

Practical 2P10

SEM and Fracture

Introduction

When a material fails by fracture, there is complete separation of the two broken halves and a new surface, the fracture surface, is formed. It is often possible to examine the fracture surface and interpret features on it in a manner similar to the examination of a metallographic cross-section specimen. Features on the fracture surface can give us information about the mechanism of crack growth and also about the nature of the crack or defect from which the fracture nucleated. Unlike metallographic cross sections, fracture surfaces often contain substantial vertical relief and scanning electron microscopy (SEM), with its much greater depth of field, is routinely used for fracture surface investigation, fractography. Fractography is an important tool of failure analysis and is often used in accident investigation to help pin-point the cause of failure. One part of this practical will use the SEM to study fracture surfaces in order to deduce the causes and mechanisms of fracture.

The energetics of the process which leads to the formation of this new fracture surface was first considered by Griffiths who developed his famous equation which defines the thermodynamic requirements for fracture

$$\sigma_f = \sqrt{\frac{2\gamma E}{\pi c}} \quad (1)$$

where σ_f is the fracture stress, γ is the surface energy, E is the Young's modulus and c is the length of some preexisting surface flaw or half the length of an internal flaw. Griffiths' equation assumes that the only process which absorbs energy during fracture is the energy required to form a new surface. Although this is a pretty good approximation for ideally brittle materials it is rather simplistic as a general rule and has been modified extensively.

From equation 1 we can see that the fracture stress of a brittle material is no longer a well defined material property. Instead it can vary significantly from specimen to specimen in the same material controlled by the size of the largest flaw present in each piece. This is very different from ductile materials which have a well defined yield stress which is a measurable material property. Thus it is much more difficult to design with materials which fail in a brittle manner than those which fail in a ductile way because of the uncertainty associated with the size of the maximum flaw and the consequent failure stress. In order to cope with this problem engineers use a set of statistically based design rules which come under the blanket name of Weibull Statistics because they are based around the use of a particular statistical distribution function the Weibull Distribution. In the first part of this practical we will explore the statistical nature of the failure strength in a brittle material and note how this is related to the inherent population of defects in a material.

The Weibull distribution is normally considered as a cumulative probability of the chance of success (P_s) of a given volume of material (V) to a given fracture stress (σ).

$$P_s = \exp \left\{ - \left(\frac{V}{V_0} \right) \left(\frac{\sigma - \sigma_c}{\sigma_0} \right)^m \right\} \quad (2)$$

where V_0 and σ_0 are normalising constants, c is a critical stress below which fracture cannot occur and m is the Weibull Modulus which defines the shape of the distribution. If m is very large the distribution shape becomes a sharper falling step and at $m = \infty$ it describes a perfect step with a probability of 1 when $(\sigma - \sigma_c) < \sigma_0$ and 0 when $(\sigma - \sigma_c) > \sigma_0$. Thus when m is large the behaviour of the material is more reproducible around a mean failure value and as m decreases the behaviour becomes more scattered. The distribution is cumulative because once a specimen has broken it has failed at the stress defined by the largest flaw present. There will be smaller flaws which could have initiated failure at a higher stress but as the specimen is now broken they will never initiate fracture themselves.

Experimental

1. Statistics of Fracture in Glass

In the first part of this experiment, we will investigate the range of failure strengths exhibited by a borosilicate glass. Select fifteen 4mm diameter glass rods cut to a size to fit in the enclosed testing box. If the rods have not already been cut to size, the Class Technician or the Junior Demonstrator will help you cut them to size. Do not attempt to do this without their supervision. Each of these rods will be tested in 3-point bend in the protective box by adding weight (sand) to the bucket attached to the mid point of the rod. The load should be increased gradually. Note the load at which each bar fails, with the help of the weighing scale. Make sure you mark each bar after failure so you can identify it later.

Always use gloves to remove the glass samples out of the protective box. Be aware of the smaller shattered pieces of glass in the box.

Do not attempt to fit the two halves of the samples back together, you may damage the fracture surface.

Due to COVID-19 restrictions, one glass sample has already been fractured and has been placed in the SEM for investigation (more about this later). You are required to use the data from this sample given to you. You will have to fracture atleast 14 additional samples (to the one already given) and proceed further. Please see the Lab demonstration video to understand this.

Now take a second batch of fifteen rods identical to the sample you have just tested. Place all these rods into the plastic container provided and approximately half fill the container with coarse silicon carbide grit. Gently shake or roll the container filled with the rods and grit for a few minutes. Carefully pour out all the grit and, wearing gloves, remove each of the rods. You should now test this second sample of glass rods, in the same manner as you tested the first set of rods, recording their failure loads.

Due to COVID-19 restriction, the data from this second set of 15 samples will be given to you. You are not expected to repeat this second batch, but you may choose to do so. Please speak to the JD or the Teaching Lab Technician about

this. A sample from this lot has already been selected and placed in the SEM for investigation (more about this later). Please see the Lab demonstration video to understand this.

Safety note: dispose of any waste glass in the broken-glass box.

Analysis

From your data calculate the Weibull Modulus, the mean fracture load and fracture stress of the glass rods in their as received state and after abrading with silicon carbide. Taking the surface energy of glass to be 1 Jm^{-2} and its Young's Modulus to be 70GPa calculate the mean flaw size which initiated fracture and the largest and smallest critical flaw tested in each batch.

2. Fractography

Preparation of Fracture Surfaces

Glass

From each batch (i.e. one as-received & one abraded) of glass specimens take one of the halves which fractured at the lowest load. With the help of the Junior Demonstrator break off the fracture surface on a short stub of glass and mount it on a SEM sample holder (using conductive paint / screw mount). In order to easily see the features on the fracture surface in the SEM, the stub will need to be coated with a very thin layer of gold. The Junior Demonstrator will help you do this.

Due to COVID-19 and time restrictions, these two samples have already been selected by the Junior Demonstrator, coated in gold and placed in the SEM for you.

Steel

We will also look at brittle and a ductile fracture surface in steel. To make these surfaces we will use an impact testing machine which uses cylindrical notched specimens. The class technician will have prepared four specimens for you. You should put two of these specimen into liquid nitrogen for about 5 minutes to make sure it is well below the ductile/brittle transition temperature. Using the impact testing machine fracture both the room temperature and liquid nitrogen

cooled specimens. **Only do this with the help of the Junior Demonstrator or Class Technician.**

Due to COVID-19 restrictions, you will not be using the impact tester or fracturing the steel samples. They have already been fractured for you by the Junior Demonstrator and have been placed in the SEM for investigation. Please see the Lab demonstration video to understand this.

Safety note: liquid nitrogen can (cold) burn the skin; wear lab coat, gauntlets and face-shield. The liquid nitrogen cooled specimen will still remain cold after they have been broken.

Mount these fractured surfaces on an SEM specimen holder (using conductive paint / screw mount).

If using conductive paint to mount, you should do all the specimen preparation the day before you use the SEM in order to make sure the conductive paint is dry before putting the specimen in the SEM.

Examination of the Fracture Surfaces

You should have four fracture surfaces ready for examination in the SEM. A fifth sample has also been loaded in the SEM to demonstrate EDX analysis (details later). Before inserting into the SEM study the surfaces with the naked eye and note any apparent details.

Due to COVID-19 restrictions and time constraints, the samples have already been placed in the SEM and you may not be able to visually note the appearance. You can check the visual appearance on another glass sample, by selection one from a batch of 14 as-received samples that you have fractured. The steel sample that has been fractured after being cooled in liquid-nitrogen has a shiny appearance under light and the one fractured at room temperature has a slightly dull appearance. Why is this ?

You should sketch the visual appearance of the fracture surface as this may help relate details found in the SEM to their position, if possible. Normally when a

material fails by fracture the fracture surface forms normal to the direction of the greatest tensile stress. However, at the scale of the microstructure local deviations may occur from this plane and the SEM is used to study these. The SEM forms an image of the surface using secondary electrons and is very sensitive to surface topography. Thus the SEM can be used to characterise the crack path from the topography of the fracture surface.

Use the SEM to take a number of photographs of the fracture surfaces at different magnifications. You will be allowed to operate the SEM in the teaching labs. The JD will help you start the instrument. In the interest of time, the JD will also give you the corresponding stage position. You can enter these details to move to the sample position in the SEM. The conditions for imaging may vary, but the JD will let you know the best imaging conditions for a sample. The students are expected to share the allotted SEM time amongst themselves to look at the samples. You will also be looking at a polished steel sample (as a 5th sample and the same material as the two fractured steel samples under impact loading) in the SEM. An Energy Dispersive X-Ray Analysis (also referred to as EDX, EDS or EDAX) will help you understand the composition of the inclusions that are generally present in steel. This is to demonstrate how analytical methods can be employed in the SEM apart from regular imaging.

The JD will help you transfer the images from the PC. Do not use external USB / other storage media on the SEM PC. In the glass specimens, try and determine the location of the initial flaw which initiated fracture and measure its size. Can Griffith's theory be used to reasonably match the measured flaw size to fracture stress? The two steel specimens will have fractured in a brittle and ductile manner; compare the two fracture surfaces. What can you deduce about the different fracture mechanisms in the steel? Compare the brittle steel specimen to the glass specimens; how can you explain the different fracture surface appearances?

Lab notebook

Do not duplicate the information given in this sheet. You are not expected to derive the Griffith equation or attempt to justify the Weibull distribution, nor

are you expected to write an essay on the ductile/brittle transition in steel. This practical concerns fracture and fractography. Keep the write up to under 1000 words: fewer would be better.

Be selective about the SEM micrographs you include in the report. Use only these that make of exemplify a definite point. Label / caption them appropriately. Please include error bars (and analysis) on the plots.

You should consider the differences in the failure strengths and Weibull moduli measured with your two glass specimen sets and try to explain the differences you measured.

If you succeed in finding the fracture-initiating flaw in the SEM, you should compare it to the values you have estimated using your results and the materials data given. If not, think about why this was not possible in your current experiment. With the steel specimens look carefully at the fracture surfaces and compare the two specimens. Try to explain the origins of all the features you can see.

Appendix: Calculation of Weibull Statistics

We intend to analyse the data to determine the Weibull modulus (m) of the glass rods. To do this we must determine the probability of survival / failure for each failure load. For each of the two sets of experimental data, rank all the measured failure loads in order of load defining the smallest load as rank 1 and the largest as rank 15. If you have found specimens with the same failure load each must be included at a different rank, e.g. if the fifth weakest specimen failed at 50g along with 2 others at the same load these would be defined rank 5, 6 and 7 but all with the same failure load. The probability of failure of each ranked position n in a total sample size of N is given by

$$P_f = \frac{n}{N + 1} \quad (3)$$

This definition is justified by arguing that until one specimen in a sample has failed we cannot define a probability of failure / survival and that no matter how

large the sample size, there is always a chance that one further specimen tested may fail at a higher load than that tested so far.

Now make a table of probability of survival against failure load. In order to simplify analysis we make two assumptions. The first is that because all the specimens tested are of the same volume we can neglect the volume terms (V and V_0) in equation 2. The second simplification is to assume that σ_c is zero. Equation 2 is now written in reduced form

$$P_s = \exp \left\{ - \left(\frac{\sigma}{\sigma_0} \right)^m \right\} \quad (4)$$

By taking logarithms of both sides of equation 4

$$\ln(P_s) = - \left(\frac{\sigma}{\sigma_0} \right)^m \quad (4a)$$

and after changing the sign of both sides to avoid negative values, taking logarithms again

$$\ln(-\ln(P_s)) = m \ln(\sigma) - m \ln(\sigma_0) \quad (5)$$

Thus a plot of the double log of survival probability against the log of the failure stress will produce a straight line of gradient m . Also, because the failure stress is related to failure load in bend by a simple linear expression, a plot of $\ln(-\ln(P_s))$ against $\ln(\text{failure load})$ will also show a gradient of m .