defined by the matrix properties, but rather by the fibre distribution in the microstructure. Since the obtained angles in figure 2.10 are within the

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expected values for the epoxies, failure most probably happened by development of a shear plastic band, so the interface has either similar or higher strength than the matrix. Furthermore, figure 2.10 shows that the angle of the fracture plane does not change with strain rate, an observation that is in line with the conclusions of other researchers (Wiegand, 2009; Vural and Ravichandran, 2004). This means that the pressure sensitivity of the resin is not affected by the strain rate.

A data reduction process was applied to obtain elastic modulus and compressive strength. Guidelines from the ASTM D695 standard were followed. The elastic modulus was calculated as the average of the tangent stiffness (derivative of stress with respect to strain) in the range of strains where it is nearly constant, which was found to be 1-1.5%. The compressive strength was obtained from the maximum test load. Both material properties exhibit a significant increase with strain rate, which is a consequence of the matrix dominating the failure mechanism. The elastic modulus is slightly lower than the one obtained in transverse compression tests of other authors who also studied the IM7-8552 system (Körber, 2010; Schaefer et al., 2014). However, the trend with strain rate was similar. The values of compressive strength and the trend with strain rate are in line too with the results of Körber (2010) and (Schaefer et al., 2014).

Test conditions	Elastic modulus [GPa]	Compressive strength [MPa]
0.2 mm/s ($\epsilon_{avg} \sim 9.2 \cdot 10^{-3} s^{-1}$)	8.1 ± 0.2	210 ± 15
20 mm/s ($\epsilon_{avg} \sim 6.8 \cdot 10^{-1} s^{-1}$)	8.6 ± 0.3	243 ± 24
1000 mm/s ($\epsilon_{avg} \sim 4.1 \cdot 10^0 s^{-1}$)	8.8 ± 0.4	260 ± 14
$\sim 1.5 \cdot 10^4$ mm/s ($\epsilon_{avg} \sim 6.9 \cdot 10^2 s^{-1}$)	10.5 ± 0.3	321 ± 26

Table 2.7: Data reduction results of the TC tests over a wide range of strain rates.

2.3.5 In-plane shear tests on composite coupons

There are several types of tests for studying the in-plane shear behaviour of fibre-reinforced polymer composites. One of the most popular configurations, recommended by the ASTM D3518 standard, is the tensile test of a ± 45 laminate. This type of samples present a uniform shear strain distribution along the gauge length and promote stable damage propagation. Samples used for the in-plane shear test were dogbone shaped, with dimensions optimized for SHPB testing. They were

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Figure 2.11: Geometry of the IPS samples for mechanical characterization over a wide range of strain rates.

extracted using CNC machining from 2 mm thick laminates with $[\pm 45]_{2s}$ lay-up. Figure 2.11 shows the dimensions and shape of the sample. Glass fibre-epoxy tabs were glued (Loctite EA 9466) for the specimens tested under low and intermediate strain rates. Steel inserts were used to fix the IPS specimens to the bars in the SHPB set-up. One end of the insert is screwed to the bar, while the other has a notch where the sample has been inserted and glued.

Low speed testing was performed in the electromechanical frame Instron 5966. Intermediate strain rate cases were tested in the hydraulic frame Gleeble 3800. The highest strain rate case was tested in the tensile SHPB set-up. Table 2.8 compiles the most relevant experimental parameters of the tests. Low and high speed cameras were used to gather data for DIC analysis.

Mechanical				Video		DIC
Test device	Test speed [mm/s]	Acq. Freq. [Hz]	ROI size [pixel]	fps	Exp. Time [μ s]	Subset/step size [pixel]
Instron	0.02	20	190x400	10	$< 10^5$	21/5
Gleeble	2	2000	85x40	2000	3	9/2
	200	$5 \cdot 10^4$	85x40	$6 \cdot 10^4$	3	9/2
SHPB	$\sim 2 \cdot 10^4$	$2.5 \cdot 10^7$	110x50	$3 \cdot 10^5$	2.3	13/3

Table 2.8: Experimental parameters of the IPS tests for characterization over wide range of strain rates. Acq. freq. means acquisition frequency and Exp. Time is exposure time.

Shear stress-strain curves were obtained following the data analysis procedures

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in the ASTM D3518 standard:

$$\tau_{12} = \frac{P}{2A_0} \quad \gamma_{12} = \epsilon_x - \epsilon_y \quad (2.5)$$

P is the force. It was measured using load cells in the electromechanical and hydraulic frames, and derived from the 1D wave analysis in the SHPB set-up. A_0 is the nominal cross-sectional area of the specimen in the gauge length. ϵ_x is the strain measured along the loading axis and ϵ_y is the strain in the direction perpendicular to the loading axis. The strains in all cases were obtained from DIC analysis of the images obtained during test. The force and strain signals were manually synchronized in time.

Figure 2.12 presents the shear stress-strain curves of the two composite materials systems under study. The strain distributions in the area used for DIC analysis for three different strain points is also shown to give insight on the failure mechanism of the samples. The curves exhibit the typical mechanical response of composite samples deformed in shear. A first linear elastic region, defined by the shear modulus of the matrix and the fibre. Then, a nonlinear region in which the continuous degradation of the tangent stiffness is caused by the initiation and propagation of damage in the fibre-matrix interface and by plastic deformation and damage of the matrix. The dominance of one mechanism over the other depends on the relative shear strength of the fibre-matrix interface and the matrix (Totry et al., 2010). At some point during the nonlinear deformation, the stress reaches a near-plateau caused by the saturation of the micro-cracking within the plies. The stored and external energies are in equilibrium with the energy necessary for fracture during this stage. During this plateau region, the fibres tend to align with the loading direction. There is a rotation of the fibres and the relative angle between fibres in adjacent plies decreases linearly with strain (Sket et al., 2014). Final failure of IPS samples is driven by the appearance of delaminations and the complete separation of the specimen in two parts.

The IM7-M91 composite exhibits a superior in-plane shear performance, which is materialized in the larger strain to failure, i. e. the material is tougher than the IM7-8552 composite. The results are in line with the behaviour of the material under impact, exemplified in figure 2.1. Riezzo et al. (2019) also studied the difference in mechanical performance of the IM7-8552 and the IM7-M91 composite systems. They showed that the interlaminar fracture toughness in Mode I is higher

Figure 2.12: Stress-strain curves of the IPS tests over a wide range of strain rates for the two studied composite systems: IM7-8552 and IM7-M91. Captions of the DIC analysis are also included for failure analysis (images correspond to the gauge area, size $\sim 10 \times 5$ mm).

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in the IM7-M91 material and attributed this to the development of intraply cracking caused by the cracks being deflected from the interlayer due to the toughening effect of the thermoplastic particles. In addition, optical inspection of the fracture surface revealed that part of the surface in the IM7-M91 material exhibited the characteristics of a ductile fracture propagating through the interleaf, combined with areas where the crack clearly propagated along the fibre-matrix interface. However, the fracture surface of the IM7-8552 consisted entirely of cracks propagating along the fibre-matrix interface. Nguyen et al. (2019) also arrived to similar conclusions in their study of a composite that contains thermoplastic particles in the interleaf region for toughening, but they focused on the in-plane shear behaviour. They found that the thermoplastic particles delay the onset of delamination and promote intraply cracking instead. Therefore, the results of figure 2.12 also seem to indicate that the thermoplastic particles in the IM7-M91 composite delay the onset of delamination, leading to a much higher strain to failure than the IM7-8552 composite. Furthermore, the DIC analysis shows an early strain localization, for a strain of 5%, in the IM7-8552 system, while the strain distribution remains uniform for larger strains in the IM7-M91 composite.

The test results show that the shear strength of the laminate increases with strain rate, while the strain to failure decreases with strain rate. Cui et al. (2016) obtained the same trend in their study of the IM7-8552 system. They concluded that the rate dependence of the IPS test is dominated by the rate dependency of the resin matrix. Their study of the fracture surfaces suggested that failure in the quasistatic tests initiated by fibre-matrix interface debonding that then led to delaminations and final failure. However, the fracture surface of the specimens tested at high strain rate mainly showed failure of the matrix, rather than fibre-matrix debonding. This result indicates that the shear strength of the fibre-matrix interface becomes larger than the shear strength of the matrix with the increase of strain rate, hence leading to matrix shear dominated failure at high speeds and fibre-matrix dominated failure at low speeds. Taniguchi et al. (2007) arrived to a similar conclusion in their study of the T700S-2500 composite material. By looking at the fracture surfaces of the specimens, they determined that the cracks propagated primarily along the fibre-matrix interface in the low speed cases, while the cracks propagated along both the matrix and the fibre-matrix interface in the dynamic case.

A data reduction procedure was carried out to determine two material properties: shear modulus and shear strength. The shear modulus was obtained as the

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	Test conditions	IM7-8552	IM7-M91
Shear modulus [GPa]	0.02 mm/s	5.3 ± 0.4	5.0 ± 0.02
	2 mm/s	4.2 ± 0.5	4.3 ± 0.1
	200 mm/s	4.7 ± 1.3	3.8 ± 1.0
	$\sim 10^4$ mm/s	5.7 ± 1.0	5.3 ± 0.7
Shear Strength [MPa]	0.02 mm/s	78 ± 1	75 ± 1
	2 mm/s	95 ± 2	95 ± 3
	200 mm/s	109 ± 4	111 ± 2
	$\sim 10^4$ mm/s	125 ± 7	135 ± 3

Table 2.9: Data reduction results of the IPS tests over a wide range of strain rates.

average tangent stiffness in the strain interval where it was nearly constant, which is from 0.3% to 0.6%. The shear strength is computed as the maximum shear stress during the test. The results are compiled in table 2.9. Overall, the elastic modulus increases with strain rate; although, the values of the two tests performed in the hydraulic system (2 mm/s and 200 mm/s cases) are lower than the value obtained in the lowest speed case. These values should be treated with care because the force and strain signals were synchronized manually and this could have induced errors in the elastic modulus values, as shown by Spronk et al. (2018). The shear strength shows the clear rate dependency of the material, as expected considering that shear deformation is dominated by the matrix mechanical behaviour.

Modification of a pendulum-based nanoindentation device for performing high rate nanoindentation

Impact nanoindentation (or nano-impact) is a technique for characterizing the mechanical behaviour of materials at high strain rates at the microscale (see section 1.4). However, its use has been limited so far due to the lack of appropriate methods to determine the dynamic hardness under impact conditions. The main limitation up to date has been the determination of the true applied force due to considerable inertia effects. This chapter describes a modification made on a commercial instrument to overcome this limitation. The test device was instrumented with force-sensing capability by means of a piezoelectric load cell. This is explained in section 3.2. The new methodology was evaluated on six materials covering a wide spectrum of mechanical behaviour. The results of this analysis are presented in section 3.3. To end with this chapter, in section 3.4 the methodology used traditionally for analysis of nano-impact test was assessed and compared with the new proposed methodology. The work shows that the approach used so far yields significant errors on the determination of dynamic hardness, and identifies the main sources of error with the aid of finite element simulations.

3.1 Introduction

3.1.1 Instrumented indentation

Hardness testing based on indentation is a widely used technique for mechanical characterization of materials because of its simplicity and fast speed of testing. However, unravelling the deformation mechanisms that take place in the material during indentation is rather complex and the technique has been the subject of extensive research for more than 100 years. In an indentation hardness test, a tip of a very hard material (normally diamond) with a controlled geometry is brought into contact with the material surface and penetrates as a result of an applied force or displacement, leaving a permanent residual mark on the material surface that can be examined with a microscope. Hardness thus measures the resistance of a material surface to be deformed permanently under the action of a contact pressure exerted by an ideally-rigid body.

Indentation hardness testing became popular in the early 1900s when the first commercial indentation machines became available. Its high demand was driven by the need of cheap and fast methods for quality testing of mass-produced items from heavy industry (Walley, 2012). The Brinell hardness test was the most popular test at the time. In this test, the material surface is indented with a ball of a hard material under a constant load. Other methods quickly appeared, all based on the same principle, but changing the indenter shape and material: the Rockwell test, which uses either a spherical or a conical probe (made of hardened steel or diamond), Vickers indentation in which the indenter has the shape of a four-sided pyramid, and the Knoop test that also employs a four-sided pyramid, in this case elongated in one direction having a 1:7 ratio between the two diagonals. The elongated shape of Knoop indenters minimizes elastic recovery during unloading and reduces cracking in brittle materials. One of the major drawbacks of the Brinell test is the load dependency of the hardness, which is caused by the variable ratio of depth to radius of the circle of contact, i. e. a spherical indentation is not self-similar. Instead, conical and pyramidal indentation presents self-similarity, hence hardness (defined as the mean contact pressure) is depth-independent because the ratio of depth to contact diagonal is constant throughout the indentation (Tabor, 1951; Fischer-Cripps, 2011). Nevertheless, spherical indentation is useful for studying elastic deformation and the elastic-to-plastic transition as the sharp shape of pyramidal and conical indenters leads to an elastoplastic indentation at the onset of contact. Another issue

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Figure 3.1: Typical force-depth curve in a pyramidal or conical indentation. In the right, schematics of the indenter-surface contact that results into material pile-up or sink-in, which is a function of the material properties and the indenter shape (Cheng and Cheng, 1998).

with four-sided pyramidal indenters is that it is difficult to ensure that all the four sides meet at one point. This is particularly critical for very small indentations. For this reason, three-sided pyramidal indenters are preferred when reducing the scale of indentation. The Berkovich indenter is a three-sided pyramid that has the same area-to-depth ratio than the Vickers indenter.

A major advance in indentation hardness testing came with the development of instrumented indentation, in which load and depth are monitored throughout the entire indentation cycle. The first instrumented indenters appeared in the late 1970s and early 1980s (Bulychev et al., 1975; Fröhlich et al., 1977; Newey et al., 1982; Loubet et al., 1984; Pethica, 1982). In addition, many of these instruments could accurately perform indentations with nanometre resolution in depth and micronewton resolution in load, thus this opened the field of nanoindentation. Unlike traditional hardness testing, instrumented indentation avoids the need of optical inspection of the indent after the test (provided the indenter area function is known), which is specially challenging for sub-micron indentations. Figure 3.1 shows the typical load-depth curve of an instrumented indentation test using a conical indenter. In the right, a schematic is shown of the two types of scenarios in an indentation. If the material piles up around the indenter during the test, the calculated contact area through the measured depth will be underestimated. The opposite happens

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when the material sinks in around the indenter, the contact area is overestimated then. The interaction of the material surface with the indenter is a function of the material parameters and the shape of the indenter (Cheng and Cheng, 1998).

A hardness value is obtained as output of an indentation hardness test. The value generally differs from method to method. There are several definitions of hardness, but the most widely used nowadays in the nanoindentation community, and that is recommended in the ISO14577 (2015) standard for instrumented indentation is given by:

$$H = \frac{P}{A_c} \quad (3.1)$$

Hardness is defined as a mean contact pressure, being P the applied load and A_c the projected contact area at that load. Traditionally, the projected contact area was estimated from optical observation of the indentation after the test. This area corresponds to the residual indent when no load is applied. However, as shown figure 3.1, the residual area after load removal may differ significantly from the contact area under load due to sink-in or pile-up phenomena. Instrumented indentation enabled the determination of the entire load and displacement history and this allowed the estimation of the contact area. Doerner and Nix (1986) proposed an estimation by assuming that the contact area during the first stages of the unloading remains constant, hence the unloading is linear. They estimated the plastic or contact depth as $h_c = h_{max} - P_{max}/S$, being S the slope of the tangent to the unloading curve in the point of maximum load. Oliver and Pharr (1992) later improved the methodology by coming up with their well known relation:

$$h_c = h_{max} - \epsilon \frac{P_{max}}{S} \quad (3.2)$$

They derived this expression from the analysis of Sneddon (1965) for the indentation with axisymmetric indenters of an elastic half-space, hence they assumed that the material around the indenter sinks in. The parameter ϵ depends on the indenter shape. $\epsilon = 0.75$ in the case of Berkovich indenters. However, in their subsequent review of the methodology, they developed a more detailed analysis for obtaining ϵ as a function of the parameters of the unloading, in which they considered that the contact area during unloading indeed changes, exhibiting a convex curvature that does not fit the shape of the indenter (Oliver and Pharr, 2004). As in the analysis of Doerner and Nix (1986), S is the stiffness of the unloading calculated as the tangent

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to the curve in the point of maximum force. However, Oliver and Pharr (1992) recommended first fitting the unloading with a power law relation $P = \alpha(h - h_f)^m$, being α , h_f and m fitting parameters. The stiffness is then computed as $S = dP/dh$ at the point of maximum force. From the estimated contact depth, the contact area, A_c , is computed with the indenter area function. Area functions are calibrated using reference materials, being fused silica the most common choice. Calibration is very important because the indenter shape normally differs from the ideal geometry. In pyramidal indenters, tip roundness due to wear induces a deviation from the ideal pyramidal shape. For a Berkovich indenter, the area function is normally approximated by $C_1 h^2 + C_2 h$. Alternative to this now traditional method for estimation of contact area from the unloading curve, another method has been proposed, the continuous stiffness measurement. An oscillatory load or displacement is superimposed to the main indenter motion and the unloading stiffness can be obtained as a function of depth (Oliver and Pethica, 1989; Oliver and Pharr, 1992).

Instrumented indentation also enabled the estimation of elastic modulus using the information obtained from the unloading curve. Shorshorov et al. (1981) and later Loubet et al. (1984) and Doerner and Nix (1986) proposed an equation based on Sneddon's solution for axisymmetric punches in an elastic half-space:

$$E_{eff} = \frac{S\sqrt{\pi}}{2\beta\sqrt{A_c}} \quad (3.3)$$

Although the expression comes from an elastic analysis, it has been demonstrated that it is also applicable to elasto-plastic contact (Cheng and Cheng, 2004; Oliver and Pharr, 2004). Furthermore, Gao and Wu (1993) have shown that the equation is also applicable to indenters that do not fulfil the axisymmetry assumption like Berkovich or Vickers. In fact, equation 3.3 includes a parameter, β , to account for non-circular projected area of pyramidal indenters. $\beta = 1.034$ for Berkovich indenters.

The modulus obtained in equation 3.3 contains the information of the elastic deformation of the material and the indenter. The effect of the elastic deformation of the indenter is eliminated using:

$$E = \frac{1 - \nu^2}{\frac{1}{E_{eff}} - \frac{1 - \nu_i^2}{E_i}} \quad (3.4)$$

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E_i and ν_i are the elastic modulus and Poisson's ratio, respectively, of the indenter. ν is the Poisson's ratio of the tested sample.

In addition to the standard hardness and elastic modulus, the elastic and plastic works of indentation are also useful metrics that can be obtained from the load-depth curve (see figure 3.1). They are computed from:

$$W_t = \int_0^{h_{max}} Pdh \quad W_e = \int_{h_r}^{h_{max}} Pdh \quad W_p = W_t - W_e \quad (3.5)$$

W_e is the elastic work of indentation computed from the area under the unloading curve. W_t is the total work of indentation (elastic + plastic) and it is calculated as the area under the loading curve. W_p is the plastic (non-reversible) work of indentation computed as the area enclosed between the loading and unloading curves.

3.1.2 State of the art of the nano-impact technique

Impact nanoindentation (or nano-impact) was the first technique for performing instrumented high strain rate nanoindentation (see section 1.4), hence most of the work on high strain rate nanoindentation has been carried out with this technique. Some of the works dealt with studying the complexities associated with the test. One of them is the strain rate of indentation. As an impact test, the strain rate rapidly changes from very high values in the moment of first contact ($10^4 - 10^5 s^{-1}$) to zero at the point of maximum depth when the indenter is arrested due to the material's resistance (Wheeler, 2009). Another challenge is the presence of a secondary oscillation superimposed on the main indenter motion due to pendulum oscillation. Jennett and Nunn (2011) first described this phenomenon and showed that it may lead to high uncertainties in the results. This oscillation or "ringing" is common for pendulum-type impactors. Some authors have proposed the modification of the instrument configuration to reduce the oscillation by stiffening the pendulum shaft to increase its natural frequency (Jennett and Nunn, 2011; Zehnder et al., 2017, 2018). Despite these works, however, there has been little clear consensus in the literature over what material metrics to extract from the single-impact nanoindentation test. Constantinides et al. focused on the direct metrics obtained from the test like maximum and residual depth, inbound and outbound velocity (Constantinides et al., 2008, 2009). They correlated the maximum-to-residual depth ratio with the parameter H/E , which is an important indicator of tribological performance. A higher H/E ratio means the surface is able to absorb more energy in the form of elastic

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deformation that does not lead to permanent damage of the surface. Other authors focused on extracting energy dissipation metrics from the direct test results, with a particular focus on biological materials and tissues (Kalcioğlu et al., 2011, 2013; Canovic et al., 2016; Qing and Vliet, 2016; Mijailovic et al., 2018). Mijailovic et al. proposed a method to directly extract parameters of a linear viscoelastic constitutive model from nano-impact testing (Mijailovic et al., 2018). Other groups calculated a dynamic hardness that, in some cases, was compared with the quasi-static hardness obtained in conventional nanoindentation tests, with unequal success (Constantinides et al., 2009; Wheeler and Gunner, 2013; Zehnder et al., 2018, 2017; Trelewicz and Schuh, 2008; Somekawa and Schuh, 2012; Arreguin-Zavala et al., 2013; Ghosh et al., 2016, 2017; Verma et al., 2016). These studies derived the value of dynamic hardness, H_E , by making two assumptions: (1) the plastic work of indentation, W_p , is equal to the difference in kinetic energy of the indenter between the start and end of the contact period; (2) the hardness is constant throughout the impact:

$$W_p = \frac{m}{2}(V_{in}^2 - V_{out}^2) = \int_0^{h_r} P dh = H_E \int_0^{h_r} A dh \quad (3.6)$$

P is the applied force in the material, h is depth and A is the indenter area function. V_{in} and V_{out} are the initial and outbound velocity, respectively, while m is the equivalent system mass and h_r is the residual depth. This leads to the following expression that can be found in literature to compute an energy-based dynamic hardness from single impact nanoindentation tests:

$$H_E = \frac{\frac{m}{2}(V_{in}^2 - V_{out}^2)}{\int_0^{h_r} A dh} \quad (3.7)$$

There are, however, two potential errors with the assumptions made in the determination of equation 3.7. Firstly, there is a missing term in the energy balance, which is significant with respect to the kinetic energy. This is the work done by the impulse (actuated) force $F_{imp}h$, which although is negligible in cases of low impulse force and high impulse distances (high impact velocities), it generally has a significant contribution to the overall energy balance. The second issue is the implicit assumption contained in the last term of equation 3.6 that assumes that hardness is constant throughout the entire indentation loading and unloading process. Considering indentation with a self-similar indenter, Cheng and Cheng (2004) dimensional

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analysis showed that hardness (defined as the mean contact pressure) is indeed constant during loading (provided the test is carried out at constant $\dot{h}/h$, which is not true during a nano-impact test). However, for the unloading part, the same does not hold true. As a consequence, the hardness calculated using equation 3.7 is prone to suffer from considerable errors.

3.2 Description of the instrument and modification

The nanoindentation and nano-impact tests have been carried out on an instrumented nanoindenter NanoTest Alpha from Micro Materials Ltd. (UK). This is a pendulum-based, force-actuated, displacement-sensing device with the in-built capability of performing energy-controlled nano/micro-impacts. The combination of conventional nanoindentation, for which a range of strain rates from 10^{-3} to 10^{-1} s^{-1} can be tested, with nano-impact provides a tool for materials characterization at the nano/microscale over 6 orders of magnitude of strain rates, from 10^{-3} to 10^3 s^{-1} .

Figure 3.2 is a schematic illustration of the most important components of the system. Force is actuated via a magnet-electromagnetic coil couple. Two capacitor

Figure 3.2: Schematics of pendulum-based nanoindentation device.

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plates in line with the indenter tip measure displacement. The pendulum rotates around a frictionless pivot (see detail 2 in figure 3.2) consisting of a beam fixed to the frame and a moveable beam, connected to the pendulum. In order to accomplish impact indentation in this system, a material of low magnetic hysteresis at the bottom of the pendulum is attracted by a solenoid, installed on the bottom plate of the instrument (see detail 3 in figure 3.2). The pendulum is brought into contact with the solenoid by switching on the solenoid voltage. At the same time, a constant current is applied in the coil in the upper part of the pendulum to supply the impulse force. Then, the sample stage is adjusted in such a way that the distance between the material surface and the tip is equal to a predefined impulse distance. The solenoid voltage is then switched off and the indenter tip swings towards the material surface, driven by the predefined impulse (coil) force.

The main challenge in nano-impact testing to compute hardness following equation 3.1 is to determine the force applied on the material. Unlike conventional low-speed nanoindentation, in nano-impact tests the applied force is not equal to the actuated force. This is due to the significant instrument dynamic contribution, especially the inertia effect. As a first approximation, the applied force can be computed by assuming a linearized damped harmonic motion for describing the instrument dynamics (Constantinides et al., 2009; Mijailovic et al., 2018; Singh et al., 2004, 2008):

$$P = F_{imp} - m\ddot{h} - \rho\dot{h} - kh \quad (3.8)$$

F_{imp} is the actuated force that serves as driving force for the impact and m , ρ and k are the effective mass, instrument damping and spring stiffness, respectively. These can be calibrated using a procedure explained elsewhere (Constantinides et al., 2009; Kalcioglu et al., 2011). Several authors used this approach to calculate the applied force in the material and derive a hardness value from it (Kalcioglu et al., 2011; Qin et al., 2019). However, although this method can produce reasonable estimations of the maximum applied force, it fails to generate an entire force-displacement curve of sufficient quality to apply the standard unloading curve analysis. This is because the high frequency noise in the measured displacement signal introduces a large uncertainty on its time derivatives. In particular, noise in the acceleration signal (second time derivative) is of particular importance because the inertia term ($m\ddot{h}$) adds the highest contribution to the total applied force. As noted by Zehnder et al. (2018), noise in the acceleration signal of the nano-impact can be so high that the real signal might be effectively obscured by noise. Therefore, signal-processing methods

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need to be applied to extract the acceleration signal, for example polynomial fitting as proposed by Qin et al. (2019). This then makes the force calculation dependent on signal processing parameters, leading to a less robust analysis.

A different approach is proposed in this study by instrumenting the nano-impact test with a force sensor to allow the direct measurement of the force applied on the material. A miniature piezoelectric force sensor ICP-209C11 from PCB Piezotronics (USA) was selected. The sensor incorporates an in-built MOSFET amplifier to convert the measured high impedance charge into a readable low impedance signal. The signal then passes to a PCB 482C05 signal conditioner before going into the National Instrument acquisition card inside the instrument controller. This sensor was selected because of its high frequency response, ability to measure in the mN range and high stiffness. The change in overall compliance after incorporation of the sensor was assessed. The resulting increase from 0.27 nm/mN to 0.345 nm/mN was considered reasonably low and could be easily accounted for by standard frame compliance calculations. The addition of a force sensor in direct contact with the specimen (see detail 1 in figure 3.2) gives direct access to the applied force on the material, thus avoiding the dependency on signal processing parameters and dynamic calibrations of the computed force approach. Besides, it allows applying procedures for zero-point determination recommended in the ISO14577 Standard for instrumented indentation. This represents an additional advantage of the use of the force sensor. In conventional nano-impact tests, it is not possible to apply the conventional slow approach contact procedure and the surface position has to be determined from a pre-contact routine prior to test, that is less accurate than in quasi-static nanoindentation. The new force sensor allows for zero-point corrections to determine the surface position more accurately.

Figure 3.3-a presents the main signals from a typical nano-impact test using the modified force-sensing device. The indenter impacts the surface causing plastic and elastic deformation of the material. This elastic deformation then leads to a spring-back of the indenter that rebounds and impacts again the surface driven by the constant impulse force applied in the actuator. This results into the typical undulating shape observed in this type of test. Figure 3.3-a also illustrates the high contribution of inertia force to the total applied force. While the total applied force was around 80 mN in this example, the actuated force was only 5 mN. Figure 3.3-b shows a comparison of the force-displacement curve obtained from the measured force and displacement signal, and that obtained from the computed force signal

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Figure 3.3: (a) Computed force (red), measured force (blue) and measured depth (green) signals for a nano-impact test on an epoxy resin using 5 mN impulse force and 20 μm impulse distance. (b) Force-depth curves, using measured and computed force, for the first impact.

using equation 3.8 in a nano-impact test made in an epoxy resin. As mentioned earlier, the computed force provides a fairly good estimation of the maximum force, but it does not provide an entire force-displacement curve of sufficient quality. On the other hand, the force-displacement curve obtained by directly measuring the force is sufficiently smooth to perform the standard unloading curve analysis. Additional benefits of directly measuring the force are that it is not necessary to calibrate the instrument dynamics, nor is to perform signal-processing procedures to filter noise.

3.3 Validation of the novel instrumentation by testing materials with a wide spectrum of mechanical properties

3.3.1 Materials and experimental details

The novel instrumentation was experimentally tested on materials displaying a wide range of mechanical behaviour in terms of yield stress, Y , and Y/E ratio. Soft and hard materials were included in the analysis, with behaviours ranging from very elastic (large Y/E ratio and therefore large elastic recovery) to very plastic (small Y/E ratios and therefore small elastic recovery). The following materials were studied:

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- Commercial grade PMMA, which is soft and elastic. This is a thermoplastic polymer with a wide range of applications and industrial interest.
- Fused silica, hard and elastic. The Spectrosil 2000 specimens from Heraeus (Germany) supplied by the nanoindenter manufacturer for instrument calibration were used.
- Polycrystalline tungsten from Goodfellow (UK), hard and very plastic. The samples for instrument calibration supplied by the nanoindenter manufacturer were used in this case too.
- Coarse-grain pure copper, soft and very plastic. This sample, referred to as Cu0P, was annealed at 600 °C for 2 hours.
- Two ultrafine grain (UFG) materials, 6082 Al alloy and pure copper, which are harder and more elastic than the coarse-grain pure copper. The UFG copper, referred to as Cu12P, was subjected to 12 passes of equal channel angular pressing (ECAP) at room temperature, as described in Moreno-Valle et al. (2013). The UFG 6082 Al alloy was subjected to 8 ECAP passes, as described in Sabirov et al. (2008).

All samples were ground and polished with 1 μm diamond paste to obtain a mirror-finished surface suitable for nanoindentation experiments.

Nanoindentation and nano-impact tests were performed with a diamond Berkovich indenter whose area function was calibrated using a standard fused silica sample. Dynamic calibrations were performed following a procedure similar to the one of Constantinides et al. (2009) resulting in: equivalent mass of 0.2918 kg, damping coefficient of 0.4333 Ns/m and pendulum spring stiffness of 20 N/m. The constant strain rate conventional nanoindentation tests were carried out using an exponential loading scheme. Strain rate was set to 0.05 s^{-1} . A dwell period of 20 s was applied in all cases, followed by a constant rate unloading at 10 mN/s. Tests were performed up to 10, 20, 50 and 100 mN of maximum force. For the two hardest materials, fused silica and tungsten, additional tests were also carried out with a maximum force of 200 mN. 5 repetitions were done per test. In the case of the nano-impact tests, they were carried out under three different conditions, by setting the impulse force to 5, 10 and 20 mN. A 20 μm impulse distance was selected in all cases. 10 repetitions were carried out per test.

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Figure 3.4: Force-displacement curves from nanoindentation (continuous red lines) and nano-impact (dashed black lines). Each nano-impact curve corresponds to each of the impact conditions (5, 10 and 20 mN impulse force). Higher impulse force leads to higher applied forces and maximum depths.

3.3.2 Direct results

Instrumentation of the nano-impact test with force-sensing capability gives direct access to the force-displacement curves in figure 3.4. These curves were obtained by averaging the results of 5 repetitions for the nanoindentation tests and 10 repetitions for the nano-impact tests. Nano-impact tests were performed with three different impulse forces. This results into three superimposed force-displacement curves for each material, with a higher impulse force (higher impact velocity) leading to a higher applied force, due to the higher inertia contribution. The force-displacement curves reveal differences in the rate sensitivity of the materials, with the rate dependent behaviour of PMMA being particularly evident. The combination of both techniques, nanoindentation and nano-impact, hence provides a tool for materials characterization over a wide range of strain rates.

The values of instantaneous indentation strain rate for each test, $\dot{h}/h$, as defined by Cheng and Cheng (2004), are shown in figure 3.5 for the case of PMMA. Since the conventional nanoindentation tests were carried out following an exponential

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Figure 3.5: Strain rate signal for nano-impact (black) and nanoindentation (red).

loading scheme, the strain rate of indentation remained constant with indentation depth. In the case of the nano-impact tests, however, the strain rate cannot be controlled and the strain rate varied from very high values ($10^4 - 10^5 s^{-1}$), at the moment of impact, to zero at the point of maximum depth, where the indenter stops and rebounds. Figure 3.5 also shows a slightly higher strain rate with increasing the impulse force, and hence the initial impact velocity, as expected.

In the nano-impact, the indenter rebounds several times in the sample surface, driven by the applied constant impulse force, until the energy dissipates and the indenter is arrested. Instrumentation with a force sensor thus enabled the study of the mechanical response during successive rebounds. Force-displacement curves in the four first rebounds after the first impact are displayed in figure 3.6. In these curves, the first anomalous part of the rebound should not be considered as it is an artefact of the averaging procedure performed to obtain the curves. They have been obtained by averaging of the ten repetitions done per material and impact condition. The curves show that the indenter loses energy in every rebound due to viscous dissipation. For this reason, the applied force in each rebound is lower than the previous one. The four metals all exhibit the same behaviour where the force-displacement curve for the successive rebounds follows the same path left by the unloading curve in the first impact. This means that the first unloading and the successive rebounds are all completely elastic. Wheeler et al. (2019) arrived to the same conclusion by studying indentation marks of nano-impacts in copper after the first impact and after several rebounds. They were able to analyse the behaviour after the first contact by capturing the pendulum immediately after the

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Figure 3.6: Force-displacement curves for several rebounds.

first rebound so that subsequent rebounds did not occur.

In contrast, the successive rebounds of the two more strain rate dependent materials, fused silica and PMMA, do not follow the path left by the first unloading, but exhibit a hysteretic behaviour suggesting a viscoelastic or viscoplastic behaviour. The trend is different for the two materials. In fused silica, all successive rebounds follow the same loading and unloading path but in PMMA the successive rebounds appear to be converging as both loading and unloading follow different paths in each of the rebounds. There is a clear energy dissipation as the maximum force and depth continuously decrease. This energy dissipation is very likely to be associated with the viscoelastic damping of the polymer.

3.3.3 Application of the conventional analysis to extract hardness and elastic modulus

Nanoindentation and nano-impact data were analysed following the standard analysis described in section 3.1. The unloading stiffness was obtained from the power law fit to the unloading curve from 90 % to 20 % of maximum force, and the contact

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area was subsequently determined. The values of hardness and elastic modulus as a function of indentation depth are presented in figures 3.7 and 3.8, respectively.

As expected from the force-displacement curves in figure 3.4, the dynamic hardness results do not vary substantially with respect to the quasi-static hardness for most of the materials under study, with the exception of PMMA and fused silica. In the case of the four metals under study, the hardness does not substantially vary within five orders of magnitude of strain rate, from $5 \cdot 10^{-2} s^{-1}$, representative of the quasi-static tests, to the $10^2 - 10^3 s^{-1}$ rates applied during the nano-impact tests. Starting with coarse grain Cu (Cu0P), the dynamic hardness was just within the experimental scatter of quasi-static hardness and reproduced well the indentation size effect expected for single crystal fcc metals (Nix and Gao, 1998), suggesting that the indentation process is still controlled by dislocation glide at impact conditions. The results agree well with previous work in fcc metals, both at the macroscopic (Khan and Huang, 1992) and microscopic (Sudharshan Phani and Oliver, 2017) scales, showing that the flow stress increase accelerates rapidly only for strain rates larger than $10^4 s^{-1}$, which is beyond the range of our study. Similar size effects were also found for coarse grain tungsten, as expected (Maier et al., 2015; Beake et al., 2018). Moreover, the increase in dynamic hardness with respect to the quasi-static hardness, albeit small, was larger than for coarse grain Cu, which agrees with the expected enhanced strain rate sensitivity in high lattice resistance bcc metals. This arises because, at low temperatures, deformation is governed by the thermally activated motion of screw dislocations (Seeger and Schiller, 1962). For fine grained fcc metals (Al6082 and Cu12P), our results show smaller indentation size effects than for the coarse grain metals, both at quasi-static and dynamic conditions, which agrees with previous observations (May et al., 2005). Moreover, a higher increase in dynamic hardness with respect to the quasi-static condition was found. This agrees with the higher strain sensitivity reported for fine grained fcc metals (Maier et al., 2011), which has been explained due to the contribution from grain boundary deformation processes.

The dynamic hardness for the two more elastic materials (PMMA and fused silica), however, presents significant differences with respect to the quasi-static hardness, but the differences are much smaller than what has been reported in other studies that rely on the use of the energy based dynamic hardness definition presented in equation 3.7 (Zehnder et al., 2017). For PMMA, the nano-impact hardness is substantially higher than the quasi-static hardness, which is not surprising for a

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Figure 3.7: Comparison of hardness from conventional nanoindentation test (black) with hardness from nano-impact test calculated by force approach (red).

polymer that has a very rate-dependent mechanical response. However, the dynamic hardness was found to be substantially size dependent, and this was not the case for the quasi-static data. A strong size effect in the dynamic hardness was also observed for fused silica, and surprisingly, the dynamic hardness was found to be slightly lower than for the quasi-static condition. This behaviour has also been reported before, both by microindentation experiments equipped with a high speed camera to measure the residual impression (Yoshioka and Yoshioka, 1995) and in nanoindentation experiments (Jennett and Nunn, 2011), and it has been related to either the formation of cracks (ISO14577, 2015) or the effects of adiabatic heating (Yoshioka and Yoshioka, 1995).

The nano-impact elastic modulus of the four metals tested exhibits significant deviations with respect to the quasi-static values. This is an experimental artifact due to the dynamic deformation of the instrument and the ringing effect (see section 3.4 for further discussion about this) and it affects more those materials with the least elastic recovery, such as tungsten and coarse grain copper, because they are more sensitive to the dynamic compliance of the instrument. Stiffening the pendulum shaft to increase its natural frequencies would be one option to overcome

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Figure 3.8: Comparison of elastic modulus from conventional nanoindentation test (black) with elastic modulus from nano-impact test calculated by force approach (red).

this problem, following previous works (Jennett and Nunn, 2011; Zehnder et al., 2017, 2018). In the case of the two more elastic materials, the nano-impact modulus of fused silica corresponds reasonably well with the quasistatic values, while the nano-impact modulus of PMMA is higher due to the viscoelastic response of the material.

One possible concern on applying the analysis in section 3.1.1 to the force-displacement curves obtained in the nano-impact tests is whether the unloading is fully elastic, which is an important requirement to determine the contact area from the unloading stiffness. In materials with time dependent deformation, this is usually achieved by ensuring that the unloading rate is at least 10 times faster than the loading rate or by introducing a dwell segment at maximum force (Briscoe et al., 1998). Otherwise, inelastic deformation during unloading can introduce artefacts, like a nose-shape unloading curve, that can compromise the stiffness measurements. In the nano-impact test, however, both the loading and unloading rates are similar, and therefore, the force-displacement curves can suffer from these effects. The force-displacement curves in figure 3.4 show that this might be the case for both the

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PMMA resin and the fused silica, and to a less extent, for tungsten.

3.4 A new energy based approach to determine dynamic hardness

The energy-based dynamic hardness computation of equation 3.7, currently used in literature, was derived based on erroneous assumptions, both on the work of indentation and the indentation volume. In this study, a new methodology to determine the energy-based dynamic hardness is proposed, supported on the novel instrumentation introduced in this work:

$$H_{E,new} = \frac{W_t}{c \int_0^{h_{max}} Adh} \quad (3.9)$$

W_t is the total work of indentation computed as in section 3.1.1. The denominator term in equation 3.9 represents the contact volume. The integral represents the total volume of the indenter at maximum depth, while the parameter c , defined as the ratio between the contact area and the indenter area function at depth h , A_c/A (Alkorta et al., 2005), can be estimated using the standard unloading curve analysis and accounts for sink-in effects. The methodology is similar to the work-of-indentation approach discussed by Jha et al. (2014) in the sense that they also calculated an energy-based hardness from the total work of indentation and considered sink-in effects.

For the sake of clarity, table 3.1 summarizes the three different approaches that were evaluated in this work to compute hardness from single impact nanoindentation tests. Thanks to the instrumentation of the test and by evaluating the methodology on six materials covering a wide spectrum of mechanical behaviour, this work demonstrates that the new energy-based computation method, $H_{E,new}$, leads to the same hardness value than that obtained using the force-based definition stated in equation 3.1, H_F . The main sources of errors in the use of equation 3.7, H_E , are also identified, based on the experimental results and finite element analysis of the impact indentation process.

The computation of hardness in the impact tests following each of the alternative methodologies described in table 3.1 leads to the results shown in figure 3.9. The figure includes the three impact conditions with impulse forces of 5, 10 and 20 mN and an impulse distance of 20 μm . For each hardness computation method,

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Equation	Description
$H_F = \frac{P}{A_c}$	Force-based definition of hardness, according to the ISO standard for instrumented indentation (ISO14577, 2015). The Oliver and Pharr (1992) method is commonly used in literature to estimate the contact area.
$H_E = \frac{\frac{m}{2}(V_{in}^2 - V_{out}^2)}{\int_0^{h_r} Adh}$	Energy-based definition of hardness commonly used in literature to determine hardness in impact nanoindentation tests.
$H_{E,new} = \frac{W_t}{c \int_0^{h_{max}} Adh}$	New energy-based definition of hardness proposed in this work.

Table 3.1: Summary of hardness definitions assessed in this section.

the computed hardness was constant with impulse condition, except for the coarse grain copper (Cu0P), which showed a decreasing hardness with increasing impulse force. Considering that an increasing impulse force results in a larger final indentation depth, as shown in figure 3.4, this was attributed to an indentation size effect. This is not surprising considering that indentation size effects are generally larger in low dislocation density coarse grain and single crystal materials (see section 3.3.3). Regarding the hardness computation method, the hardness calculated with the energy-based definition commonly used in literature, H_E , was always larger than that calculated with the accepted force-based definition, H_F , indicating that the former can lead to extremely large errors in the estimation of hardness. Furthermore, the discrepancy was larger for the more elastic materials, i.e., the error could be as high as around 1000% for fused silica, the material with the highest Y/E ratio, while the errors remained substantial but decreased down to 50% in the case of Cu0P, the softest material with the lowest Y/E ratio. On the contrary, the new energy-based definition of hardness proposed in this work, $H_{E,new}$, leads to values that correlate extremely well with the force-based hardness, H_F , irrespective of the type of material.

3.4.1 Numerical analysis

Finite element method (FEM) analysis was employed to numerically assess the differences encountered between the traditional energy-based hardness computation method found in literature, H_E , and the energy-based hardness computation method

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Figure 3.9: Nano-impact hardness calculated by (1) force approach, (2) energy-based approach in literature and (3) proposed energy-based approach. Results for the three tested impact conditions. Impulse distance is 20 μm in all cases and impulse forces are 5, 10 and 20 mN.

proposed in this work, $H_{E,new}$. FEM models of nanoindentation and nano-impact tests were set up using the commercial FEM software Abaqus. In both cases, the Berkovich indentation was modelled as a cone of 70.3° semiapex angle (an equivalent cone with same area-to-depth ratio than the Berkovich), so that a 2D model with radial symmetry could be used. The indenter was considered as a rigid analytical solid with frictionless contact. The domain size was set large enough to avoid boundary effects and it was meshed with 4-noded quadrilateral axisymmetric elements (CAX4), or the same element with reduced integration (CAX4R), when explicit analysis was carried out. An area of refined and structured mesh of size $6h_{max}$ was set beneath the indenter, since this is the area where larger deformations are expected. The Abaqus Implicit solver was used for the simulations of nanoindentation and the Abaqus Explicit solver for the simulations of the nano-impact tests. In both cases, non-linear, large deformation analysis was used.

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Figure 3.10: Application of energy-based definitions of hardness to simulation results and comparison with force-based hardness (reference).

The nanoindentation simulations were carried out by driving the indenter into the material in displacement-controlled mode, using a linear scheme. The indentation depth was set to $2 \mu\text{m}$ in all cases. For the nano-impact simulations, only one condition was simulated, by setting an impulse force of 5 mN and an initial velocity of 0.75 mm/s, and by modelling the indenter as a rigid body with the same effective mass, damping and spring stiffness than the real instrument.

An isotropic linear elastic material with J_2 -plasticity and no hardening was used as the constitutive law of the material. The elastic modulus, E , and Poisson ratio, ν , were fixed at 100 GPa and 0.3, respectively, and the yield stress, Y , was varied between 200 and 15000 MPa. Since the simulated materials were rate independent, the outcomes of the conventional indentation and impact indentation simulations must be identical. This provided a framework to evaluate the different methods used in this work to evaluate hardness from impact indentation tests, and to elucidate the origin of the discrepancies found, as a function of the Y/E ratio of the material.

The nanoindentation simulations were used to compute the hardness values for each of the simulated material cases, applying the definition in equation 3.1. These values were used as reference, H_{ref} in figure 3.10, to compare with the hardness values obtained in simulations of nano-impact tests by applying the two energy-based definitions of hardness in table 3.1. The results are presented in figure 3.10, together with an alternative computation of H_E , named H_E (real W_p). The alternative approach uses the plastic work of indentation, W_p , instead of the change in kinetic energy of the indenter, to compute the energy balance in the numerator of equation

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3.7. Figure 3.10 shows that the hardness computed using the newly proposed energy-based method, $H_{E,new}$, is essentially the same as the reference hardness, validating the new approach. However, hardness values calculated using the energy-based computation method commonly used in literature deviate significantly from the reference hardness, irrespective of whether the energy is computed from the change in kinetic energy of the indenter, H_E , or from the real work of indentation, H_E (real W_p). In line with the experimental results (see figure 3.9), the discrepancy was higher for the more elastic materials (increasing Y/E ratio). However, the discrepancy was smaller than that found experimentally, which suggests that there are additional experimental factors that contribute to the unbalance between the change in kinetic energy of the indenter and the real work of indentation, that are not accounted for in the simulations. Finally, the simulations also showed that the discrepancies increase too for very plastic materials ($Y/E = 10^{-3}$). This trend was not observed in the experiments because it is due to pile-up effects, which are captured by the real contact area measured in the FEM simulations, but cannot be estimated experimentally using the standard unloading curve analysis, which assumes sink-in behaviour only.

3.4.2 Discussion

As discussed in section 3.1.2, the energy-based computation of hardness adopted in previous studies, represented by equation 3.7, is based on two main hypotheses: (1) the calculation of plastic work of indentation can be estimated from the change in kinetic energy of the indenter throughout the impact; and (2) the hardness is constant during the entire indentation process. The experimental and simulation results summarized in figures 3.9 and 3.10 show that these assumptions are incorrect most of the time, and they lead to large errors in the computed hardness values, if compared with the accepted definition of hardness, based on equation 3.1.

One of the sources of deviation, linked to the first hypothesis, is the energy balance in the numerator of equation 3.7. There are several reasons for this. Equation 3.10 considers all possible contributions to the energy available and the energy consumed in the impact indentation process:

$$\begin{aligned} \frac{mV_{in}^2}{2} - \frac{mV_{out}^2}{2} + F_{imp}h_r = W_p + \frac{k}{2}h_{max}^2 - \frac{k}{2}(h_{max} - h_r)^2 + \\ + \int_0^{h_r} \rho|\dot{h}|dh + W_d \simeq W_p + W_d \end{aligned} \quad (3.10)$$

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The total energy available is the sum of the balance in kinetic energy of the indenter before and after indentation plus the contribution of the work done by the impulse force, $F_{imp}h_r$. This energy is consumed on the non-reversible work of indentation W_p and on other parts of the instrument. Considering the instrument as a linearized damped harmonic oscillator, $\frac{k}{2}h_{max}^2 - \frac{k}{2}(h_{max} - h_r)^2$ and $\int_0^{h_r} \rho|\dot{h}|dh$ represent the spring and dissipative terms of the harmonic oscillator, while W_p accounts for other energy dissipation contributions. The dissipative and spring energy terms are sufficiently small to be negligible with respect to kinetic energy, as noted by Chen et al. (2016) and equation 3.10 can be rewritten to account only for $W_p + W_d$. However, the rest of the terms neglected in the numerator of equation 3.7 are not, by any means, negligible. Firstly, the work done by the impulse force is not as significant as the kinetic energy, but it has an important contribution, as can be seen in the simulation results of figure 3.10, where the difference between W_E and $W_E(\text{real } W_p)$ is solely due to the contribution of the work done by the impulse force, which is only accounted for in the latter. Secondly, and more importantly, there is a substantial amount of available energy that is dissipated in the instrument, W_d , and hence, does not contribute to the work of indentation. Thanks to the instrumentation of the nano-impact test with force-sensing capability, it was possible, in this work, to compute the non-reversible work of indentation, W_p , from the enclosed area of the force-displacement curves between the loading and unloading segments and compare it with the total energy available in the experiment, $m/2(V_{in}^2 - V_{out}^2) + F_{imp}h_r$. Indeed, as shown in figure 3.11, W_p was, in all cases, substantially lower than the total available energy, indicating a substantial amount of energy dissipated elsewhere. Moreover, the fraction of energy loss remained constant with impact energy for each material, and it was higher for the harder materials. While the energy loss for PMMA was around 25%, the loss for tungsten and fused silica was higher than 50%.

These results suggest that part of the energy available for the nano-impact test is dissipated and hence not spent on material elastic and plastic deformation. The origin of this dissipated energy could be associated with losses through the boundary conditions in the form of dynamic deformation of the instrument. As it was discussed in section 3.1.2, several authors have identified the presence of a secondary oscillation or “ringing” in the output signals, especially velocity, of the nano-impact test (Jennett and Nunn, 2011; Wheeler and Gunner, 2013). This vibration is typical of pendulum-like strikers and it is caused by loading of the mechanism at a

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Figure 3.11: Comparison of available energy for permanent deformation and measured non-reversible work of indentation (W_p). Mean values are displayed.

frequency near its natural frequency of oscillation. The energy dissipated could be then associated with the energy consumption from the pendulum oscillation. Ghosh et al. (2017) also carried out a similar study. They calculated the difference between the plastic energy calculated as the difference in kinetic energy from inbound to outbound, and the plastic work computed as area beneath the force-displacement curve. Their observations are in agreement with the results presented in figure 3.11, but they associated this with the energy spent on creation and transmission of plastic waves in the material. In any case, the results in figure 3.11 make clear the existence of an additional energy dissipation term in the whole energy balance and support the use of the force approach (with the use of a force sensor) over the energy approach in the calculation of hardness for nano-impact test as these problems are directly avoided. Nevertheless, it should be pointed out that the force-displacement curves might be affected by additional dynamic compliance effects introduced by the “ringing” phenomenon, that cannot be accounted for due to the lack of appropriate reference materials for calibration of dynamic compliance at impact conditions. The latter should be born in mind, especially when testing hard materials.

The last source of deviation in the literature energy-based definition of hardness comes from the divisor term in equation 3.7. Experimental and numerical results, in figures 3.9 and 3.10 respectively, show that the deviation between H_E and H_F increases for the more elastic (higher Y/E) materials. This is due to the fact that there is a higher difference between the residual and contact volumes in the more elastic materials, because they have a larger recovery, as it can be clearly seen in figure 3.4.

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Figure 3.12: Comparison of different indentation volumes calculated from the finite element simulations. All simulated cases have a maximum depth of $2 \mu\text{m}$.

The simulation results in figure 3.12 provide a deeper insight on the importance of considering the indentation contact volume, and not the residual volume. Since all indentation simulations were carried out to the same value of maximum depth, $2 \mu\text{m}$, the nominal volume was the same in all cases. The contact volume was computed directly from the FEM simulations and also applying the standard unloading curve analysis (Oliver-Pharr approach) to the simulated force-displacement curves, because this is the method used in the analysis of the experiments. Indentation volumes larger than the nominal volume indicate pile up effects, while lower values correspond to sink in behaviour. Starting from the left (very small Y/E ratios typical of very plastic materials), elastic recovery is negligible and hence the correction proposed in this work is not important, as both the residual volume and the contact volume predicted using the Oliver and Pharr method converge to the nominal contact volume. In this limit, no single method provides a good estimate of the contact volume because they all neglect pile up effects, which can be quite large as revealed by the contact volumes measured directly on the FEM simulations. Nevertheless, the simulations carried out here represent an upper bound to the amount of pile up that can be expected because they were carried out assuming a simple J_2 -plasticity model with no strain hardening and it is well known that strain hardening effects tend to decrease the amount of pile up (Cheng and Cheng, 2004; Oliver and Pharr, 1992; Bolshakov and Pharr, 1998). However, the correction proposed in this work has a large impact for materials with Y/E ratios larger than $2 \cdot 10^{-2}$. In this case,

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the Oliver and Pharr method provides a very good estimate of the contact volume and the results agree reasonable well with the contact volumes measured directly on the FEM simulations, while errors if the residual indentation volume is considered become substantial. Finally, the contact volume converges to the Sneddon solution, as expected in the limit for a fully elastic material (very large Y/E). This is the reason why the errors of using equation 3.7 to estimate H_E are maximum for PMMA and fused silica in figure 3.9, while the errors, although substantial, are reduced for the four tested metals, with the coarse grain copper, being more plastic, showing the smallest difference with the traditional definition of hardness H_F .

3.5 Concluding remarks

The nano-impact test is a technique for material characterization at the nano-/microscale at high strain rates (10^2 - 10^3 s^{-1}). However, the test could not provide the typical output of a nanoindentation test, a force-displacement curve, because the applied force is much higher than the actuated force due to inertia effects. The work presented in this chapter has resolved this issue by instrumenting the nanoindentation device with a force sensor. This has enabled the use of the ISO14577 (2015) standard analysis of instrumented indentation that makes use of the unloading curve to obtain hardness and elastic modulus. Furthermore, the traditional energy-based hardness definition found in literature for the analysis of nano-impact tests and the standard force-based hardness were compared by testing materials covering a wide spectrum of mechanical behaviour. In combination with FEM simulations, the comparison revealed a significant deviation of the values obtained with the literature energy-based definition, with respect to the values computed using the force-based hardness. For plastic materials (metals), this was attributed to a missing energy dissipation term and to the effect of the work of the impulse force that were not considered in the energy-based method. For elastic materials (ceramics and polymer), the deviation was even higher because the difference between the contact and residual volumes of indentation is larger in these materials. Finally, this work proposes a new energy-based definition that provides values equivalent to the standard force-based hardness.

Analysis framework for the extraction of rate dependent constitutive behaviour from nanoindentation

The determination of mechanical properties from indentation tests is not direct because of the complex stress and strain fields that develop under the contact area. Nevertheless, the versatility and simplicity of the technique have inspired researchers to establish methodologies to solve the inverse analysis problem in indentation, i. e. the extraction of elastoplastic properties from indentation data. Section 4.1 briefly reviews some of these methodologies. One of the most popular approaches is the use of dimensional analysis to obtain sets of dimensionless functions that relate mechanical properties with the direct outputs of the instrumented indentation test. These functions are then calibrated using FEM simulations. Section 4.2 contains the details of the dimensional analysis used in this work to set up an analysis methodology to extract viscoplastic properties from nanoindentation. In section 4.3, the different parametric studies based on FEM are explained. The outcome of this combination of dimensional analysis and FEM is a set of master curves, which are also presented in section 4.3. Furthermore, the procedure to extract properties over a wide range of strain rates using the modified nano-impact instrument presented in chapter 3 is presented in section 4.4. Finally, in section 4.5 the inverse analysis methodology is validated against experimental results. The predictions of the inverse analysis agree fairly well with the experimental macroscale results, and the deviations are attributed to the limitations imposed by the hypotheses of the material model used in the FEM-based inverse analysis.

4.1 Introduction

The extraction of elastoplastic properties of materials from indentation testing has been an attractive research line since the early stages of hardness testing. This has been motivated by the simplicity and low cost of the indentation technique. In addition, only a relatively small volume of material is tested. In fact, in some cases it can be considered a nondestructive testing technique. Furthermore, indentation testing is applicable over a wide range of length scales (from nm to mm), which enables the multiscale characterization of mechanical behaviour. In some cases, nanoindentation may be the only feasible technique, for example in thin films (Pharr and Oliver, 1992), for mechanical property mapping at the microstructure scale (Zhu et al., 2007), or for *in situ* testing of the matrix in fibre-reinforced polymer composites (Herráez et al., 2017).

The extraction of mechanical properties from nanoindentation is challenging because of the complex stress and strain fields beneath the indenter that do not allow the direct determination of mechanical properties. Even so, some authors have attempted the determination of simple analytical approximations. For sharp indentation, Johnson (1985) derived a set of equations based on the so called expanding cavity model, which was originally proposed by Marsh (1964). The volume of material beneath the indenter is assumed to be encased in a hemispherical core where the hydrostatic component of the stress is constant. The core is subjected to a pressure that is related to the mean contact pressure or hardness. This formalism allowed Johnson (1985) to relate the mean contact pressure, p , with the yield stress, Y , by $p/Y = 2/3(1 + \ln(E/3Y)\cot\theta)$, being θ the semi-included indenter angle. Other authors have improved the accuracy of this method and expanded it to include, for example, the hardening exponent. Clausner and Richter (2015) proposed two modified expressions for perfectly plastic and power-law hardening materials and validated their analysis with a large database of experimental and numerical FEM results. Tabor (1951) followed a different approach, based on the slip-line solution, and came up with its well-known expression that relates hardness, H , with yield stress, Y , by $H=3Y$, for a rigid-perfectly plastic solid in sharp (self-similar) indentation. Tabor (1951) also expanded its analysis for strain hardening materials by defining the relation $H=3\sigma_r$, where σ_r is the flow stress at a certain representative strain which is an average of the strain field beneath the indenter, and established that it is around 0.08 for Vickers indenters. Since then, other definitions of representative strain have been proposed. Johnson (1985) proposed a generalized expression

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for the representative strain in sharp indenters, which is $\epsilon_r = 0.2 \cot \theta$. However, Bucaille et al. (2003) carried out an extensive FEM-based analysis of sharp indenters with different indenter angles and found significant deviations from Johnson's formula.

Similar analytical treatments were also developed for spherical indentation. Although, unlike self-similar sharp indenters, in spherical indentation the strain field beneath the indenter changes with penetration so the representative strain is expected to depend on the indenter depth. Tabor (1951) suggested a relation between the mean contact pressure in spherical indentation, p_m , and the representative flow stress, σ_r , given by $p_m \approx 3\sigma_r$ and an expression for the representative strain given by $\epsilon_r = 0.2a/R$, where a is the contact radius and R is the radius of the indenter. A priori, this meant that a full stress-strain relation could be approximated from spherical indentation at several depths. Herbert et al. (2001) compared the stress-strain curve estimated from spherical indentation with the stress-strain curve obtained by conventional testing, but found that the shape of the predicted curve differed significantly. In addition to the determination of plastic properties, the extraction of elastic modulus from spherical indentation has also been the subject of analytical treatment. Hertz's solution has been often used to fit the response at very low depths, which is assumed to be fully elastic (Herbert et al., 2001; Alcalá et al., 1998).

While analytical approaches offer simple and fast means of deriving mechanical properties from indentation, the complex stress and strain fields that develop under the indenter and the variety of existing material behaviours question the real applicability of these approaches. In addition, these methods do not take into account highly nonlinear phenomena like the piling up of material around the indenter. More recent studies have benefited from the advances in computational power and numerical methods like the Finite Element Method to devise more advance inverse analysis methodologies. Furthermore, the introduction of instrumented indentation entailed great advances in this field. In fact, the determination of elastic modulus from indentation is now an industry standard (ISO14577, 2015). The new information that instrumented indentation provides, i.e. continuous monitoring of load and displacement, has stimulated new research works to solve the inverse problem in indentation testing. Some researchers have proposed semi-analytical or phenomenological expressions calibrated with FEM simulations that relate the metrics obtained in a instrumented indentation test with the elasto-plastic properties (Alkorta et al.,

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2005; Giannakopoulos and Suresh, 1999; Larsson et al., 1996). In most of the cases, a power-law hardening material is assumed. Other researchers have used dimensional analysis to derive sets of dimensionless functions that relate the instrumented indentation metrics with the material properties of the bulk material (Cheng and Cheng, 2004; Dao et al., 2001) and substrate and coating properties in coated systems (Tunvisut et al., 2001). Other studies in literature employed iterative FEM approaches to match the simulation and experimental results by manually tuning the model parameters (Wang and Cheng, 2017), or by making use of a "goodness of fit" parameter that evaluates the level of agreement of the predicted and measured results in a material property space (Dean and Clyne, 2017).

One of the challenges in these inverse analyses that relate the load-displacement curve of the instrumented indentation test with the elastoplastic properties of the material is the issue of uniqueness. In most of the cases, different material property sets can lead to the same load-displacement curve. Therefore, in the inverse extraction of mechanical properties, the solution may not be unique. The hardening exponent is usually the most challenging to extract. Furthermore, the load-displacement curves are not very sensitive to changes in the material properties. This makes inverse analyses very susceptible to experimental errors and uncertainties in the measurement of the curves (thermal drifts, instrument compliance, zero-load point, etc.). To this end, some authors have suggested the use of additional information in the analysis, like for example the residual imprint (Dean and Clyne, 2017), or the use of multiple sharp indenters (Chollacoop et al., 2003; Bucaille et al., 2003). Other authors have preferred to take advantage of the last advances in deep learning to propose inverse analyses based on neural networks that are trained with multifidelity data (2D and 3D FEM simulations and experimental results) to predict mechanical properties from the indentation load-displacement curve (Lu et al., 2020).

The extraction of time dependent mechanical constitutive behaviour from instrumented indentation has also attracted the interest of researchers over the past years. Most of the studies focused on the determination of the strain rate sensitivity (SRS) of the hardness, assuming a material in which the flow stress is a power law function of the strain rate, leading to a constant SRS value (Lucas and Oliver, 1999; Maier et al., 2011; Alkorta et al., 2008a; Guillonneau et al., 2018; Wehrs et al., 2015). Some researchers made then estimations of the strain rate dependent flow stress by using the SRS of the hardness and applying an appropriate constraint factor, usually 2.8-3 for metallic materials. Most of these studies made use of a popular technique

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called strain-rate jump test. The indentation test is carried out at a constant strain rate and its value is varied abruptly during delimited periods of time. These tests were performed under the assumption that the hardness value is path independent, i.e. its value only depends on the instantaneous value of strain rate. Indentation creep tests have been also used for the study of strain rate sensitivity, although their reliability is compromised by thermal drift (Alkorta et al., 2008a). In all these studies, the indentation strain rate is computed as $\dot{h}/h$. Cheng and Cheng (2004) have shown through dimensional analysis of sharp indentation, and assuming a power law creeping material ($\sigma = k\dot{\epsilon}^m$), that hardness is proportional to $k(\dot{h}/h)^m$ and to a dimensionless function $\Pi(m, \theta)$; hence, the quantity $\dot{h}/h$ can be indeed considered a representative indentation strain rate.

One of the main assumptions in all these studies of strain rate sensitivity is that the value of SRS of the hardness is equal to that of the flow stress. However, while this is valid for most metals in which the indentation is almost fully plastic, it is not the case for materials in which the elastic work of indentation is not negligible. Several authors have shown that for materials in which the elastic deformation cannot be neglected, which is the case of most polymers and hard ceramics, the SRS of the hardness is significantly lower than that of the flow stress (Alkorta et al., 2008b; Elmustafa et al., 2007; Stone et al., 2010; Jakes et al., 2012; Kermouche et al., 2006). Nevertheless, Alkorta et al. (2008b) and Elmustafa et al. (2007) have developed methods to obtain the SRS of the flow stress from the SRS of the hardness. They found that the ratio of SRS of hardness to SRS of flow stress is approximately a unique function of the parameter H/E_{eff} . Elmustafa et al. (2007) have attributed the lower rate sensitivity of the hardness in materials with high H/E_{eff} (above 0.01) to the fact that in this regime the constraint factor (relation of hardness and flow stress) changes with the plasticity index (relation of flow stress and elastic modulus).

Most polymers are remarkably rate dependent and elastic materials (σ_y/E_{eff} between 0.01 and 0.1), thus the SRS of the hardness is also lower than that of the flow stress, according to the aforementioned argument (Jakes et al., 2012). Moreover, adding to their complex mechanical behaviour is the fact that their yield stress is pressure sensitive, a feature that is shared by other amorphous solids like metallic and ceramic glasses. This type of materials can be classified as cohesive-frictional materials, which are characterized by an asymmetric plastic behaviour and fracture (brittle in tension and ductile with large deformations in compression). Several authors have carried out studies on the inverse extraction of properties of

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these materials, making use of the Mohr-Coulomb (Ganneau et al., 2006) and the Drucker-Prager (Keryvin, 2008; Giannakopoulos and Zisis, 2013; Rodríguez et al., 2012a; Seltzer et al., 2011) yield criteria, which are the two most popular material models to simulate plasticity in cohesive-frictional materials. Rodríguez et al. (2012a) performed an extensive inverse analysis study in which they showed that pressure sensitivity tends to increase the constraint factor.

This literature review shows that researchers have devoted significant efforts to the extraction of mechanical properties in rate sensitive materials, and to a less extend in pressure sensitive materials. However, to date there are no studies that aim at extracting mechanical properties from materials that combine both behaviours, which is the case of polymers. The inverse analysis presented in this chapter focuses then on extracting mechanical properties for pressure and rate sensitive materials.

4.2 Dimensional analysis

Dimensional analysis is a powerful tool for establishing relationships between the material metrics obtained in an instrumented indentation test and the material parameters of a certain constitutive model. Buckingham Pi theorem is one of the most popular approaches to perform dimensional analysis. Cheng and Cheng (2004) carried out an extensive review of dimensional analysis of indentation, mainly assuming sharp (self-similar) indenters, considering different types of material models. Self-similar indenters are preferred over spherical indenters because the hardness (defined as the mean contact pressure) does not depend on depth. This review contains a dimensional analysis assuming a material model in which the flow stress is a power law function of the strain rate. However, they assumed that the elastic deformation in the material is negligible, an assumption that is not valid for many materials (see section 4.1). Alkorta et al. (2008b) extended Cheng's analysis to include the effect of elastic deformation. The methodology presented in this chapter is an extension of Alkorta's analysis by introducing the effect of pressure sensitivity using a Drucker-Prager yield criterion.

4.2.1 Materials in which the flow stress is a power law function of strain rate

The dimensional analysis of instrumented indentation of Alkorta et al. (2008b) is based on the these assumptions:

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- Rigid, self-similar indenter (conical or pyramidal). Self-similar indenters are defined by just one parameter, the included semiangle θ .
- The contact between the material surface and the indenter is frictionless.
- The material is homogeneous and isotropic, with the elastic deformation defined by elastic modulus and Poisson's ratio (combined in E_{eff} as in equation 3.4), and the plastic deformation characterized by a yield stress which is a power-law function of the strain rate (no strain hardening):

$$\sigma = k\dot{\epsilon}^m \quad (4.1)$$

The load and contact area in the instrumented indentation test are then a function of these variables:

$$P = P(k, m, E_r, \theta, h, \dot{h}) \quad A_c = A_c(k, m, E_r, \theta, h, \dot{h}) \quad (4.2)$$

The application of dimensional analysis results in these two dimensionless functions:

$$P = k \left(\frac{\dot{h}}{h} \right)^m h^2 \Pi_P \left(m, \theta, \frac{k(\dot{h}/h)^m}{E_r} \right) \quad A_c = h^2 \Pi_{A_c} \left(m, \theta, \frac{k(\dot{h}/h)^m}{E_r} \right) \quad (4.3)$$

Hardness can be obtained by applying equation 3.1 to the previous expressions for load and contact area. As it happens in Cheng's dimensional analysis, the magnitude $\dot{h}/h$ can be considered a representative strain rate of indentation because hardness is constant if this magnitude is held constant throughout the indentation, independently on depth. Unlike Cheng's analysis, hardness is a function of the elastic modulus in this case. However, Cheng's analysis can be recovered if the ratio $k(\dot{h}/h)^m/E_r$ approaches zero, which is the case in most metals.

4.2.2 Extension to cohesive-frictional materials following a Drucker-Prager yield criterion

Cohesive-frictional materials present an asymmetric plastic behaviour, being the compressive yield strength greater than the tensile yield strength. These materials generally fail in a brittle manner in tension while they exhibit large deformation prior to fracture in compression. In addition, the yield strength is pressure-dependent. Granular-like materials used in civil and geotechnical engineering fall

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under this category: soils, rocks and concrete. Furthermore, many amorphous solids like metallic and ceramic glasses, and polymers also exhibit the characteristics of cohesive-frictional materials. Polymers, in addition, present rate dependent elastic and plastic behaviour.

Under appropriate assumptions, it is possible to reproduce the deformation under loading of these complex materials. The accuracy of the predictions and the complexity of the models depend on these assumptions, that in turn depend on the available experimental methods for model calibration. The Mohr-Coulomb model (Coulomb, 1776; Mohr, 1900) and the Drucker-Prager model (Drucker and Prager, 1952) are two typical yield criteria used for simulation of the mechanics of cohesive-frictional materials. The calibration parameters can be transferred from one model to the other. The Drucker-Prager model generally leads to better numerical convergence because of the smooth shape of the yield surface in the deviatoric plane (π -plane), unlike the Mohr-Coulomb model. Besides, the Drucker-Prager model has proven effective in the extraction of mechanical properties from cohesive-frictional materials through instrumented nanoindentation (Rodríguez et al., 2012a; Rodríguez, 2012; Seltzer et al., 2011). The yield surface is defined by:

$$F(\boldsymbol{\sigma}, \beta) = q - p \tan \beta - d = 0 \quad (4.4)$$

The parameters β and d are the friction angle and the cohesion strength, respectively. The friction angle is related to the pressure sensitivity of the material, while the cohesion is related to the yield stress. $\boldsymbol{\sigma}$ is the Cauchy stress tensor, and q and p are the Mises equivalent stress and hydrostatic pressure stress, respectively. They are defined by:

$$q = \sqrt{\frac{3}{2}(\boldsymbol{\sigma}' : \boldsymbol{\sigma}')} \quad p = -\frac{1}{3}tr(\boldsymbol{\sigma}) \quad (4.5)$$

$\boldsymbol{\sigma}'$ is the deviatoric part of the stress tensor, defined as:

$$\boldsymbol{\sigma}' = \boldsymbol{\sigma} + p\mathbf{I} \quad (4.6)$$

The cohesion strength of the material can be defined as a function of the compression yield stress and the friction angle by:

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$$d = \sigma_{yc} \left(1 - \frac{1}{3} \tan \beta \right) \quad (4.7)$$

The Drucker-Prager model is represented by a cone in the meridional (p-q) plane. This means that the dependency of the Mises equivalent stress with hydrostatic stress follows a linear relation and the slope is defined by the friction angle. If the friction angle were zero (no pressure sensitivity), the classical Mises yield criterion would be recovered.

The Drucker-Prager model thus needs the input of two parameters, the compressive yield stress and the friction angle. Following the assumptions in section 4.2.1, the compression yield stress is assumed to be a power-law function of the strain rate and the friction angle is rate independent. The dimensional analysis provides this expression for the hardness:

$$H = k \left(\frac{\dot{h}}{h} \right)^m \Pi_H \left(m, \theta, \frac{k(\dot{h}/h)^m}{E_{eff}}, \beta \right) \quad (4.8)$$

Pressure sensitivity is therefore uniquely represented by the value of the friction angle, β . Equation 4.8 shows that there are two independent material metrics from indentation, hardness (H) and the effective modulus (E_{eff}), but there are three material parameters to be extracted: k, m and β . A unique solution to the inverse analysis problem can be found only if one of the parameters was known beforehand. The methodology developed in this chapter assumes previous knowledge of the pressure sensitivity parameter, β , and focuses on the determination of m and k.

4.3 Numerical analysis

Equation 4.8 is the central function in the inverse analysis methodology proposed in this chapter. FEM simulation was used to calibrate the equation. The inverse analysis is a two-steps procedure. The strain rate sensitivity of the flow stress, m, is first determined. Then, the constraint factor (H/σ_y) is computed from the values of m, β and the parameter H/E_{eff} . The FEM-based calibration is based on a series of parametric analyses, which are separated into two groups: (1) studies to determine the master curve of strain rate sensitivity, and (2) studies to obtain the master curve of constraint factors.

Figure 4.1: Schematics of the Abaqus FEM model used for simulation of conical indentation assuming axisymmetry.

4.3.1 Details of the finite element model

FEM simulations were carried out in the framework of the commercial FEM software package Abaqus. The entire analysis is specific to Berkovich indentation and considers the following assumptions:

- Berkovich indentation is modelled as an equivalent cone of same area-to-depth ratio with an included semiangle of 70.3° . This simplifies the problem to a 2D domain by assuming radial symmetry. This is a popular procedure usually found in studies in literature dealing with FEM modelling of pyramidal indentation (Rodríguez et al., 2012a; Cheng and Cheng, 2004).
- The indenter is modelled as a rigid analytical solid with frictionless contact. The effect of friction on the indentation response has been studied by several authors that concluded that it is negligible for Berkovich indenters, while it has a more pronounced effect on sharper indenters like the cube corner (Rodríguez et al., 2012a; Bucaille et al., 2003).

The domain is a square of side $300h_{\max}$. The size was set large enough to avoid boundary effects. The optimum size was chosen from a parametric study using the worst case scenario, which is a very elastic material ($\sigma_y/E > 0.01$). In very plastic

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materials, the deformation is mostly confined beneath the indenter, so the size of the domain does not need to be as large. The domain was divided in two areas, as in figure 4.1. An area of refined and structured mesh beneath the indenter, where the higher plastic strains are expected. The size of the mesh is such that around 40 elements are in contact at the point of maximum displacement (Rodríguez et al., 2012a). The size of this area is $6h_{max}$. Outside the refined region, the element size gradually increases to save computational time. 4- and 3-noded quadrilateral axisymmetric elements (CAX4 and CAX3) are selected for the mesh. Reduced integration elements were chosen when the Explicit solver was used.

Particularities of the parametric analysis for determination of strain rate sensitivity

FEM simulations used to build a master curve to obtain the strain rate sensitivity of the flow stress consisted of series of seven constant strain rate indentations covering a wide range of strain rates: 10^{-3} , 10^{-2} , 10^{-1} , 10^0 , 10^1 , 10^2 and 10^3 s^{-1} . This series of seven indentations were applied to each of the material sets. The constant strain rate indentations were realized by driving the indenter into the material in a displacement-controlled mode using an exponential scheme. The indentation depth was set to 2 μm in all cases. The Abaqus Implicit solver was used, imposing a nonlinear, large deformation analysis. The time increments were scaled with the total duration of the displacement scheme, being $\Delta t_{max} \sim 10^{-2}$ ·step time.

Two types of material models were implemented. In the construction of the master curves, an isotropic linear elastic material with J_2 -plasticity and no hardening was used. Rate dependency is introduced in the model through the yield stress assuming a power law behaviour, as in equation 4.1. A summary of the different material sets that were simulated is presented in table 4.1. In addition, another set of simulations using a Drucker-Prager yield criterion was prepared. The objective was to assess if the implementation of a different plasticity model had an influence on the master curve of strain rate sensitivity. The specifics of the model are described in section 4.2.2. In addition, perfect plasticity was assumed, and a non-associated flow rule with dilation angle equal to zero. The cohesion, d , of the Drucker Prager model was defined in terms of the compression yield stress, which is assumed to be a power-law function of the strain rate, as in equation 4.1. A summary of the material sets that were simulated is presented in table 4.2

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Elastic properties	Viscoplastic properties	
	m	k [GPa·s ^m]
$\nu = 0.3$, E = 5 GPa	m = 0.02	k = 100, 200, 300
	m = 0.04	k = 100, 200, 300
	m = 0.08	k = 200, 400
	m = 0.12	k = 300, 500
	m = 0.16	k = 300, 500
	m = 0.12	k = 200
$\nu = 0.3$, E = 30 GPa	m = 0.04, 0.08, 0.12, 0.16, 0.2	k = 200
$\nu = 0.3$, E = 70 GPa	m = 0.02, 0.04, 0.08, 0.12, 0.16, 0.2	k = 200

Table 4.1: Material parameters used in the parametric FEM analysis for determination of the master curve of strain rate sensitivities using a J_2 -plasticity model. 24 cases by combination of all parameters. 7 cases of strain rate are performed for each material set, resulting in a total of 168 simulations.

Elastic properties	Viscoplastic properties	
	Friction angle (β)	Compression yield stress
$\nu = 0.3$, E = 5, 10, 20, 30, 40, 50 GPa	$\beta = 15, 30$	k = 200 GPa·s ^m , m = 0.08, 0.16

Table 4.2: Material parameters used in the parametric FEM analysis for determination of the master curve of strain rate sensitivities using a Drucker-Prager plasticity model. 24 cases by combination of all parameters. 7 cases of strain rate are performed for each material set, resulting in a total of 168 simulations.

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Particularities of the parametric analysis for determination of the constraint factors

The master curves of constraint factor as a function of friction angle of the Drucker-Prager model, β , and the SRS of the flow stress, m , were obtained from a set of FEM simulations of constant strain rate indentation. However, in this case only one case of strain rate, 10^3 s^{-1} , was simulated. The Abaqus explicit solver was used in all cases to avoid convergence problems. Convergence of the Drucker-Prager yield criterion is more difficult to achieve than in the classical J_2 -plasticity. Nonlinear, large deformation analysis was imposed. An Arbitrary Lagrangian-Eulerian (ALE) adaptive mesh option was used in the refined area beneath the indenter to reduce element distortion. Mass scaling was employed to reduce the computational time, setting up a Δt_{target} of 10^{-8} s . For the material model, isotropic linear elasticity was applied, together with a Drucker-Prager yield criterion with no hardening, rate dependent flow stress as in equation 4.1 and non-associated flow rule with dilation angle equal zero. Table 4.3 shows a compilation of the simulated material cases.

Elastic properties	Viscoplastic properties	
	Friction angle (β)	Compression yield stress
$\nu = 0.3$, $E = 2, 5, 7, 10, 20, 40, 60, 80, 100, 150, 200 \text{ GPa}$	$\beta = 0, 10, 20, 30$	$k = 200 \text{ GPa} \cdot \text{s}^m$, $m = 0, 0.04, 0.08, 0.12, 0.16, 0.2$

Table 4.3: Material parameters used in the parametric FEM analysis for determination of the constraint factor master curve. 264 cases simulated by combining all parameters with each other.

4.3.2 Results: strain rate sensitivity

The first step in the inverse analysis methodology to extract viscoplastic properties of materials is the determination of the strain rate sensitivity of the flow stress, m . This first part of the analysis was inspired by the work of Alkorta et al. (2008b) and Elmustafa et al. (2007). They found that there exist an approximately unique relation between the ratio of SRS of the hardness to SRS of the flow stress and the parameter H/E_{eff} . This is crucial for the analysis because, as it is explained in section 4.1, the SRS of the hardness is not equal to that of the flow stress when the

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elastic deformation in the indentation test cannot be neglected, which is the case for most polymers and ceramics.

Furthermore, following the recommendations given by Alkorta et al. (2008b), the analysis makes use of an additional hardness definition, the apparent hardness H_{ap} . It is defined as the hardness assuming that there is no pile-up or sink-in, thus the contact area is simply the area function of the indenter applied to the displacement directly measured during the test. The FEM simulations in this study use a conical indenter equivalent to a Berkovich, so the apparent hardness was calculated as:

$$H_{ap} = \frac{P}{A} = \frac{P}{\pi h^2 \tan^2 \theta} \quad (4.9)$$

The strain rate sensitivities of the hardness and apparent hardness are defined by:

$$m_H = \frac{\partial \ln H}{\partial \ln(\dot{h}/h)} \quad m_{H_{ap}} = \frac{\partial \ln H_{ap}}{\partial \ln(\dot{h}/h)} \quad (4.10)$$

Figure 4.2-a shows the results of the parametric FEM analysis, together with the literature data from Alkorta et al. (2008b) and Elmustafa et al. (2007). The results correlate well with the trends in literature. Each point corresponds to a simulated material case. The strain rate sensitivities of the hardness and apparent hardness were computed using central differences. The values for all cases of m line up in a unique curve, meaning that the ratio of SRS of the hardness to SRS of the flow stress is a unique function of the parameter H/E_{eff} . Alkorta et al. (2008b) showed that actually there is a slight dependency on m , but it can be considered negligible.

The important implication of the master curve in figure 4.2-a is that more elastic materials exhibit less sensitivity to changes in the strain rate of indentation independently on the SRS of the flow stress. If two materials had the same m value, but one of them were much more elastic than the other (higher H/E_{eff}), the SRS of the hardness would be reduced in the case of the more elastic material. Furthermore, when the ratio of m_H/m approaches zero, in the case of extremely elastic materials, no rate sensitivity would be measured, even if the material were actually strongly rate dependent (high m).

4.2-a also suggests that the SRS of the hardness is higher than that of the apparent hardness as the curve of SRS ratio using the apparent hardness is steeper.

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Figure 4.2: Ratio of strain rate sensitivity of the hardness (and apparent hardness) and the strain rate sensitivity of the flow stress obtained as a function of the dimensionless parameter H/E_{eff} . (a) Values obtained from a FEM-based parametric study using J_2 -plasticity, and compared with previous studies in literature (Alkorta et al., 2008b; Elmustafa et al., 2007). (b) Values obtained from a FEM-based parametric study using Drucker-Prager plasticity, and compared with master curves obtained in (a) and literature values.

The determination of m is thus easier from the strain rate sensitivity of the hardness because this value will always be higher than that of the apparent hardness. However, in the determination of the SRS of the hardness, the contact area needs to be estimated and this could lead to other experimental challenges.

A second set of simulations was carried out implementing a Drucker-Prager yield criterion to assess if there were changes in the master curves. Figure 4.2-b shows that there is indeed a modest deviation of the trend when using a Drucker-Prager yield, and this deviation is higher for higher friction angles, β . Alkorta et al. (2008b) indicated that the ratio of SRS of the hardness to SRS of the flow stress as a function of H/E_{eff} depends on a parameter, c_{rig} , which is a function of the SRS of the flow stress, m . Although they show that the variation of this parameter with m has a negligible influence on the master curve of figure 4.2-a, it could be the cause for the slight change in trend with β when using a Drucker-Prager model. c_{rig} is the pile-up parameter for a quasi-rigid material ($E_{eff} \gg Y$). The pile-up parameter

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Figure 4.3: (a) Pile-up parameter, defined as h_c/h_{max} , as a function of work ratio, W_e/W_t , and strain rate sensitivity of the flow stress, m (for $\beta=0$). The Oliver and Pharr (2004) estimation is also shown for comparison. (b) Pile-up parameter for quasi-rigid materials ($E_{eff} \gg Y$) as a function of strain rate sensitivity of the flow stress, m , and friction angle, β , and (*) comparison with the results of Alkorta et al. (2008b).

is defined as the ratio of contact depth, h_c , to the measured depth, h . Figure 4.3-a presents the values of the pile-up parameter as a function of m and the work ratio, in the case of no pressure sensitivity ($\beta=0$). The Oliver and Pharr (2004) estimation is also showed for comparison. Only for predominantly elastic materials ($W_e/W_t > 0.5$), i. e. dominated by sink-in, the Oliver-Pharr approach provides a reasonable estimation of the pile-up parameter, as it was also shown by Cheng and Cheng (2004). The values of pile-up parameter for a quasi-rigid material can be estimated by extrapolating the curves to $W_e/W_t = 0$. This operation is repeated for the other sets of curves corresponding to different values of β . Figure 4.3-b shows the value of c_{rig} as a function of friction angle and m . Indeed, there is a significant change in this parameter with the friction angle, which can be the cause for the slight deviation shown in figure 4.2-b. However, the effect is so small that it can be safely neglected in the analysis.

4.3.3 Results: constraint factor

The second step in the inverse analysis methodology to extract viscoplastic properties of materials is the determination of the constraint factor, i. e. the relation

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Figure 4.4: Constraint factor (for apparent hardness) as a function of H_{ap}/E_{eff} , strain rate sensitivity of the flow stress, m , and friction angle, β .

between hardness and yield stress. The dimensional analysis in section 4.2.2 has shown that there are three parameters to be determined (m , β and σ_y), but only two independent material metrics can be extracted from indentation (H and E_{eff}). One of the material parameters needs to be known beforehand to arrive to a unique solution. To this end, in the inverse analysis methodology developed in this work, it was assumed that the pressure sensitivity parameter was known and it is, hence, an input of the analysis.

The parametric FEM analysis carried out to determine the master curves of con-

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straint factor consisted of four sets of simulations, each one for a specific value of friction angle (0, 10, 20 and 30). The master curves for apparent hardness and real hardness are presented in figures 4.4 and 4.5, respectively. Both curves exhibit a similar trend. The constraint factor decreases with the increase in the parameter H/E_{eff} , which is directly proportional to the plasticity index, σ_y/E_{eff} , and the elastic-to-total work ratio, W_e/W_t . This result is in line with previous studies that have used dimensional analysis for inverse extraction of mechanical properties (Cheng and Cheng, 2004). Only in the limit of very plastic materials ($H/E_{eff} < 10^{-2}$), the constraint factor becomes nearly independent on the elastic deformation of the material through H/E_{eff} . In this case, the analysis of Cheng and Cheng (2004) is recovered (only for $\beta=0$) in which the constraint factor is a function of m and θ . Also in this limit of very plastic materials, and in the case of $\beta=0$ and $m=0$, the well-known Tabor's relation is recovered, i.e. $H/Y \approx 3$. Furthermore, figures 4.4 and 4.5 indicate a decrease in the constraint factor with the increase of SRS of the flow stress, which is opposite to the trend found with friction angle. The constraint factor increases for higher friction angles. This is in line with the observations made by Rodríguez et al. (2012a).

These master curves also exhibit the decrease in SRS of the hardness for very elastic materials, in line with the master curve in figure 4.2. This can be noticed in the trend curves of figure 4.4. The curves are closer together for higher values of H_{ap}/E_{eff} . In the limit of a fully elastic material (Sneddon's solution), the material is likely to show no rate sensitivity in the hardness, even if the material has a strongly rate dependent flow stress.

In certain occasions, it may be more convenient to employ the ratio of elastic-to-total work, W_e/W_t , instead of the parameter H/E_{eff} as dimensionless indentation metric. Cheng and Cheng (2004) showed that there exist a unique relation between these two dimensionless material metrics. This implies that either the elastic modulus or the hardness can be calculated if the other value is known, without the need of estimating the contact area. In the case of materials in which the yield stress is a power-law function of strain rate, Alkorta et al. (2008b) showed that the relation between H/E_{eff} and W_e/W_t depends on the SRS of the flow stress, m , through the parameter c_{rig} , the pile-up parameter for quasi-rigid materials. Both dimensionless metrics have been compared to evaluate if the friction angle or the exponent m have an effect. The curves are shown in figure 4.6. In line with the observations of Cheng and Cheng (2004), it seems that the work ratio is a unique function of the parameter

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Figure 4.5: Constraint factor (for real hardness) as a function of H/E_{eff} , strain rate sensitivity of the flow stress, m , and friction angle, β .

4.4. Inverse analysis procedure and special considerations in the use of combined nanoindentation and nano-impact testing

Figure 4.6: Relation of H/E_{eff} (and H_{ap}/E_{eff}) factor and elastic-to-total work ratio W_e/W_t for all simulated cases ($\beta=0, 10, 20$ and 30 , and $m=0, 0.04, 0.08, 0.12, 0.16$ and 0.2).

H/E_{eff} . A slight dependency with m is observed in the detail of the curve for real hardness, but as a first approximation it can be considered negligible. Taking the average value in the curve for real hardness will only induce an error of about 5 %. In the other hand, the relation between work ratio and the parameter H_{ap}/E_{eff} in the case of apparent hardness is reasonably unique. Therefore, the relations in figure 4.6 can be used to swap between both dimensionless metrics, which gives more versatility to the inverse analysis methodology presented in this chapter.

4.4 Inverse analysis procedure and special considerations in the use of combined nanoindentation and nano-impact testing

The FEM simulations involved in the development of the inverse analysis methodology presented in section 4.3 assumed constant strain rate of indentation, which in the case of self-similar indenters is defined as $\dot{h}/h$. This implies that experiments should be carried out also under conditions of constant strain rate in order to fulfill the assumptions of the inverse analysis. However, this is not the case when using the nano-impact technique presented in chapter 3. Figure 3.5 showed that the strain rate of indentation continuously decreases throughout the contact in a nano-impact test, and this is because of the ever decreasing speed during the test as the indenter

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Figure 4.7: Outputs of a set of FEM simulations of combined nanoindentation and nano-impact. The material set is $E = 5 \text{ GPa}$, $k = 300 \text{ GPa} \cdot \text{s}^m$ and $m = 0.12$.

is arrested due to the material resistance. In this section, FEM simulations are used to show that it is possible to employ the nano-impact test together with conventional constant strain rate nanoindentation to extract rate dependent mechanical behaviour of a material.

In addition to the series of seven constant strain rate indentations described in section 4.3.1, FEM simulations of the nano-impact test were carried out applying the same material models. The Abaqus explicit solver was used. Three impact conditions were implemented, as in the experiments of section 3.3. The dynamic parameters (m , ρ and k) of the real instrument were applied.

Figure 4.7 presents the results of one of the simulated material cases. The trend followed by the strain rate with depth in the three nano-impact cases correlates well with the shape observed in the experiments (see figure 3.5). Interestingly, the plots of hardness and apparent hardness indicate that the instantaneous value of hardness in the nano-impact test also decreases with depth, in line with the decrease in strain rate. If the values of instantaneous strain rate and hardness for each depth point are brought together, one obtains the cloud of points in the right-hand side plot

4.5. Validation of the methodology

of figure 4.7. The cloud of points follows to a great extent the trend defined by the constant strain rate indentations in the range of $10^2 - 10^3 \text{ s}^{-1}$. The cloud of points for the real hardness case presents a higher dispersion, but this is because of the higher noise induced by the mesh size. A finer mesh would lead to lower dispersion. This plot then probes that the cloud of hardness points obtained in the nano-impact test can be used in the extraction of rate dependent behaviour from combined nanoindentation and nano-impact tests.

A power law function was fit to the points of hardness and apparent hardness as a function of strain rate to show that the SRS changes with strain rate (i. e. the points do not fit properly to the power law), in agreement with the trends revealed in figure 4.2. Higher strain rates lead to higher hardness and higher H/E_{eff} parameter, hence lower ratio of SRS of the hardness to SRS of the flow stress. Being m constant, this means that the SRS of the hardness decreases with strain rate, as indicated by figure 4.7.

4.5 Validation of the methodology

4.5.1 Using numerical data

Two sets of FEM simulations were used to verify the numerical analysis presented in section 4.3. The outputs of the simulations were post-processed and the inverse analysis was applied in the same way as it would be done in an experiment. The two material sets are:

- Linear elastic isotropic material with Young's modulus of 30 GPa and Poisson's ratio of 0.3. Plasticity is defined by a von Mises yield criterion with no hardening and the flow stress is given by a power law function of the strain rate: $\sigma = 300\dot{\epsilon}^{0.06}$. This material model represents the typical mechanical behaviour of some metals.
- Linear elastic isotropic material with Young's modulus of 5 GPa and Poisson's ratio of 0.3. Plasticity is defined by a Drucker-Prager yield criterion with no hardening, friction angle of 20° and the flow stress is given by a bilinear power law function of the strain rate: $\sigma = 150\dot{\epsilon}^{0.03}$ for $\dot{\epsilon} < 1$ and $\sigma = 150\dot{\epsilon}^{0.06}$ for $\dot{\epsilon} > 1$. This material model could easily represent the typical mechanical behaviour of certain polymers.

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a) Material set 1

b) Material set 2

Figure 4.8: Predicted flow stress by application of the inverse analysis in section 4.3 to the FEM simulation results used for validation. Plots also include the evolution of the SRS of the apparent hardness with strain rate.

Figure 4.8 contains the results of the application of the inverse analysis to the two simulated material cases. The predicted flow stress values agree perfectly with the input. Furthermore, this verification exercise extends the capability of the methodology presented in this chapter. The inverse analysis was used to extract a bilinear power-law function in which the strain rate sensitivity of the flow stress, m , changes from 0.03 to 0.06 at $\dot{\epsilon} = 1$. Therefore, even if a power-law function with a single value of m was assumed for the analysis (as in equation 4.1), these results probe that different functions of flow stress with strain rate can be obtained.

Plots showing the evolution of SRS of the apparent hardness with strain rate have been also embedded in figure 4.8. The material set 2 presents a lower SRS of the hardness and a more abrupt change with strain rate compared to the trend in the material set 1. This agrees with the curves in figure 4.2. The material set 2 has a higher H_{ap}/E_{eff} , hence the ratio of SRS of the hardness to SRS of the flow stress is lower.

4.5.2 Using experimental data

Three of the materials studied in section 3.3 were selected for validation of the inverse analysis methodology: PMMA, coarse grain copper (Cu0P) and ultrafine grain copper (Cu12P). In addition to the constant strain rate nanoindentation tests

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at 0.05 s^{-1} included in section 3.3, two more strain rate cases were tested: 0.005 and 0.5 s^{-1} . The rest of experimental procedures were kept constant.

Test conditions		PMMA	Cu0P	Cu12P
Nanoindentation	E_r [GPa]	4.76 ± 0.003	110.6 ± 5.0	139.6 ± 4.7
$\dot{h}/h = 0.005 \text{ s}^{-1}$	W_e/W_t	0.392 ± 0.0004	0.040 ± 0.001	0.085 ± 0.003
Nanoindentation	E_r [GPa]	4.84 ± 0.01	115.1 ± 0.0	137.1 ± 4.0
$\dot{h}/h = 0.05 \text{ s}^{-1}$	W_e/W_t	0.419 ± 0.0003	0.041 ± 0.002	0.089 ± 0.005
Nanoindentation	E_r [GPa]	4.84 ± 0.01	111.5 ± 0.0	131.7 ± 3.0
$\dot{h}/h = 0.5 \text{ s}^{-1}$	W_e/W_t	0.447 ± 0.003	0.041 ± 0.0005	0.098 ± 0.004
Nano-impact	E_r [GPa]	7.64 ± 0.51	65.8 ± 9.4	96.9 ± 39.3
$5 \text{ mN}, 20 \mu\text{m}$	W_e/W_t	0.369 ± 0.027	0.017 ± 0.016	0.039 ± 0.027
Nano-impact	E_r [GPa]	7.55 ± 0.28	76.8 ± 10.2	117.4 ± 13.3
$10 \text{ mN}, 20 \mu\text{m}$	W_e/W_t	0.367 ± 0.012	0.017 ± 0.013	0.022 ± 0.014
Nano-impact	E_r [GPa]	7.37 ± 0.18	82.1 ± 26.0	188.0 ± 47.7
$20 \text{ mN}, 20 \mu\text{m}$	W_e/W_t	0.352 ± 0.008	0.010 ± 0.014	0.015 ± 0.010

Table 4.4: Values of reduced modulus and elastic-to-total work ratio obtained from constant strain rate nanoindentation (three speeds and maximum applied force of 100 mN) and nanoimpact (impulse force and impulse distance specified in the table) of the three tested materials used for validation of the analysis methodology: PMMA, coarse grain copper (Cu0P) and ultrafine grain copper (Cu12P).

Table 4.4 compiles the outputs of the application of the standard nanoindentation analysis (see section 3.1.1) to the three tested materials. Only the highest load cases (100 mN) are included for the nanoindentation tests because the obtained maximum depths are closer to the ones of the nano-impact tests. The reduced modulus values of the two copper samples agree reasonably well with the typical modulus obtained in macroscopic test, around 120 GPa. The agreement is better in the case of the coarse grain copper, and a higher deviation is observed for the UFG copper. This is probably caused by the higher hardening rate of coarse grain copper compared to the near-zero work hardening of the nanocrystalline material (Gray et al., 1997). Work hardening is caused by the formation of dislocations that move and interact with each other. The applied stress for additional plastic deformation becomes higher because the dislocations act as obstacles for further dislocation motion. However, in nanocrystalline materials the grain size is below the characteristic length scale for the nucleation and interaction of dislocations, hence plastic deformation is con-

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trolled by diffusional grain boundary sliding (Champion et al., 2003). This leads to the low hardening rate observed in nanocrystalline materials. Previous studies have shown that a high hardening exponent leads to lower pile-up (Cheng and Cheng, 2004; Bolshakov and Pharr, 1998). The coarse grain copper adjusts better to the assumptions of the standard unloading analysis because of the lower pile-up induced by the higher hardening, hence the better correspondence of the indentation modulus with the macroscopic value. In the case of PMMA, the indentation modulus, around 4 GPa if the elastic deformation of the diamond indenter is removed, deviates significantly from the typical macroscopic value, around 3 GPa. This is in line with the observations made by other authors who also obtained indentation modulus higher than the macroscopic value in nanoindentation tests on polymers. There is no consensus in literature about the reason for this unexpected size effect. Some authors have attributed it to the fact that polymers do not fulfil the assumptions of the standard unloading analysis, which is based on elastic contact mechanics (Hardiman et al., 2017).

The values of elastic-to-total work ratio are also included in table 4.4. There is a significant deviation in the material metrics obtained in the nano-impact tests compared to the nanoindentation values. The reduced modulus of PMMA is almost twice as high. While the elastic modulus is expected to rise with strain rate in polymers and the unloading is indeed much faster in the nano-impact, this increase could be also caused by the effect of creep in the first stages of the unloading. This is a common phenomenon in indentation of polymers and occurs when the material is unloaded right after loading, as it happens in the nano-impact, and it is materialized as a "nose" in the unloading curve, which is actually evident in figure 3.4. In addition, the lower values of work ratio in the nano-impact tests of PMMA support this hypothesis. Creep (viscoplastic deformation) during the unloading leads to lower elastic work. The nano-impact reduced modulus and work ratio values in the two metals are also rather off from the nanoindentation values. In this case, the deviation is most probably caused by the effect of dynamic compliance or ringing in the unloading curve, which is also translated into a loss of energy in the impact as it is explained in section 3.4.2.

Capturing the strain rate sensitivity of the elastic modulus using the standard unloading analysis is thus a difficult task, as demonstrated from the results presented in table 4.4. In fact, one of the hypothesis of the standard unloading analysis is that the unloading should be completely elastic, with no time dependent effects.

4.5. Validation of the methodology

Therefore, the results presented in this section relied on literature to extract the strain rate sensitivity of the elastic modulus of the materials under study:

- A constant reduced modulus was assumed for the two copper samples because the elastic behaviour of metals is generally assumed to be rate independent. Therefore, the reduced modulus of the nano-impact cases used as input for the inverse analysis was obtained from the average reduced modulus of the three quasistatic cases.
- Unlike metals, the elastic modulus of most polymers is significantly rate dependent. The mechanical behaviour of PMMA has been extensively studied in the past, hence there is a great amount of publications regarding the rate sensitivity of its mechanical properties. The strain rate sensitivity of the elastic modulus was obtained from a selection of previous studies (Richeton et al., 2005; Moy et al., 2011; Wu et al., 2004). Then, the reduced modulus obtained for the 0.005 s^{-1} case was used to scale the values obtained from literature. The values used as input in the inverse analysis are: 4.76 GPa for the 0.005 s^{-1} case, 5.46 GPa for the 0.05 s^{-1} case, 6.26 GPa for the 0.5 s^{-1} case and 8.79 GPa for the nano-impact case.

The second set of outputs from the combined nanoindentation and nano-impact tests are the distributions of apparent hardness and strain rate as a function of depth, included in figure 4.9. The PMMA and UFG copper samples do not exhibit a significant size effect. The nano-impact curves exhibit more size effect. In the UFG copper, this could be due to uncertainties in the zero-load determination, which is necessary in the nano-impact tests (see section 3.2). However, in the PMMA most of it comes from the rate dependent behaviour of the material that leads to higher hardness for higher strain rates. The coarse grain copper presents a significant size effect. The same trend is followed by the nano-impact and nanoindentation results. This pronounced size effect is typical in metallic materials and is generally represented by the Nix and Gao (1998) model based on the concept of geometrically necessary dislocations. This size effect is strongly reduced in the UFG material due to the much lower internal length scale and higher dislocation density (Maier et al., 2011; Durst et al., 2005).

The inverse analysis methodology presented in section 4.3 was applied to the experimental results to extract the flow stress as a function of strain rate in the three

4. Analysis framework for the extraction of rate dependent constitutive behaviour from nanoindentation

Figure 4.9: Distributions of apparent hardness as a function of depth (left) and distributions of strain rate as a function of depth (right) for three of the tested materials in section 3.3.

4.5. Validation of the methodology

materials. The master curve in figure 4.2 was applied first. The strain rate sensitivity of the apparent hardness was computed by central differences, and forward and backward differences for the first and last hardness points, respectively. Figure 4.10-a shows the distributions of apparent hardness as a function of strain rate for the three materials. An average value was computed in the nano-impact cases and it was used for the inverse analysis instead of the cloud of points, for simplicity. Finally, the master curves of figure 4.4 were used to derive the constraint factor. In addition to the experimental inputs, the inverse analysis of PMMA required an extra parameter, the friction angle that was assumed to be 20° . Rittel and Brill (2008); Bardia and Narasimhan (2006); Quinson et al. (1997) all obtained a pressure sensitivity parameter equivalent to 20° friction angle in a Drucker-Prager model. Furthermore, Rittel and Brill (2008) found that the pressure sensitivity parameter slightly decreases with strain rate, but argued that there are not many studies in literature regarding the strain rate sensitivity of the pressure sensitivity parameter and, therefore, did not make any conclusions from the dependency they found.

Figure 4.10 presents the results of the inverse analysis. The estimations of flow stress in PMMA agree reasonably well with the literature values. The results show a substantial rate dependent behaviour. This is typical in polymers and it is attributed to the characteristic times for activation of the different motions (rotations, chain alignment, etc.) of the molecular chain that constitutes the polymer. The estimated value of flow stress for the nano-impact is slightly off from the trend defined by the three low speed values. This could be due to uncertainties in the reduced modulus used for the inverse analysis, which was estimated from literature.

The estimated flow stress for the two metals is also close to the literature values, although the deviations are higher compared to the PMMA. In the case of coarse grain copper, it is not surprising that the estimations do not agree perfectly well with literature. This material presents a high hardening rate; however, the inverse analysis methodology was built around a FEM model in which perfect plasticity has been assumed. Secondly, the literature values shown in the plot correspond to a specific value of strain, 15 %. Tabor (1951) postulated that the representative strain for Vickers indenters (equivalent to Berkovich) is about 8 %, so it makes sense that the estimation is below the literature value. In any case, the trend of flow stress with strain rate follows the expected behaviour. The flow stress is nearly independent on strain rate up to around $10^3 - 10^4 \text{ s}^{-1}$, when it start increasing abruptly. This has

4. Analysis framework for the extraction of rate dependent constitutive behaviour from nanoindentation

Figure 4.10: (a) Apparent hardness as a function of strain rate obtained in the combined nanoindentation and nano-impact tests of PMMA, coarse grain copper (Cu0P) and ultrafine grain copper (Cu12P). (b), (c) and (d) show the results of applying the inverse analysis presented in this chapter to the tested materials. Results are compared with literature (Mulliken and Boyce, 2006; Richeton et al., 2006; Rittel and Brill, 2008; Jordan et al., 2014; Follansbee and Kocks, 1988; Croteau et al., 2019; Nemat-Nasser and Li, 1998; Mishra et al., 2008; Gray et al., 1997). Literature results for coarse grain copper correspond to the flow stress at 15% deformation, and 10% in the case of ultrafine grain copper.

4.6. Concluding remarks

been attributed to the increase in the dislocation accumulation rate when the strain rate reaches those high values, leading to a higher strain rate sensitivity (Follansbee and Kocks, 1988).

The estimated flow stress in the UFG copper correlates better with literature, as compared with the coarse grain material. The mechanical behaviour of the UFG copper fits better the assumptions of the material model used in the FEM models of the inverse analysis because the material presents near-zero hardening. The trend with strain rate also agrees with previous studies. The UFG material has a higher strain rate sensitivity compared to the coarse grain material. Many authors have attributed this to the decrease in activation volume when decreasing the grain size, which is caused by the change in deformation mechanism from forest dislocation hardening (dislocations act as obstacles to each other) in coarse grain material to a deformation mechanism dominated by grain boundary diffusion in the UFG material (Wei et al., 2004; Mishra et al., 2008). As in PMMA, in this case the estimated flow stress value of the nano-impact also deviates to a certain extent from the trend defined by the three low speed cases. Although, in this case it is caused by the apparent hardness value in the nano-impact being almost equal to the value from nanoindentation at 0.5 s^{-1} . Rather than representing a real material behaviour, this could have been caused by some experimental uncertainty.

4.6 Concluding remarks

Inverse analyses are often used for the extraction of elastoplastic properties in instrumented nanoindentation. In the case of strain rate and pressure sensitive materials, which is the typical behaviour of polymers, the studies found in literature focused on one effect or the other, but there is no study considering the combined effect of pressure and strain rate. To this end, the work presented in this chapter has proposed a FEM-based inverse analysis methodology that captures the pressure sensitivity with a Drucker-Prager yield criterion and strain rate sensitivity by assuming that the flow stress is a power-law function of the strain rate. The analysis has shown that the ratio of SRS of the hardness to SRS of the flow stress is a nearly-unique function of the H/E_{eff} parameter, in line with the findings of previous works (Alkorta et al., 2008b). In addition, the pressure sensitivity parameter has a marginal effect on the SRS ratio compared to the effect of H/E_{eff} . Furthermore, the constraint factor (H/σ_y) was found to depend equally on the pressure sensitivity and strain rate sensitivity parameters; although, their effects are opposite. The constraint factor

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increases with the increase in pressure sensitivity parameter, while it decreases with the increase in strain rate sensitivity parameter. The inverse analysis was validated by testing three materials (coarse grain copper, ultrafine grain copper and PMMA) with a well-known rate dependent mechanical behaviour. The predictions of the analysis agreed well with macroscale results from literature.

Microscale characterization of composite constituents from low to high strain rates

The modified nanoindentation instrument presented in chapter 3 in combination with the inverse analysis methodology introduced in chapter 4 were used to determine the elastoplastic properties of the resin matrix in the two composite systems under study: IM7-8552 and IM7-M91. The results are presented in section 5.1. The predicted values of elastic modulus and flow stress are higher than the values obtained from macroscale compression in section 2.3.3. This is further explained in section 5.1. As an alternative to the nanoindentation technique, micropillar compression tests were used to characterize the elastoplastic properties of the matrix. In this case, the results showed good agreement with the macroscale compression tests. The results of the micropillar compression tests are compiled in section 5.2. Finally, section 5.3 presents the fibre-matrix interface characterization of the two composite systems under study. The fibre push-in technique was selected because of the simplicity in the sample preparation. The technique has been used for the first time to test the fibre-matrix interface at high speed, in the order of mm/s, around six orders of magnitude higher than the typical test speeds of 1-100 nm/s.

5.1. Matrix characterization using combined nanoindentation and nano-impact tests

5.1 Matrix characterization using combined nanoindentation and nano-impact tests

5.1.1 Experimental details

Samples for nanoindentation were extracted from plates of neat resin material fabricated following the same procedure used in the samples for macroscale testing (see section 2.1), making sure that the surface for testing was parallel to the back surface. The surface for testing was grounded using fine grade SiC paper, followed by polishing with an alumina suspension.

Three cases of constant strain rate nanoindentation were tested: 0.005, 0.05 and 0.5 s^{-1} . Two nano-impact conditions were tested in the 8552 resin, using an impulse force of 5 and 10 mN and an impulse distance of $20\text{ }\mu\text{m}$. In the M91 resin, the matrix and the two thermoplastic particles were tested separately. The nano-impact test on the matrix was carried out with an impulse force of 5 mN and an impulse distance of $20\text{ }\mu\text{m}$. The two thermoplastic particles were tested with an impulse force of 5 mN and an impulse distance of $5\text{ }\mu\text{m}$, to avoid having an effect of the matrix surrounding the particles. The rest of experimental parameters were the same as those explained in section 3.3.1.

5.1.2 Application of inverse analysis

The standard unloading analysis was first applied to the experimental results to obtain the elastic modulus and the elastic-to-total work ratio. The results are compiled in table 5.1. As in PMMA, the computed elastic modulus of the epoxies (around 5.2 GPa for the 8552 and around 4.2 GPa for the matrix of the M91 when subtracting the effect of the diamond indenter) is higher than the modulus obtained in the macroscale compression tests of section 2.3.3. Opposite to the results of PMMA, the obtained elastic modulus in the nano-impact cases does not increase substantially compared to the nanoindentation values. This is because the epoxy resins present a lower degree of creep upon unloading during the nanoindentation test.

The same apparent hardness (equation 4.9) and strain rate distribution plots as in section 4.5.2 were computed for the four tested materials in this section, but they are not shown. The trends were similar to the one shown by the PMMA sample. The application of the inverse analysis followed the same procedure explained in section 4.5.2, except for the estimation of the reduced modulus. The strain rate sensitivity of the reduced modulus was derived from the results presented in section 2.3.2. The

5. Microscale characterization of composite constituents from low to high strain rates

Test conditions		8552	M91-matrix	M91-P1	M91-P2
Nanoindentation $\dot{h}/h = 0.005s^{-1}$	E_r [GPa]	5.89 ± 0.01	4.96 ± 0.01	4.19 ± 0.48	5.17 ± 0.03
	W_e/W_t	0.495 ± 0.0005	0.513 ± 0.001	0.323 ± 0.030	0.404 ± 0.007
Nanoindentation $\dot{h}/h = 0.05s^{-1}$	E_r [GPa]	5.98 ± 0.02	4.94 ± 0.04	4.16 ± 0.33	5.03 ± 0.28
	W_e/W_t	0.509 ± 0.0005	0.534 ± 0.007	0.334 ± 0.022	0.415 ± 0.010
Nanoindentation $\dot{h}/h = 0.5s^{-1}$	E_r [GPa]	6.00 ± 0.02	5.28 ± 0.12	4.03 ± 0.09	5.49 ± 0.16
	W_e/W_t	0.514 ± 0.004	0.534 ± 0.003	0.316 ± 0.013	0.415 ± 0.009
Nano-impact $5mN, 20\mu m$	E_r [GPa]	6.59 ± 0.35	5.69 ± 0.39	-	-
	W_e/W_t	0.505 ± 0.022	0.501 ± 0.010	-	-
Nano-impact $10mN, 20\mu m$	E_r [GPa]	6.42 ± 0.27	-	-	-
	W_e/W_t	0.488 ± 0.009	-	-	-
Nano-impact $5mN, 5\mu m$	E_r [GPa]	-	-	4.9 ± 0.49	5.75 ± 0.46
	W_e/W_t	-	-	0.331 ± 0.040	0.399 ± 0.018

Table 5.1: Values of reduced modulus and elastic-to-total work ratio obtained from constant strain rate nanoindentation (three speeds) and nanoimpact (impulse force and impulse distance specified in the table) of 8552 and M91 resin systems.

modulus was scaled with the reduced modulus values at $0.005 s^{-1}$. The estimated reduced moduli for the 8552 are: 5.89 GPa for the $0.005 s^{-1}$ case, 6.03 GPa for the $0.05 s^{-1}$ case, 6.18 GPa for the $0.5 s^{-1}$ case and 6.64 GPa for the nano-impact case. The estimated reduced moduli for the M91 matrix are: 4.96 GPa for the $0.005 s^{-1}$ case, 5.07 GPa for the $0.05 s^{-1}$ case, 5.18 GPa for the $0.5 s^{-1}$ case and 5.5 GPa for the nano-impact case. The estimated reduced moduli for the M91 particles, P1 and P2 respectively, are: 4.19 and 5.17 GPa for the $0.005 s^{-1}$ case, 4.29 and 5.29 GPa for the $0.05 s^{-1}$ case, 4.38 and 5.4 GPa for the $0.5 s^{-1}$ case and 4.64 and 5.73 GPa for the nano-impact case.

Figure 5.1 presents the results of the inverse analysis. The two thermoplastic particles present in M91 are considerably softer than the matrix, as revealed in the estimations of figure 5.1, hence they lead to a lower macroscopic flow stress. The estimated flow stress values for the 8552 resin and the matrix of the M91 resin deviate significantly from the values obtained in the macroscale compression tests of section 2.3.3 and the deviation increases with strain rate. The fact that the deviation increases with strain rate may suggests that the estimated reduced modulus used as input in the analysis does not capture properly the real viscoelastic behaviour of

5.1. Matrix characterization using combined nanoindentation and nano-impact tests

Figure 5.1: (a) Apparent hardness as a function of strain rate for the two tested neat resins 8552 and M91 (matrix and thermoplastic particles). (b), (c) and (d) are the results of the application of the inverse analysis devised in chapter 4 to the combined nanoindentation and nano-impact tests. The estimations of the inverse analysis are compared against the macroscale compression results presented in chapter 2.

the resins. The estimation of the flow stress of PMMA in section 4.5.2 agreed well with the literature value, but in that case the strain rate sensitivity was obtained from literature values obtained from conventional mechanical testing. However, the estimations here for the two epoxies rely on the results of section 2.3.2 that were obtained from the application of the TTSP to DMA results and are subjected to many assumptions and experimental uncertainties. In the other hand, the increasing deviation with strain rate could be due to limitations of the inverse analysis. The analysis does not account for viscoelastic effects in the material and it assumes perfect plasticity.

5.2 Matrix characterization using micropillar compression

5.2.1 Introduction

Section 4.1 highlights the challenges of extracting constitutive behaviour from instrumented nanoindentation using pyramidal indenters. The determination of constitutive behaviour has proven to be especially challenging due to the large strain gradients and complex stress states that develop in the material during indentation. This issue was overcome with the pioneering work of Uchic et al. who demonstrated the possibility of fabricating micron-size pillars in a material surface that could be tested with a conventional nanoindentation system (Uchic et al., 2004; Dimiduk et al., 2005). This way, the stress and strain are uniform along the tested volume and the uniaxial constitutive behaviour can be directly probed, without the need of a complex inverse analysis. Since then, the micropillar compression technique has been applied to study the mechanical behaviour of all classes of materials: from the study of size-dependent deformation in metals (Maaß et al., 2009; Lee and Nix, 2012), ceramics (Michler et al., 2007; Östlund et al., 2009) and polymers (Wang et al., 2012; Xu et al., 2016; Guruprasad et al., 2016), to the calibration of micromechanical models of polycrystalline metals (Cruzado et al., 2015) and fibre-reinforced polymer composites (González et al., 2017; Herráez et al., 2017; Naya Montáns, 2017). An area that has received less attention is the study of high strain rate behaviour, due to the absence of suitable instrumentation for obtaining reliable data. Recently, several authors have demonstrated the capabilities of a novel nanoindentation device to perform high strain rate micropillar compression (see Section 1.4). The modified nanoindentation device presented in section 3.2 was used here to carry out impact micropillar compression tests.

5.2.2 Simulation of the micropillar compression test

Finite element analysis (FEA) was used to study the influence of experimental defects in the extraction of mechanical properties from the micropillar compression tests. In particular, the effects of taper and pillar misalignment were analysed. To this end, finite element models were developed within the framework of the commercial software package Abaqus. 2D axisymmetric models were used for the study of taper effect and 3D models for assessing the effect of misalignments. CAX4 elements were used for the axisymmetric models and C3D6/C3D8 for the 3D models. In both cases, the pillar was modelled with a refined mesh while coarser elements

5.2. Matrix characterization using micropillar compression

Figure 5.2: Effect of taper in the mises stress distribution along the pillar.

were used for the rest of the domain. The domain is large enough to avoid boundary effects. Simulations of constant low displacement rate micropillar compression and impact micropillar compression were carried out using the implicit and explicit solvers, respectively. Three displacement rate cases were simulated in the case of low speed compression and one impact condition for the high speed case. Nonlinear, large deformations analysis was set up in both cases. For the formulation of the contact problem in the 3D models, a slight penalty friction coefficient of 0.1 was employed to improve the convergence of the models and avoid premature buckling of the pillars.

Regarding the material model, isotropic linear elasticity was implemented together with a Drucker-Prager yield and flow rule with no hardening and compression yield stress dependent on strain rate (see section 4.2.2 for further information about the material model). A power law strain rate dependence was assumed.

Ideal micropillars should have a constant cross-sectional area along their length. This results into an homogeneous stress distribution and the stress-strain relation can be easily extracted from the direct test results. However, the manufacturing of perfectly straight micropillars is very challenging and normally they present a tapered shape, being the bottom diameter generally bigger than the top diameter. This leads to an inhomogeneous stress field in the pillar, being the stress at the top higher than at the bottom, as it is shown in figure 5.2. The simulations were thus used to assess the effect of taper on the extracted mechanical properties. The computation of mechanical properties was carried out using the top diameter, which is the conventional approach. Figure 5.3 shows the effect of taper angle on the two main extracted mechanical properties, which are the maximum compression

5. Microscale characterization of composite constituents from low to high strain rates

Figure 5.3: Effect of taper angle on the extracted mechanical properties when using top diameter and correction by using the average diameter.

stress as a function of strain rate and the elastic modulus. Both values are known because they are an input of the FEM simulations, so they can be compared with the values obtained by applying the conventional data reduction method to obtain stress and strain in micropillar compression (see section 5.2.4). The extracted values of maximum compression stress and elastic modulus increase with higher taper angles. This is due to the higher stresses at the top of the pillar. The deviation can be as high as 30% in elastic modulus and around 50-100 MPa in maximum compression stress for 3° taper, which is a typical taper obtained experimentally.

There are several approaches to correct for the effect of taper angle in the data reduction process. One way is to use an average diameter of the pillar, computed as $(D_T + D_B)/2$ where D_T and D_B are the top and bottom diameters, respectively.

5.2. Matrix characterization using micropillar compression

Figure 5.4: Effect of misalignment on the stress-strain curves obtained in micropillar compression.

Figure 5.3 shows the extracted mechanical properties if the average diameter is used instead of the top diameter. In this case, the extracted properties correlate well with the input material behaviour. This proves the validity of using an average diameter instead of the top diameter for tapered pillars.

FEA simulations were also employed to study the effect of misalignment between the flat punch and the top surface plane of the micropillar. As for the taper, misalignments are common in the micropillar compression tests and difficult to avoid. Figure 5.4 presents the effect of misalignments of the pillar plane with respect to the punch plane. Overall, misalignment results into an underestimation of flow stress and elastic modulus, but the deviation is not significant up to around 3° . Higher misalignment angles lead to large deviations on the extracted stress-strain relations.

5.2.3 Experimental procedures

Micropillars of the two resins under study, 8552 and M91, were prepared from the material in neat resin form. The micropillars were carved on the same type of polished plates prepared for nanoindentation (see section 5.1.1). Neat resin plates for micropillar compression were manufactured following the same procedure used in the plates for macroscale testing specimens (see section 2.1). The specimens were coated with a few nm thick gold layer to avoid charging effects during the fabrication of the micropillars. The micropillars were manufactured using a Helios NanoLab DualBeam 600i of Thermo Fisher Scientific (USA), equipped with a Focused Ion Beam (FIB). Cylindrical pillars were milled by annular milling in a 3-steps procedure

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detailed in table 5.2. This procedure resulted in pillars of about 7 μm top diameter, aspect ratio around 3 and a slight taper angle of 1-2°. Pillars in the M91 resin were manufactured in particle-free areas to capture the mechanical behaviour of the resin matrix, without the influence of the softer thermoplastic particles.

	Beam parameters	Milling type	Geometry parameters
Step 1	30 kV, 21 nA	Ga^+ ion beam in annular configuration	I:10 μm , O:35 μm
Step 2	30 kV, 9.3 nA	Ga^+ ion beam in annular configuration	I:9 μm , O:30 μm
Step 3	30 kV, 0.43 nA	Ga^+ ion beam in annular configuration and assisted by $(\text{MnSO}_4) \cdot 7\text{H}_2\text{O}$ gas	I~7 μm , O~15 μm

Table 5.2: Steps for fabrication of epoxy micropillars in 8552 and M91 resins. "I" stands for inner diameter and "O" for outer diameter.

Micropillar tests were performed on the modified NanoTest instrument described in section 3.2. A diamond flat punch with a diameter of 10 μm was used. Low strain rate micropillar compression tests were carried out at two low speed conditions, 10 and 100 nm/s, for the 8552 resin and three conditions 10, 100 and 500 nm/s for the M91 resin. A closed-loop displacement-controlled scheme up to a maximum displacement of 5 μm was used. High strain rate micropillar compression tests were carried out with the impact configuration of the instrument setting the impulse force to 2 and 5 mN and impulse distance to 20 μm . This produced an initial impact velocity of about 0.5 and 0.9 mm/s, respectively.

5.2.4 Results and discussion

True stress and strain were calculated assuming that the tested volume is kept constant:

$$\sigma_{true} = \frac{4P}{\pi D^2} \left(1 - \frac{u - u_b}{L} \right) \quad (5.1)$$

$$\epsilon_{true} = -\ln \left(1 - \frac{u - u_b}{L} \right) \quad (5.2)$$

5.2. Matrix characterization using micropillar compression

Figure 5.5: Stress-strain curves from micropillar compression test over a wide range of strain rates (10^{-3} to 10^2 s^{-1}) and true strain rate registered throughout the test.

P is the applied force and u is the total displacement measured at the top surface of the pillar. u_b is the displacement of the pillar base and was estimated using Sneddon's correction, which assumes elastic deformation of the base, $u_b = (1 - \nu_s^2)P/(E_s D)$ (Sneddon, 1965; Zhang et al., 2006). E_s and ν_s are the sample's Young's modulus and Poisson's ratio, respectively, L is the pillar length and D is the pillar diameter. The top diameter is commonly used for the calculations; however, the simulations in section 5.2.2 have shown that this overestimates the stress in tapered pillars, a phenomenon that has been described too in literature (Zhang et al., 2006). This was corrected in this work by using an average diameter, computed as $D_t + L \tan \theta$, being D_t the top diameter, an approach that has been followed before (Xu et al., 2016; Fei et al., 2012).

Figure 5.5 plots the stress-strain curves and strain rates for the micropillar in all loading conditions and for all repeats. The strain rate achieved when using the impact configuration, of the order of 10 - 100 s^{-1} , was about four orders of magnitude

5. Microscale characterization of composite constituents from low to high strain rates

Figure 5.6: SEM images of a pillar before testing (a), the same pillar after low speed test (b) and a pillar after impact test (c).

larger than the strain rate obtained for the low speed cases. Besides, the strain rate was fairly constant throughout the strain range of interest. Even higher strain rates can potentially be achieved in the impact configuration by impacting the pillar with higher velocities; however, this was not feasible due to limitations in the data acquisition rate of the current configuration. In addition, the curves in figure 5.5 show a significantly noisier response at high strain rates. This is because the measurement was performed near the sensitivity limit of the force sensor (the noise floor was of the order of ± 0.5 mN for the current setup), while in the low speed cases, the force is actuated and not measured. Nevertheless, the curves were of sufficient quality for the extraction of mechanical properties. Regarding the reproducibility of the results, there was a significant scatter in the stress-strain curves performed at the same strain rate. The scatter is inherent to any microscale testing technique and is consistent with the behaviour observed in other epoxy micropillars fabricated by FIB milling (Wang et al., 2012; Naya Montáns, 2017). The major potential challenges to reduce the scatter of micropillar compression tests in a homogenous material like an epoxy resin are the control of misalignment during the compression tests and the precise measurement of the micropillar dimensions. Based on the FEM simulations (see section 5.2.2), alignment errors are not the major source of scatter. As a matter of fact, it was found that scatter due to alignment errors is not very significant, of the order of 10%, for a realistic maximum misalignment angle between the flat punch axis and the pillar axis of 5° . On the contrary, the precise measurement of the micropillar dimensions remains experimentally elusive, due to the irregularities produced by FIB milling at the pillar base, it is difficult to measure the bottom diameter and the micropillar height precisely (see for instance the example of an as-prepared

5.2. Matrix characterization using micropillar compression

micropillar in figure 5.6-a). Using lathe FIB milling instead of an annular strategy could potentially facilitate a more accurate description of the micropillar geometry; however, this method is much more time consuming. Other authors have proven an excellent reproducibility of results in polymer micropillars fabricated by other methods, like lithography (Xu et al., 2016) or by direct moulding of the polymer at the microscale (Guruprasad et al., 2016). However, the FIB process has the advantage of fabricating pillars *in situ* in selected locations within a desired microstructure, such as in the resin pockets of fibre-reinforced polymer composites (Naya Montáns, 2017), which cannot be done by any other microfabrication methods.

The stress-strain curves in figure 5.5 exhibited the typical deformation behaviour of epoxy resins under compression loading. The first part was linear, corresponding to the elastic deformation of the material, up to the onset of plastic deformation. The deformation then became nonlinear and the stress increased up to the maximum compressive stress, followed by a slight strain softening. The epoxy specimens tested in compression at the macroscale exhibited the same behaviour, as shown in the stress-strain curves presented in section 2.3.3. Furthermore, the shape of the low speed curves was in good agreement with the work of Naya Montáns (2017) who tested one of the epoxy resins analysed in the present study, the 8552.

The two important mechanical properties that can be extracted from the stress-strain curves obtained from the micropillar compression tests are the elastic modulus and the maximum compressive strength, which are presented in figure 5.7 as a function of true strain rate. The elastic modulus was obtained by fitting a linear polynomial to the first part of the curve (in the strain range from 1 % to 1.5 %) whereas the strength was obtained as the average stress in the near-plateau region after the maximum stress (in the strain range from 10 % to 20 %).

The mechanical properties obtained in the micropillar compression tests of the 8552 resin correlated well with those obtained from the macroscale compression tests. The maximum compression stress obtained with the macroscale compression tests exhibits a variable strain rate sensitivity with strain rate. At quasi-static strain rates, the strain rate sensitivity was 0.03. However, the results at higher strain rates indicated a higher rate sensitivity of 0.05. The transition in strain rate sensitivity occurred at strain rates of around $10^{-1}s^{-1}$. As explained in section 2.3.3, this bilinear behaviour has been reported before in epoxy resins and has been attributed to the effect of molecular motions on the mechanical response (Jordan et al., 2008). Regarding the elastic modulus, results correlated well too, but did not

5. Microscale characterization of composite constituents from low to high strain rates

Figure 5.7: Comparison of maximum stress and elastic modulus versus true strain rate obtained from micropillar compression and macroscale compression.

capture any substantial effect of strain rate in elastic modulus, while the macroscale data exhibits certain increase in the modulus. This could be caused by a lack of resolution in the nano-impact results in the significantly small strain range in which modulus is computed.

The results of the M91 resin revealed the clear softening effect of the thermoplastic particles on both the elastic modulus and maximum stress when testing the material at the macroscale. Nanoindentation testing in section 5.1 showed that the two types of thermoplastic particles in the M91 resin are significantly softer than the matrix. The micropillar compression tests hence enabled the extraction of the constitutive behaviour of the matrix in the M91 resin, which is the one needed for the micromechanical simulations of the composite ply in chapter 6. These results highlight the potential of the technique to test small volumes that could not be tested otherwise by macroscale test techniques.

The close correlation found between results obtained from both micropillar and macroscale compression tests in the 8552 resin, for which macroscale results are not affected by thermoplastic particles softening, raises the question about whether the comparison of mechanical properties at the two scales is valid due to size effects.

5.3. Fibre-matrix interface characterization

Wang et al. (2012) studied the issue of size effects in epoxy resins and found that they only manifested at the submicron scale and attributed this to the lower probability of percolations of weak bonds in the epoxy molecular network. Naya Montáns (2017) studied size effects for micrometer size micropillars on the 8552 epoxy and found that the maximum compressive strength was significantly size dependent. They attributed this to the confining effect of a stiff skin around the pillar formed due to Ga⁺ ion implantation during the FIB milling process. This stiff skin also appeared in the micropillars made in the present study, as revealed by the surface wrinkles that appeared on the micropillar surface after low speed deformation (red arrows in figure 5.6-b). At high speeds, the skin spallation was not perceived because the maximum displacement could not be prescribed, as it is an impact test, and therefore, the pillars were severely deformed, as shown in 5.6-c. In any case, Naya Montáns (2017) determined that the skin-induced size effect only introduced size effects for pillar diameters below 6 μm . Since the diameter of the pillars manufactured in this study was around 7 μm , the current micropillar results are free of size effects, and hence, their close correlation with the macroscale tests obtained in figure 5.7 constitutes a further experimental validation of the new instrumentation for impact micropillar compression presented in this work.

5.3 Fibre-matrix interface characterization

5.3.1 Overview of techniques for fibre-matrix interface characterization

There is no standard for testing the mechanical behaviour of the fibre-matrix interface in FRP composites. However, several techniques have been proposed over the years to test the fibre-matrix interface. These techniques can be classified in two main groups. *Ex situ* techniques that test the fibre-matrix interface using a specific test set-up or specimen that do not come from the real composite laminate. *In situ* techniques, in which the test specimen is extracted from the composite laminate, hence the obtained interface properties are likely to be more representative of the real interface behaviour.

The fibre fragmentation test is one of the *ex situ* techniques (Kelly and Tyson, 1965; Zhou et al., 2001). A single fibre is embedded on a neat resin specimen shaped as a dog-bone sample for tensile test. Since the matrix is more ductile than the fibre, the fibre will break in several fragments upon loading in tension. For the analysis of this test, it is required to measure the complete stress-strain history and to monitor

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the fibre breaks and the length of the fibre fragments. This can be done using optical techniques in the case of a transparent matrix, or acoustic emission techniques in the case of an opaque matrix. Another *ex situ* technique is the fibre pull-out test (Favre and Perrin, 1972). In this test, a single fibre is partially embedded on a resin block. The fibre is loaded in tension while the matrix block is gripped. Force applied to the fibre is continuously monitored and decohesion occurs when the fibre interface strength is reached. This technique, however, is difficult to apply to fibres of small diameter due to the limitation on the length of the embedded fibre. For this reason, a variant of this method was proposed, the microbond test (Miller et al., 1987). In this case, a droplet of resin is deposited on a single fibre and cured. The fibre is then pulled while the cured resin droplet is constraint. Applied force in the fibre is continuously monitored and the data is analysed using a similar procedure than in the classical pull-out test.

Despite the large amount of *ex situ* techniques that have been proposed, the test set-ups are normally difficult to carry out. In addition, it is unsure if the interface behaviour obtained with these techniques is really representative of the interfaces in the real composite laminate. This is because only a single fibre is being tested. The influence of surrounding fibres or thermal residual stresses from the manufacturing process is not taken into account. For this reason, *in situ* techniques have been also developed. There are two: fibre push-in test (Rodríguez et al., 2012b) and fibre push-out test (Marshall, 1984; Canal Casado, 2011).

The fibre push-in and push-out tests are both based on the use of an instrumented indentation device that continuously monitors force and displacement, while the indenter pushes the fibre into the cross-section of a composite laminate. The test specimen is extracted from the composite laminate by preparing a cross-section surface of the laminate. In addition, for the push-out test a very thin cross-section surface of the composite laminate is prepared, the thickness being in the order of magnitude of the fibre diameter. The test finishes with the complete debonding of the fibre, that comes out the other end of the thin specimen. The specimen preparation is complex and time consuming because the thickness of the composite laminate slice needs to be reduced to few μm . This massive reduction of laminate thickness might have an effect on the residual stress created in the fibre-matrix interface after cooling down from curing temperature. Furthermore, bending of the very thin specimen may trigger the early initiation of damage in the face opposite to the indentation due to radial tensile stresses, or overestimate the strength due

5.3. Fibre-matrix interface characterization

to high compressive stresses in the surface in contact with the indenter. The fibre push-in test does not present this problem because the specimen thickness is that of the laminate from where it was extracted. However, as the fibre is pushed into the surface, there is no information about the length of debonded surface. This makes the analysis more difficult. The fibre push-out test does not present this problem because the entire interface fails in the test, and the interface strength can be easily computed from the maximum force and the interface area. Recently, Ghaffari et al. (2021) have developed a novel fibre push-out test in which a cave is carved in the composite cross-section using a femto-s laser. The hole span covers several fibres and it is deep enough to allow the full decohesion of the pushed fibre. Unlike the traditional push-out test, this technique does not need the preparation of a few μm thick specimen, which is the main advantage of this novel technique.

Most of the studies dealing with fibre-matrix interface characterization in composites have focused on quasistatic loading conditions. The study of dynamic conditions was limited, mostly due to the lack of suitable experimental techniques. Even so, the important role of the fibre-matrix interface as energy absorbing mechanism in composites subjected to high speed events (e. g. impacts) has motivated several researchers to conceive techniques for characterizing the fibre-matrix interface at high strain rates. Greenfield et al. (2000) carried out high speed fibre fragmentation tests using a pendulum-type impact tester. One of the sides of the dogbone specimen was fixed to the pendulum. The other was free and had a crosspiece attached that hit a stationary anvil when the pendulum swung towards its equilibrium position. They could achieved strain rates in the order of $50\text{-}100\text{ s}^{-1}$ with this novel technique. Tanoglu et al. (2000) proposed a methodology to perform fibre push-out tests at speeds up to about 3 mm/s using a custom-built apparatus that incorporated a piezo-actuator and a high frequency response load cell. They observed an increase in interface shear strength with displacement rate. Tamrakar et al. (2016) presented a method to test the interface under shear loading at speeds up to 1 m/s . They modified a Split-Hopkinson bar in tension configuration in which they attached a microdroplet specimen (microbond test) to the incident bar and the opposite fibre end to a load cell. In line with the observations of Tanoglu et al. (2000), they found that the interface shear strength increased by a factor of around 2 over six orders of magnitude of displacement rate. Other authors have followed a similar approach, i. e. using a modified Kolsky bar, to study the fibre-matrix interface. Chu et al. (2018) performed the fibre pull-out test at speeds of about $2\text{-}5\text{ m/s}$, combined with

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synchrotron X-ray imaging to study the initiation and evolution of damage in the interface. In a later study, the authors used the same set-up to study the normal interface strength using a cruciform specimen (Chu et al., 2020).

After review of the main techniques used for fibre-matrix interface characterization of composite materials, the fibre push-in test was selected. Sample preparation is easier to perform than for the push-out test and there are already analysis methodologies in literature that make computation of interface shear strength more straightforward (Naya Montáns, 2017; Rodríguez, 2012). In the present study, the nano-impact test is applied for the first time to the fibre push-in technique. This is to assess if, in combination with the traditional nanoindentation test, the fibre push-in technique can be performed over a wide range of displacement rates (from nm/s to mm/s).

5.3.2 Push-in test for characterization of fibre-matrix interface shear strength

Experimental procedures

Specimens for fibre push-in test were extracted from laminates of IM7-8552 and IM7-M91. The laminates were cut in such a way that the surface to be tested contained several plies with fibres perpendicular to the surface. The surface was ground/polished to a fine degree using SiC paper and an alumina suspension.

Push-in tests were performed with a diamond flat punch tip of 5 μm . It is slightly smaller than the fibres, which have an average diameter of 5.2 μm . A punch of size near the fibre diameter reduces the chances of plastically deforming the fibre surface during indentation. Figure 5.8 shows the schematics of the push-in test. When indenting the fibre, the surrounding matrix also deforms elastically due to the large mismatch of properties between the fibres and the polymeric matrix. Indented fibres were always in a close-packed configuration consisting of the central indented fibre and six surrounding fibres in a hexagonal pattern, as it is specified in figure 5.8. This way the indented fibre had always a constant surrounding environment. Furthermore, this configuration has been used in FEM simulation of the push-in test aiming at extracting properties of the interface (Naya Montáns, 2017; Rodríguez, 2012).

Tests were carried out on the two composite systems in a Hysitron TI-950 Tri-

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Figure 5.8: Schematics of the push-in test.

boIndenter of Bruker (USA). A displacement-controlled linear scheme at 30 nm/s was applied. Around ten indentations were performed in each material.

Results and discussion

The analysis of the push-in test is based on the extraction of two material metrics from the load-displacement curve: the stiffness and the critical load that marks the onset of nonlinearity. There is no consensus on how to extract stiffness and critical load from the curves because there is no standard for analysis of the push-in test. In this study, the curves were analysed following the method developed in Naya Montáns (2017). The stiffness was computed by an iterative linear least squares fit of the force displacement curve, increasing the amount of points taken for the fit at each step (i. e. performing the smooth numerical derivative). A distribution of slopes was obtained and the stiffness was taken as the maximum of that curve. For the critical load, an iterative polynomial fitting of the force displacement curve was performed, considering several orders of the polynomial and several ranges of points for the fit. Then, the derivative of the polynomial is obtained and the critical load is defined as the point where the derivative deviate 1% from the previously determined value of stiffness.

The analysis of the fibre push-in test is not straightforward because there is no knowledge about the length of debonded interface during the test. As a first approximation, several authors have proposed the use of an analytical method, the shear-lag model (Cox, 1952; Kelly and Tyson, 1965). The interface shear strength is calculated as (Rodríguez et al., 2012b):

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$$\tau_c = \frac{SP_c}{2\pi^2 r^3 E_1^f} \quad (5.3)$$

S and P_c are the stiffness and critical load, respectively. r is the fibre radius and E_1^f is the fibre elastic modulus in longitudinal direction.

While the shear-lag model provides an easy approximation, the estimation does not take in to account several important factors that influence the value of interface strength: (1) the local environment of the tested fibre is not properly considered; (2) the model considers isotropic fibres, while carbon fibres are significantly anisotropic; and (3) it does not account for thermal residual stresses from the curing process. To this end, inverse analysis based on 3D FEM simulations has been often used to extract the interface strength from the direct results of the push-in test. Naya Montáns (2017) and Rodríguez (2012) developed a 3D FEM model of the push-in test considering the central tested fibre, the surrounding fibres, the resin matrix, the interface, and a surrounding area with the composite homogenised behaviour. The interface was modelled using a cohesive interface governed by a linear traction-separation law, which is the same approach used for modelling the fibre-matrix interface in the micromechanical models of the composite ply (see section 6.2.2). The fibres surrounding the tested fibre were assumed to be arranged in hexagonal close-packed configuration, as in figure 5.8. For this reason, experiments were performed on fibres with this type of configuration. Naya Montáns (2017) proposed a correction of the critical shear stress obtained from the shear lag model (equation 5.3) based on the FEM simulations, which is:

$$t_{t1}^0 = 1.6329\tau_c - 0.0578\Delta T \quad (5.4)$$

t_{t1}^0 is the interface shear strength in longitudinal direction and ΔT is the temperature gradient between the curing temperature and the temperature at which the test is performed.

Equations 5.3 and 5.4 were applied to the metrics obtained from the push-in test to estimate the longitudinal shear strength of the interface. A temperature gradient of 160 °C was applied. The results are compiled in table 5.3. Overall, the test results exhibit considerable scatter. This could be caused by the uncertainties in the calculation of critical load and stiffness. The identification of the onset of nonlinearity is challenging because there is not a clear indicator in the force-displacement curve.

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Therefore, the computed critical load is significantly sensitive to experimental uncertainties that may have an effect on the shape of the force-displacement curve: thermal drifts, instrument compliance, plastic deformation of the fibre surface, inhomogeneity in the indented surface surrounding, among others.

Experimental conditions	IM7-8552	IM7-M91
30 nm/s	74.5 ± 12	77.3 ± 16.2

Table 5.3: Shear strength (t_{t1}^0) values (in MPa) obtained by applying equations 5.4 and 5.3 to the push-in test results.

The results in table 5.3 show that the interface shear strength of both materials is nearly the same. Considering that both composite systems share the same fibre (IM7) and the resin chemistries are very much alike (see section 2.1), the interface strength of both composite systems was expected to be similar.

5.3.3 Assessment of the applicability of the nano-impact test set-up to the push-in technique

Experimental procedures

Tests were performed on the modified Nanotest Alpha of Micro Materials Ltd. (UK) described in section 3.2. Low speed tests were performed on a displacement-controlled mode, applying a linear scheme. Three cases were tested: 1, 10 and 100 nm/s. The impact configuration was used for the high speed cases. Only one impact condition was performed: impulse force of 5 mN and impulse distance of 20 μm . This resulted in an impact velocity around 1 mm/s. Ten repeats were performed per material and test condition, for statistical analysis.

Results and discussion

The direct outcome of the push-in test is a force-displacement curve like the ones shown in figure 5.9. The typical push-in force-displacement curve is linear up to the onset of nonlinearity, located at the onset of interface failure. As described in the previous section, there are two metrics to be extracted from this result: stiffness and critical load. The stiffness is related to the first linear elastic part and it is related

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Figure 5.9: Force-displacement curves obtained in the push-in tests of the two composite systems under study, IM7-8552 and IM7-M91. The curves correspond to the tests performed with the Nanotest at 3 speeds in the conventional nanoindentation configuration and one condition using the impact configuration.

to the elastic deformation of fibre, indenter and the surrounding environment. The critical load defines the onset of debonding of the interface.

The analysis of the push-in test requires a sufficiently smooth curve to be able to identify the onset of nonlinearity, which is not visually apparent. The resulting force-displacement curve from the push-in test under impact conditions does not exhibit sufficient quality to extract the critical load because of the significant oscillations in the curve. Nevertheless, these tests probe the capability of the nano-impact technique to extend the interface characterization to the mm/s range, enabling the testing of the interface with a single instrument over six orders of magnitude of displacement rate (nm/s to mm/s). Next steps should go into seeking to smooth

5.4. Concluding remarks

the force-displacement curve, or attempting the use of other techniques, for example the push-out test, where the point of interface failure is easier to perceive in the force-displacement curve.

5.4 Concluding remarks

The matrix and fibre-matrix interface of the two composite systems under study (IM7-8552 and IM7-M91) were tested using the modified instrument presented in chapter 3. For the matrix, the inverse analysis devised in chapter 4 was applied to extract the compression yield stress and was compared with the macroscale compression test results of chapter 2. The inverse analysis provided an accurate prediction of the yield stress at low strain rates; however, the predictions deviated at higher strain rates. This has been attributed to the strain rate sensitivity of the reduced modulus input in the analysis, which was estimated from the DMA results of chapter 2. For this reason, an alternative micromechanical testing technique for the matrix was proposed, the micropillar compression test. The micropillars were compressed at low speeds and impact conditions. In this case, the results agreed well with the macroscale compression values. Furthermore, the micropillar compression tests could successfully isolate the mechanical behaviour of the M91 matrix, without the effect of the thermoplastic particles. Regarding the fibre-matrix characterization, the push-in test was selected and the modified instrument presented in chapter 3 was used to perform tests at low speeds (1-100 nm/s) and impact conditions (~ 1 mm/s). The interface shear strength was obtained at low speed, but the force-displacement curve under impact conditions was not of enough quality to extract mechanical properties because of the large oscillations.

Computational micromechanics and correlation with experiments

This chapter presents the computational micromechanics models used to predict the stiffness and strength properties of a fibre-reinforced polymer composite ply over a wide range of strain rates. The micromechanical models have been developed in the framework of the Finite Element Method, making use of the Representative Volume Element approach to simulate the mechanical behaviour of the ply. Two loading cases were selected, in-plane shear and transverse compression, because the failure mode under these conditions is mainly dominated by the matrix and fibre-matrix interface. Matrix-dominated failure modes are the ones that show a more significant strain rate sensitivity (see section 1.3).

Section 6.1 contains a brief overview of the different homogenization techniques. Section 6.2 presents the details of the FEM models used in the simulations. It also describes the material models applied to each of the composite constituents. A sensitivity analysis was performed to study the effect that changes in the mechanical properties of the constituents have in the mechanical behaviour of the composite ply. The two matrix-dominated failure modes studied throughout this chapter, in-plane shear and transverse compression, were included in the sensitivity analysis. The results are presented in section 6.3. Finally, the results of the FEM simulation are presented in section 6.4. They are validated against the experimental results presented in section 2.3. Section 6.4 also includes a summary of all the inputs needed for the material models. Some of these inputs have been obtained through the micromechanical testing results presented in chapter 5.

6.1 Introduction

Composite materials consist of two or more constituents with significantly different properties. In the case of fibre-reinforced polymers, fibres are generally much stronger and stiffer than the matrix material. Therefore, for the representation of the material in a continuum mechanics framework, one has to go to the microstructure level, so that the variation in stress and strain fields across the constituents can be captured. The aim of micromechanics is to provide a prediction of the average (effective) properties of the heterogeneous material from the properties of its constituents. This is a process called homogenization. There are several micromechanical approaches and they can be classified in two main groups: analytical micromechanics and computational micromechanics.

Analytical micromechanics presents the advantage of providing fast predictions, without the need of large computing power; however, it is restricted to linear elastic behaviour and it cannot capture the behaviour of anisotropic microstructures. There are two main groups of analytical methods. One set of methods is based on the mean-field approach, in which it is assumed that the stress and strain fields of each of the constituents of the composite material is represented by their volumetric averages. Assuming linear elastic behaviour for all the constituents, the mechanical response of the composite is reduced to determining the so called concentration tensors that contain information about volume fraction, shape and spacial distribution of the reinforcement phase. The Mori-Tanaka model (Mori and Tanaka, 1973) is an example of a mean-field method. The other set of analytical approaches is based on variational bounding methods, like the Voigt (assumes uniform strain throughout the material) and Reuss (assumes that the phases are subjected to an uniform stress) bounds (Hill, 1952). The Voigt-Reuss bounds are simple and easy to apply, but provide a wide window of effective properties, especially for phases with very dissimilar properties or when the volume fractions of the constituents are similar. Narrower bounds are given by other models like the Hashin-Shtrikman bounds (Hashin and Shtrikman, 1963), which is based on principles of minimum potential energy.

Computational micromechanics approaches extend the predictions to the nonlinear regime, and enable the addition of manufacturing effects (like thermal residual stresses or porosity) and anisotropic constituents. In addition, the stress and strain fields are fully resolved in the microstructure, so it is possible to study the early stages in the initiation of damage. There are two main approaches: the method of

Figure 6.1: Examples of the two approaches for computational micromechanics: (a) Representative Volume Element (RVE) and (b) Embedded Cell.

periodic RVEs and the method of embedded cells.

In the method of periodic RVEs, the mechanical behaviour of the composite material is predicted from a volume containing a representation of the composite microstructure (see figure 6.1-a). The size of the RVE should be big enough so that the homogenised response is not size dependent, but not too large because it would increase the computational cost. In addition, the boundaries of the representative volume are subjected to periodic boundary conditions that link the displacement of the nodes in opposite sides of the volume. One of the main challenges of the RVE approach is the generation of the representative microstructure of the composite. One option is to directly reproduce the microstructure from an optical image of the cross section of the composite (Trias et al., 2006). However, this methodology can be time consuming and complex. Therefore, a more popular option is to use random generation algorithms like the Random Sequential Absorption (RSA) (Segurado and Llorca, 2002) or the Nearest Neighbour Algorithm (NNA) (Vaughan and McCarthy, 2010). Recently, Herráez et al. (2020b) proposed a method based on computing an overlapping potential that quantifies the amount of overlapping in between fibres of an RVE. The optimum microstructure is found by minimizing this overlapping potential. The fibres are allowed to move (translation and rotations) and impact each other (elastic contacts) so that the microstructure can converge to the solution that provides the optimum overlapping potential.

The method based on periodic RVEs provides a good prediction of the ho-

6.2. Set-up of micromechanical model

mogenised nonlinear response of the composite ply, but it is limited to predictions up to the initiation of damage. Once cracks initiate and propagate, the validity of the prediction is not ensured because crack propagation is a local phenomenon. In this case, the embedded cells approach is a more suitable tool, although it is limited to cases where the crack propagation path is known beforehand. The embedded cell approach consists of having a domain formed by a detailed microstructure of the composite embedded in a bigger domain, as it is shown for example in figure 6.1-b. The mechanical behaviour of the outer domain is defined by the homogenised properties of the composite. Analytical micromechanics can be used for determining the average properties of the outer domain, and the properties of the constituents are input in the embedded cell. In this case, no periodic boundary conditions are needed as the entire microstructure is resolved in the embedded cell. However, it is necessary to have prior knowledge about the path that the crack will follow.

6.2 Set-up of micromechanical model

6.2.1 FEM model details

The in-plane ply properties were simulated using RVEs of the composite microstructure. The first step was the generation of random microstructures of the composite ply. A Python-based tool called VIPER was used (Herráez et al., 2020b). Among the several random microstructure algorithms implemented in the tool, the algorithm developed by Herráez et al. (2020b) was used. FEM models of the RVEs were then created to be analysed in Abaqus. Three RVEs were created to account for the effects of different fibre distributions. Figure 6.2 shows the three RVEs. The fibre volume fraction was set to 60% in all cases, and the fibre diameter to $5.2\ \mu\text{m}$, as specified in the product datasheet of the IM7 fibres (Hexcel, b). All the RVEs have the same size, $40\times 40\times 0.5\ \mu\text{m}$. This size results in an RVE with a total of 40-50 fibres, which is enough to ensure that the mechanical response of the ply is size independent (González and LLorca, 2007a; Rodríguez, 2012). The domain was meshed with C3D8 and C3D6 elements, with an average element size of $0.5\ \mu\text{m}$. This means that there is only one element in the thickness direction. For modelling the fibre-matrix interface, cohesive interactions were used. This links the degrees of freedom of the nodes that belong to the fibres with the nodes that belong to the matrix. The interaction is defined by a traction-separation law, which is defined by the material properties of the interface (see section 6.2.2).

Figure 6.2: Representative volume elements used to predict the strain rate dependency of the in-plane properties of the IM7-8552 and IM7-M91 composites.

Figure 6.3: Schematics of the location of the master nodes in the RVEs.

The periodicity of the generated microstructure is ensured by having continuity of the fibres cut by the domain borders. This means that if a fibre is sectioned by an edge, the remaining piece will be located in the opposite edge, as it can be seen in figure 6.2. In addition, the nodes in opposite edges are located at the same position along the edge, which is a requirement for the periodic boundary conditions (PBCs). The PBCs establish a continuity of displacements between neighbouring RVEs by ensuring that the relative displacement between nodes in opposite faces of the RVE is equal to the relative displacement between the master nodes located at each of the surfaces. Figure 6.3 describes the location of the master nodes in the RVEs used for the simulation of the in-plane ply properties. Considering this set of master nodes, the PBCs can be expressed as:

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$$u(L, y, z) - u(0, y, z) = U^{MN_x} - U^{MN_0} \quad (6.1)$$

$$u(x, L, z) - u(x, 0, z) = U^{MN_y} - U^{MN_0} \quad (6.2)$$

$$u(x, y, W) - u(x, y, 0) = U^{MN_z} - U^{MN_0} \quad (6.3)$$

PBCs were implemented in the FEM model automatically, using a python code that generates equations that relate the degrees of freedom of the nodes in opposite surfaces. The load cases are introduced in the RVE by imposing displacements to the master nodes. The deformation of the RVE will then lead to reaction forces in each of the master nodes. From the imposed displacements and the resulting reaction forces, one can extract the homogenised stress-strain response of the RVE. In this work, three load cases have been simulated: transverse compression, shear perpendicular to the fibres and shear parallel to the fibres. Table 6.1 compiles the displacements imposed in the master nodes for each of the load cases.

Load case	Master node X	Master node Y	Master node Z
Shear parallel to fibres ($\tau_{ }$)	(0, 0, -)	(0, 0, 2.002)	(0, 0, 0)
Shear perpendicular to fibres (τ_p)	(-, 0, 0)	(0, 0, 0)	(0, 0.025, 0)
Transverse compression	(-1.4, -, -)	(0, -, -)	(0, 0, -)

Table 6.1: Displacements imposed in the master nodes for each of the simulated load cases.

The data reduction procedure consisted of obtaining the stress-strain relations from the input displacements and reaction forces in the master nodes. The in-plane shear test was simulated following the recommendations of Totry et al. (2010), i.e. the shear stress is computed as the average stress of two shear loading cases: shear parallel to the fibres, $\tau_{||}$, and shear perpendicular to the fibres, τ_p . The stress is thus computed as:

$$\tau_{12} = \frac{\tau_{||} + \tau_p}{2} \quad (6.4)$$

Stress and strains were computed from the input displacement and reaction forces in the master nodes, using the following equations:

$$\gamma_{||} = \tan^{-1} \left(\frac{U_z}{L} \right) \quad \tau_{||} = \frac{R_z}{L \cdot t} \quad (6.5)$$

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$$\gamma_p = \tan^{-1} \left(\frac{U_y}{t} \right) \quad \tau_p = \frac{R_y}{L \cdot L} \quad (6.6)$$

Stress and strains in the case of shear parallel to fibres were obtained from the displacement and reaction force in z-direction, U_z and R_z respectively, in the master node Y. In the case of shear perpendicular to fibres, the displacement and reaction force in y-direction, U_y and R_y respectively, in the master node Z were used.

Stress and strains in the simulation of the transverse compression test were obtained from the displacement and reaction force in x-direction, U_x and R_x respectively, in the master node X:

$$\epsilon = \frac{U_x}{L} \quad \sigma = \frac{R_x}{L \cdot t} \quad (6.7)$$

Before the mechanical loading step, the RVEs were subjected to a thermal step that emulates the cooling process that the material undergoes after curing. A thermal gradient of -160 °C was applied to the RVEs, while rigid body motions were constraint.

6.2.2 Constitutive models for fibre, matrix and fibre-matrix interface

Constitutive model for the fibres

The fibres were modelled as transversally isotropic solids because the composites under study are made with carbon fibres, which are very anisotropic. The mechanical behaviour is thus defined by five elastic constants. The calibration process was detailed in section 2.2. The mechanical properties of the carbon fibres were assumed to be rate independent, in line with the findings of previous works (Zhou et al., 2007).

Constitutive model for the matrix

The polymer matrix of composite materials exhibits a complex mechanical constitutive behaviour. Their elastic and plastic behaviour is generally rate dependent. Yielding is pressure sensitive, so the yield stress in both tension and compression changes when the material is subjected to confinement. In addition, they behave brittle when tensile loads are applied, while large deformation prior to fracture is observed in compression. While there are many constitutive models in literature

6.2. Set-up of micromechanical model

to represent the mechanical response of polymers, previous works on computational micromechanics of polymer composites have revealed the advantages of assuming isotropic linear elasticity and plasticity defined by a Drucker-Prager yield and flow rule (Naya Montáns, 2017; Rodríguez, 2012). This model provides fair accuracy, while being sufficiently simple to be calibrated with *in situ* micromechanical testing techniques. Section 4.2.2 contains further details about the Drucker-Prager model. This was the model used for the matrix material in the RVEs. The model implemented in the Abaqus' material library was used. The material model requires the input of four material parameters. Two elastic properties: elastic modulus as a function of strain rate and Poisson's ratio. Two plastic properties: friction angle and compression yield stress as a function of strain rate. The friction angle is assumed to be independent on strain rate. The compression yield stress is assumed to be a function only on the strain rate, so there is no plastic strain hardening.

Constitutive model for the fibre-matrix interfaces

The mechanical behaviour of the fibre-matrix interface is represented by a cohesive zone model. The separation of the fracture surfaces under the action of a traction vector is defined by a traction-separation law and damage progressively advances along the cohesive zone. Damage evolution is controlled by a scalar damage variable, d , that goes from 0 (no damage) to 1 (fully damaged). There are several ways to implement this approach into a FEM code. One way is the use of cohesive elements, which constitutive behaviour is defined by the traction-separation law. This method is convenient when the material and geometric properties (thickness) of the interface are known, for example in adhesive joints. However, for interfaces of negligible thickness it is best using cohesive interactions between the two adjacent surfaces involved in the fracture process. In this case, the traction-separation law links the degrees of freedom of the nodes in the two surfaces involved.

In the presence of several failure modes acting simultaneously, which is the case in a 3D simulation, the traction stress vector (t_n, t_{t1}, t_{t2}) and the separation vector $(\delta_n, \delta_{t1}, \delta_{t2})$ have three components: one normal to the surface and two shear components (longitudinal and transversal). In this case, the traction-separation law can be expressed in terms of effective traction and separation:

$$\bar{t} = \sqrt{\langle t_n \rangle^2 + t_{t1}^2 + t_{t2}^2} \quad \bar{\delta} = \sqrt{\langle \delta_n \rangle^2 + \delta_{t1}^2 + \delta_{t2}^2} \quad (6.8)$$

Figure 6.4: Linear traction-separation law that defines the constitutive behaviour of the cohesive interactions used to model the fibre-matrix interface.

The normal components use the Macaulay brackets to ensure that there is no damage advancement when the interface is in compression.

There are several types of traction-separation laws, depending on the material under study and the level of desired accuracy. In the present study, a linear law like the one in figure 6.4 is used, which is one of the most popular options. Before damage initiates, the behaviour is linear and defined by a slope K (three in the case of 3D problems). Rather than being related to an elastic modulus of the interface, this constant is an arbitrary parameter that should be large enough so as to have displacement continuity through the interface, but not too large because it can cause numerical instabilities. The onset of damage is at the point where the traction-separation relation reaches its maximum. For mixed-mode fracture, there are several criteria available. The quadratic interaction criterion is selected in this case:

$$\left\{ \frac{\langle t_n \rangle}{t_n^0} \right\}^2 + \left\{ \frac{t_{t1}}{t_{t1}^0} \right\}^2 + \left\{ \frac{t_{t2}}{t_{t2}^0} \right\}^2 = 1 \quad (6.9)$$

t_n^0 , t_{t1}^0 and t_{t2}^0 are the normal and the two shear strengths of the interface, respectively. Once damage is initiated, the stiffness of the interface degrades as damage develops. Since a linear softening law is imposed (see figure 6.4), the damage variable can be estimated from:

$$d = \frac{\bar{\delta}_f(\bar{\delta} - \bar{\delta}_0)}{\bar{\delta}(\bar{\delta}_f - \bar{\delta}_0)} \quad (6.10)$$

6.3. Effect of the constituents mechanical properties on the matrix dependent ply properties

$\bar{\delta}_f$ is the displacement jump at the point where the interface has failed completely, and it can be estimated from the effective fracture energy and strength as $2\bar{G}_c/\bar{t}_c$. For mixed mode fracture, the effective fracture energy is a function of the fracture energy of each of the fracture modes (normal and two shear modes). There are several approaches for computing the effective fracture energy. The one used in this study is the Benzeggagh and Kenane (1996) model, which is already implemented in Abaqus (Simulia, 2016).

In this study, the cohesive zone model for simulation of interface debonding is coupled with a Coulomb friction model to reproduce the frictional interaction between the debonded surfaces after failure. More information about the coupling of the cohesive zone model with friction can be found in the work of Naya Montáns (2017).

6.3 Effect of the constituents mechanical properties on the matrix dependent ply properties

A parametric study was carried out to study how sensitive the mechanical response of the ply is to changes in the properties of the constituents. To this end, a number of material property sets was applied to one of the RVEs presented in section 6.2.1, RVE2. Three cases have been analysed:

- Matrix and fibre properties were kept constant. The matrix yield stress in shear is about 70 MPa and the friction angle of the Drucker-Prager model is 30°. The shear strength in longitudinal direction of the interface was varied, together with the value of interface fracture energy. For simplicity, the interface strengths in all directions were assigned the same value. The same applies for the energies. A case of interface with no damage (perfect interface) was implemented too.
- Matrix and fibre properties were kept constant. The interface strength was varied in this case too (including the perfect interface case), together with the friction coefficient. The interface fracture energies were kept constant.
- The matrix yield stress was varied and all the other material properties were kept constant. Two extreme cases of interface strength were considered: perfect interface (stronger than the matrix) and interface weaker than the matrix.

6. Computational micromechanics and correlation with experiments

The RVE was analysed under the two loading cases of interest: in-plane shear and transverse compression. The results are shown in figure 6.5.

The debonding of the fibre-matrix interface has a strong effect on both the in-plane shear and transverse compression behaviour, as long as the interface is weaker than the matrix. In the case of materials with fibre-matrix interface stronger than the matrix itself, the nonlinear deformation will be exclusively caused by the plastic deformation of the matrix. This corresponds to the curves in magenta of figure 6.5. In the case of an interface weaker than the matrix, the stress-strain curves start deviating from the perfect interface case (magenta) at the onset of interface debonding. Higher interface strength leads then to higher overall stress carried by the ply. In the material model implemented for this study, the matrix yield stress in shear is about 70 MPa. Therefore, one would expect the 90 MPa case (cyan) to behave like the perfect interface case. However, since a Drucker-Prager yield was implemented (being the friction angle 30°), the shear yield stress of the matrix increases due to the confining effect of the fibres, hence triggering interface damage.

Fracture energy has a less significant effect on the mechanical behaviour of the ply. The influence of fracture energy becomes relevant when the displacement to full failure in the cohesive zone model of the interface is small compared to the fibre diameter (5.2 μm in this study). In the case of $0.05 \text{ nJ}/\mu\text{m}^2$, the separation at full failure is around 2 μm for the 50 MPa case and around 1 μm for the 90 MPa case. This is thus the reason why there is a slight loss of load bearing capability for the $0.05 \text{ nJ}/\mu\text{m}^2$ case.

Friction is activated upon debonding of the fibre-matrix interface, so it does not affect the location of the onset of nonlinearity in the stress-strain curve. Once damage is initiated, the friction effect ramps up proportionally with the evolution of the damage parameter. At the point where the damage variable is one, the only stress transfer in the interface is due to friction. The effect of friction is certainly more pronounced in the transverse compression case because of the higher compression stress acting in the interface. In fact, in the cases with higher friction coefficient (0.4 and 0.6), the response is almost as the perfect interface case. In the in-plane shear case, the residual stress generated during the thermal step that simulates the cooling from curing temperature plays an important role in the friction forces.

Changes in the matrix yield stress have significantly different effects on the in-plane shear and the transverse compression response. The two plots at the bottom of figure 6.5 show the effect that a $\pm 20\%$ change in the matrix compression yield

6.3. Effect of the constituents mechanical properties on the matrix dependent ply properties

Figure 6.5: Sensitivity analysis of the mechanical properties of the composite constituents on the ply mechanical response under in-plane shear and transverse compression loading: (a) effect of the interface shear strength and fracture energy, (b) effect of the interface shear strength and friction coefficient and (c) effect of matrix yield stress.

6. Computational micromechanics and correlation with experiments

stress has on the stress-strain curves. Under in-plane shear loading, the effect of changes in the matrix yield is certainly higher when the interface is stronger than the matrix. However, when the interface is weaker, changes in the matrix yield will not affect much the stress-strain response because the nonlinear deformation is mostly dominated by the interface debonding.

6.4 Simulation of the in-plane shear and transverse compression tests and correlation with experiments

6.4.1 Summary of inputs of the material models

Tables 6.2, 6.3 and 6.4 compile all the material constants used in the simulation of the micromechanical models presented in this section.

Table 6.2 contains the elastic constants of the IM7 fibres. The constants were estimated from an inverse analysis of macroscale tests carried out in composite laminates and neat resin coupons. The results were presented in section 2.2. The Chamis rule of mixtures was used for the prediction of the properties, following the guidelines of previous works in this area (Rodríguez, 2012; Naya Montáns, 2017). The thermal expansion coefficients were taken from the work of Kaddour and Hinton (2012).

IM7 fibre material properties						
E_1^f [GPa]	ν_{12}^f	E_2^f [GPa]	G_{12}^f [GPa]	G_{23}^f [GPa]	α_1^f [K^{-1}]	α_2^f [K^{-1}]
275.7	0.23	12.2	18.3	4.7	$-4 \cdot 10^{-7}$	$5.6 \cdot 10^{-6}$

Table 6.2: Inputs for the material model of the fibres.

Table 6.3 collects the material properties needed for the material model of the matrix material. The elastic modulus is given as a power law function of the strain rate. These functions were obtained by fitting the elastic modulus values in figure 2.5. In the case of the M91 resin, the results in figure 2.5 were scaled with the elastic modulus values obtained in the micropillar compression tests (see figure 5.7) before performing the power law fitting. Scaling with the elastic modulus obtained in the micropillar compression tests ensures the removal of the effect of the thermoplastic particles. The values of Poisson's ratio were obtained from tensile testing of neat resin coupons. The values of friction angle were obtained from literature

6.4. Simulation of the in-plane shear and transverse compression tests and correlation with experiments

(Rodríguez, 2012; Naya Montáns, 2017). The compression yield stress is given as a power law function of the strain rate. The compression yield stress function of the 8552 resin was obtained from the combined macroscale compression and micropillar compression results. Using the combined results enabled the calibration of a bilinear law, which is typical in epoxy resins (see section 5.2). In the case of the M91 resin, only the micropillar compression results were used for the extraction of the power law function to remove the effect of the thermoplastic particles. In this case, there were not enough points for fitting a bilinear law. The thermal expansion coefficient was taken from the work of Naya Montáns (2017) for the 8552 resin, and the same value was assumed in the M91 resin, given its similar chemistry (see section 2.1).

Property	8552 matrix	M91 matrix
Elastic modulus, E_m [MPa]	$4585\dot{\epsilon}^{0.012}$	$4174\dot{\epsilon}^{0.0104}$
Poisson's ratio, ν_m [-]	0.3738	0.3716
Friction angle, β [°]	30	30
Compression yield stress, σ_{yc} [MPa]	$\sigma_{yc}(\dot{\epsilon}) = \begin{cases} 191\dot{\epsilon}^{0.0298}, \dot{\epsilon} < 1 \\ 189\dot{\epsilon}^{0.0536}, \dot{\epsilon} \geq 1 \end{cases} \quad \sigma_{yc}(\dot{\epsilon}) = 187\dot{\epsilon}^{0.0418}$	
Coefficient of thermal expansion, α^m [K^{-1}]	$5.2 \cdot 10^{-5}$	$5.2 \cdot 10^{-5}$

Table 6.3: Inputs for the material model of the matrix.

Table 6.4 contains the properties of the material model of the fibre-matrix interface. The value of the penalty stiffness was set to 100 GPa/ μm . This value is sufficiently high so that there is displacement continuity through the interface when there is no damage. Higher values would increase the computational time. The longitudinal shear strength is the only value that could be calibrated experimentally through *in situ* micromechanical testing (see section 5.3). Following the guidelines of the work of Naya Montáns (2017), the transverse shear strength was assigned the same value. However, for the interface strength in normal direction Naya Montáns (2017) recommended the application of a 2/3 factor to the value obtained in shear, based on the findings of Ogihara and Koyanagi (2010). Regarding the interface fracture energies, they had to be taken from literature because there are no suitable experimental methods to calibrate them. The values used in the

6. Computational micromechanics and correlation with experiments

work of Naya Montáns (2017) were used in this case, together too with the recommended exponent of the Benzeggagh and Kenane (1996) model. The friction coefficient was adjusted to get an optimum fit of the transverse compression results, as the response in this loading mode is more sensitive to changes in the friction coefficient (see section 6.3).

Property	IM7-8552	IM7-M91
Penalty stiffness, K [GPa/ μm]	100	100
Normal strength, t_n^0 [MPa]	49	51
Shear strength (longitudinal), t_{t1}^0 [MPa]	74	77
Shear strength (transversal), t_{t2}^0 [MPa]	74	77
Fracture energy (Mode I), G_n [$nJ/\mu\text{m}^2$]	0.002	0.002
Fracture energy (Mode I), G_{t1} [$nJ/\mu\text{m}^2$]	0.1	0.1
Fracture energy (Mode I), G_{t2} [$nJ/\mu\text{m}^2$]	0.1	0.1
B-K exponent	1.45	1.45
Friction coefficient	0.1	0.1

Table 6.4: Inputs for the material model of the fibre-matrix interface.

The simulation of different strain rates was carried out by properly adjusting the material properties, instead of running simulations with different step times. Using different step times would be the most realistic approach; however, using different step times and material models with rate dependent properties resulted in poor convergence of the simulations. Instead, the material models were made rate independent and the properties were changed for each of the simulated strain rate cases. The compression yield stress, elastic modulus of the matrix and the interface strengths were the properties adjusted for simulating strain rate dependent behaviour. The validity of this approach was checked by comparing results of models run with several step times and rate dependent material models, with models run at a constant step time (1 s, which is the default in Abaqus) and rate independent material models in which the properties were adjusted in each case of strain rate. Both approaches delivered the same results, but convergence was better in the analysis with no rate dependent material models.

6.4. Simulation of the in-plane shear and transverse compression tests and correlation with experiments

Figure 6.6: Comparison of experimental and simulation results for the IPS tests of the IM7-8552 composite. Two sets of simulation results are included: RVE with perfect interface and RVE with interface value obtained from fibre push-in test.

6.4.2 In-plane shear

Figure 6.6 presents the outcomes of the FEM simulations using the material set of the IM7-8552 composite described in section 6.4.1. The results of FEM simulations using a perfect interface (no damage) have been also included. The experimental results presented in section 2.3.5 were included for comparison. The mechanical response of the ply predicted by the FEM simulation correlates well with the experimental result for the lower speed case. The simulation using a perfect interface shows that the ply could still carry higher loads if the interface had not failed. This means that damage initiation under in-plane shear loading for the lowest strain rate case is dominated by fibre-matrix interface debonding. The other two higher strain rate cases show a significant deviation between the experimental trends and the simulation results using the interface properties in table 6.4. Furthermore, the gap increases for higher strain rates, suggesting that the strain rate dependent mechanical behaviour of the interface is not properly represented by the material properties in table 6.4. In particular, no rate dependency was implemented in the material model of the interface. This is because the push-in test results presented in section 5.3 were not conclusive regarding the strain rate dependency of the interface. Therefore, no rate dependency was assumed due to the lack of calibration data.

Interestingly, the simulation results using a perfect interface model reproduce accurately the experimental trends up to a certain strain point in the two higher strain rate cases. This suggests that not only the mechanical behaviour of the interface is rate dependent, but that the shear strength of the interface actually

6. Computational micromechanics and correlation with experiments

increases at a higher rate with strain rate than the shear strength of the matrix. This then leads to a change in the damage initiation mechanism from damage initiated by combined interface debonding and matrix plasticity at the lowest strain rate case to damage initiated by matrix plasticity exclusively for the other two higher strain rate cases.

In addition to the predicted homogenised stress-strain response of the ply, the FEM simulations allow a more in depth analysis of the damage initiation mechanisms by looking at the distributions of stress and strain in the RVE. Figure 6.7 shows contour plots of the equivalent plastic strain at the point of maximum simulated deformation for the two more extreme strain rate cases. The RVEs of the high strain rate case correspond to simulations using a perfect interface because this seems to be the representative behaviour of the ply at this strain rate, according to the results of figure 6.6. In the case of shear parallel to fibres at low strain rate, the matrix exhibits plastic deformation along the entire RVE, but there is a band of higher plastic deformation parallel to the shear loading. This area of localized plastic strain was induced by the debonding of the fibre-matrix interface. When the interface fully fails, the fibre cannot carry loads anymore, thus increasing the stress in the matrix. On the other hand, at high strain rate there is no band of localized plastic deformation and the plastic strains are uniformly distributed along the microstructure. This is because there is no interface debonding that may trigger localization of damage. As expected, the plastic strains in the high strain rate case are higher than at low strain rate because the stress carried by the ply is higher. The case of shear perpendicular to fibres exhibits a similar response than the shear parallel to fibres; although, the strain localization at low strain rate is not as obvious under this loading condition. Plastic deformation is generally more widespread along the microstructure in the case of shear perpendicular to the fibres (Totry et al., 2009). At high strain rate, the response is nearly the same as the shear parallel to fibres.

Figure 6.8 presents the comparison of experimental trends and simulation results for the in-plane shear test of the two composites under study: IM7-8552 and IM7-M91. The simulation results using a perfect interface were included for the two higher strain rate cases because these are the predictions that better represent the experimental trends. The intermediate strain rate case of the IM7-M91 presents a slight deviation from the experimental trend, meaning that, although the interface strength increases with strain rate, still interface damage is triggered at this speed

6.4. Simulation of the in-plane shear and transverse compression tests and correlation with experiments

Figure 6.7: Distribution of equivalent plastic strain at the maximum simulated stress in the cases of shear parallel to fibres (two contour plots at the top) and shear perpendicular to the fibres (two contour plots at the bottom). The high strain rate cases correspond to the FEM simulations with perfect interface. These results correspond to the simulations with the IM7-8552 material set.

6. Computational micromechanics and correlation with experiments

Figure 6.8: Comparison of experimental and simulation results for the IPS tests of the IM7-8552 and IM7-M91 composites. The simulation results of the lowest speed case correspond to the case of interface with strength obtained from push-in test, while the two higher speed cases correspond to the case of perfect interface.

in this material. In any case, these results show that there is a change in damage initiation mechanism with strain rate under in-plane shear loading. At low strain rate, damage is controlled by combined plastic deformation of the matrix and fibre-matrix interface debonding. However, at high strain rates damage initiation is caused entirely by matrix plasticity. These findings are actually in line with the observations made by other authors regarding the change in damage initiation mechanism in in-plane shear over a wide range of strain rates. By looking at the fracture surfaces, both Cui et al. (2016) and Taniguchi et al. (2007) concluded that cracks propagated primarily along the matrix at high strain rates; while at low strain rate the cracks propagated mainly along the fibre-matrix interface. Further proof of this conclusion would come from being able to test the fibre-matrix interface over a wide range of strain rates. The results of section 5.3 showed that the nano-impact technique can be used to test the interface at high strain rates, but the load-displacement curves were not of enough quality to extract mechanical information. Therefore, the instrumentation needs to be improved, or a different testing technique should be applied, for example the push-out test.

Although figure 6.8 shows a good correlation between the FEM simulations and the experimental trends, the predicted ply response starts deviating from the experiments at a certain deformation. After this strain point, the FEM predicts higher load bearing capability as compared with the expected behaviour represented by the

6.4. Simulation of the in-plane shear and transverse compression tests and correlation with experiments

experiments. There may be two reasons for this: (1) a matrix damage model was not included in the simulations, just the plastic deformation; and (2) the RVE simulations are valid as long as they represent a periodic feature of the microstructure. Damage initiation can be predicted by this type of models, but once damage starts propagating and becomes a local phenomenon, it cannot longer be simulated with periodic RVEs. In fact, the DIC analysis of figure 2.12 shows that the samples exhibit damage localization around 3-5% of deformation for the IM7-8552 composite. In the IM7-M91 composite, damage localization happens at a later stage, around 5-7%. This is the reason why, for example, the simulation of the highest strain rate case shows a good correlation up to around 5% in the IM7-M91, while the high strain rate case in the IM7-8552 deviates from the experiment at around 3% strain.

6.4.3 Transverse compression

Figure 6.9 presents the results of the FEM simulations, which are compared with the experimental trends obtained from the transverse compression test results presented in section 2.3.4. The results of FEM simulations considering a perfect interface have been also included. At low strain rate, the simulations featuring an interface with the properties in table 6.4 exhibit a close match with the experimental trends, meaning that at low strain rate damage initiation is controlled by combined matrix plasticity and fibre-matrix debonding. Nevertheless, the stress-strain response of the ply is just a bit deviated from the perfect interface case, so mainly plastic deformation dominates the nonlinear deformation. The high strain rate results show the opposite behaviour. The prediction of the perfect interface model matches better the experimental trend in this case. This is in line with the in-plane shear results in the previous section (section 6.4.2). Therefore, these results provide additional evidence to support the idea that there is a change in the damage initiation mechanism from combined matrix plasticity and interface debonding at low strain rates to just matrix plasticity at high strain rates.

The contour plots in figure 6.10 give deeper insight on the damage mechanisms of the ply under transverse compression. At low strain rate, the microstructure exhibits several bands of localized plastic deformation. Some of these bands form an angle with the perpendicular to the loading direction of about 55° , which is a reasonable orientation of the fracture angle for an epoxy resin (González and LLorca, 2007a). This angle is related to the pressure sensitivity of the polymer matrix. There are also other bands in the microstructure that form a lower angle. This is because

6. Computational micromechanics and correlation with experiments

Figure 6.9: Comparison of experimental and simulation results for the TC tests of the IM7-8552 composite. Two sets of simulation results are included: RVE with perfect interface and RVE with interface value obtained from fibre push-in test.

Figure 6.10: Distribution of equivalent plastic strain at the maximum simulated stress in the case of transverse compression. The high strain rate case corresponds to the FEM simulation with perfect interface. These results correspond to the simulations with the 8552 material set.

6.5. Concluding remarks

the fibre-matrix interface debonding triggers the plastic deformation of the matrix. Then, the bands initiated by the interface debonding all coalesce in a shear band. The contour plot of figure 6.10 for the high strain rate case corresponds to the simulations with a perfect interface because this is the case that best represents the experimental trends (see figure 6.9). The contour plot shows that the deformation is nearly linear elastic for the high strain rate case. Only a few areas of high stress concentration in between fibres exhibit plastic deformation.

6.5 Concluding remarks

Computational micromechanics simulations based on the RVE approach have been used to predict the composite ply response under in-plane shear and transverse compression. These are two loading modes dominated by the matrix and thus sensitive to the strain rate of testing. First, a sensitivity analysis was employed to study the effect of the constituents properties on the ply response. It was found that the ratio of strength of the fibre-matrix interface and the matrix is the main parameter that defines damage initiation at the microscale. An interface stronger than the matrix leads to nonlinear deformation and damage initiation controlled by matrix plasticity. However, weaker interfaces lead to nonlinear deformation driven by the combined effect of interface debonding and matrix plasticity. In the second part of this chapter, the RVE models were calibrated with the micromechanical testing results of chapter 5, and complemented with the macroscale results of chapter 2 and literature. The simulations correlated closely with the macroscale results of chapter 2. Furthermore, the comparison revealed a change in the damage initiation mechanism that corresponds to previous observations in literature. At low strain rates, nonlinear deformation and damage is caused by interface debonding and matrix plasticity. At high strain rates, matrix plasticity dominates the ply behaviour. This suggests that the fibre-matrix interface strength increases with strain rate at a higher rate than the matrix yield stress.

Conclusions and future work

7

7.1 Conclusions

The work presented in this thesis provides a simulation tool and experimental methodologies that contribute to the understanding of the dynamic behaviour of fibre-reinforced polymer composite materials. The main conclusions drawn in this work with respect to each of the intermediate objectives presented in section 1.5 are:

7.1.1 Novel micromechanical testing methodology: instrumentation

An existing nano-impact instrument was selected for extending the micromechanical testing of composites constituents to the high strain rate regime. The existing instrument relied on the displacement over time signal during the impact of the indenter tip against the material and the determination of a dynamic hardness value based on a simplified energy balance. The limitations of the existing instrument were overcome by instrumenting it with a piezoelectric force sensor in direct contact with the sample so that the essential output of a nanoindentation test, a force-displacement curve, could be obtained. The following conclusions were obtained:

- The new instrumentation was tested in several materials covering a wide spectrum of mechanical behaviour. As a result, the nanoindentation force-displacement curve of these materials could be obtained for the first time at high strain rates (10^2 - 10^3 s^{-1}). This enabled the application of the ISO14577 (2015) standard analysis of instrumented indentation to obtain hardness and elastic modulus from the nano-impact test.
- The hardness calculated with the conventional analysis of the nano-impact test, which is based on a simplified energy balance, deviates significantly from

7.1. Conclusions

the standard hardness based on the analysis of the force-displacement curve. This was attributed to a missing energy dissipation term that could be estimated thanks to the new instrumentation, and to the effect of the work of the impulse force, both not accounted for in the simplified analysis. Furthermore, the deviations might be even higher in very elastic materials because of the larger difference between the residual volume and the contact volume of indentation.

- Based on these results, a new energy-based hardness definition was proposed that delivers equivalent results than the force-based standard analysis.

7.1.2 Novel micromechanical testing methodology: inverse analysis

Nanoindentation testing by itself cannot be used to extract material parameters needed for the calibration of a material constitutive model. Therefore, the second step in the development of the micromechanical testing methodology involved the implementation of a FEM-based inverse analysis methodology to extract material properties from the direct outputs of the nanoindentation test. The main outcomes of the analysis are:

- The strain rate sensitivity (SRS) of the hardness is in certain cases not equal to the strain rate sensitivity of the flow stress. They are the same for very plastic materials (low H/E_{eff}), but the ratio of SRS of the hardness to SRS of the flow stress decreases with increasing H/E_{eff} , i. e. for more elastic materials. This has important experimental implications because it means that measuring the SRS in very elastic materials with rate dependent behaviour (e. g. hard ceramics or glasses) using nanoindentation may be complex or even impracticable. This conclusion is in line with the results of previous works (Alkorta et al., 2008b), but it was here extended to materials with pressure sensitive yield. It was found that the pressure sensitivity parameter has a marginal effect on the ratio of SRS of the hardness to SRS of the flow stress, in contrast with the effect of the H/E_{eff} parameter.
- The inverse analysis was tested with three materials with a well-known strain rate dependent plastic flow behaviour (coarse and ultrafine grain copper, and PMMA). The predictions of the inverse analysis were in good agreement with the literature trends.

7.1.3 Micromechanical characterization of composite constituents, computational micromechanics and comparison with experiments

The following conclusions were drawn from the application of the instrumentation and methodologies implemented to two composite materials (IM7-8552 and IM7-M91) of aeronautical interest:

- The nanoindentation testing and proposed inverse analysis does not provide a reliable prediction of flow stress as a function of strain rate for epoxy resins. The predictions are good at low strain rates, but the results deviate at higher strain rates. This was attributed to the values of elastic modulus used as input in the analysis and the viscoelastic behaviour of the epoxy resins.
- Micropillar compression tests constitute an alternative micromechanical testing methodology that can be implemented on the modified nanoindentation equipment, both at low and high strain rates. The tests provide an accurate determination of the flow stress of the resins as a function of strain rate, provided that the micropillar diameter is bigger than approximately 5 μm . Furthermore, micropillar compression test enabled the extraction of the constitutive behaviour of the M91 matrix without the effect of the toughening thermoplastic particles that this resin contains. This is an effect, however, that cannot be avoided using macroscale tests of neat resin coupons.
- The new high velocity setup also allows for the characterization of the fibre-matrix interface using the push-in test. However, in this case, further improvements in the data acquisition system are needed for a reliable determination of interface properties at high strain rates, due to the inherent noise of the load-displacement curves at high displacement rates.
- The IM7-M91 composite system offers an enhanced mechanical behaviour with respect of the IM7-8552 system, which is materialized in a higher strain to failure under in-plane shear deformation. This is attributed to the toughening effect of the thermoplastic particles that deflect the interlaminar cracks from the interleaf and promote intraply cracking. The superior in-plane shear performance of the IM7-M91 was shown by the macroscale tests; however, the RVE simulations could not capture this phenomenon because it is passed the point of failure initiation. Mesoscale simulations would be more appropriate to study the toughening effect of the thermoplastic particles.

7.2. Proposal of future research

- The computational micromechanics simulations based on the RVE approach revealed a change in the dominant failure mechanism with strain rate. At low strain rates, nonlinear deformation and failure initiation is controlled by the combined effect of the fibre-matrix debonding and matrix plasticity. At high strain rates, matrix plasticity is entirely responsible for the initiation of failure.

7.2 Proposal of future research

The conclusions drawn in this work reveal that there are several aspects of the presented research that can be improved or extended. The following paragraphs describe proposals for further research in line with the two main topics of this work: (1) the development of novel micromechanical testing techniques for material characterization over a wide range of strain rates at the nano-/microscale; and (2) the development of simulation tools based on computational micromechanics to study the dynamic (high strain rates) behaviour of FRP composites.

Regarding the micromechanical testing methodologies for high strain rates, the work of chapter 3 demonstrated that the instrumentation of a pendulum-based nanoindenter with load sensing capability solved the issue of obtaining a force-displacement curve in its impact configuration. However, the assessment of the novel instrumentation revealed some unexpected hardness and elastic modulus results of the tested materials. In particular, the elastic modulus at high strain rates of the four tested metals deviated significantly from the quasistatic values. Very plastic materials present an unloading curve almost perpendicular to the displacement axis; hence, they are very sensitive to the compliance of the instrument. The deviating high strain modulus obtained with nano-impact suggests that there may be a dynamic compliance induced by the instrument being used near its natural frequencies. This issue has been discussed in previous works and several authors proposed the stiffening of the pendulum shaft to increase its natural frequencies (Jennett and Nunn, 2011; Zehnder et al., 2018). However, these authors could not assess the effect in the force-displacement curve because the device was not instrumented with a load cell. Therefore, it would be certainly interesting to try a stiffened pendulum shaft in a device with force-sensing capability, to check if that would bring the elastic modulus of the four tested metals closer to the quasistatic values. Alternatively, a 3D FEM simulation of the pendulum-based device with sufficient level of detail could be used to study the dynamic deformation of the instrument and, if possible, extract a correction factor for the dynamic compliance. Furthermore, the development of the

nano-impact-based methodology in this work was carried out in parallel with the development of other techniques for high strain rate characterization of materials at the nano-/microscale (Sudharshan Phani and Oliver, 2017; Guillonneau et al., 2018). It would be interesting to apply these techniques to the the matrix and interface of the FRP composites studied in this work so that the results can be compared with the ones obtained with the combined nanoindentation and nano-impact tests in the pendulum-based nanoindenter.

Concerning the work on techniques for fibre-matrix characterization, the results presented in section 5.2 have shown that the nano-impact technique might not be suitable for application to the push-in test. This is because the analysis of the push-in test relies on the calculation of the critical load at the onset of nonlinearity. The change in slope at this point is almost undetectable and even more considering the oscillatory response obtained in the nano-impact (see figure 5.9). Therefore, future research should go into improving the data acquisition system of the pendulum based instrument or trying other techniques, for example the push-out test, in which the change in slope at failure is easier to detect. Alternatively, the push-in test could be applied in other high strain testing devices like the one presented by Guillonneau et al. (2018), that exhibits a better control over the displacement rate at high strain rates.

A logical extension of the proposed micromechanical test methodologies would be the study of combined strain rate and temperature effects because polymers are equally sensitive to both variables (see section 1.3). Besides, many instrumented nanoindenters offer the possibility of performing experiments over a wide temperature range (Fischer-Cripps, 2011). Of course, these tests are challenging because they add up the complexities of high strain rate and high/low temperature testing. However, recently Rohbeck et al. (2020) have shown promising results in this area.

The inverse analysis could be subject of further development, mainly by changing the assumptions of the materials in which is based. For example, a material with hardening rate and strain rate sensitivity could be considered. Another line of research could go into using a coupled viscoelastic-viscoplastic material model, which is a better representation of the mechanical behaviour of a polymer than the purely viscoplastic model assumed in this work. In line with this, techniques for viscoelastic characterization at the microscale could be considered as candidates for model calibration. Progress in this regard has been already achieved with the nanoindentation tests based on continuous stiffness measurement in which an oscillatory load

7.2. Proposal of future research

is superimposed to the main indenter motion (White et al., 2005).

The work on computational micromechanics for the study of high strain rate behaviour could be improved and extended in several ways. First, the convergence of the simulations carried out with rate dependent models should be improved in such a way that the RVEs can be simulated at different rates by changing the step time. The approach followed in this work considered a constant step time and rate independent materials models, the properties adjusted for each strain rate case. Regarding the material models, future research could go into implementing a coupled viscoelastic-viscoplastic model for the matrix, which represents better the mechanical behaviour of composites. In the case of the fibre-matrix interface, the results of chapter 6 have proven necessary the consideration of strain rate sensitivity of the interface strength. Therefore, future works could look into implementing a rate-dependent traction-separation law for the interface.

Finally, an extension of the work performed in this thesis could be the set up of simulations one scale up in the multiscale simulation strategy, i. e., mesoscale simulations, with which damage propagation can be studied. The results of chapters 2 and 6 regarding the comparison of the IM7-8552 and IM7-M91 materials show that computational micromechanics cannot reproduce the toughening effect of the thermoplastic interleaved particles in the IM7-M91 because their toughening effect emerges once failure has initiated.

Publications and list of contributions in conferences

8

Part of the outcome of this work has resulted in the publication of two articles in scientific journals:

- *Mario Rueda-Ruiz, Ben D. Beake, Jon M. Molina-Aldareguia, New instrumentation and analysis methodology for nano-impact testing*, Materials and Design 192 (2020) 108715.
- *Mario Rueda-Ruiz, Miguel A. Monclús, Ben D. Beake, Francisco Gálvez, Jon M. Molina-Aldareguia, High strain rate compression of epoxy micropillars*, Extreme Mechanics Letters 40 (2020) 100905.

Throughout the development of this thesis work, part of the results have been presented in scientific conferences in the field of nanomechanics and the field of mechanics of fibre-reinforced polymer composites:

- Poster: *Mario Rueda-Ruiz, Ben D. Beake, Jon M. Molina-Aldareguia, Measuring strain rate sensitivity of aerospace epoxies by nanoimpact testing*, Advanced nano-mechanical techniques for academic and industrial research, December 2017 in University of Warwick (Coventry, UK).
- Oral presentation: *Mario Rueda-Ruiz, Ben D. Beake, Jon M. Molina-Aldareguia, Nanoindentation impact testing methodologies for the high strain rate characterization of materials at the microscale*, 55th Annual Technical Meeting of the Society of Engineering Science, October 2018 in Universidad Carlos III (Madrid, Spain).

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- Oral presentation: *Mario Rueda-Ruiz, Ben D. Beake, Jon M. Molina-Aldareguia*, **Force-sensing nano-impact testing for the high strain rate characterization of materials at the microscale**, Advanced nano-mechanical techniques for academic and industrial research, December 2018 in TU Aachen (Aachen, Germany).
 - Oral presentation: *Mario Rueda-Ruiz, Ben D. Beake, Jon M. Molina-Aldareguia*, **High strain rate micromechanical testing of composite materials**, 9th International Conference on Composite Testing and Model Identification, May 2019 in Lulea University of Technology (Lulea, Sweden).
 - Oral presentation: *Mario Rueda-Ruiz, Ben D. Beake, Jon M. Molina-Aldareguia, Carlos González*, **Micromechanical modelling of composite ply incorporating a strain rate dependent constitutive model informed by a novel micromechanical testing technique**, 7th ECCOMAS Thematic Conference on the Mechanical Response of Composites, September 2019 in University of Girona (Girona, Spain).
 - Poster: *Mario Rueda-Ruiz, Ben D. Beake, Jon M. Molina-Aldareguia*, **New instrumentation and analysis methodology for nano-impact testing**, Nanomechanical Testing in Materials Research and Development VII, October 2019 (Malaga, Spain).
 - Oral presentation: *Mario Rueda-Ruiz, Ben D. Beake, Jon M. Molina-Aldareguia*, **High strain rate micromechanical testing of FRP composite materials**, Advanced nano-mechanical techniques for academic and industrial research, December 2019 in University of Southampton (Southampton, UK).

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