Supplementary Document

Optimization Strategies and Challenges in Inverse Finite Element Analysis

Inverse finite element analysis within ANSYS is a powerful tool for determining material properties by fitting numerical simulations to experimental data, such as load-displacement curves from nano-indentation. Several optimization strategies are available in ANSYS to perform this fitting, each with distinct advantages and limitations:

- **Gradient-Based Optimization (e.g., Sequential Quadratic Programming)**: This method efficiently converges to optimal solutions when the objective function is smooth and differentiable. However, it is highly sensitive to initial parameter estimates and may converge to local minima rather than the global solution.
- **Global Optimization Methods (e.g., Genetic Algorithms (GA) and Particle Swarm Optimization (PSO))**: These methods explore a broader solution space and reduce the risk of being trapped in local minima. While they offer improved robustness, they are computationally expensive and require a significantly higher number of function evaluations.
- **Bayesian Optimization and Response Surface Methods**: These techniques approximate the response function based on a limited number of FEA simulations, reducing computational cost while maintaining reasonable accuracy. However, the quality of results depends on the appropriateness of the surrogate model used for approximation.

Non-Uniqueness Issue in Inverse FEA

A major challenge in inverse FEA is the **non-uniqueness** of the extracted material properties. Multiple sets of elastic moduli, yield strength, and creep coefficients can produce similar load-displacement curves, leading to ambiguity in the determined properties. This issue arises because different combinations of parameters can influence the indentation response in a compensatory manner.

Mitigation Strategies for Non-Uniqueness

To improve the reliability and uniqueness of the extracted properties, several approaches can be implemented:

1. **Multi-Objective Optimization**: Instead of fitting only nano-indentation data, inverse FEA can be enhanced by incorporating additional experimental datasets, such as tensile or shear test results, to provide independent constraints on the material properties.
2. **Regularization Techniques**: Physical constraints or prior knowledge of material behaviour can be imposed to eliminate unrealistic solutions. For instance, upper and lower bounds for stiffness and yield strength can be set based on literature data for similar materials.
3. **Sensitivity Analysis**: By analysing the influence of each parameter on the load-displacement response, it is possible to identify which parameters are most sensitive to changes in material behaviour. Parameters with low sensitivity can then be constrained to reduce ambiguity.
4. **Cross-Validation with Independent Experimental Data**: The properties obtained from inverse FEA should be compared against mechanical properties measured from tensile and shear tests using digital image correlation (DIC). This cross-validation ensures that the extracted properties are physically meaningful and not merely numerical artifacts.

Ensuring Reliability of Extracted Properties

To validate the robustness of the inverse FEA approach, numerical sensitivity studies were conducted to determine how variations in material parameters affect the load-displacement curve. The finite element simulations were iteratively refined by adjusting material parameters until a strong agreement between experimental and numerical curves was achieved. Additionally, mechanical properties obtained from inverse FEA were compared with experimental values derived from tensile and shear tests, showing less than 8% deviation for in-plane elastic properties and approximately 12.5% deviation for out-of-plane properties. These differences are primarily attributed to imperfections at filament interfaces in the physical specimens, which are assumed perfectly bonded in the FE model.
Furthermore, published studies on inverse FEA applications in composite materials support the validity of this methodology. Previous research [1] has demonstrated that combining nano-indentation with inverse FEA significantly improves accuracy in estimating micro-scale elastic and plastic properties. The approach adopted in this study aligns with these findings and further enhances reliability through multi-experiment validation. By integrating these strategies, the present study minimizes the risk of non-uniqueness and enhances confidence in the extracted mechanical properties of 3D-printed composites.

Advanced Correction Methods for Nano-Indentation: Inverse FEA and Microstructural Analysis of 3D-Printed Composites

Several correction methods have been proposed to address the limitations of the Oliver-Pharr method in nano-indentation measurements, particularly in materials with pile-up effects and viscoelastic behaviour. Alternative approaches include:

1. **Direct Measurement Techniques**: Atomic Force Microscopy (AFM) or Scanning Electron Microscopy (SEM) can be used to directly measure the contact area post-indentation. However, these methods are limited by resolution constraints and the difficulty of accurately capturing the true pile-up profile.
2. **Empirical Correction Models**: Some studies propose empirical correction factors based on material properties and indentation depth. While useful for certain materials, these models often lack general applicability and require extensive calibration.
3. **Finite Element-Based Methods**: These approaches simulate the indentation process to estimate material properties, allowing for corrections to be made by aligning experimental and numerical load-displacement curves. Unlike empirical models, FEA-based techniques account for material-specific behaviour, such as viscoelasticity, plasticity, and fibre-matrix interactions in composites.

The **Inverse Finite Element Analysis (FEA)** method was chosen in this study because it allows for precise calibration of material parameters by iteratively adjusting them until the numerical and experimental load-displacement curves converge. This approach not only corrects for pile-up effects but also incorporates viscoplastic properties, which are critical for accurately characterizing polymer-based composites. Additionally, inverse FEA enables a more comprehensive understanding of stress transfer mechanisms in heterogeneous materials, such as fibre-reinforced composites.

Strategic Selection of Nano-Indentation Locations for Accurate Composite Characterization

The selection of two distinct locations within the representative volume element (RVE) for nano-indentation simulations—the polyamide composite matrix and the in-situ glass fibre—was guided by the need to capture the fundamental mechanical behaviour of the composite constituents while maintaining computational efficiency. This approach aligns with prior studies on micromechanical characterization of fibre-reinforced composites, where nano-indentation is commonly performed on the polymer matrix and reinforcing fibre as the two primary load-bearing components of the composite structure [2-5]

The polyamide composite matrix was selected because it governs the deformation and load transfer characteristics of the composite and exhibits time-dependent viscoelastic behaviour that must be accurately characterized. Similarly, the in-situ glass fibre was chosen as it contributes to the overall stiffness and strength of the composite and experiences significant mechanical constraints due to the fibre-matrix interaction. Performing nano-indentation on these two key locations allows for an accurate estimation of their elastic-plastic and creep properties, which serve as input parameters for finite element homogenization.

Additionally, studies have shown that nano-indentation at fibre-matrix interfaces is highly sensitive to local heterogeneities and may not provide reliable property measurements due to the small contact area and stress concentration effects [3, 4]. Therefore, focusing on the bulk matrix and fibre regions ensures consistency in the mechanical characterization while avoiding potential errors associated with measurements at transitional interfaces.

If necessary, additional indentation locations could be considered in future work to capture local variations within the composite microstructure, such as interfacial regions or boundary effects within the RVE. However, for the scope of this study, the selected locations provide a representative assessment of the composite's mechanical behaviour with minimal computational cost and experimental uncertainty.

Ensuring Accuracy and Clarity in Data Presentation: Standard Deviation, Error Consideration, and Scientific Illustration

Standard Deviation in figures and tables where experimental works involved

- The standard deviation has been calculated for all presented data points to indicate the variability in the results. Please note that Error bars cannot be added to Figures where FEA was involved as using FEA there is no deviation to be discussed and analysed. Also figures involving experimental were updated (in the caption) with confidence limit and number of tests/locations for tests. The standard deviation values are derived from repeated measurements conducted under identical experimental conditions. This ensures statistical reliability and provides a clear understanding of data consistency.

Error in Calculation Consideration

- The sources of possible errors in the calculations have been carefully analysed, including instrumental accuracy, measurement uncertainties, and data processing limitations.
- Where applicable, confidence intervals are reported to further quantify the uncertainty in the derived results. See the highlighted sections

Scientific Illustration of Curves

- The graphical representation of the data has been refined to enhance clarity and scientific accuracy. This includes the addition of improved axis labelling, and a clearer distinction between different data sets.
- The choice of colours, line styles, and markers has been optimized to ensure the readability of the figures, even in grayscale printing.
- A detailed caption has been provided for each figure, explaining the significance of the presented data and any observed trends.

1. Gonabadi, H., et al., *Investigation of the effects of environmental fatigue on the mechanical properties of GFRP composite constituents using nanoindentation.* Experimental Mechanics, 2022. **62**(4): p. 585-602.
2. Gregory, J.R. and S. Spearing, *Nanoindentation of neat and in situ polymers in polymer–matrix composites.* Composites science and technology, 2005. **65**(3-4): p. 595-607.
3. Hardiman, M., T. Vaughan, and C. McCarthy, *Fibrous composite matrix characterisation using nanoindentation: The effect of fibre constraint and the evolution from bulk to in-situ matrix properties.* Composites Part A: Applied Science and Manufacturing, 2015. **68**: p. 296-303.
4. Hardiman, M., T. Vaughan, and C. McCarthy, *The effect of microscale residual stress from thermal cooldown on the nanoindentation properties of fibre-reinforced composites.* Journal of Composite Materials, 2016. **50**(29): p. 4147-4158.
5. Hardiman, M., T.J. Vaughan, and C.T. McCarthy, *The effects of pile-up, viscoelasticity and hydrostatic stress on polymer matrix nanoindentation.* Polymer Testing, 2016. **52**: p. 157-166.