

Scanning Electron Microscopy

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

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]

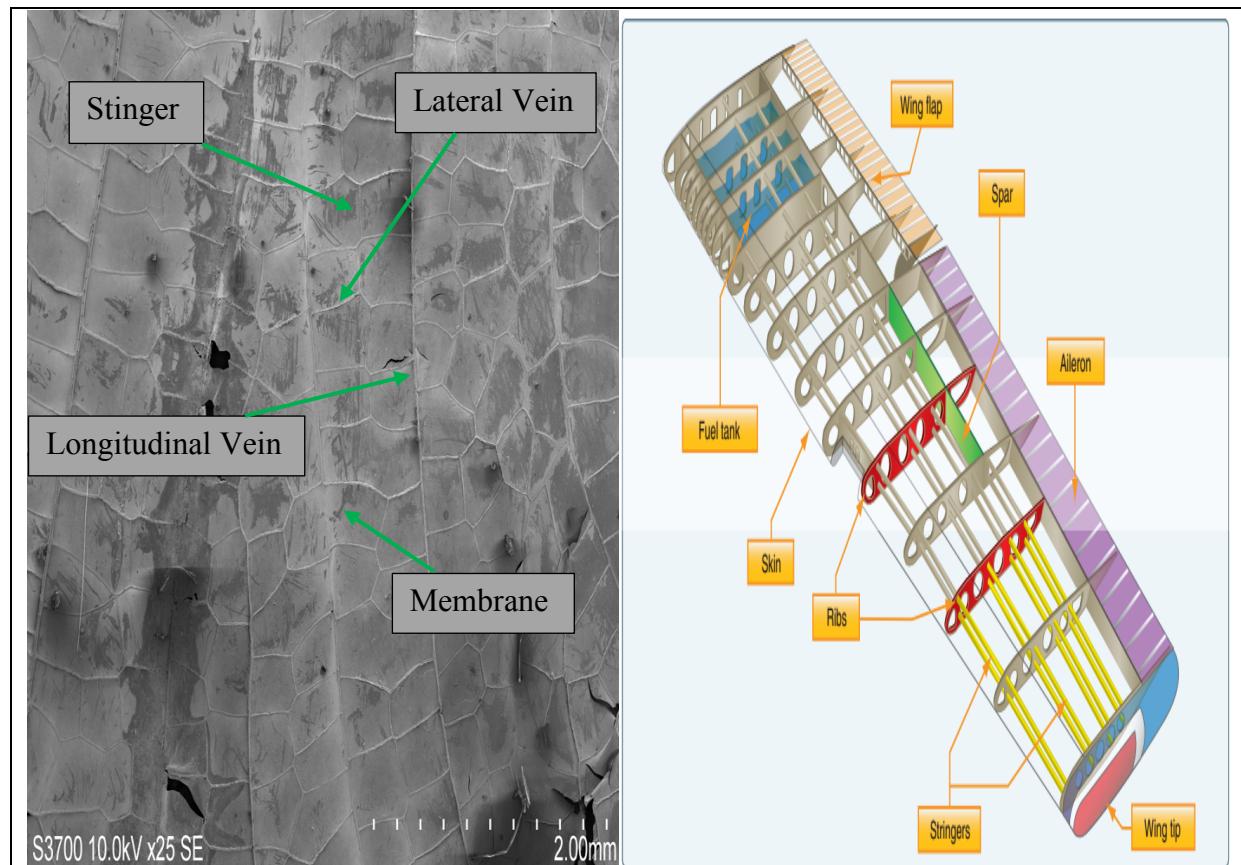


Figure 1: Comparison of Dragonfly Wing and Aircraft Wing.

As it can be seen in Figure 1 the dragonfly wing and an aircraft wing have similar features. Firstly, Ribs, Spar and Skin on an aircraft can be compared with the Lateral Veins, Longitudinal Vein and Membrane, respectively, on the dragonfly wing. Secondly, both wings have thin longitudinal so-called Stingers which locate parallel to the length of the wing. To improve damage tolerance ribs are not continuous across the spar. Lastly, just like the aircraft wing has skin, the dragonfly wing is also covered in a membrane which serves as an aerodynamic surface and ensures good aerodynamic performance as well as transfers loads arising from pressure distribution and skin friction. Both structures are designed to withstand significant loads and ensure stable and trimmable flight. The lateral veins and the ribs give shape to the wing and carry shear stresses, whereas the spars and the longitudinal threads provide rigidity and stiffness to the structure while undergoing bending. Spars and longitudinal veins bear both tensile and compressive loads.

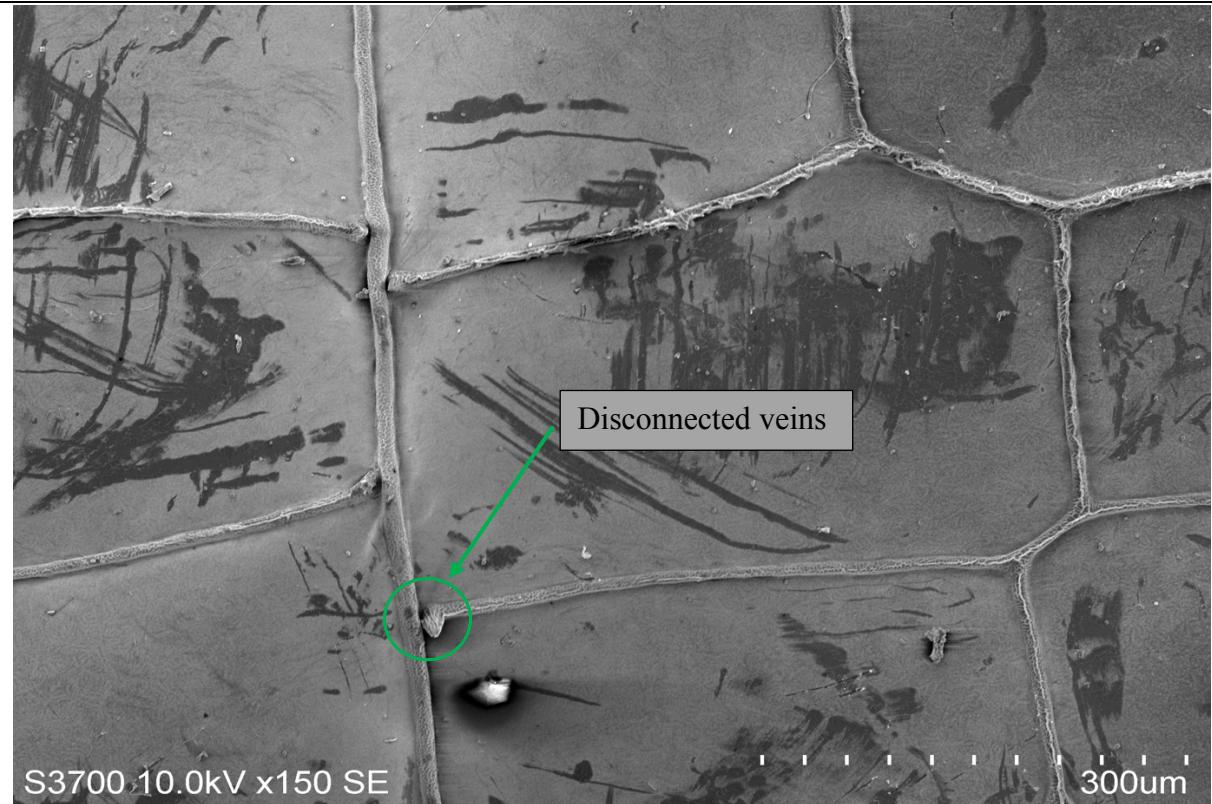


Figure 2: Disjoints on the Dragonfly wing.

As per the Figure above, unlike an aircraft where the spar and ribs are jointly connected the dragonfly wing's lateral and longitudinal veins are not entirely connected. Flight mechanics of a dragonfly is different to that of an aircraft, the former flies by flapping its wings. Therefore, disjointed lateral veins are required to allow the dragonfly to turn and twist its wings during the flight; however this connection provides lower stiffness, hence making the wing more vulnerable to deformations [1].

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]

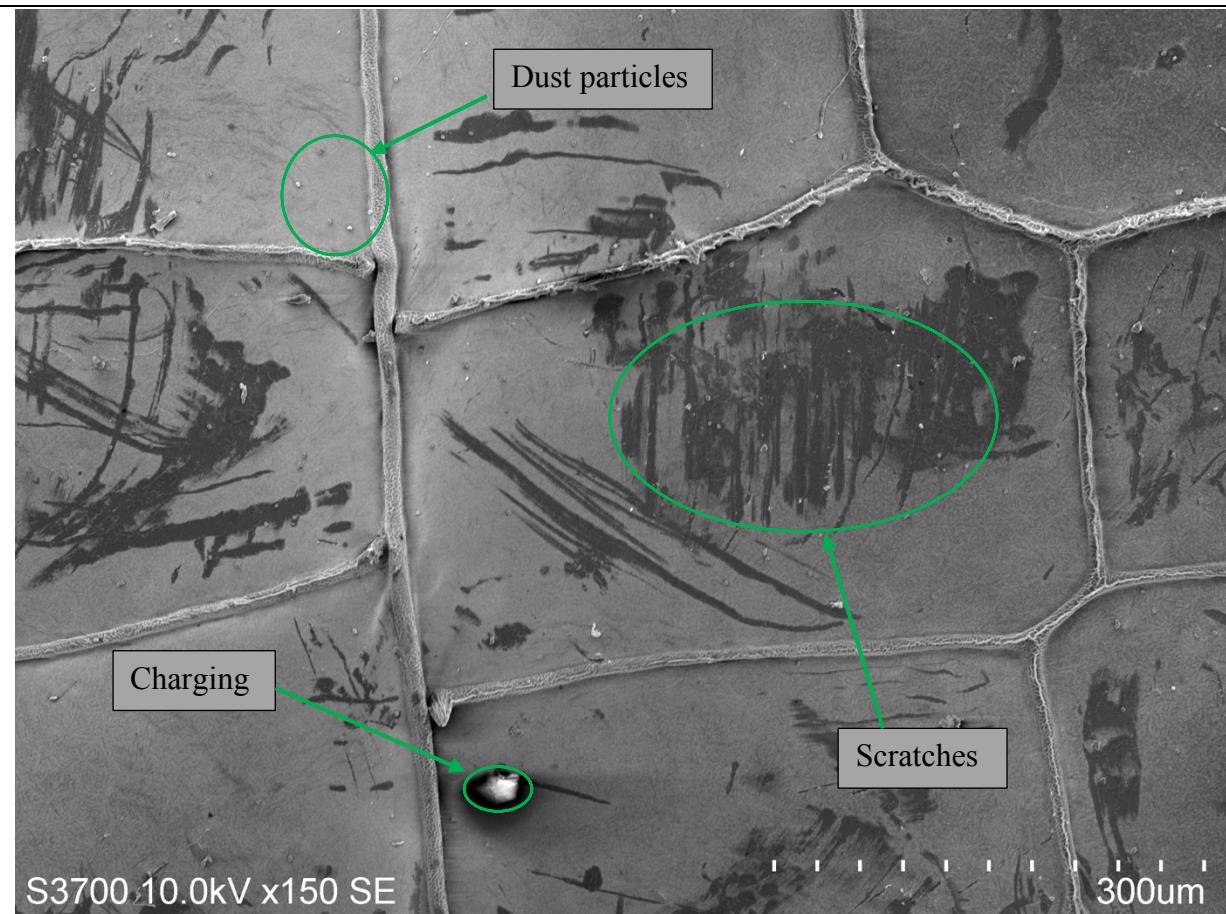


Figure 3: Artefacts on the Dragonfly wing.

SEM artefacts are unexpected and undesired features which can appear due to various reasons. Artefacts are a side-effect of microscope conditions, and it is important not to be fooled that artefacts are part of your specimen.

Various artefacts appear in Figure 3. Scratches are natural for such species; the dragonfly could have obtained them for example during its lifecycle. Scratches could have also appeared due to tweezers that the lab supervisor used to move the specimen. Nothing can be done about the former reason, however, to mitigate the latter one maybe one has to use smoother surfaces for specimen transportation.

Dust particles are impurities which probably present due to insufficient pre-experiment cleaning or due to the laboratory environment where the particles attached to the surface of the wing after the samples had been extracted to be shown to the students. Dust particles are normally not conductive they display as shiny spots at high voltages (just like in our case) and dark spots at low voltages. To mitigate them, conducting the pre-lab preparations in a dust-free environment as well as cleaning the wing thoroughly before coating would be an excellent solution to this problem.

Lastly, charging effects are due to an increased electron build-up which triggers charging. When the number of incident electrons is greater than the number of escaping electrons the negative charge builds-up on the surface when the beam hits the sample. To mitigate charging, reduce the beam voltage or increase the conductivity if the sample during its preparation. The latter can be achieved by increasing the concentration of heavy metals in the sample [2].

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]

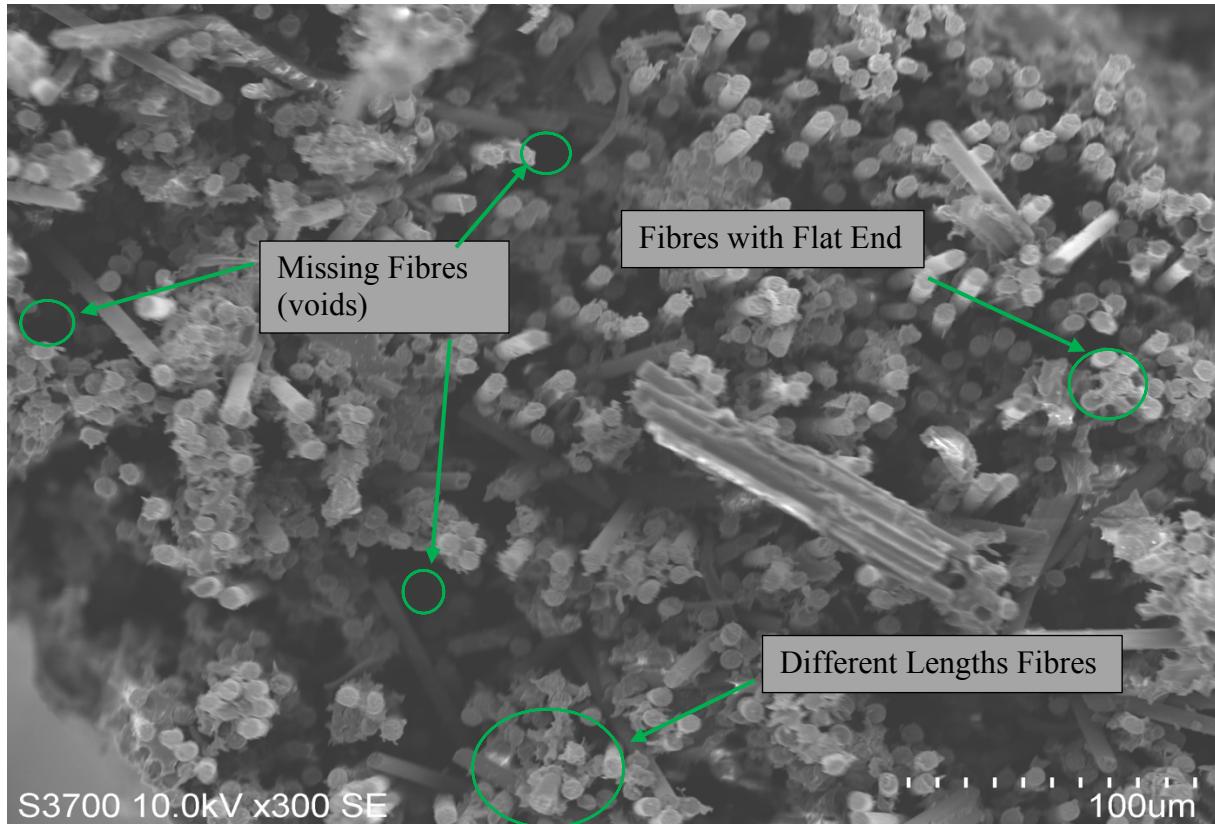


Figure 4: Tension failure of unidirectional CFRP.

The specimen fractured by Tension and Compression. In both tension and compression, failure is dictated by similar mechanisms, i.e. gliding of crystalline planes.

As seen in Figure 4, there are regions where the clusters of smooth fibres have flat ends. These are normal to the direction of loading. Moreover, one can see missing fibres, i.e. voids where the fibres were pulled out by the applied load. Besides, some groups of fibres have different lengths indicating the features of a tensile fracture.

Some fibres were pulled out by direct tension field, and others fractured inside the matrix, this phenomenon leads to an open hole on one side of the material and sticking out fibres on another side. In the picture above one can see the missing fibres (voids) as well as fibres with different lengths. Pulling out fibres from the matrix causes friction which requires more energy to be spent. The pullout lengths are mostly minimal; hence they exhibited uniform fracture. When a composite material is loaded under a tensile load, an open crack propagates through a matrix and causes stresses to exist ahead of the tip of the crack accompanied by the shear stresses at the crack tip. To identify the direction, as well as the origin of the crack, the macroscopic analysis of the radial marks and the individual fibre fracture ends have to be examined.

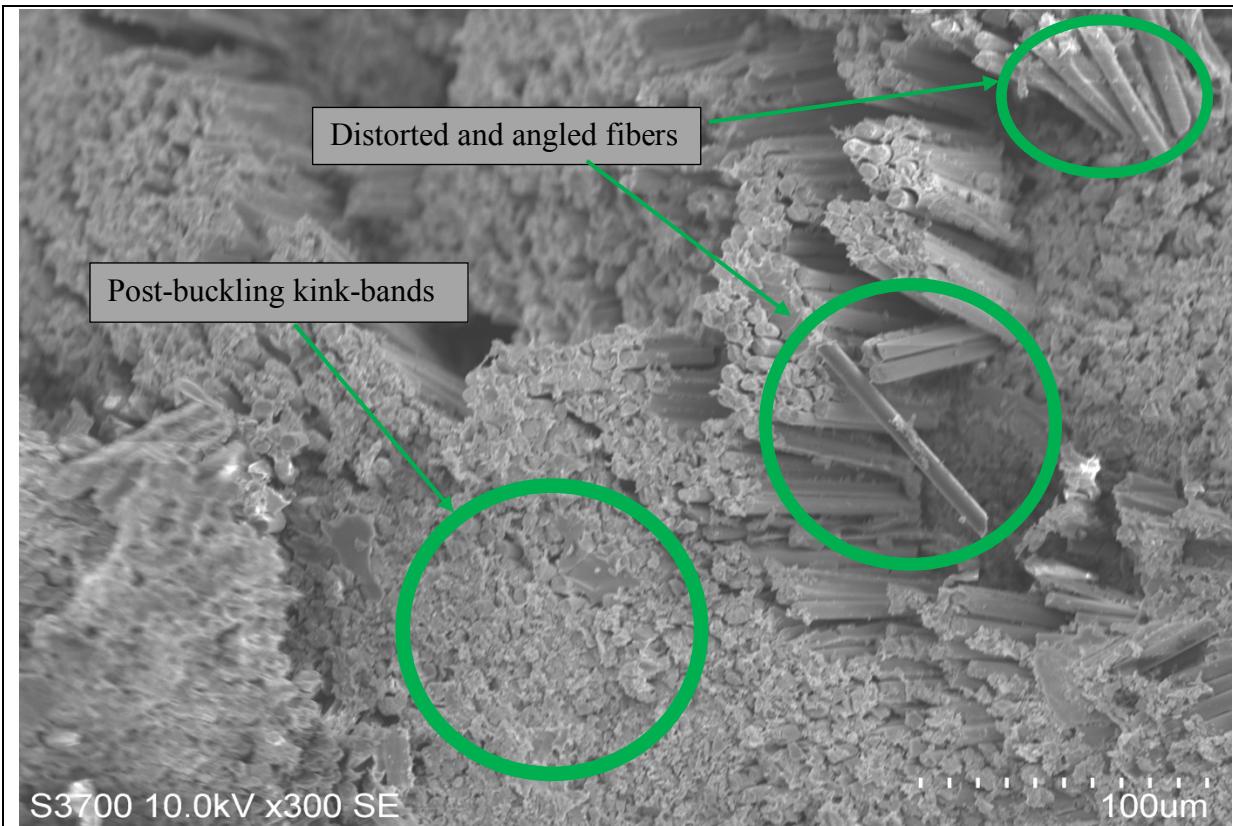


Figure 5: Compression failure of unidirectional CFRP.

Figure 5 displays the regions where the fibres are slightly tilted and the areas where the fibres buckled. Following the Weibull controlled fracture, the fibres shed load on the adjacent fibres. When the specimen was compressed and unable to tolerate the applied stresses, fibres underwent plastic deformation and got affected by buckling dramatically. This fact inevitably led to distorted, rough-ended and angled fibres which can be identified on the image above. The regularly spaced, equal length in-plane kink-bands are phenomena indicative of compression failure described by Vogler and Kyriakides. These kink-band formations are a post-buckling event and start from the local imperfections of the sample.

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]

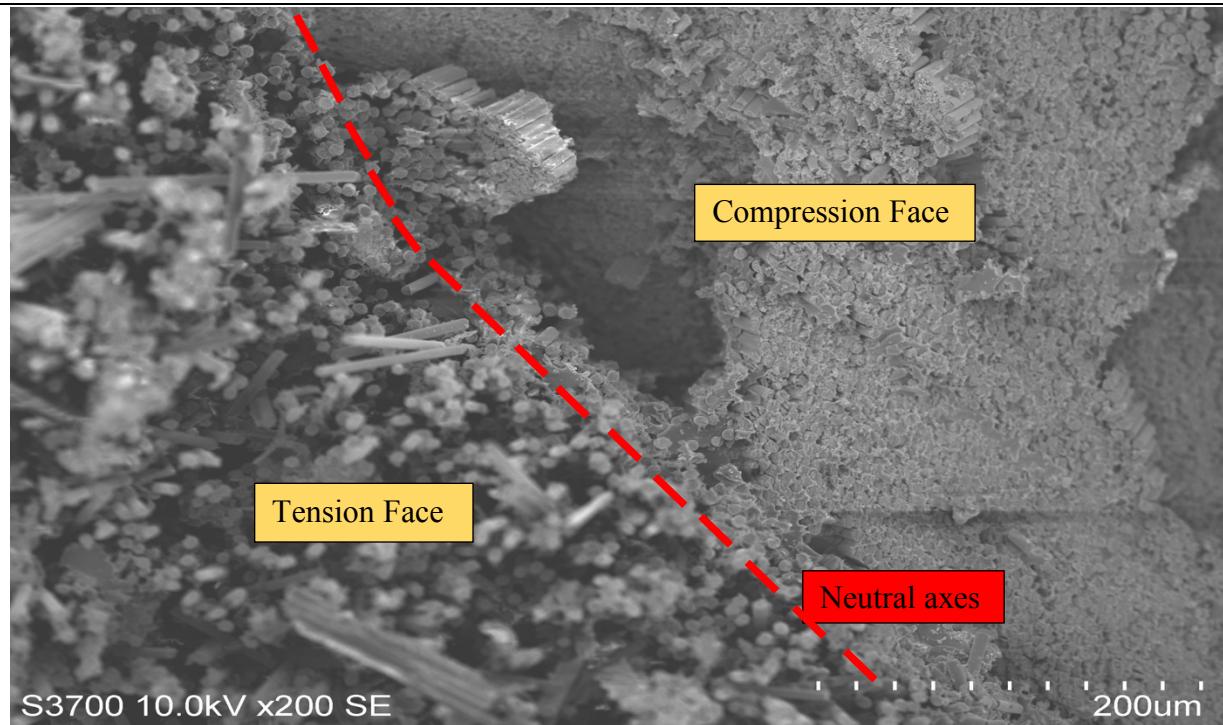


Figure 6: Fracture of unidirectional CFRP due to bending.

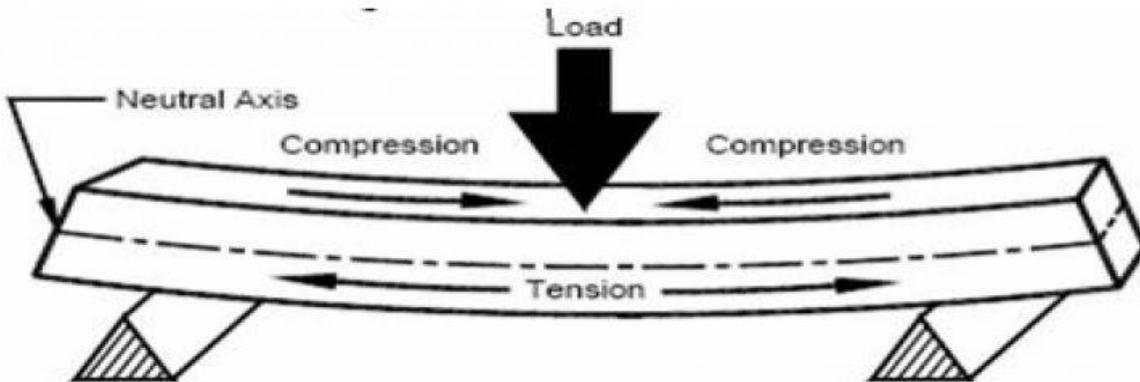


Figure 7: Illustration of 3-point bending.

Carbon Fibre has a higher tensile strength than compressive strength. Besides, Carbon Fibre is unidirectional which implies that this specimen is strong in the direction in which the fibres point. The fibres undergoing deformations in the direction of fibres would look different. Cook-Gordon mechanism controls the degree of matrix/fibre debonding.

As seen in Figure 6 there is a region of compression and a region of tension with clear neutral axes which separate the faces. From the Structural Analysis lectures, one knows that no direct stresses are acting along the Neutral Axis. As per Figure 7, the surface below the neutral axis is identified to be in tension with the upper surface in compression. The presence of these two modes in Figure 6 indicates that the specimen underwent static flexural failure due to 3-point bending. However, there is more than a pair of compression and tension faces, which would be the case for solid bulk material, but there are many such faces. This phenomenon is called interlaminar shear delamination.

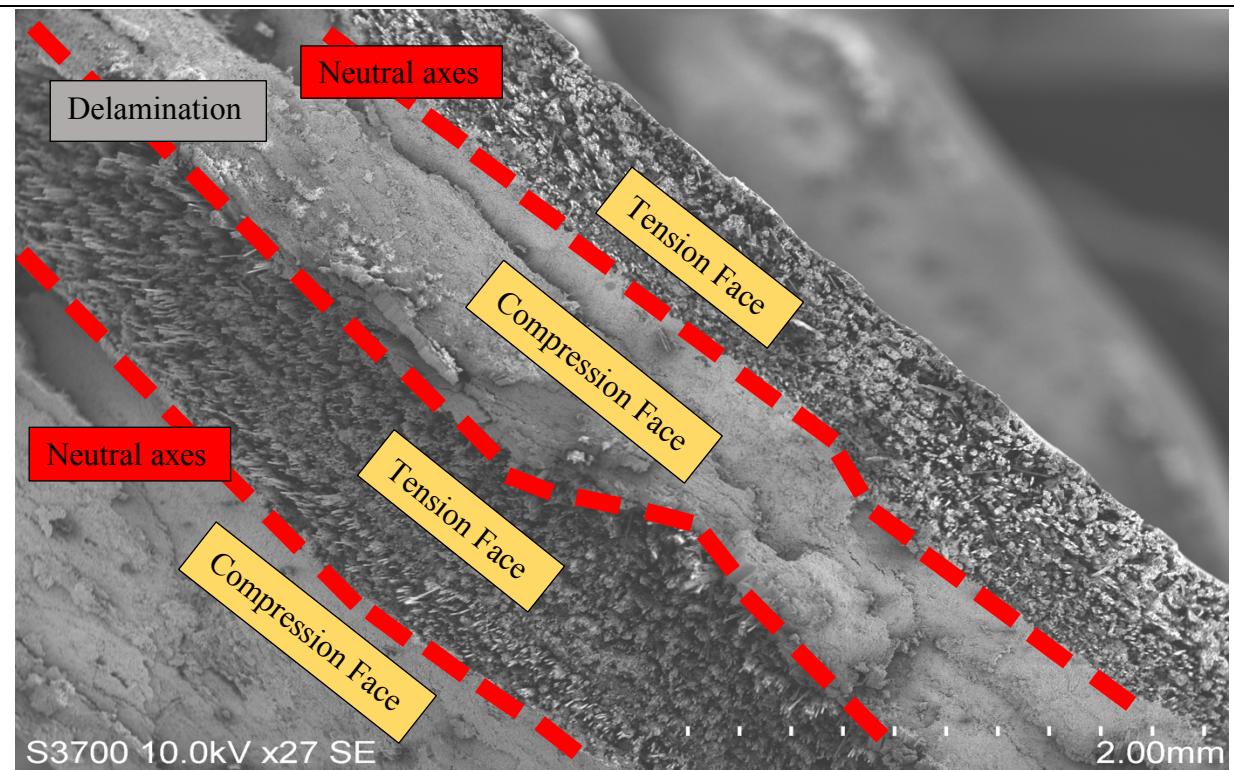


Figure 8: Delamination fracture of unidirectional CFRP.

Figure 8 shows alternating regions of compressive and tensile failure surfaces, and as described above, this occurred due to delamination. When the bending moment was applied, and the shear stresses were induced, the latter acted parallel to the load (perpendicular to the fibres direction). Effectively a sandwich of independent fibre layers was generated due to the 3-point bending [3].

Specimen 3 – Interlaminar Fracture of Unidirectional CFRP

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

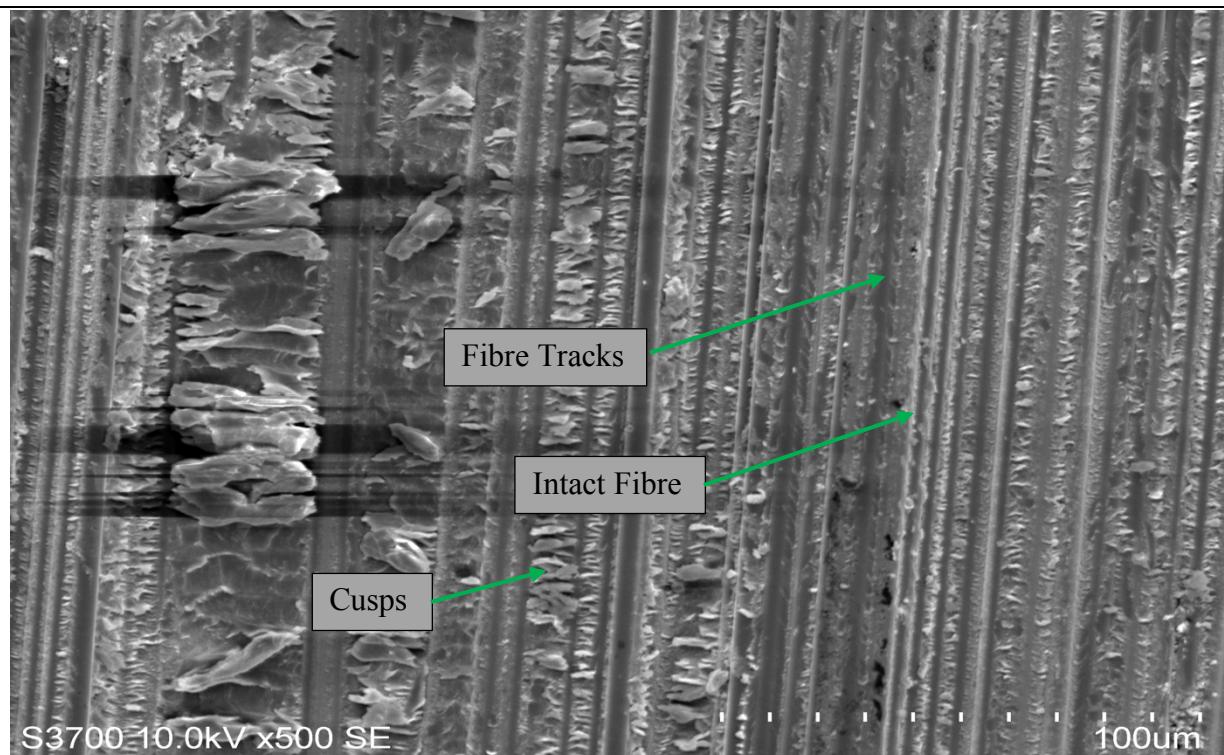


Figure 9: Interlaminar fracture of unidirectional CFRP.

Modes of Failure

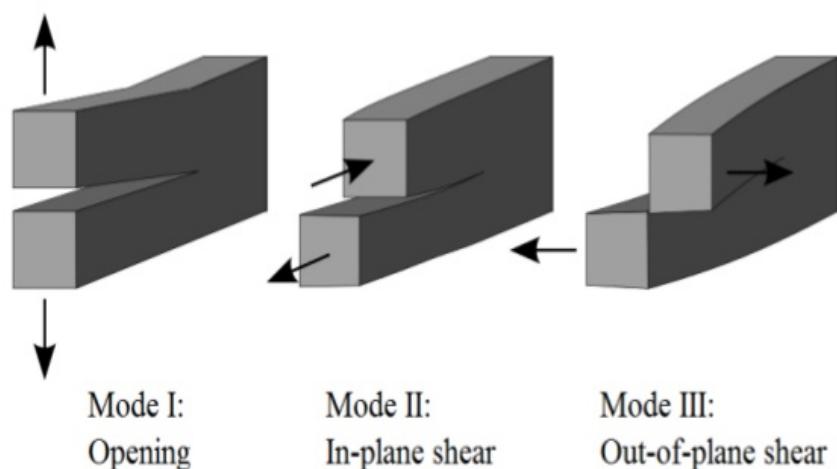


Figure 10: Illustration of different failure modes.

As per Figure 9, it is crucial to distinguish between the intact fibres and the fibre tracks. The presence of both implies that the specimen was most likely to fracture due to a matrix failure rather than a fibre fracture. There are also several rows of cusps which lie perpendicular to the orientation of the intact fibres. Taking into consideration these factors, it becomes clear that the specimen's local failure mode is Mode II where the top surface is sheared inwards and parallel to the bottom surface.

The reason for why the local failure mode is neither Mode I nor Mode III is as follows:

We would expect the specimen to have broken fibres as well as fibre tracks in the first case. Similarly, for Mode III we would expect to see curved and broken fibres oriented in the same direction with cusps. Neither of these features, however, can be observed in Figure 9.

Effectively, a CFRP specimen affected by shear loads will generate cusps like the ones seen in Figure 9. The creation of cusps leads to the formation of cracks spreading into adjacent matrices extending their length. These deformed matrices will inevitably fail in tension leaving a trace of shear cusps on the fracture surface.

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]

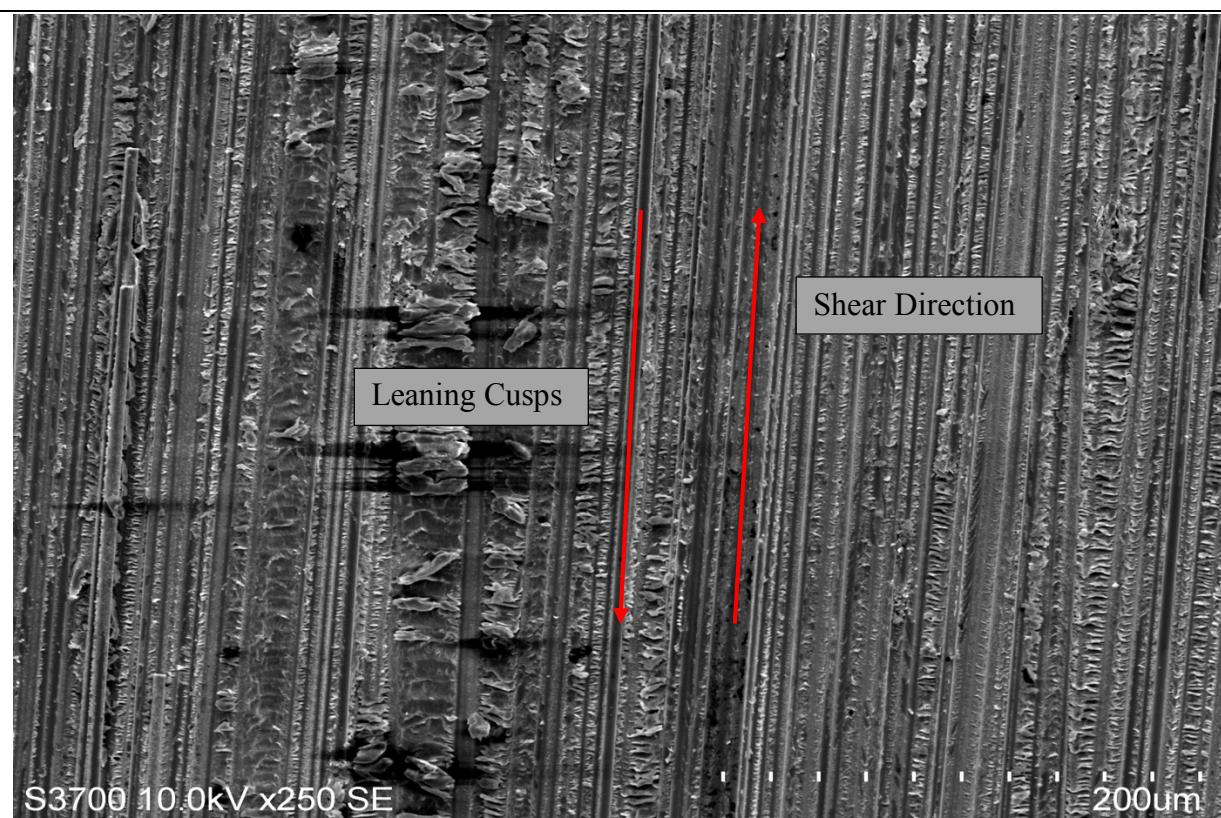


Figure 11: Shear direction of interlaminar fracture of unidirectional CFRP.

Due to the leaned cusps and their respective orientation, the specimen underwent an in-plane shearing (Mode II).

Cusps on the picture above are tilted in the same direction and at the same angle that is why the direction of the applied shear can be deduced. The shear will act directly opposite to the cusps' orientation. As per Figure 11, the specimen was sheared bottom to top. As this sliding occurs, the cusps will go through plastic deformations and subsequently elongate in the direction parallel to the shear force until the final fracture.

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 12: Brittle fracture of Zinc after Charpy Impact test.

To find out more about Charpy Impact refer to Leis [4]. There is no evidence of plastic flow. Small roughness zones observed on the picture above are due to plastic zones at the tip of the crack. In brittle materials, atomic bonds are broken rapidly extending the size of a crack. The crack propagates along a crystallographic plane or more specifically along a structural cleavage plane, the plane in which crystalline material splits along. Cleavage can be seen in cases when the tip of a crack is sharp. This fractural mechanism typically associates with a quick crack propagation with a low energy release rate, hence not allowing the material to deform plastically by the motion of dislocation or otherwise. Atomic bonds are progressively broken, extending crack. Creation of new cracked surface necessitates energy spending. This tensile or compressive deformation in such cases leaves a faceted surface which can be observed on the image above.

The surface of Zinc on Figure 12 is solid, crystalline, spiky and angular. The herringbone pattern is detected which indicates the direction of crack growth and its origin.

Because of the rapid crack growth across the grains, cleavage faces formed. Crystallographic cleavage is characterised by cracks growing parallel to low-index crystallographic planes. Crack nucleation starts at regions where the movement of dislocations is hindered, such as grain boundaries, impurities et cetera. Also, as zinc has a hexagonal close-packed (hcp) crystal, the slip is much more limited than in hcp or bcc. Moreover, the motion is complicated due to the different orientation of grains, hence forcing a crack to form highly stepped regions whenever it crosses a grain boundary (shown above).

The so-called river line (or level difference line) can be observed in Figure 12, and they represent the direction of crack propagation. The further from the origin (delta), the less branched the river becomes. The lines do not always propagate along grain boundaries and therefore can only be used to spot the cracks orientation [5].

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

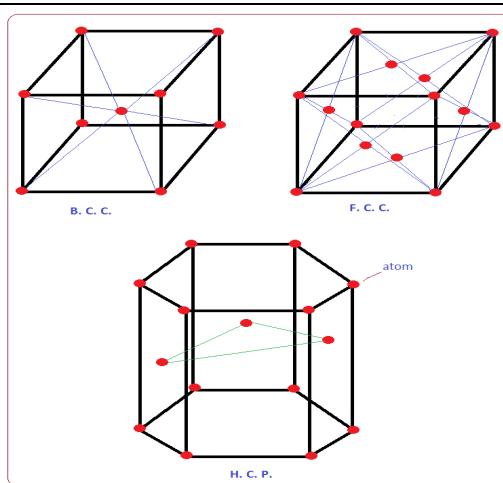


Figure 13: Close packed structures and their slips.

The behaviour of material directly depends on its crystal lattice structure. Ductility and brittleness are associated with the number of slip systems that the material has. A slip system consists of a slip plane and a slip direction. Both BCC and FCC have 12 slip systems; however, the BCC system is less packed which implies a lack of active slip systems. Therefore, BCC slip systems can only be active at elevated temperatures. Hence, out of these two, FCC structure is generally more ductile for the same temperature ratio. HCP has only three slip

systems which make it the most brittle and has a higher tendency to fail through the brittle fracture.

Now, on the atomic scale temperature is a measure of the average kinetic energy, according to $E = \frac{3}{2}kT^2$. The higher the temperature, the more vibrations atoms have, hence making it easier for them to slip through slip systems, i.e. for dislocations to move. Whereas, at lower T/T_m atoms generally vibrate less frequently; therefore, atoms are more likely to break the atomic bonds (brittle fracture). To conclude, at lower temperatures crack propagation and brittle fracture are more probable, whereas at elevated temperatures ductile fracture in combination with dislocation motion is more likely. Therefore, in HCP and BCP at low values of T/T_m, the brittle fracture occurs.

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]

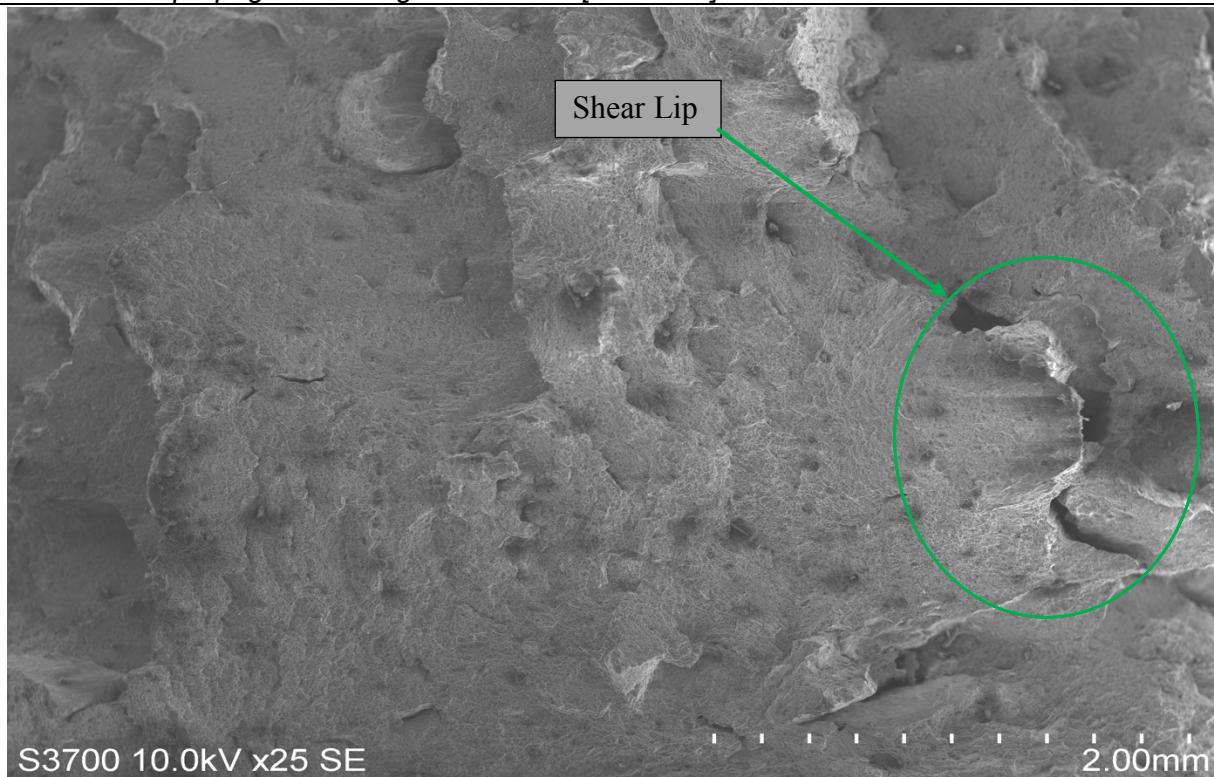


Figure 14: Ductile Aluminium fracture after Charpy Impact test.

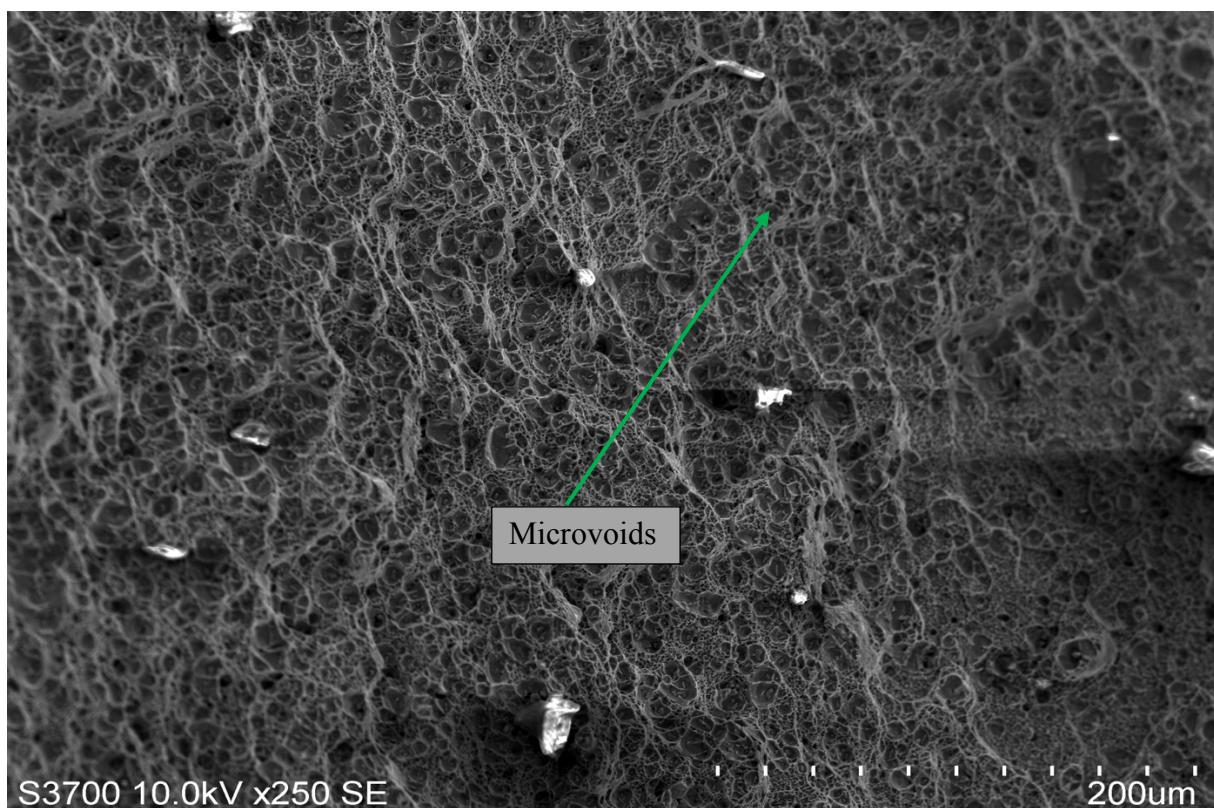


Figure 15: Ductile Aluminium fracture after Charpy Impact test at a smaller scale.

The fracture patterns observed in Aluminium differs from those in Zinc. In Figure 15, one can notice a high level of porosity of the specimen accompanied by microvoids as well as uneven and rounded surfaces. These cavities (microvoids) are the primary feature of the sample; they characterise ductile behaviour where plastic deformation had occurred before the material fractured. In ductile fracture, the work is spent to deform plastic zones and to create new surfaces by tearing. Besides, the smooth surface of the Shear Lip can be seen in Figure 14 which is an indication of a necked area formation at the fractured region.

The specimen underwent an internal plastic strain in the Charpy Impact test. Plastic deformation is associated with microvoids which initiate their growth at different imperfections, such as precipitates, inclusions, impurities and manufacturing defects. Crack tip then becomes rounded. Then crack proceeds by microvoid growth and coalescence. Ultimately, the specimen, therefore, will fail by shearing and separation around the necked area once the UTS of the material exceeds [6].

Most work is spent on heat, and the rest goes into dislocation structures that improve yield strength of the metal (i.e. work hardening), essentially requiring more work to be done.

Stress concentration mainly accumulated at plastic zones because the fracture occurred at high T/T_m . At high-temperature ratio, dislocations have a sufficient amount of energy to propagate through the material's structure. As a result, dislocations passing through more active slip planes absorb more energy to deform plastically instead of initiating crack growth, implying that the ductile fracture has a higher probability to occur.

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]

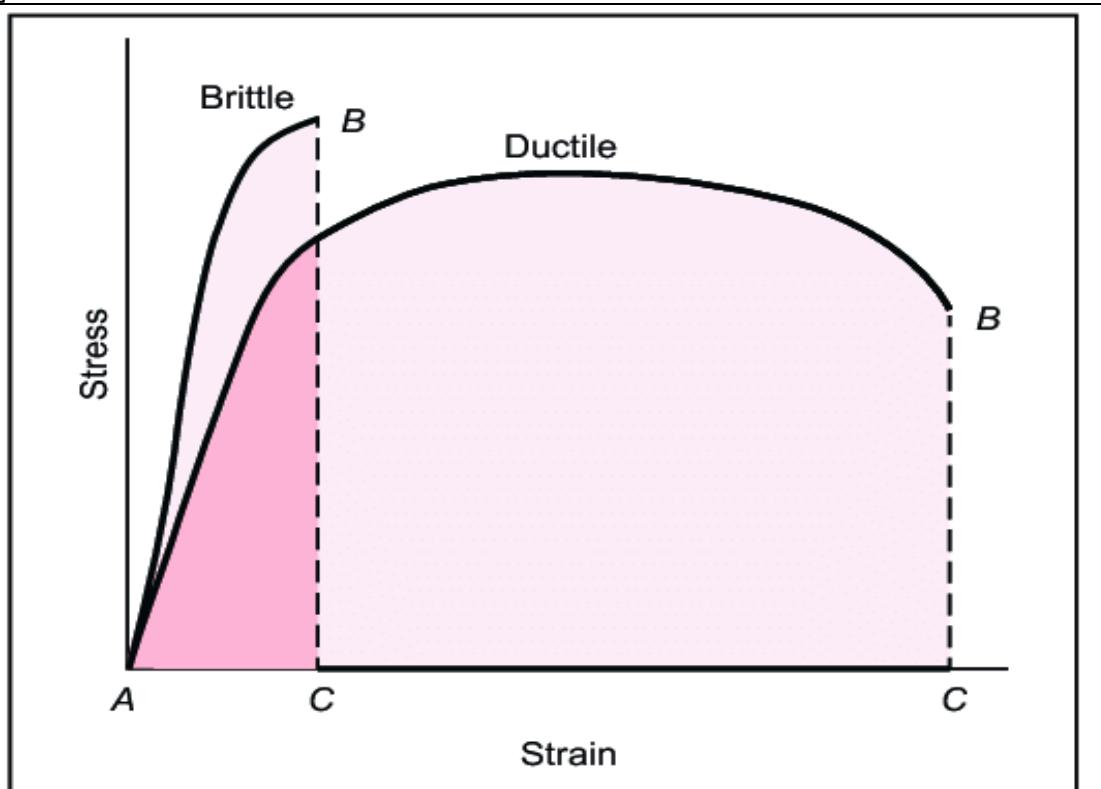


Figure 16: Stress-strain curves of Brittle and Ductile materials.

Toughness, by definition, is the ability of a material to absorb energy and plastically deform without fracturing or material's resistance to fracture when stressed [7]. From Figure 16, the amount of energy stored before material fractures is the area under the stress-strain curve.

The area under the ductile curve is larger than the area under the brittle one. This observation signifies that a ductile fracture leads to a much higher toughness in metals.

In ductile materials, most of the absorbed energy is restored as plastic deformation heating. The fraction of this energy is consumed by dislocation that improves the yield strength of a material and subsequently requires more work to be done. More work means a larger area under the curve as the material plastically deforms.

Now, brittle materials fail through crack propagation. Therefore, the strain energy absorbed is aimed to break the atomic bonds instead of leading to dislocations motion. Also, for a crack to start propagating less energy is required compared to the amount of energy needed to initiate dislocation motion. Hence, brittle materials will store less energy before fracture. Finally, a brittle fracture will lead to a lower toughness than that of a ductile fracture.

Dr E S Greenhalgh
08 March 2019

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