Chapter 1. Introduction

"The part of imagination in scientific work is the same as in the work of a painter or writer. It consists of cutting the real and recombining the pieces in order to create something new."

—François Jacob, Nobel Laureate (1965) in Medicine.

1.1. Background

In recent years, researchers have been exploring the utilization of emerging material systems to enhance existing engineering systems. The objective is to improve system performance, reduce the number of components, minimize maintenance expenses, achieve weight reduction, enhance corrosion resistance, increase design flexibility, improve fatigue resistance, leverage thermal and electrical properties, enhance impact resistance, achieve cost-effectiveness, ensure biocompatibility, and enhance overall reliability.

Composites are materials that are created by combining two or more chemically distinct phases on a macroscale. These phases retain their individual qualities while also exhibiting superior properties that neither constituent possesses alone. Typically, the reinforcement phase, which is stronger and stiffer, is discontinuous and embedded within the continuous and less stiff matrix phase. Although the materials retain their physical identities within the composite, they do not blend or dissolve into each other. The composite material is employed as a promising material to satisfy the aforementioned qualities. The classification of composites includes Fibre-reinforced polymer (FRP) composites, nanocomposites ..etc. This thesis specifically concentrates on the investigation of nanocomposites.

1.1.1. FRP Composites

FRP composites are composite materials comprised of a epoxy matrix reinforced with fibers such as glass, carbon, or aramid (**Fig. 1.1**). Fabricating FRP composites involves a series of steps. Initially, the fibers are impregnated with the resin using techniques like hand lay-up, vacuum infusion, or filament winding. Subsequently, the composite is shaped into the desired form utilizing processes such as molding, curing, or pultrusion. This fabrication procedure guarantees the uniform distribution of fibers within the epoxy matrix, thereby enhancing the mechanical properties of the composite.

Fabricating FRP composites involves employing various commonly used methods. The selection of a specific fabrication method is based on factors like the desired shape, size, complexity, and mechanical properties of the end product. Here are several types of fabrication methods for FRP composites: Hand Lay-up, Spray-up, Filament Winding, Resin Transfer Molding (RTM), Vacuum Infusion, and Pultrusion.

FRP composites have diverse applications (as some examples in **Fig. 1.3**) in industries such as aerospace, automotive, construction, marine, sporting goods, renewable energy, electrical, medical, and defense. Their unique properties, including lightweight, high strength, corrosion resistance, and design flexibility, make them suitable for a wide range of industries.

The failure analysis of FRP composites aims to investigate the reasons and mechanisms behind their failure or deterioration. This analysis provides insights into the limitations of FRP composites and enables improvements in design and manufacturing processes to enhance the safety and reliability of structures made from these composites. Common failure modes observed in FRP composites include Fiber Breakage, Delamination, Matrix Cracking, Fiber-Matrix Debonding, and Environmental Degradation. FRP composites are subject to limitations such as cost, manufacturing complexity, challenges in detecting damage, sensitivity to high temperatures, and constraints in design allowances. The presence of these failure mechanisms, limitations, and the need for additional functional properties drive our attention towards nanocomposites.

1.1.2. Nanocomposite

A nanocomposite is a material composed of a bulk matrix and nanoparticles. Nanoparticles, which are extremely small particles mean it's at least one dimension should be less than 100 nm, are dispersed within the matrix, altering its properties (**Fig. 1.2**). This can enhance mechanical, thermal, electrical, or optical characteristics. For instance, adding nanoparticles to a polymer can increase its strength, while incorporating them into a ceramic can enhance thermal conductivity. Nanocomposites can exhibit superior properties like electrical conductivity, transparency, or magnetism compared to bulk materials. Nanocomposites are synthesized by uniformly dispersing nanoparticles into the matrix using techniques like solution mixing or in-situ synthesis. The choice of nanoparticles and matrix material depends on the desired properties and applications. Nanocomposites find use in electronics, aerospace, automotive, energy, medicine, and environmental remediation. Ongoing research focuses on exploiting their unique properties and tailoring functionalities for specific purposes. Various nano reinforcements, such as Carbon Nanotubes (CNT), Graphene, Nanoclays, Metal Nanoparticles (e.g., silver, gold, platinum), Ceramic Nanoparticles (e.g., alumina, titania, zirconia), Quantum Dots, Nanowires, Nanofibers, rubber, Nanoplatelets, Nanoporous Materials, Magnetic Nanoparticles, Silica Nanoparticles, Nanodiamonds, Layered Double Hydroxides, Nanosheets (e.g., boron nitride, molybdenum disulfide), Metal-Organic Frameworks (MOFs), Carbon Nanofibers can be integrated into a matrix material to create nanocomposites. Each type of nano reinforcement has unique properties that enhance specific characteristics of the resulting nanocomposite. The selection of a nano reinforcement depends on desired properties, intended applications, and compatibility with the matrix material[1][2].

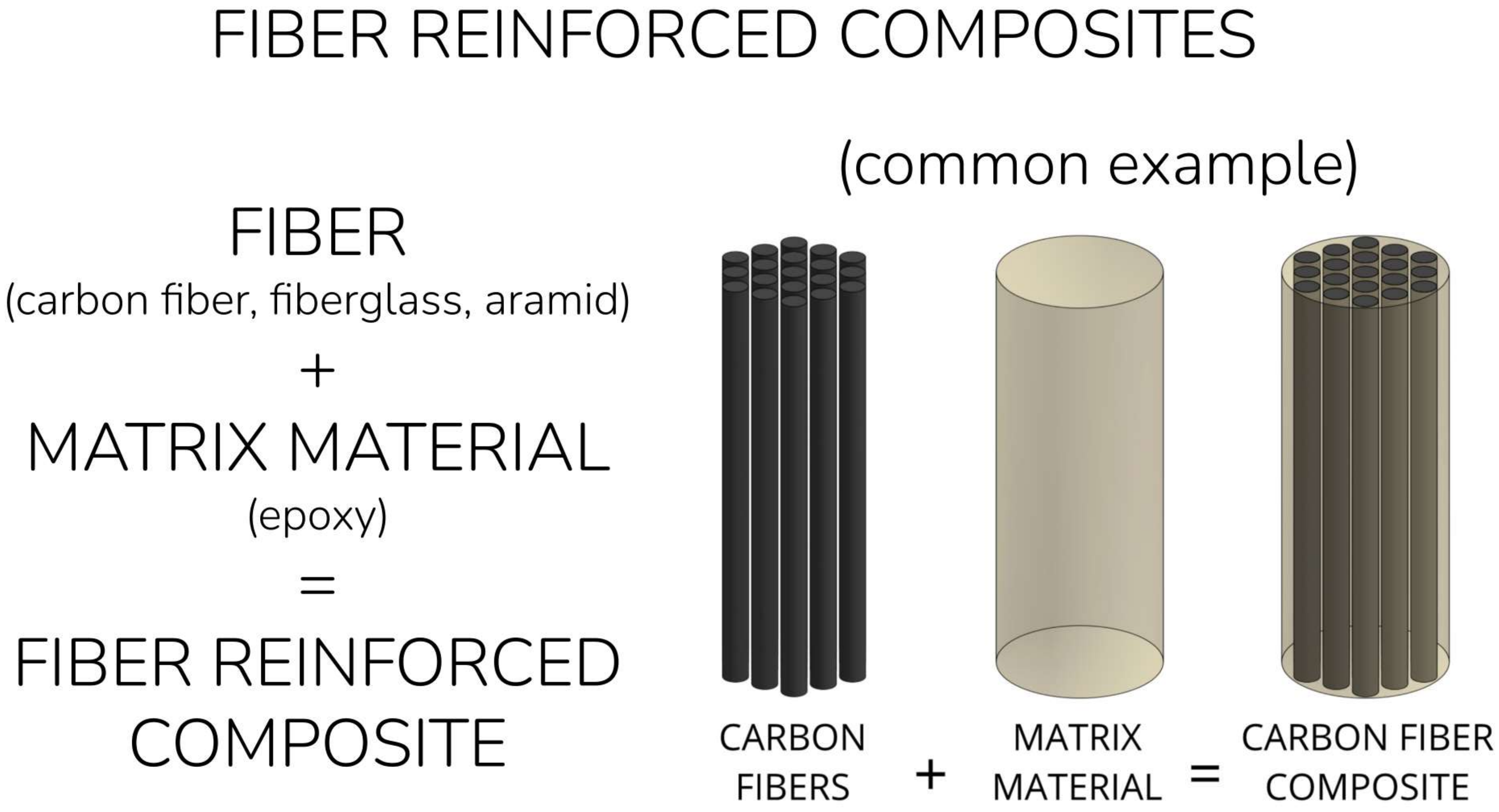

Fig. 1.1 Shows the exampleof FRP composite ([73] and source Internet)

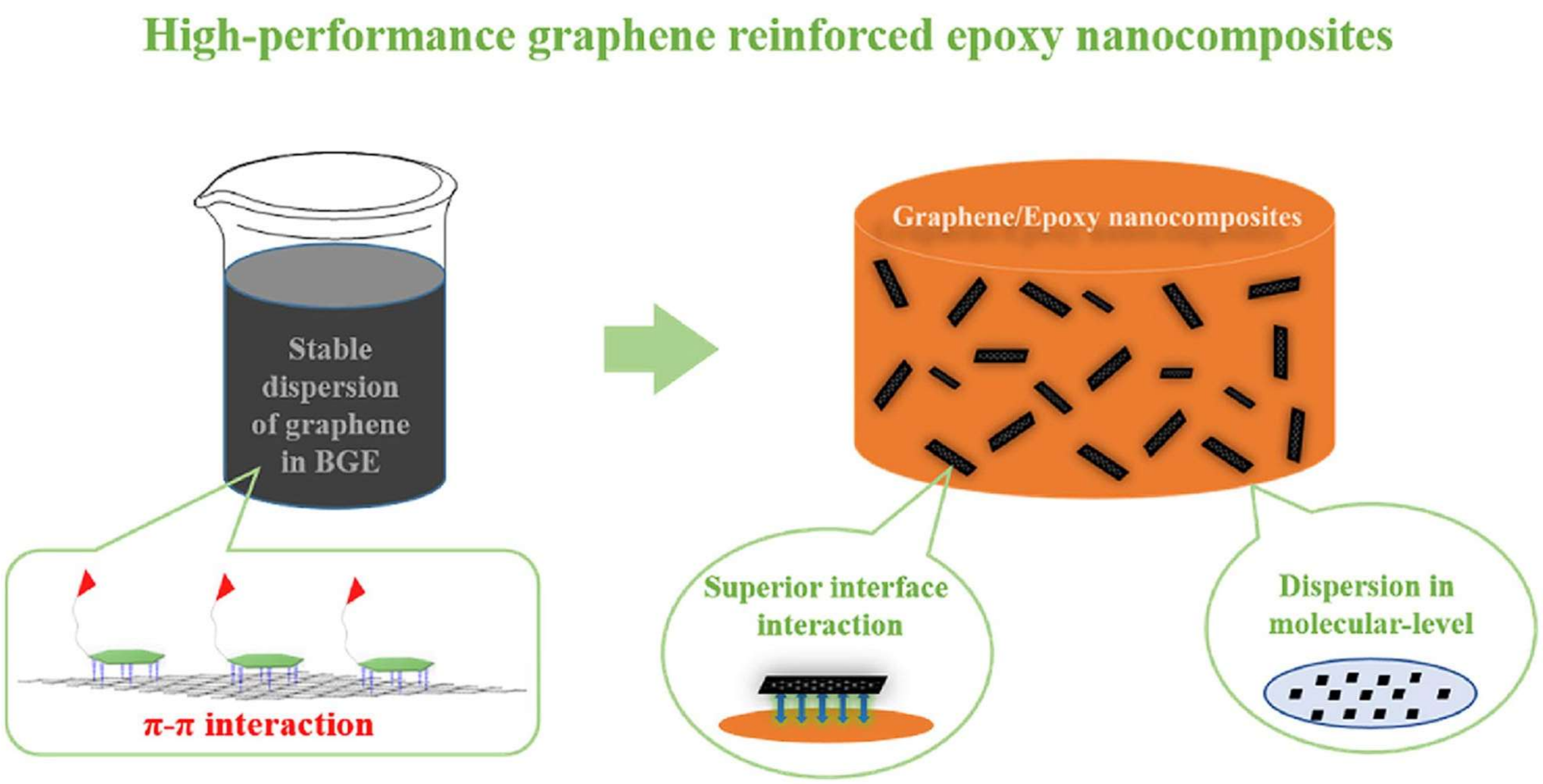

Fig. 1.2 Depicts the example of nanocomposites[73]

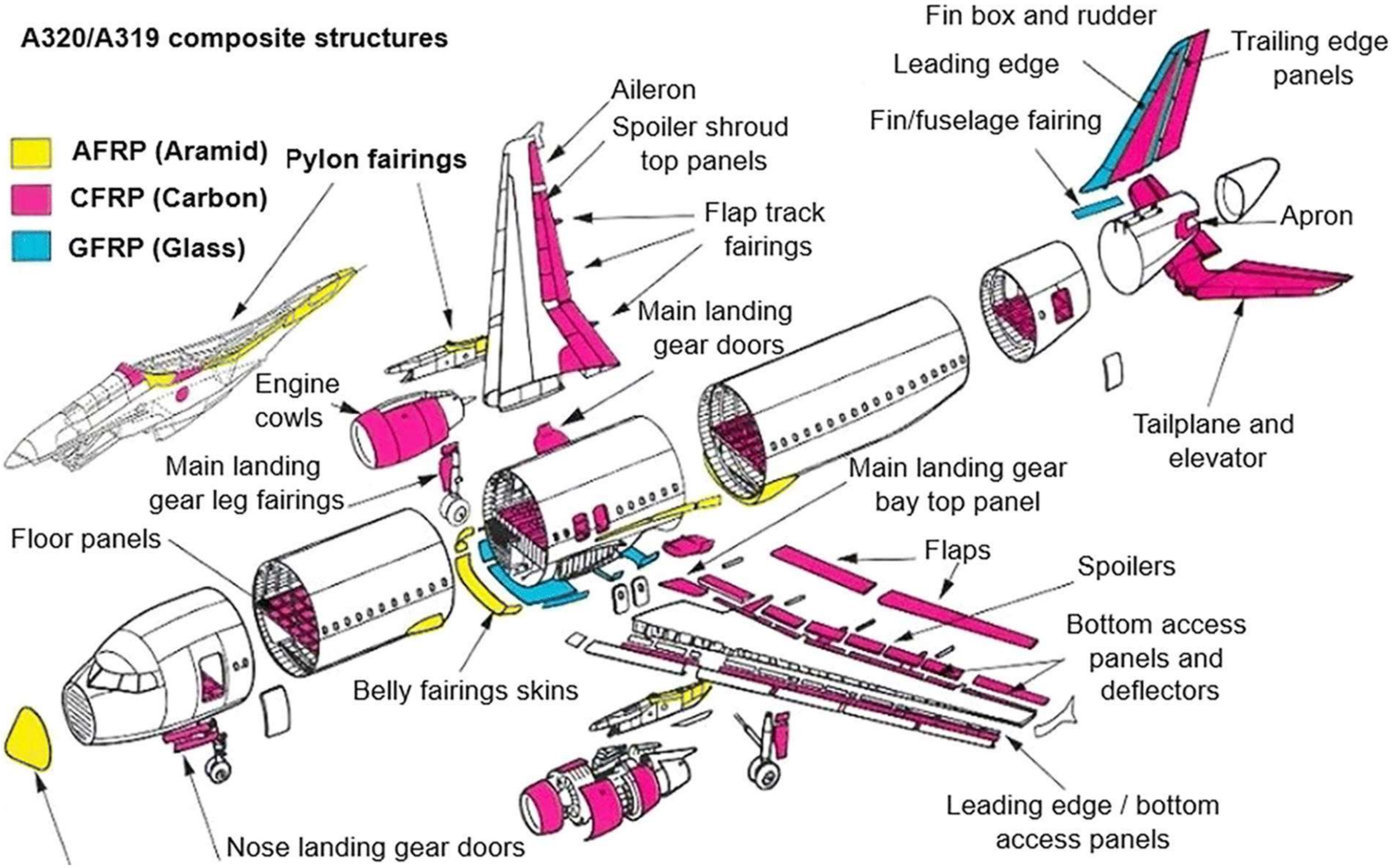

Fig. 1.3 Shows components made of FRP composite used in Airbus[284]

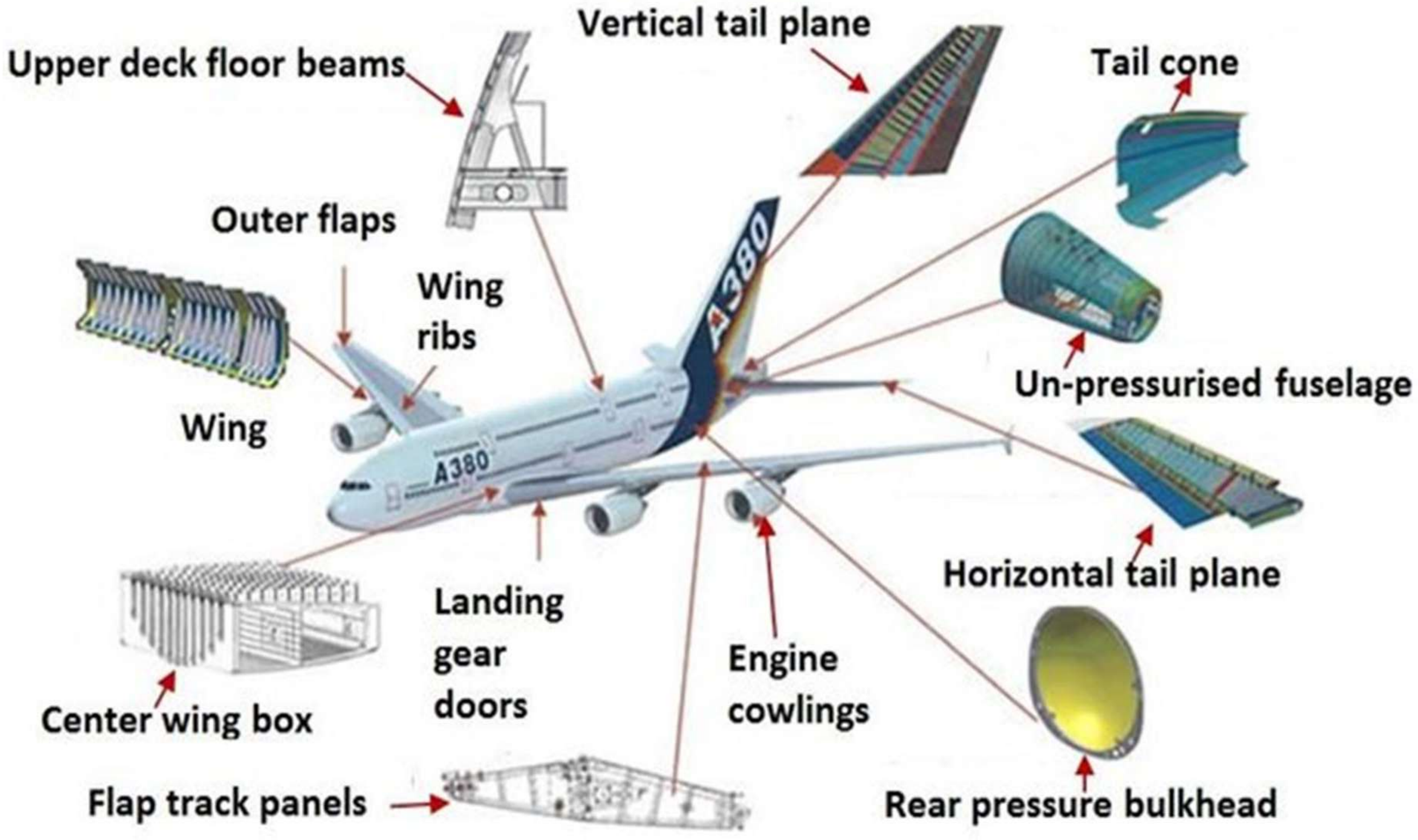

Fig. 1.4 Shows components made of nanocomposite used in Aerospace [285].

1.2.Motivation

For structural composite material applications, epoxy have attracted significant interest from researchers. Epoxy is extensively used as a matrix in aerospace, automotive, marine, construction, shipbuilding, and electronics composite materials. Because of their densely cross-linked structure, they have a high glass transition temperature, stiffness, modulus, creep resistance, low shrinkage at higher temperatures, dimensional stability, and chemical resistance.Leveraging these distinctive attributes, researchers have successfully developed reinforced composites characterized by exceptional specific strength, corrosion resistance against various chemicals, and dimensional stability at higher temperatures [3–17].

Composite materials are widely used in various applications. However, their brittleness and low resistance to crack propagation make them unsuitable for load-bearing applications, where fracture and fatigue crack growth rate (FCGR) resistance could lead to catastrophic failure or partial loss of functioning in the parent structure. Instead, composite materials are commonly used as adhesives or matrix materials for fibre composites. Epoxy which are widely used as matrix materials, have poor mechanical, chemical, and thermal qualities when uncured. Upon curing with an appropriate agent, they form a thermoset 3-D cross-linked structure[18], significantly improving their properties. However, cured epoxy has lower strength and fracture toughness, limiting its application in mechanical parts. Robust measures are needed to prevent catastrophic failures[13]. To enhance these qualities, researchers have explored reinforcing various type of nanoparticles and microparticles as : rubber, rigid core-shell and hollow glass spheres/bead, silica [5–9,14–17,19–23] ZnO nano-particle ,titanium dioxide (TiO2), aluminium oxide (Al2O3) [11,24–26]epoxidized hyperbranched polymer[27], carbon black ,Carbon nanotubes/nanofibers, Graphene, SBR ,NR[3,4,28–44]with varying wt% as well as its synergistic ratio epoxy with different types of nanoparticles, and fabricating nanocomposites.

Researchers have reached a consensus that carbon nanomaterials, including Graphite, carbon black, CNT, and Graphene, exhibit superior characteristics as nanofillers in comparison to other reinforcement particles. This leads to improved strength, fracture properties, fatigue properties like fatigue thresholds and resistance to fatigue crack growth rate, and various other functional properties. Moreover, researchers have discovered that Graphene, which is a two-dimensional (2D) carbon nanomaterial, stands out as the most remarkable among these carbon nanomaterials due to its distinct properties. These properties set Graphene apart as a superior nanofiller compared to other types of carbon nanoparticles. However, its instability limits structural applications. Efforts to modify the properties of epoxy through graphene nanocomposites have not yielded the anticipated results as per theoretical predictions. However, addressing challenges related to exfoliation, dispersion, the spatial orientation (e.g., alignment) of graphene in epoxy, and interfacial bonding can lead to significant improvements in graphene epoxy nanocomposites, making them more suitable for structural applications[10,45–54].

Consequently, there are various methods available for the alignment of carbon-based 1D and 2D nanoparticles before the gelling process of the hosting thermosetting polymer, including those that utilize an electric field [55–58]. Mechanical stretching has also been employed as a means of alignment [59,60]. Additionally, a magnetic field has been suggested as another possible alignment method[61,62]. Graphene and ceramic particles have been uniformly oriented with the help of an electrical medium in targeted areas of polymer composite[63]. Donglu Shi and Peng He et al [64] research focused on the magnetic alignment of carbon nanofibers in polymer composites. Shuying Wua and Raj B. Ladani et al [57] proposed method for aligning GNP in epoxy involves exposing the material to an AC electric field. The alignment process was investigated through a combination of experimental and theoretical approaches. Erin et al.[61]conducted a study on the alignment of carbon nanotubes (CNT) through the use of magnetic fields up to 25 T. Their findings indicated that the orientation of CNT has a significant impact on the mechanical properties of the resulting nanocomposite. However, due to the low magnetic susceptibility of carbon-based nanomaterials, a relatively high magnetic field is typically necessary, which greatly limits its practical applications. Therefore, achieving magnetic alignment of carbon nanomaterials for the cost-effective production of nanocomposites has remained a challenging endeavor [65].

The aforementioned discussion serves as a motivation to pursue this thesis further. In this thesis, The aim was to develop a mathematical model that optimizes the alignment parameters of GNP and Fe_3O_4-GNP within an epoxy matrix. This optimization is achieved by utilizing a weak DC magnetic field (0.05T) during the alignment process. The characteristic magnetic, viscosity and hydrodynamic parameters required by the mathematic model are determined experimentally by synthesizing the magnetite ferric oxide (Fe_3O_4) and attaching them to GNP to increase its magnetic susceptibility. A highly aligned Fe_3O_4-GNP nanocomposite is fabricated at 0.05T magnetic field and 40Pa-s dynamic viscosity of epoxy. The aligned Fe_3O_4-GNP nanocomposite undergoes a thorough characterization utilizing various techniques such as XRD, FTIR, Raman spectroscopy, TGA, DSC, AFM, XPS, BET, TEM, SEM, EDXMA, and VSM. Subsequently, the effect of incorporating aligned Fe_3O_4-GNP nanoplates into epoxy nanocomposites is examined. The investigation focuses on evaluating parameters including crack growth resistance, K_{IC}, G_{IC}, $CTOD_c$ fracture mechanisms, fatigue thresholds (ΔK_{th} and ΔG_{th}), and fatigue crack growth rate (FCGR) resistance. A comparative analysis is conducted, considering different weight percentages of both aligned and randomly oriented GNP nanoparticles. The findings indicate a notable improvement in the properties of the nanocomposites.

The exceptional properties of CNT make them ideal nanoparticles for reinforcing epoxy, enhancing its stiffness, strength, and fracture toughness[66,67]. Theoretical models, such as molecular dynamics, analyse fracture toughness, while CNT orientation impacts Mode I toughness[68–71]. Interphase properties and CNT weight fraction influence fracture toughness in the nanocomposite system[72]. Further research is needed to fully comprehend the multi-phase mechanism and the impact of multi-scale physical constituents. This motivates the additional analysis in this thesis, which includes a multi-scale model and a comprehensive mechanism model. These models consider progressive interphase debonding, cavitation, and plastic deformation of nanovoids near crack tips during open-mode loading. Stress distributions across nano, micro, and macro scales are considered using a cylindrical RVE comprising CNT, interphase, and neat resin. Variables like CNT weight fraction and interphase thickness influence debonding stress and fracture toughness.

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