Module 8: Composite Testing

Lecture 36: Quality Assessment and Physical Properties

Introduction

In the previous lecture we have introduced the needs, background and societies for mechanical testing of composites. In this lecture and subsequent lectures we will see principles for the measurement of physical and mechanical properties of composite. In the present lecture we will see the methods to assess the quality of the composite and physical properties.

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Primary Mechanical Properties of a Long-Fibre Composite

There is a large set of properties of a long fibre composite that one needs in design and analysis. However, it is generally agreed that the minimum requirement to assess the three main properties – modulus, strength and ductility, are the parameters listed below:

1. Tensile modulus
2. Compressive modulus (uniaxial)
3. Flexural modulus
4. Shear modulus (in plane)
5. Lateral contraction ratios
6. Tensile strength
7. Compressive strength (uniaxial)
8. Flexural strength
9. Apparent interlaminar shear strength
10. Fracture toughness (various modes)

The requirement of property evaluation listed above is based upon the tests for an isotropic homogeneous sample. However, this minimum requirement is not sufficient to completely quantify the strength and stiffness tensors. Further, it neglects the viscoelastic behaviour aspect of the composites.

All the tests included in the minimum requirement of the property evaluation are not carried out by most of the industries working in composites. Their objective can be different and carry out some of the tests. For example, most of the fibres manufacturing companies give the properties of composite which are fibre properties dominant. In such cases, the properties like axial tensile and flexure are given more significance. However, the resin manufacturers tend to give more significance to compression and shear properties of the composite. In this case, these properties are dominated by matrix properties.

The call for ‘open hole’ tests reflects reservations about the reliability of the theories of failure and about the relevance and relative paucity of the empirical evidence from conventional fracture toughness tests. The protagonists of such tests sometimes seem to be preoccupied with a search for authentic and/or definitive data which is perpetually frustrated by a preponderance of mixed-mode failures in their experiments.

The following are the primary engineering properties for preliminary selection of composite materials in a commercial aeroplane industry:

1. Tensile strength at room temperature
2. Uniaxial compression at room temperature
3. Interlaminar shear at room temperature
4. Open hole tension at room temperature
5. Open hole compression at 93°C
6. Hot/wet compression strength
7. Edge-plate compression strength after impact at room temperature

The tests mentioned above are the essential tests in the ***initial testing*** phase.

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Quality Assessment

The quality assessment of the composites used in the specimens to be tested must be done prior to the testing whenever possible. If the composite used in the specimens is of low quality with defects then the property measured are spurious. It can mislead the design and analysis procedure and result in a premature and catastrophic failure of the structure. Hence, the quality assessment of composites before it is used in specimen or actual structure fabrication is essential.

The following quality assessment in composites is essential:

- quality of bond between fiber and matrix,
- voids,
- broken fibers,
- matrix cracks,
- delaminations.

In the following we will see the various methods by which we can assess quality of the composites.

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1. Microscopy

The microscopy is one of the best methods that provide the first hand information on the form of damage. The microscopy can provide the information like:

1. Shape of the fibers,
2. Geometry and uniformity of the fiber spacing,
3. Presence of voids
4. Regions rich or poor in matrix,
5. Fibre alignment.

The microscopy method has limitations like that it can give the information inside the composite. It can give the information on the surface as mentioned above. It cannot give the information like fibre-matrix bond, broken fibres, matrix cracks and delaminations inside the composite.

2. Ultrasonic Inspection

Ultrasonic inspection is a non-destructive method of testing. Using this method one can assess the quality of the composite. The ultrasonic testing method includes the propagation of mechanical waves through the object to be inspected. The mechanical waves propagated are in the range of 100 kHz to 25 MHz. Some of the waves propagated are reflected or transmitted at the other end. The intensity of the waves at the other end is measured by a receiving transducer.

There are two types of waves: Longitudinal and transverse waves. In longitudinal waves direction of oscillation of atoms and the direction of propagation of the wave is along same direction. In the transverse waves the direction of oscillation of atoms is perpendicular to the direction of propagation of wave. The longitudinal waves propagate in all materials whereas the transverse waves propagate only in solid materials. Further, due to Due to the different type of oscillation, transverse waves travel at lower speeds.

When the wave propagating in the material is intercepted by a defect and interfaces (like change from fibre to matrix material and vice a versa or a foreign particle) the energy transmitted through the material also gets reduced due the effect of reflection and attenuation. Thus, one can use both reflection and transmission form of energy for ultrasonic inspection.

The ultrasonic beam requires a transfer medium. In general, water is used as a transfer medium. This is a disadvantage of this method. Further, use of water during the test process can lead to absorption water by composite.

In the recent years the new developments in the ultrasonic testing have made this process very sophisticated and attractive. One can get the complete map or intensity distribution corresponding to the discontinuity in the material. Such a map is called C-scan.

The detailed information on the ultrasonic inspection can be found in ASTM E114-90 for Pulse-Echo method, E214-68(91) for Reflection method, E317-93 for Pulse-Echo and E494-91 for ultrasonic velocities.

3. X-Ray

X-ray technique is a very useful technique. It uses the electromagnetic waves of extremely of short wavelength. These waves are capable of penetrating solid substances and are affected by discontinuities much as other waves. It should be noted that the polymer composites X-rays. A X-ray opaque penetrant is introduced in the damaged area as a liquid solution or suspension so that it fills the cracks and delaminations and makes them clearly visible on X-ray films as a dark region. Opaque dye penetrant such as tetra-bromo-ethane (TBE) is used in these processes.

It is cautioned that the frequent use of dye penetrant should be avoided. This is because the penetrant actually enhances the crack growth. Thus, under loading the frequent use of penetrant will increase the growth rate. Hence, this technique is treated as effectively destructive.

The regions with lower density such as voids, defects and cracks absorb less radiation. This result in higher intensity of the radiation that reaches a photographic film or plate placed on the far side of the sample. The higher intensity causes the darkening of the film or the plate. Thus, the darker areas of the film indicate the outline of the low density region.

ASTM Standards related with this technique are E 94-93, E 142-92 and E 1316-94.

4. Thermography

This is one of the sophisticated techniques that are used in infrared thermography. The advantage of this technique is that it does not require any interruption for inspection. Thus, it is well suited for fatigue testing.

This technique is based on the principles that the infrared thermography detects the heat generated from a source. In case of damage in composite there are two types of such heat sources. The first one is hysteresis evolving from resin and interface. The second source is heating due to friction between the cracks and delaminated interfaces. Thus, the area which appears hot on the thermographs is the area of damage. Once the area of damage is detected, one can zoom into it and get more details.
ASTM Standard guide for nondestructive testing of polymer matrix composites used in aerospace applications is E2533-09.

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Physical Properties of the Composite

The physical properties of the composite play an important role in the measured mechanical properties. There is a direct dependence of mechanical properties on the physical properties. For example, the mechanical properties are directly dependent on fibre volume fractions. Here, we will consider the following physical property measurements.

1. Density

From our basic knowledge, the density of a material is defined as mass of the material per unit its volume. A test method to determine the density of a material is detailed in ASTM standard D792-91. This method is used determine the density of the composite and its constituents. The key points of the test procedure are explained in the following paragraphs.

The density of a material is determined using its weight in air W_a and in water W_w. The densities of air (negligible) and water $\rho_w (= 0.9975 g/cm^3 \text{ at } 23°C)$ are taken as known parameters in this test. The volume of the specimen is determined from the difference between the weight of the material in air and the weight in water and using the known density of water. Then the composite density ρ_C is given as

$$\text{Composite density } \rho_C = \frac{W_a}{\frac{W_a - W_w}{\rho_w}} = \frac{\rho_w W_a}{W_a - W_w} \qquad (8.3)$$

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2. Fiber Volume Fraction

The fibre volume fraction is an important factor in composites as it governs the properties of a composite. The usual fibre volume fraction ranges from 30% to 65%. As we know, the lower end depends upon the significance of property contribution of the fibres where as the upper depends upon the effective, defect-free packing.

In the following we will see some methods to determine the fibre volume fractions.

***1st Method*:** In this method the number of fibers is counted in several measured representative areas of a polished surface of the composite under magnification. Then measure the diameter or the cross sectional area of one or more fibers. Then calculate the average fiber volume fraction as the percentage of area that is fiber. The advantage of this method is that it is simple and provides information about type and uniformity of fiber spacing as well as indication of the void content. However, it should be noted that it is a crude method.

***2nd Method*:** In this method the matrix material is digested or dissolved by putting a measured volume of composite in an acid bath. Then weigh the (dry) fibers remaining after digestion. Thus, knowing the density of the fibers, the volume of fibers and the fiber volume fraction can be determined. One should be careful to choose the liquid for digestion such that the fibers are not digested. Generally, hot nitric acid is used for carbon/epoxy. The ASTM standards used for digestion method are D3171-76 (1990) for polymeric composites and D3553-76 (1989) for metal matrix composites.

***3rd Method*:** In this method one determines the density of the composites and then calculates the fiber volume fraction knowing the density of the fiber and the matrix. This method makes an assumption that the void content is negligible. However, which is not true for any composite. Hence, the results of this method may vary with the results of earlier two methods.

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3. Void Content

Unlike in other conventional materials, polymer and ceramic matrix materials have to be tested for one more physical property like void content. These composites have voids after fabrication. A composite with voids can affect the mechanical; thermal properties, strengths and resistance to fatigue and corrosion. A composite with less than 1% void content is treated as a well fabricated composite. Further, a composite upto 7% void content is regarded as a poorly fabricated composite.

The void content V_c is measured from experimental composite density ρ_e and theoretical composite density ρ_t. The void content in percent is simply the ratio of difference between experimental and theoretical densities to theoretical density. If one knows the densities of the constituents and resin content then, theoretical density can be found. The methods are described in ASTM standard D2534–91, which requires use of ASTM standard D2584–68(1985) for determination of the resin content.
The theoretical density of the composite is of weight of the composite per unit its volume. The volume of the composite is sum of the volume of the fibre and resin. The volume of the fibre and resin can be found from their weights and respective densities. Thus,

$$\text{Theoretical Composite Density, } \rho_t = \frac{\text{Weight of the Composite}}{\text{Composite Volume}} \quad (8.4)$$

Thus, using the volumes of fibre and resin, V_f and V_m

$$\rho_t = \frac{W_c}{V_m + V_f} = \frac{W_c}{\frac{W_m}{\rho_m} + \frac{W_f}{\rho_f}} \quad (8.5)$$

where, W_f is the weight of the fibre, W_m is the weight of the resin, ρ_f is the density of the fibre and ρ_m is the density of resin. Now, percent the void content is given as:

$$\text{Void Content in \% } V_c = \frac{100(\rho_t - \rho_e)}{\rho_t} \quad (8.6)$$

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4. Moisture Content:

The composite materials when exposed to the environment or water absorb moisture. The absorption of the moisture results in the expansion as we have seen already. This also affects the degradation of the mechanical as well as thermal properties.

The moisture content in a polymeric composite is given in terms of percent of moisture by weight. Hence, to measure the moisture content a sample is weighed at the ambient conditions. Then the sample is dried and weighed again. The difference in these two weights per unit weight of the dry sample gives the weight change due to moisture content. Figure 8.1 shows a qualitative variation in weight change over time due to two different drying conditions for a polymer composite. From the figure it can be seen that the drying in vacuum increases the desorption rate significantly. That is why the vacuum ovens are used in laboratory for drying the specimens.

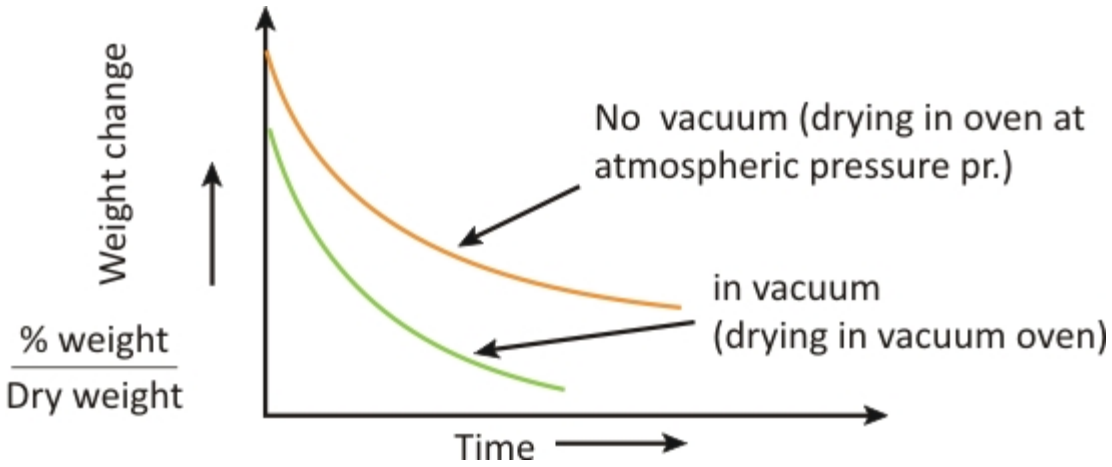

Figure 8.1: Effect of moisture on weight change (Qualitatve)

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Home Work:

1. List the parameters needed to assess the three main properties – modulus, strength and ductility.
2. What are the ways of assessing the quality of the composite?
3. What the physical properties of composite that needs to be quantified?
4. Explain the methods in short to measure the physical properties of a composite.

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