Practical 2P5
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

$$\sigma_f = \frac{2\gamma E}{\pi c}$$

where \(\sigma_f\) is the fracture stress, \(\gamma\) is the surface energy, \(E\) is Young's modulus and \(c\) is the length of some pre-existing 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 survival (\(P_s\)) of a given volume of material (\(V\)) to a given fracture stress (\(\sigma\)).

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

where \(V_0\) and \(\sigma_0\) are normalising constants, \(\sigma_c\) 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 (trade-name Pyrex). Select fifteen 3mm 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 weights to the scale pan attached to the mid point of the rod. The load should be increased using increments of 0.05N (or approximately 5g). Note the load at which each bar fails. Make sure you mark each bar after failure so you can identify it later. Do not attempt to fit the two halves of the samples back together, you may damage the fracture surface.

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.

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 (see Appendix). Taking the surface energy of glass to be 1Jm$^{-2}$ 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 conducting paint. 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. Again the Junior Demonstrator will help you do this.

Steel
We will also look at a 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 two specimens for you. You should put one 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.

Safety note: liquid nitrogen can (cold) burn the skin; wear lab coat, gauntlets and face-shield.

Note the amount of energy absorbed for each specimen and retain the two broken fracture surfaces in each case. Mount one fracture surface from each specimen on an SEM specimen holder with conducting paint.

In order to make sure the conducting paint is fully dry before putting the specimen into the SEM vacuum chamber you should do all the specimen preparation the day before you use the SEM.

Examination of the Fracture Surfaces
You should have four fracture surfaces ready for examination in the SEM. Before inserting into the SEM study the surfaces with the naked eye and note any apparent details. You should sketch the visual appearance of the fracture surface as this may help relate details found in the SEM to their position. 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 Class
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.

Using the SEM take a number of photographs of the fracture surfaces at different magnifications. The film on which the SEM images are recorded will be developed by the Photographic Unit. The JD will show you where to take the film. In the glass specimens try and determine the location of the initial flaw which initiated fracture and measure its size. 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? Using the impact test results estimate the energy absorbed per square metre of fracture surface and compare the value to the surface energy of steel (about 1Jm$^{-2}$).

**Writing your Report**

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 2000 words: fewer would be better.

Be selective about the SEM photographs you include in your report, and stick them in. DO NOT just attach a sheaf of loose photographs. Use only those that make or exemplify a definite point. Print the photographs at a useful size - maybe half a page width so that you can put a caption / explanation beside them.

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. 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, or $P_s$, in equation 2, 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 survival of each ranked position $n$ in a total sample size of $N$ is given by

$$P_s = \frac{n}{N + 1}$$  \hspace{1cm} (3)

This definition is justified by arguing that until one specimen in a sample has failed we cannot define a probability of 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 $\sigma_c$ is zero. Equation 2 is now written in reduced form

$$P_s = \exp\left\{- \frac{\sigma}{\sigma_o}^m \right\}$$  \hspace{1cm} (4)

By taking logarithms of both sides of equation 4

$$\ln(P_s) = -\left(\frac{\sigma}{\sigma_o}\right)^m$$  \hspace{1cm} (4a)

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

$$\ln(-\ln(P_s)) = m \ln\sigma - m \ln\sigma_o$$  \hspace{1cm} (5)

Thus a plot of the double log of failure 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 $\ln$(failure load) will also show a gradient of m.