Practical 2P10

SEM and Fracture

Introduction

When a material fractures, there is complete separation of the two broken halves and new surface, the fracture surface, is formed. Examination of the fracture surface yields information about the mechanism of crack growth, the nature of the crack or defect from which the fracture originated and the stress state responsible for the fracture. Unlike metallographic cross sections, fracture surfaces often contain substantial vertical relief, so low magnification optical methods and scanning electron microscopy (SEM), with its good depth of field, are 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 visual examination and SEM to study fracture surfaces in order to deduce the causes and mechanisms of fracture. EDX microanalysis will also be demonstrated.

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

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

where σ_f is the fracture stress, γ 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. Griffith's equation assumes that the only process which absorbs energy during fracture is the energy required to form the new surface. Although this is a pretty good approximation for ideally brittle materials it is rather simplistic as a general rule and has since been modified extensively.

From equation 1 we can see that the fracture stress of a brittle material is not a well defined material property. Instead it can vary significantly from specimen to specimen in the same material, as it is controlled by the size of the single largest flaw that happens to be 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.

One way of coping with this problem is to design such that components have a certainly probability of survival. In the first part of this practical we will explore the statistical nature of the strength of a brittle material and describe it using the Weibull distribution, which is the most commonly used strength distribution.

The Weibull distribution describes the probability of survival (P_s) of a volume of material (V) when subjected to a 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, σ_c is a critical stress below which fracture cannot occur and m is the Weibull Modulus which defines the shape of the distribution. The equation directly describes the strength distribution but it is fundamentally related to the distribution of flaw sizes. Although the Weibull modulus m is essentially a fitting parameter, it has a useful physical interpretation. 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.

Metallic materials can also fracture in a brittle manner under some conditions, though local plasticity occurring around the crack tip raises the energy required for fracture compared with glass and ceramics. Metallic materials can also fracture in a ductile manner, in which general plasticity absorbs even more energy. Brittle and ductile fracture of a steel tested under different conditions will also be investigated in this practical.

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 4 mm 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 to do this. **Do not attempt to do this without their supervision**.

Each of these rods will be tested in 3-point bending 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. Note how many pieces the sample broke into and where along the length of the bar the fracture occurred. (Where would you expect it to occur? Why might it fracture somewhere else? What is the implication for the actual fracture stress?) Make sure you mark each bar after failure so you can identify it later.

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

Do not attempt to fit the broken pieces of the samples back together: you may damage the fracture surface, spoiling your fractography results.

Now take a second batch of fifteen rods nominally 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 waste glass in the broken-glass box. **Do not** dispose of anything else in this box!

Analysis

From your data calculate the Weibull Modulus (see Appendix) and the mean strength of the glass rods, both in their as-received state and after abrading with silicon carbide grit. [The second moment of area I of a circular cross-section rod with diameter d is $\pi d^4/64$]. Use the *nominal* stress at failure – i.e. the maximum tensile stress in the specimen – for these analyses, even if the failure did not take place at the point in the sample where this maximum stress would be located. It can be shown that the Weibull equation in the Appendix still returns that correct Weibull modulus.

Taking the surface energy of glass to be 1 J m^{-2} and its Young's Modulus to be 70 GPa, calculate the mean flaw size which initiated fracture and the largest and smallest critical flaw in each batch tested. Here it *would* be appropriate to use the actual stress at the point of fracture.

2. Fractography and Ductile-Brittle Transition in Steel

Preparation of Fracture Surfaces

Glass

From the as-received batch of glass specimens, take one of the pieces of the specimen that fractured at the median load (or close to it). For the abraded batch, use the specimen with the lowest strength. With the help of the Junior Demonstrator break off the fracture surface on a short stub of glass and mount the stub on a SEM sample holder (grind the end to be attached to the holder flat on an abrasive disc, use compressed air to blow debris off the fracture surface, and use conductive paint as adhesive). In order to make the specimen electrically conductive for examination in the SEM, it will need to be coated with a very thin layer of gold. The Junior Demonstrator will help you do this.

Steel

We will also look at brittle and ductile fracture surfaces in steel. To make these surfaces we will use an impact testing machine which uses cylindrical notched specimens. As well as breaking the specimens, the machine records the energy absorbed during fracture, which should be recorded and included in your report. The class technician will have prepared four specimens for you. You should put two of these specimens into liquid nitrogen for about 5 minutes to make sure they are 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. The liquid nitrogen cooled specimens 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

Before inserting into the SEM, study the surfaces with the naked eye and note the shape of the fracture path. For the glass specimens, do this before and after coating with gold. Examine the fracture surfaces with a magnifying glass or stereomicroscope and record your observations. If you have a good phone-camera, take pictures of the fracture surface from different orientations and with various directions of illumination. This evidence can give information about the location of the fracture origin, the stress state causing fracture, the direction of travel of the crack, the shape of the crack front, and other factors involved in fracture. It will also be a useful reference for what you see in the SEM.

As well as your own four fracture surfaces a fifth sample has also been loaded in the SEM to demonstrate EDX analysis (details later).

When a material fails by brittle fracture, the crack usually travels normal to the direction of the greatest tensile stress and you should consider the macroscopic crack path in the glass specimens in this light. 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 sensitive to surface topography. Thus the SEM can be used to characterise the crack path from the topography of the fracture surface. For the glass specimens, it also has the resolution to be able to observe the critical defect from which fracture originated.

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 5^{th} sample and the same material as the two fractured steel samples under impact loading) in the SEM.

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 to determine the location of the flaw which initiated fracture and measure its size. (Is the flaw at the expected position on the cross section? Why might it be somewhere else and again, what is the implication for your strength results? Can Griffith's theory be used to reasonably match the measured flaw size to fracture stress?) As well as the defect itself, various features on the fracture surface may (or may not) be visible, e.g. mirror, mist, hackle, Wallner lines. Which, if any of these are present? The two steel specimens should have fractured in a brittle and ductile manner according to their testing temperature; compare the two fracture surfaces. What can you deduce about the different fracture mechanisms in the steel? How do they correlate with the energy absorbed? Compare the brittle steel specimen to the glass specimens; how can you explain the different fracture surface appearances?

Assessment

This lab is assessed through your **laboratory notebook**, which should document your practical following the guidance given in the Teaching Laboratory Guide available on Canvas, and in particular Appendix A, "Keeping a good lab notebook: some advice".

Note that for this practical, SEM micrographs are "Results". Any that might be useful if someone were to write a report based on your notebook should be included, along with relevant information concerning the micrograph.

Regarding analysis of the bend tests on glass rods: failure stresses and estimates of the corresponding critical defect sizes, Weibull plots and Weibull moduli should be extracted from the results.

Appendix: Analysis of Weibull Statistics

To determine the Weibull modulus (m) of the glass rods, we must allocate probabilities of survival for each failure stress. The simplest way of doing this for a set of experimental data is first to rank all the measured strengths, defining the lowest load as rank 1 and the largest as rank 15. If you have found specimens

with the same strength, each must be included at a different rank, e.g. if the fifth weakest specimen failed at 50 MPa along with 2 others at the same stress, these would be allotted ranks 5, 6 and 7 but all with the same strength. The probability of survival of each ranked position n in a total sample size of N is then given by

$$P_s = 1 - \frac{n}{N+1} \tag{3}$$

This corresponds to the assumption that the measured strengths are equally separated in the range of allowed probabilities, 1 (for zero stress) to 0 (for infinite stress). (Other, statistically more rigorous approaches are available).

Now make a table of probability of survival against strength. We can simplify eq. (2) in two ways. 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, which is almost always found to be justified in ceramics ans glasses. (What would you see in your analysis if it were not justified?). Equation 2 is now written in reduced form

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

By taking logarithms of both sides of equation 4

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

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

$$ln(-ln(P_s)) = m\ ln(\sigma) - m\ ln(\sigma_0) \tag{5}$$

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.