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From Surface Activation to Delamination Resistance: Nitrogen APP Jet Effects on UD Carbon Fiber Composites for Sustainable Mobility Applications

By

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Declaration

I hereby declare that, the contents and organization of this dissertation constitute my own original work and does not compromise in any way the rights of third parties, including those relating to the security of personal data.

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Fiber-reinforced epoxy composites represent the foundation of lightweight structures for sustainable mobility applications, where delamination is often the primary failure mode limiting structural performance and durability. This thesis investigates nitrogen-based Atmospheric Pressure Plasma Jet (APP Jet) surface treatments applied to industrial-grade unidirectional (UD) carbon fiber textiles through a multiscale experimental framework that spans surface treatment design, surface characterization and mechanical testing. The work is structured around the progressive assessment of the treated reinforcements at micro-, meso- and macro-scale, with the aim of clarifying how processing parameters affect surface state and how these changes translate, when possible, into interfacial and laminate-level response.

The experimental campaign begins with the definition of a suitable APP Jet process window, based on primary and secondary plasma parameters namely jet power, stand-off plasma-substrate distance, exposure time and gas flow, in order to balance cleaning, etching and functionalization while preserving fiber integrity. Surface characterization by Optical Emission Spectroscopy (OES), Fourier Transform Infrared in Attenuated Total Reflectance (FTIR-ATR) spectroscopy, X-ray Photoelectron Spectroscopy (XPS), SEM-EDS and micro-Raman spectroscopy show effective removal of peel-ply residues and sizing contaminants, an increase in oxygen-to-carbon ratio and the introduction of polar functional groups ($-$OH, $-$C=O, $-$COOH), while preserving graphitic order and fiber morphology. These observations provide a surface-level basis for evaluating the treatment effectiveness and for defining the most suitable processing conditions.

To asses the practical value of the treatment across scales, the thesis combines microbond tests, transverse fiber bundle tensile tests (TFBT), Mode I Double Cantilever Beam (DCB) tests, and dynamic impact testing. Microbond testing is used as a first screening tool for local interfacial response, while TFBT provides a mesoscale assessment of bundle-level behavior and failure modes. At the laminate scale, DCB testing reveals an increase in Mode I fracture toughness for the treated system, whereas dynamic impact tests show a less pronounced treatment effect under high

strain-rate loading. Taken together, these results indicate that the plasma treatment is effective in quasi-static delamination-dominated conditions, but that its influence becomes less detectable when bulk damage mechanisms dominate.

A key element of the work is the critical evaluation of micro- and meso-scale testing as quantitative predictors of interface strength in industrial UD textiles. The study shows that these methods are useful for ranking surface states and for identifying qualitative changes in failure behavior, but their quantitative predictivity is limited by specimen geometry, treatment sensitivity, and the complexity of the failure mechanisms involved in industrial UD reinforcements. In this sense, the work does not treat small-scale testing as a substitute for laminate validation, but as a hierarchical screening route that helps reduce material consumption, testing time, and experimental effort before committing to full-scale mechanical characterization.

By integrating multiscale mechanical data with detailed surface analysis, the thesis defines an APP Jet process window that delivers reproducible gains in quasi-static delamination toughness on UD laminates, while maintaining compatibility with industrial constraints. Compared to traditional wet oxidation or solvent-based surface treatments, the nitrogen APP Jet approach is dry, solvent-free and inline-compatible, reducing process steps and drying times and offering clear opportunities for energy and environmental impact reduction. These surface treatment technology effects are hence presented in the thesis in terms of interfacial improvement under quasi-static conditions and according to its methodological limits of inferring laminate performance form smaller-scale tests alone. The resulting multiscale framework supports a more cautious and structured interpretation of plasma-assisted interface modification in industrial composites.