Scanning Electron Microscopy

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<u>You only have to answer the questions in this handout,
there is no need for a full write-up.</u>

Specimen 1 - Dragonfly Wing

Q1. *Comment on the structure of the wing of the dragonfly, in particular, how does it compare with conventional aerostructures (think about the components in an aircraft wing). [5 marks]*

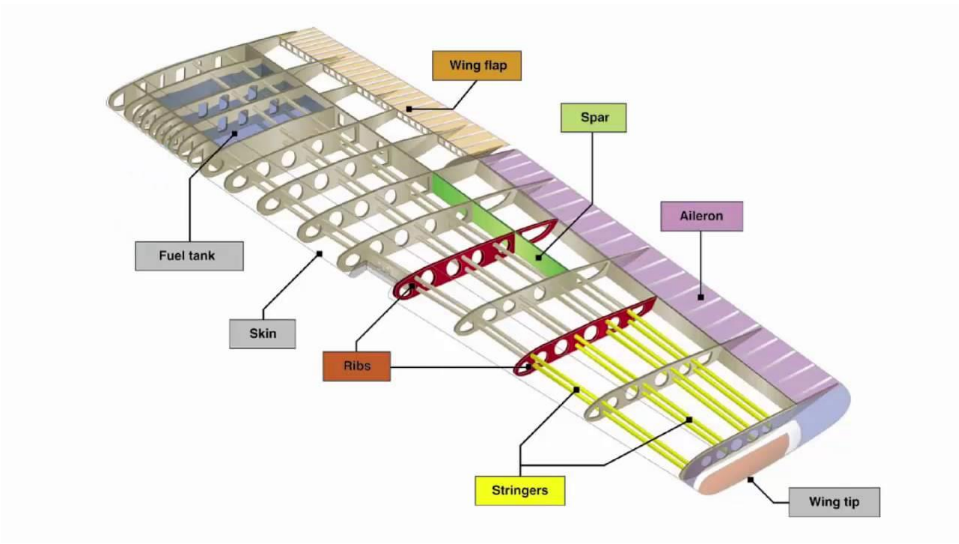

Figure 1. A typical skeleton view of a modern aircraft wing (1).

There are distinct spanwise veins along the wing, with shorter lateral veins in between supporting the membrane of the dragonfly's wing. This layout bears close resemblance to the spars and ribs respectively, found in a modern aircraft wing in Figure 1. These are structural members that resist bending and torsional forces to maintain the wing's shape under loading. Likewise, these veins serve the same function.

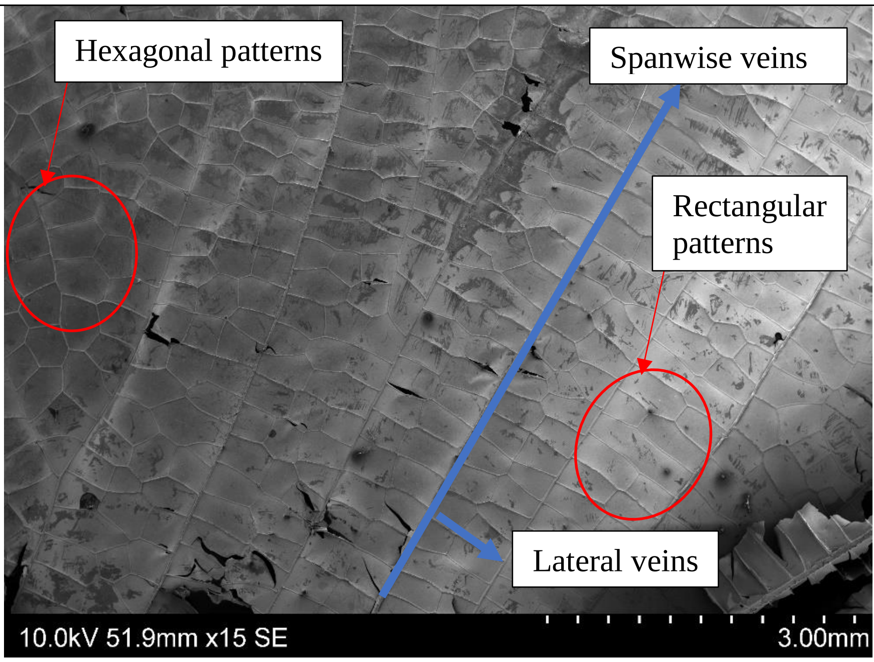

Figure 2. Dragonfly wing at 15X magnification. Note the change in the patterns from hexagonal to rectangular from left to right

While ribs of an aircraft are typically perpendicular to the spar, the dragonfly's wings have curved and angled lateral veins or "ribs", running between. Shorter spanwise veins connect the lateral veins. From Figure 2, a change in pattern of the lateral veins can be observed, from hexagons to long rectangles. The complex flapping cycle and manoeuvrability of the dragonfly results in multiple loading directions. This pattern of lateral veins will accommodate the different loadings across the wing.

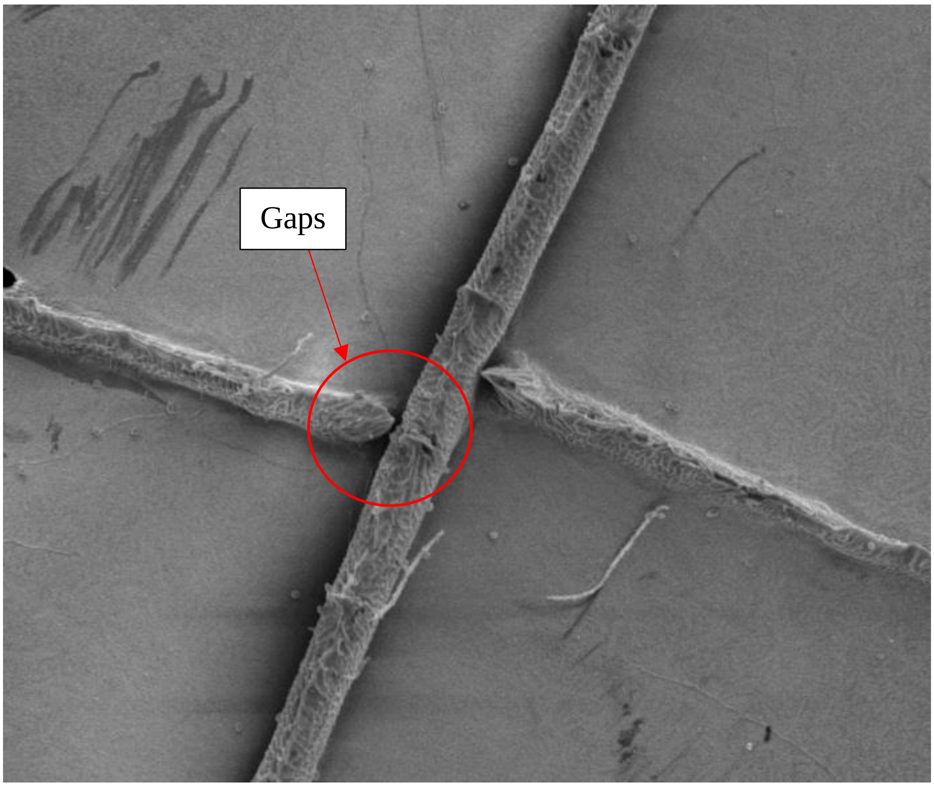

Figure 3. A cropped view of the dragonfly wing at 100X magnification. Note the gaps between the spanwise veins and the lateral veins

At higher magnifications in Figure 3, gaps between at the joints of 2 membranes can be seen. Soft and thin wings of insects such as the dragonfly are known to experience aeroelasticity during flight (2) . Each stroke of the flight cycle generates different loadings on the wing, causing them to deform. Changes in temperature of the surroundings (night to day) can cause thermal contraction and expansion of the wing structure. Thus, these gaps facilitate these forms of elasticity. Such allowances for movement are seen in the SR-71 Blackbird, whose titanium-alloy body panels had gaps built in to allow for eventual expansion by aerodynamic heating due to the high operational Mach numbers.

This corrugated structure acts as a skeleton for the membrane to form a light weight but effective lift generating surface. Once again, this is like the wing structure of early gliders such as the Wright Glider, which had a cotton fabric covering the wing and forming the lifting surfaces. However, these thin membranes will be prone to punctures that will destroy the lift generating capabilities of the surface.

Q2. *Although the images from the electron microscope look very similar to those from optical (and visual) images, there are a number of 'artefacts' particular to this technique for examining surfaces. Comment on what they are, what could be done to mitigate for them. [5 marks]*

The nature of the SEM technique and the handling and preparation of specimens play a role in the appearance of such artefacts. To the inexperienced, these artefacts appear to be part of the specimen, but it requires much experience to discern them, understand their source and to learn to mitigate them.

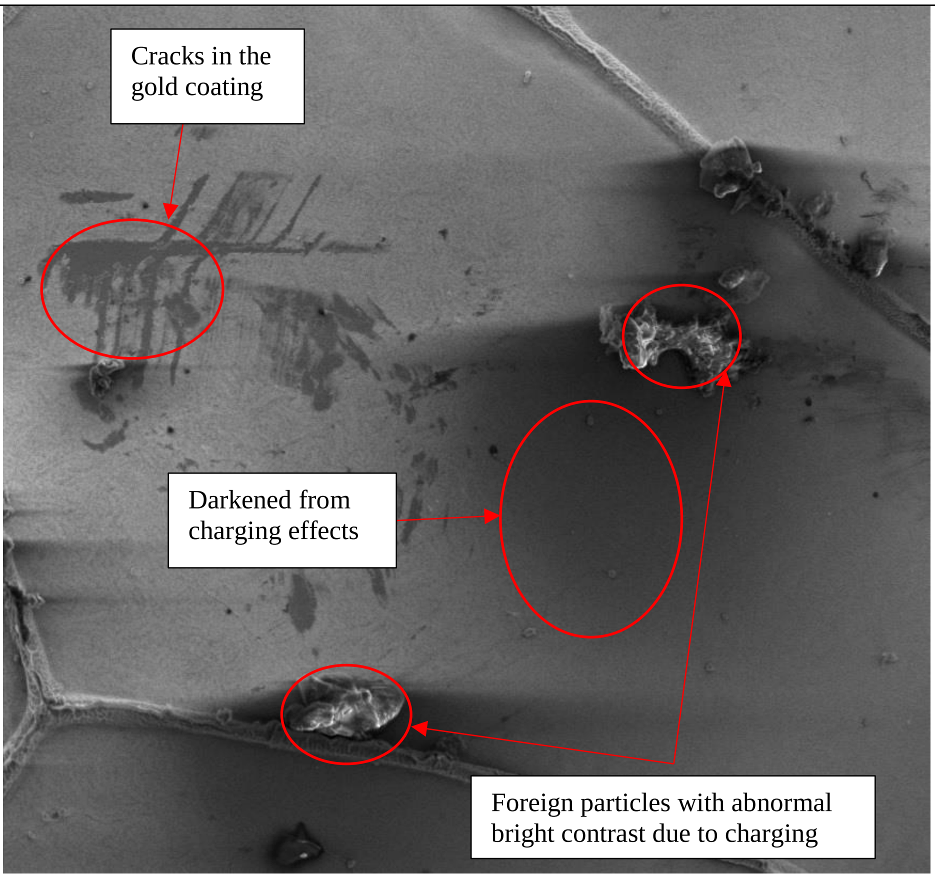

Figure 4. The cropped image of the dragonfly wing at 100X magnification with some artefacts

Artefacts from charging effect

Charging effects are a result of the built-up of electrons on the specimen. During the operation of the SEM, an electron beam bombards the specimen with electrons. If the number of incident electrons exceeds the number escaping the specimen, charging occurs. The charging effect causes a wide range of artefacts; from smear lines, black holes and spots, white and black halos or bands of white and black. Bright flashes on the image are a result of the sudden release of built-up electrons on the specimen. Dark spots and abnormal contrast were observed in the dragonfly wing specimen in Figure 4.

Methods to mitigate these include re-sputtering with gold or platinum, a pre-treatment of tetroxide vapour to enhance the conductivity of regions (such as joints) not effectively coated by the metals (3). Alternatively, the user may wish to reduce the power of the electron beam.

Artefacts from handling, preparation and storage

Specimens are treated in the process known as sputtering, where they are coated with a fine layer of metal (gold or platinum are some examples). In the case of the dragonfly wing, it was coated with a layer of gold. If the powder is not applied evenly or portions of it has worn or cracked off, the effect manifest itself in dark spots on the image. An example of a crack surface can be seen in Figure 4. The thickness of the gold layer must be precise; too thick and metal particles may appear in the specimen, too thin and the specimen may not be adequately coated. Care must be taken during application and expectations managed due to the complex topography of certain specimens.

If the specimen chamber is not kept clean or the storage method inappropriate, foreign particles such as dust, insects and other debris can contaminate the specimen. Ideally, the specimens should be stored in a vacuum to mitigate contamination. It will be ideal to take upmost care during the initial preparation and testing to obtain clean images of the specimen, which can be referred to in the event of artefacts turning up over the course of the specimen's life.

As the electron beam loses energy in the form of heat to the specimens (3), considerations must be taken when placing heat-sensitive specimens under an SEM. Polymers, proteins and some volatilise waxes may melt when exposed to the electron beam, contaminating the specimen chamber. The energy of the electron beam can be lowered to mitigate this. Alternatively, the working distance of the beam can be increased to enlarge the beam's spot size, with a trade-off in resolution of the image.

<u>Artefacts due to edge effects</u>

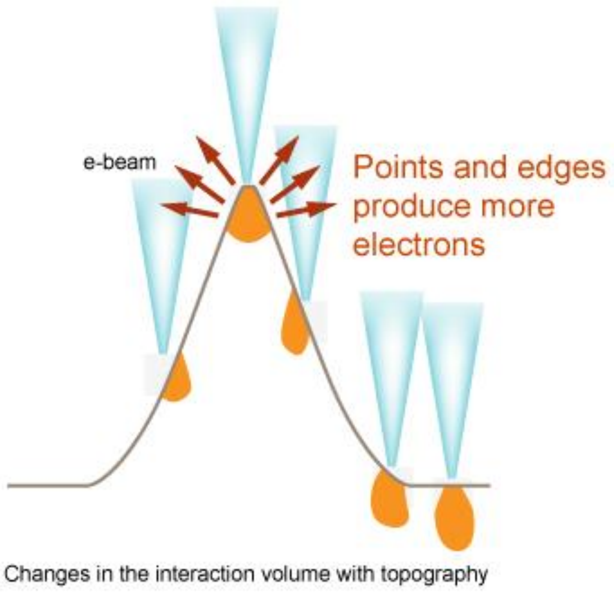

Figure 5. Edges and peaks are where electrons naturally flow to and emit from (3)

The topography of a specimen may involve edges and peaks, to which electrons preferentially flow to and are emitted from. This leaves areas, such as depressions, shielded by the edges and peaks with a poor signal intensity. This is illustrated in Figure 5. This effect, in moderate amounts, gives form and outline to the images produced by the Secondary Electron detector. Lowering of the beam energy will help to reduce this effect when it is too pronounced.

Specimen 2 – Translaminar Fracture of Unidirectional CFRP

Q3. *The fracture of the specimen is characterised by two types of fibre failure mode. Comment on these fracture modes, explaining the processes by which the fibres have failed. [15 marks]*

From examining the fracture surface, the specimen is concluded to have failed by a combination of tension and compression.

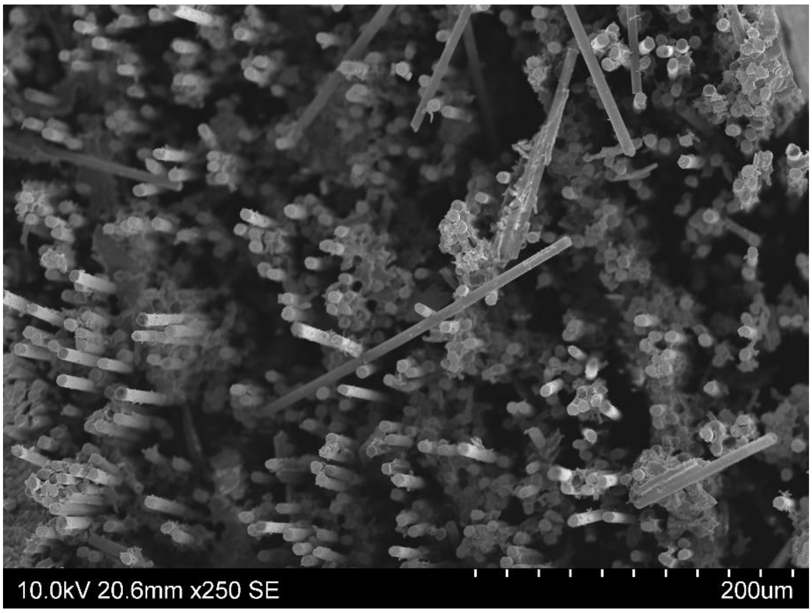

Figure 6. Fracture surface at 250X magnification. The fibres in this region have failed by tension

Failure under tension

From Figure 6, the fibres at various lengths are seen with a flat and smooth fracture surface. The dark regions indicated that the fibres are at different heights due to the edge effects of the SEM. These features suggest the fibres in this region failed due to tension. The orientation of the fracture surface will indicate the direction of tensile loading.

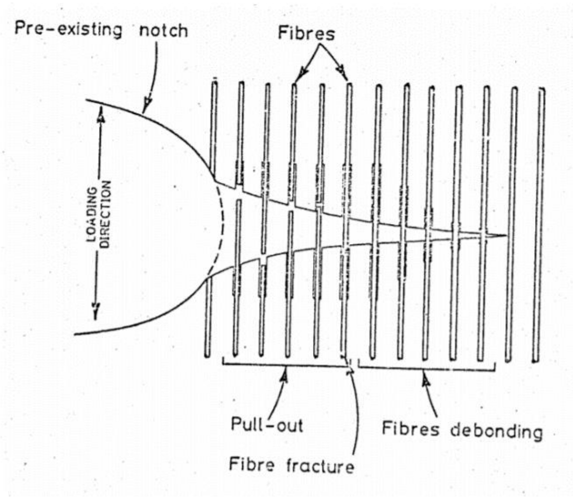

Figure 7. An illustration of how fibres are pull out as the crack propagates (4)

When material is loaded in the tension and a crack propagates through the material, shear stresses will be experienced parallel to the fibre direction. As the crack propagates through the matrix, the fibres will have to be pulled out in order to extend the crack. The friction between the fibres and the matrix creates non-uniform stress distributions along the length of the fibre. The fibres will fail when loaded to their fracture strength, with the exact location of failure determined by the Weibull distribution of flaws along the length (4). This would occur at high fibre/matrix interface strength. If the strength is low, there will be a high degree of fibre debonding. Additional work must be done by the crack to overcome the friction needed pull and separate the fibres from the matrix. This process is known as fibre pull-out or fibre debonding as is describe by the Cook-Gordon mechanism (5).

The combination of fibre fracture and fibre pull-out resulted in the different heights of fibres seen in Figure 6. The additional work done needed to; fracture the fibres, pull out the fibres and/or shear the matrix parallel to the fibres in order to propagate the crack are energy-absorbing mechanisms that increase the fracture toughness of the composite.

Failure under compression

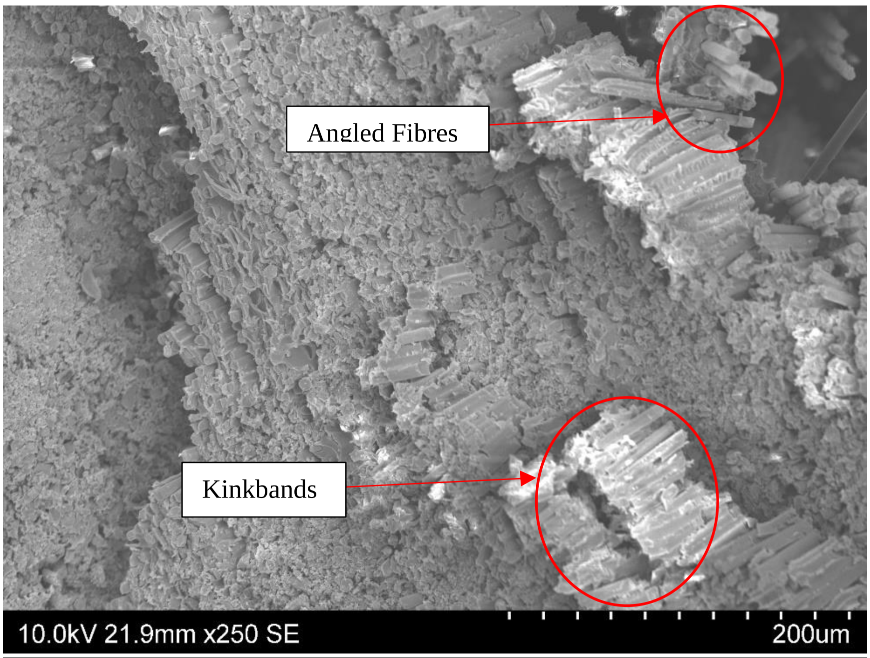

Figure 8. Fracture surface at 250X magnification. The fibres in this region have failed by compression

The surfaces of a compression fracture are often angled to the loading direction due to the in-plane shear loading during fracture. Bundles of fibres of equal length can be seen in Figure 8, indicating a collective failure at the same length, leading to the formation of “kinkbands”. The appearance of kinkbands characterises compression failure. 2 methods describe the failure mechanisms that form kinkbands.

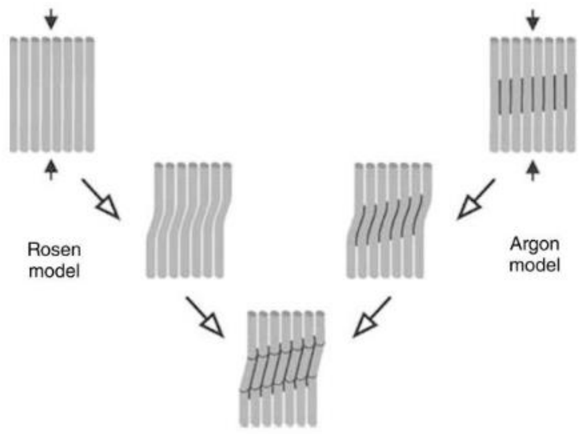

Figure 9. The Rosen Model and Argon Model describe the formation of kinkbands during compression failure (5)

The Rosen Model suggest the micro-buckling of the fibres occurs without prior fracture. It describes the domino effect due to the load shedding from the failure of the initial fibre. The initial fibre would fail when the lateral stress within the fibre exceeds the support providing by the surrounding matrix. In this model, the matrix stiffness would heavily influence the kinking process. The Argon Model suggest that ply-splitting occurs ahead of micro-buckling. A zone of ply-splits would develop ahead of the kinkbands. Here, the shear strength of the matrix influences the kinking process. It is likely that compression failure is a mixture of both models (5).

Q4. *Comment of the distribution of the two fracture modes over the specimen. Based on these observations, suggest the global loading mode and direction by which the specimen failed. [10 marks] + [10 additional marks]*

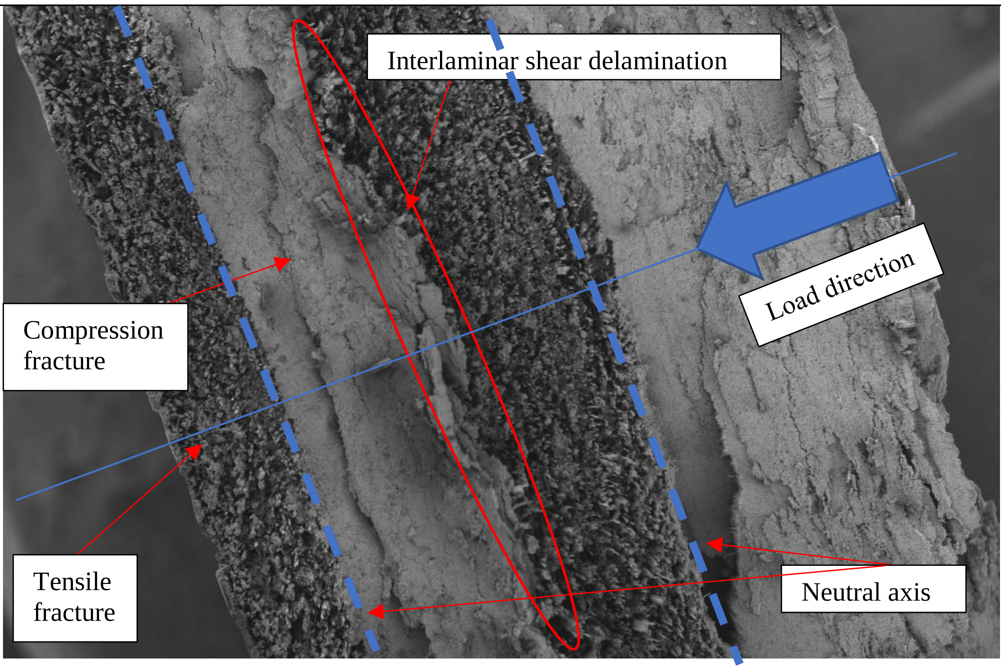

Figure 10. Cropped view of the fracture surface at 16X magnification. 2 different types of fracture surfaces are clearly visible

From Figure 10, alternating regions of a fibrous fracture surface and a flatter and smoother surface can be observed. These correspond to the tensile fracture and compressive fracture regions discussed in Question 3. This fracture surface is characteristic of flexural failure (5).
Since the outer layer on the left failed by tension and by compression on the right, the specimen is concluded to have been subject to a 3-point bending test seen in Figure 11. The loading direction is indicated by the blue arrow in Figure 10. The neutral axis divides the 2 failure regions and is a region of no direct stress. The tensile fracture area is noticeably smaller than that of the compression.

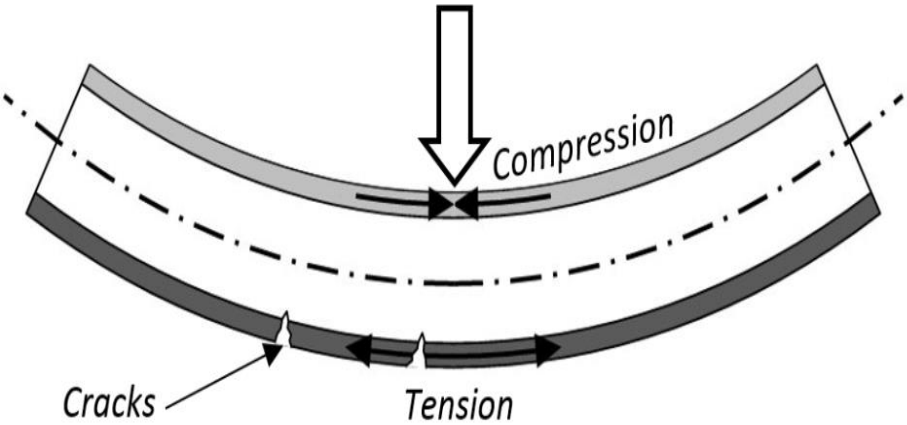

Figure 11. A beam during a bending test. Note the compression region is within the concave side of the beam and the tension region is in the convex. Photo courtesy of the Fraunhofer Institute.

When the load is applied, the crack begins to propagate from the compression side due to the comparatively lower strength in compression than in tension. It propagates towards the neutral axis. Meanwhile, the tensile crack initiates and propagate towards the neutral axis. At the neutral axis, both compressive and tensile stresses are decreasing with an increase in interlaminar shear stresses. This increases the chances of interlaminar shear delamination. This has occurred in the specimen provided as indicated by the delamination region in Figure 10. This delamination appears to have “split” the specimen, causing it to behave as 2 separate composite beams, each with regions of tension and compression. This will explain alternating region of tension and compression fracture, with each set of tension and compression fracture surface divided by the delamination region.

Specimen 3 – Interlaminar Fracture of Unidirectional CFRP

Q5. *Delamination is a very important fracture mode in laminated composites, and the specimen shows the associated fracture morphology. Comment of local fracture mode of the matrix between the fibres, and suggest a process by which such a morphology could form. [10 marks]*

Delamination of a laminated composite occurs due to excessive out-of-plane or interlaminar stress at the interfaces between plies and is dominated by matrix fractures. Delamination can be split into 3 different modes shown in Figure 12.

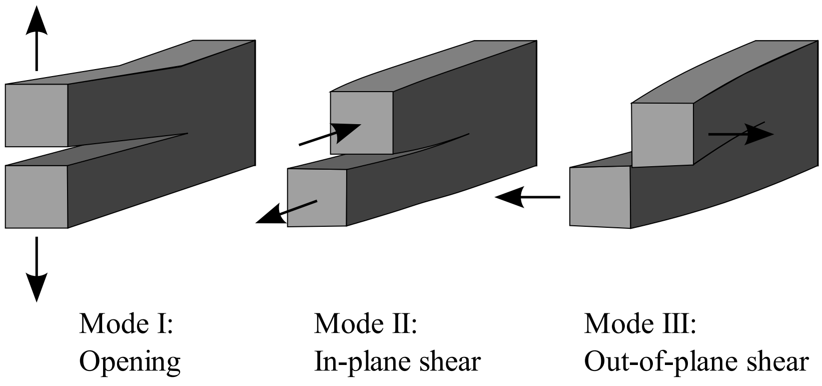

Figure 12. 3 modes of crack propagation. Mode 3 is negligible during delamination of laminated composites (5).

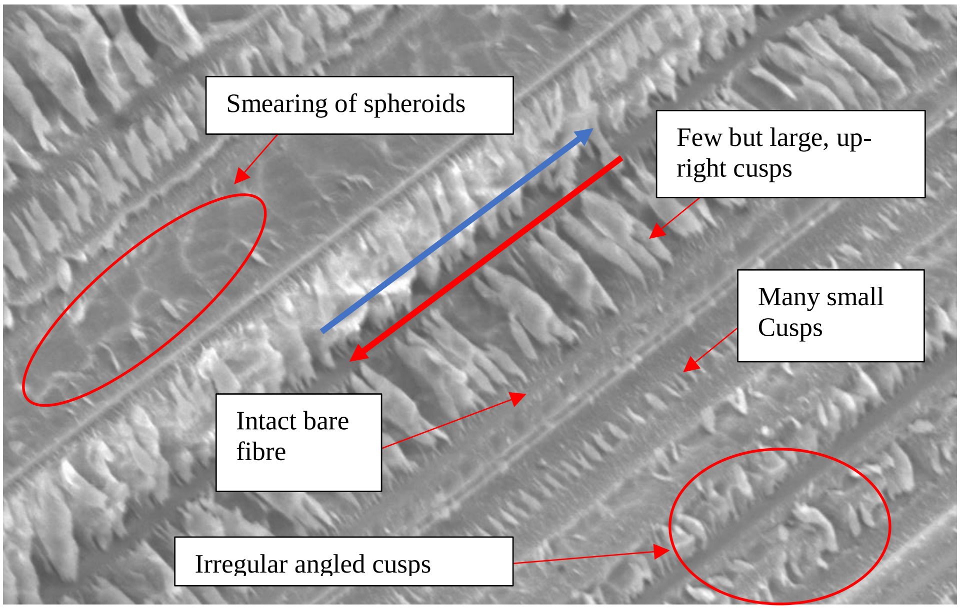

Figure 13. The fracture surface due to delamination at 500X magnification.

The clear and intact fibres indicate a matrix failure. From Figure 13, raised curled platelets of material can be seen in the regions between the intact fibres. These are known as cusps and their presence is indicative of a Mode II type fracture. The directions of their tilts indicate the relative shear directions of the matching fracture surfaces. In Figure 13, the concave side of cusps are leading towards the bottom-left of the picture, indicated by the red arrow. For this Mode II fracture, the surface shown has moved towards the bottom left while its matching fracture surface has moved to the top-right indicated by the blue arrow. An example of how the shear directions would have resulted in the surface shown in seen in Figure 14.

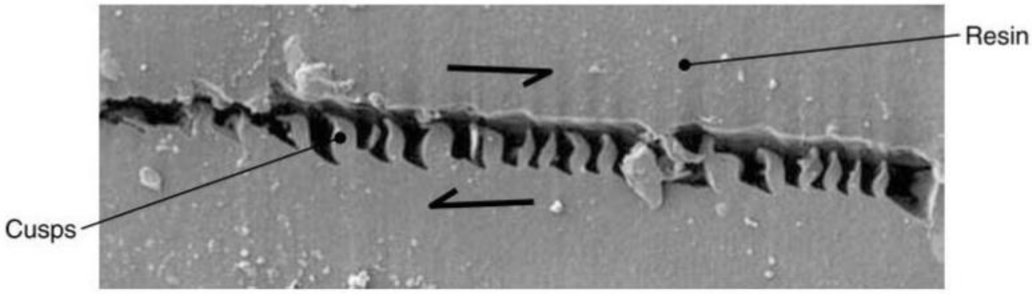

Figure 14. How shear directions influence the directions of cusps (5)

As the shear load on the composite specimen increases, micro voids nucleate at the interface of the matrix and fibre and grow larger with increased plastic deformation. They grow across the fibre to the adjacent matrix, which is why they are perpendicular to the fibre direction. The separated portion of the matrix “curls” back as the shear load increases, eventually coalescing into a fracture surface upon failure, creating cusps.

Cusps sizes are affected by moisture content, temperature and processing conditions and the matrix toughness. Larger and thicker cusps are indicative of greater plasticity. Distribution of cusp also describe the resin content of the regions. Regions with large few cusps have high resin content while many small cusps indicate low resin content. This distribution can be seen in Figure 13.

Notably, the existence of thin, irregularly angled cusps suggests some Mode I type failure is present (5). Therefore, the specimen failure is probably a mixture of Mode I and II failure, with Mode II being the dominating failure.

Q6. *The matrix features on this fracture surface provide an indication of the global loading and direction on the specimen. Can you deduce what that was? [5 marks]*

The global loading was an in-plane shear loading and the direction of shear can be determined by the direction of tilt of the cusp.

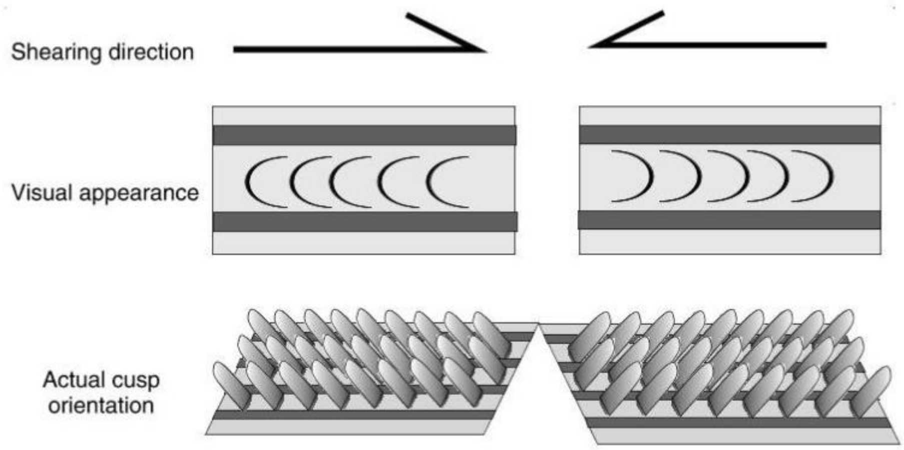

Figure 15. Direction of cusps tilt indicates the shear direction (5)

From Figure 13, the cusps are tilted to the bottom-left due to the direction of shear. Referencing Figure 14 and Figure 15, it can be concluded that the direction of shear would be opposite to the direction of tilt of the cusps, towards the top-right.

Specimen 4 – Zinc Charpy Impact test at $T/T_m = 0.1$; Brittle Fracture

Q7. *What are the key features of this fracture surface? Based on these features, explain how the crack has propagated through the metal? [10 marks]*

Figure 16. Fracture surface at the zinc specimen at 500X magnification

From Figure 16, the surface on the fracture consists of many smooth angular planes, many of them parallel to each other. There is little sign of major plastic deformation. As the impact test temperature was conducted at 10% of the specimen's melting temperature, the specimen had a reduced ductility. Based on this, it is deduced the failure happened due to a brittle fracture.

Brittle fractures are sudden, and the crack propagation occurs by a process known as cleavage. The smooth angular surfaces are known as cleavage facets and are marked by river patterns along them, which are the characteristic feature of brittle fractures (6). Cleavage facets indicate parallel planes of weak chemical bonding between the layers of atoms. The crack propagates along these planes by breaking the atomic bonds through planar sectioning. The crack changes direction as it travels between crystals as each crystal has a unique orientation of these planes. This results in the different orientations of cleavage facets seen. Local river lines indicate the direction of crack propagation and of the local crack origin. Cleavage initiate from tiny defects such as crack and inclusions.

This specimen was subject to a Charpy Impact Test. A notch was created on the specimen before applying an impact force. The crack would propagate from the notch tip along the cleavage plane, breaking the atomic bonds through planar sectioning like in Figure 17.

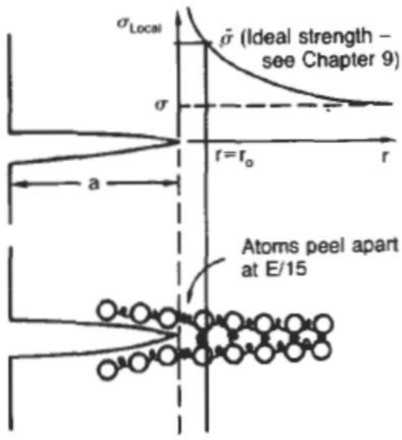

Figure 17. Illustration of the breaking of atomic bonds at the crack tip of a brittle material (7)

Q8. *Why does this type of fracture occur in HCP and BCC metals at low values of T/T_m? [10 marks]*

HCP (Hexagonal Close-Packed) and BCC (Body-Centred Cubic) packing structures have a limited number of close-packed slip systems, having only 12 and 3 respectively, as compared to an FCC (Face-Centred Cubic) structure which as 12. The more slip systems a structure has, the more ductile the structure.

These close-packed slip systems involve planes and directions in which all the atoms are in direct contact. Dislocation movement, which are carriers of plastic deformation, “seek out” slips systems as they require the lowest energy.

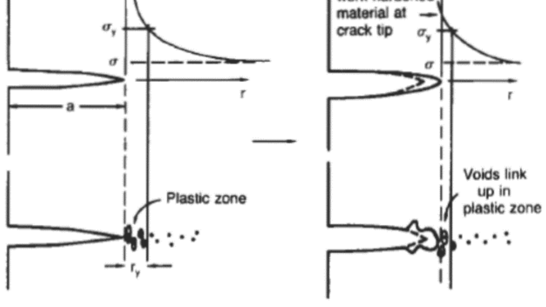

Figure 18. Illustration of the breaking of atomic bonds at the crack tip of a ductile material (7).

In a ductile material such as aluminium and copper, the large number of slip systems means it is easy to plastically deform. The ductile crack tip has a large plastic zone and the crack progresses by ductile tearing. This absorbs a lot of energy and hence the crack progresses slowly, performing plastic work as it progresses and resulting in a rough surface.

In HCP and BCC structures, the limited availability of slip systems means little plastic work is done. The lack of blunting of the crack tip means a high local stress at the tip, which can break the interatomic bonds along a slip plane, creating the cleavage surface.

In addition, low temperatures make metals with a BCC and HCP structures even more brittle. Thermal agitation assists the motion of dislocations in BCC and HCP structure (7). The lower the temperature, the less the thermal agitation and the more impeded the dislocation. This increases the intrinsic lattice resistance (7). The result is an even smaller plastic zone at the crack tips and increased local stress, making the material even more susceptible to brittle fracture. This effect is known as the ductile-to-brittle transition temperature of metals.

Specimen 5 – Aluminium Charpy Impact test at T/T_m = 0.5; Ductile Fracture

Q9. *What are the key features of this fracture surface? Based on these features, explain how the crack has propagated through the metal? [10 marks]*

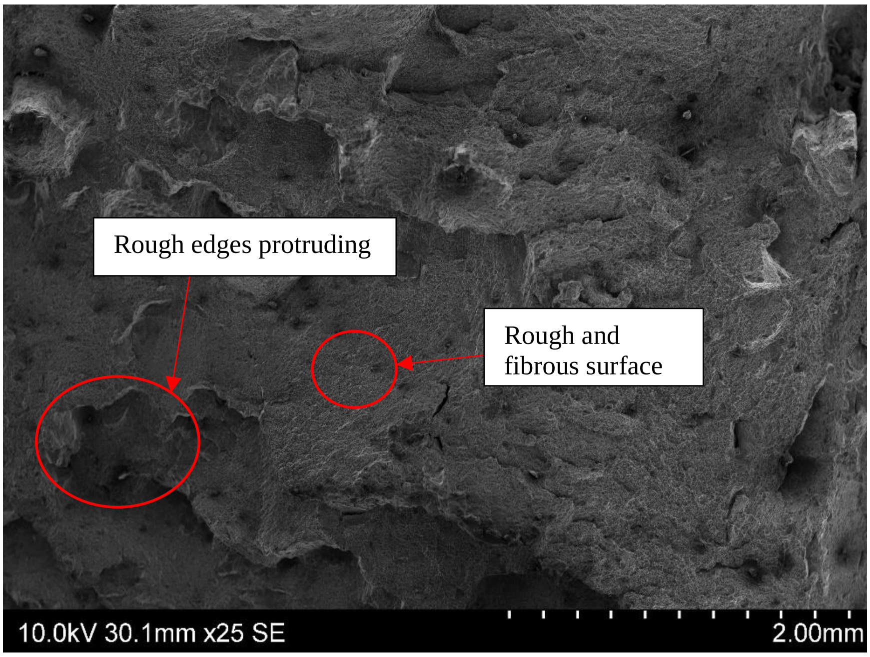

Figure 19. Fracture surface of aluminium specimen at 25X magnification

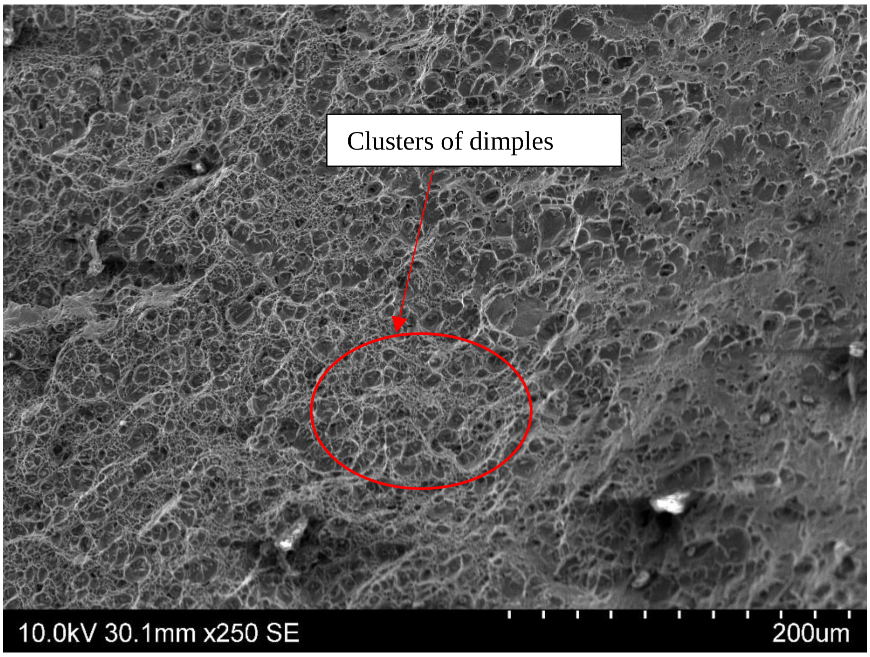

Figure 20.Fracture surface of aluminium specimen at 250X magnification

The surface is mostly rough and fibrous. Closer inspection at 250X magnification in Figure 20 shows dimples with rounded edges on the surface of the material. Also observed at 25X magnification in Figure 19 are rough edges protruding from the surface. Those rough edges indicate that larger than normal plastic deformation has occurred in those regions. The combination of the dimpled rough surface and rough edges indicate that large amounts of plastic deformation have occurred over the fracture surface, this is indicative of a ductile material. The dimples are characteristic of ductile fractures as they relate to the method of crack propagation in ductile materials.

From Figure 18, the crack tip leads to high stress concentration that create a plastic zone just at the tip. The crack tip for this specimen is in the notch made during the Charpy Impact test. The plastic strain at the tip promotes void formation around microscopic inclusions such as precipitations. These voids grow larger and eventually coalesce with the crack tip, allowing it to grow. This is called plastic flow, which blunts the initially sharp crack tip. The crack advances, creating plastic zones deeper into the material and promoting more micro-void formation until the specimen fails. The dimples seen on the fracture are formed from the micro-voids.

Q10. *Based on your observations of brittle and ductile fracture surfaces in Specimens 4 and 5, explain why ductile fracture leads to much higher toughness than brittle fracture in metals? [10 marks]*

Toughness is the measure of a material's ability to absorb energy and plastically deform without fracturing. The key difference observed between the brittle and ductile fractures surfaces is the amount of plastic deformation that has occurred. In ductile fractures such as Specimen 5, the surface is rough from plastic flow while the brittle fracture surface of Specimen 4 is smooth and flat. This is due to the different mechanism of crack propagation.

In a ductile material, the existence of a plastic zone blunts the tip of the crack. This decreases the local stress, ensuring that the surrounding material merely deforms plastically. The method of micro-void growth and coalescence and growth of the crack by ductile tearing means more of the applied energy is consumed for plastic flow (7). Blunting the crack tip means more energy is needed to break the interatomic bonds by raising the local tip stress.

Comparatively, brittle materials have little plastic deformation at the tips, causing high local stress. Without a plastic zone to reduce the local stress, it easily exceeds the strength of the interatomic bonds and fracture occurs by planar sectioning of these bonds, creating the smooth flat surfaces seen on the Specimen 4. This means that low energy is required to break the interatomic bonds in a brittle fracture.

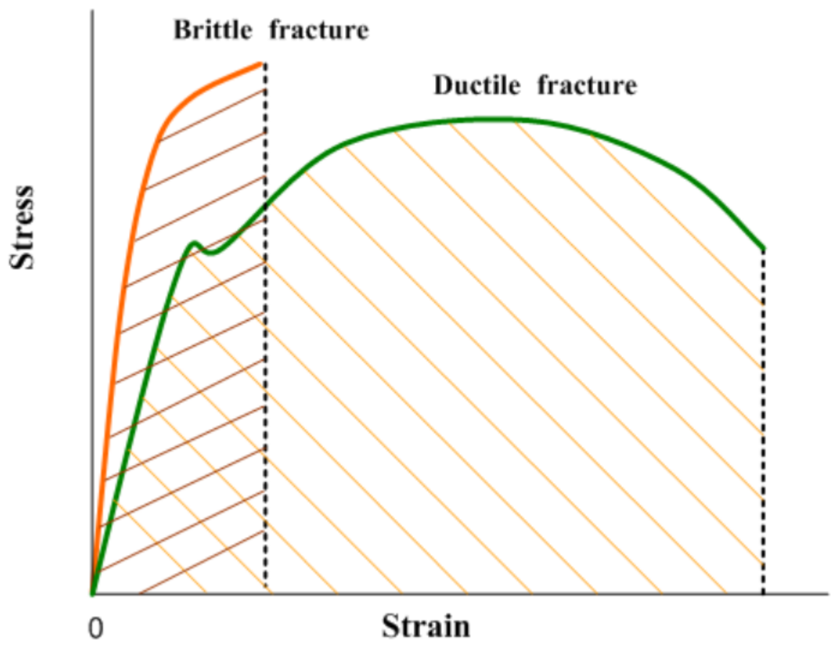

Figure 21. Stress vs Strain curve for Brittle and Ductile fractures. The area under each curve represent the amount of energy absorbed by the fracture mode (8).

In conclusion, ductile fractures promote plastic deformation by facilitating dislocation movement, therefore they have a larger energy absorption capacity before failure compared to brittle fractures as seen graphically in Figure 21. Hence, a ductile fracture leads to higher toughness than a brittle fracture.

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13 December 2018

Bibliography

1. **Liebhaber, Will.** *Screengrab from video "Major Aircraft Components".* s.l. : KL Aviation.
2. **Jongerius, S.R and Lentink, D.** *Structural Analysis of a Dragonfly Wing.* s.l. : Springerlink.com, 2010.
3. Troubleshooting: edge effect, charging, specimen damage. *Myscope.* [Online] Microscopy Australia, May 12, 2014. [Cited: 11 25, 2018.] https://myscope.training/legacy/sem/practice/principles/troubleshooting.php.
4. **Wells, J. K. and Beaumont., P. W. R.** *Debonding and Pull-Out Processes in Fibrous Composites.* Department of Engineering, Cambridge University. Cambridge : Cambridge University, 1984.
5. **Greenhalgh, Emile.** *Failure Analysis and Fractography of Polymer Composites.* Cambridge : Woodhead Publishing Limited, 2009.
6. **D.Hull.** *Fractography: Observing, Measuring and Interpreting Fracture Surface Topography.* Cambridge : Cambridge University Press, 1999.
7. **Ashby, M.F. and Jones, D.R.H.** *Engineering Material 1: An introduction to their properties and applications.* Oxford : Butterworth-Heinemann, 1996.
8. **Kopeliovich, D.** *Stress vs Strain Curve for Brittle vs Ductile Materials.* s.l. : Subtances and Technologies, 07 10, 2015.
9. *Brittle Fracture Surface.* Sydney : University of New South Wales, School of Materials Science and Engineering, 2013.