Unit 2: Fracture of Glass

MSE 360 Materials Laboratory I- Fall 2007

I. Objectives

Mechanical properties are key attributes of materials. In this unit we will consider the simplest type of mechanical material – an ideally brittle solid, which is very closely represented by ordinary soda lime silicate glass. We will determine the fracture strength of glass using flexural testing rods, and examine how the strength depends upon surface flaws. We will do simple fractography to identify the features on the fracture surface of the rods. We also will use the indentation cracking method to examine the fracture toughness of glass. Since glass is photoelastic, we an visualize stress with polarized light. We have four activities.

ACTIVITY SCHEDULE

Activity 1 flexural testing; Activity 2 fractography; Activity 3 indentation toughness; Activity 4 residual stress imaging

ACTIVITY SCHEDULE

Date/Time	Group 1	Group 2	Group 3	Group 4
Nov 13-15 day1 first half 1:30-3:30	activity #1 flexural testing	activity #2 fractography	activity #3 indentation toughness	activity #4 residual stress
Nov 13-15 day1 second half 3:30-5:30	activity #2 fractography	activity #3 indentation toughness	activity #4 residual stress	activity #1 flexural testing
Nov 27-29 day 2 first half 1:30-3:30	activity #3 indentation toughness	activity #4 residual stress	activity #1 flexural testing	activity #2 fractography
Nov 27-29 day 2 second half 3:30-5:30	activity #4 residual stress	activity #1 flexural testing	activity #2 fractography	activity #3 indentation toughness
Dec 4-6 Day 3	Finish tasks	Finish tasks	Finish tasks	Finish tasks

II. Experimental Procedures

In this unit we will determine the fracture strength and toughness of soda lime glass flexural testing of glass rods. Determine the role of surface flaws by testing abraded specimens, and compare results with expectations of Griffith theory. Results from several groups will be combined to make a large enough data set for analysis of fracture statistics. Learn the utility (and limitations) of the indentation technique for inferring the fracture toughness by analyzing the cracks associated with a Vickers indent in glass. Conduct a fractographic analysis of fracture surfaces of the the specimens to identify the fracture origin and relate fracture mirrors to fracture toughness. Use polarized light methods to observe applied and residual stresses.

There will be four major activities, scheduled as below to minimize bottlenecks. These are: flexural testing on MTS Insight 10kN load frame ; examination of fracture surfaces on the Nikon SMZ binocular microscope and the Philips XL30 SEM; producing a series of Vickers microhardness indents at several loads on the Clark Vickers Microhardness tester and measuring the indentation cracks on the Nikon Optiphot microscopes. You also will examine residual stresses using the photoelastic effect, using polarized light.

Activity 1 Flexural Testing

The laboratory instructor will supply each group with 20 rods of soda-lime glass rods. These have had an anneal to remove most of the residual stress. The surface condition is "as-received"[1]. Each group will test 10 rods in the as-received conditions and 10 rods in an abraded condition. Store all your data in files accessible by your classmates, since each student will conduct statistical analyses of the combined results for all lab groups.

The flexural test is conducted according to MIL-SND 1942, using a fully 4-point flexural fixture (Instron model 2810-400 5 kN) using the 10 kN loadframe (MTS Insight 10kN). The lab instructor will point out to you the features of this fixture and the details of conducting the test with the MTS Testworks4 software. Identify and measure each specimen. Be careful to protect the fracture surfaces for later fractographic analysis. Calculate the strength from the breaking load and dimensions.

Abraded specimens

We will examine the effect of surface condition by testing glass rods after a light abrasion treatment. Each group will test 5 rods abraded with longitudinal scratches, and 5 abraded with transverse scratches. Abrade the rods with SiC grinding paper of your assigned grit size: Group 1 uses 240 grit, group 2 uses 400 grit, group 3 uses 600 grit, and group 4 uses 1200 grit paper. With very light pressure, scratch 5 bars along the length to make longitudinal scratches, and 5 additional bars along the diameter to make transverse scratches. Test the rods, again preserving their fracture surfaces.

REPORT—Activity 1

1) Flexural strengths of your group's as-received rods
2) Use the combined as-received strength data for the entire class [there should be about 120 specimens]. Examine the combined data set and infer if the various group's data can lumped together. If lumping the data is justified, fit a 2-parameter Weibull distribution and calculate the Weibull modulus and characteristic strength. Can the data be fit to a 3-parameter Weibull?
3) Flexural strengths of your group's abraded rods

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[1]Very freshly made glass has a fire-polished "pristine" surface which is virtually defect free. A few moments of ordinary handling will decorate the surfaces with a population of defects. The "as-received" condition means nothing more than what it says-- the defect population is simply what it happens to be.

4) Combine the flexural strengths for the abraded of the four groups for your lab day. Test the assumption that the dominent flaw size is proportional to the size of the abrasive particles[2], assuming that the strength and flaw size obey a Griffith relationship.

5) Use the combined abraded strength data for the entire class [there should be about 16 specimens for each grit size]. Examine the combined data set an infer if the various group's data can lumped together. If lumping the data is justified, fit a 2-parameter Weibull distribution and calculate the Weibull modulus and characteristic strength for each grit size.

Does the abrasive-size dependence of the characteristic strength fit your expectation? Do you expect the Weibull modulus to change with grit size? Use the combined as-received strength data for the entire class [there should be about 60 specimens]. Examine the combined data set an infer if the various group's data can lumped together. If lumping the data is justified, fit a 2-parameter Weibull distribution and calculate the Weibull modulus and characteristic strength.

Activity 2 Fractography

Here you will examine the fracture surfaces of your glass rods (If you have not tested your glass yet, obtain some samples from another group, carefully noting the strength of the indiviual specimens you borrowed.) Use the Nikon stereomicroscope in the Van Vlack Lab, using a variety of illumination angles, and use the Philips XL30 SEM. The SEM gives much higher resolution, but there is information at all stages of magnification. Reflected light also provides better contrast for certain features which are not easily visible with secondary electrons. Take a few stereomicroscope and SEM images for your report.

Using the diagrams from Michalske and Varner (below), identify the fracture origin, the fracture mirror, the mist region, and the hackle zone. Use the "shear lip" to infer the orientation of the rod when it broke, and determine if the fracture origin was at the point of peak tensile stress[3]. If possible, determine the size of the fracture origin (it is not always distinct), and relate the fracture origin size to the strength for the individual rods.

Each person in your lab group should individually measure the radius of the fracture mirrors. The location (or even the existance!) of the mirror is rather subjective-- so it will be of interest to compare what each person sees.

REPORT – Activity 2

1) Accurate photograph of one specimen, identifying origin, mirror, mist, hackle, and shear lip
2) Fracture origin size plotted against strength, to test for Griffith behavior.

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[2]use the average SiC particle size in microns, not grit size. The relationship between particle size and grit size is available in standard handbooks.

[3]The most severe flaw might not be at the point of maximum stress.

3) Mirror radii for each specimen, with discussion of discrepancies between yourself and other observers. Estimate the fracture mirror constant of the glass with the Mecholsky equation (eq. 5, p. 655 of Michalske handout).

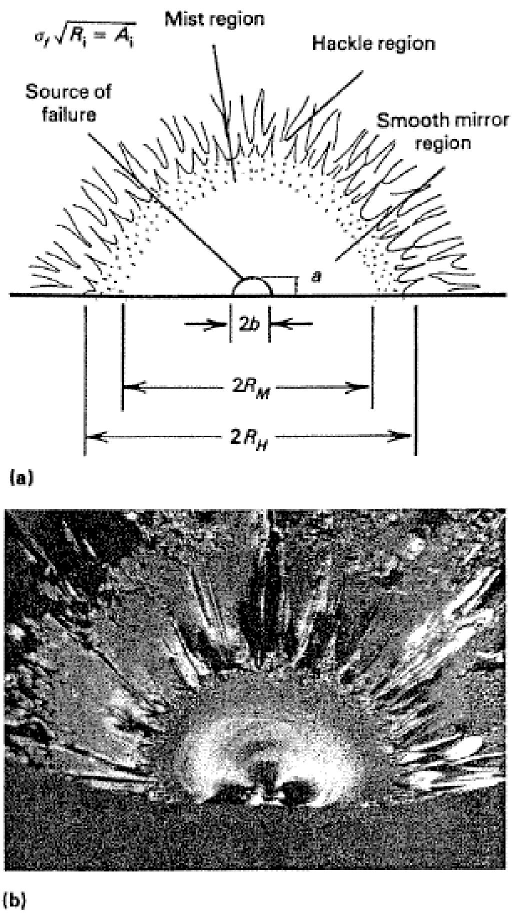


Fig 7 (a) Schematic representation of a fracture surface showing the formation of mist and hackle that define the mirror radius. (b) Photomicrograph of fracture surface in glass showing mirror, mist, hackle, and crack branching radii. Reflected light, 10×

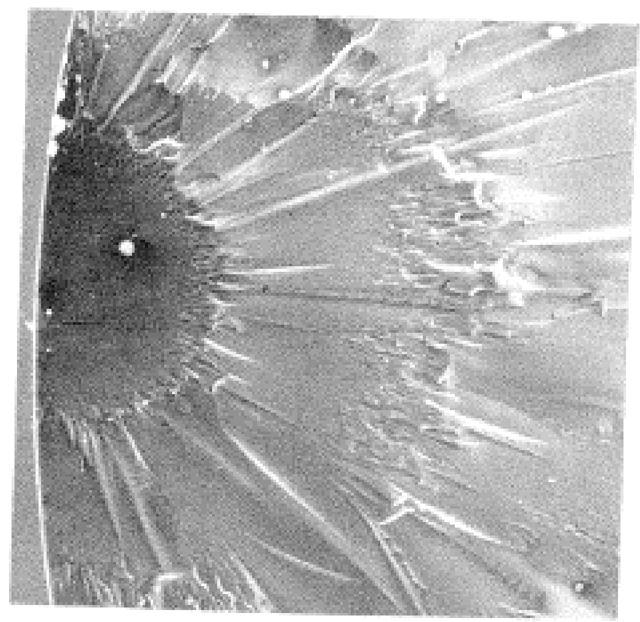


Fig 1 Fracture surface of a glass rod broken in bending. Fracture origin is at left; nearly semicircular region is the fracture mirror, bordered by mist and velocity hackle. Note the second region of mist and velocity hackle. SEM. 47×

Left: T.A. Michalske, "Quantitative Fracture Analysis", Right: J.R. Varner "Descriptive Fractography" from Vol 4, *Engineered Materials Handbook*, ASM International (1991) (see resources)

Activity 3 Vickers Indentations

In brittle materials, a Vickers diamond indentor makes a square impression of diagonal 2a, which is related to the hardness. A pattern of cracks -- the "radial/median crack system" -- also develops. The surface traces of the cracks appear along the diagonals of the indent. Lawn and his coworkers have related the length of these cracks to the fracture toughness [K_c] with the following expression[4]

[4]G.R. Anstis, P. Chantikul, D.R. Lawn, and D.B. Marshall, *J. American Ceramic Society* **64** [9] p. 533-538 (1981)

$$K_c = 0.016\left(\frac{E}{H}\right)^{1/2}\left[\frac{P}{c^{3/2}}\right]$$

where P is the indentation load [in Newtons], "c" is the distance from the center of the indent to the tip of the crack, E is the modulus [70 GPa for soda lime glass] and H is the hardness [5.5 GPa for this glass]. It is common to use this to infer fracture toughness from simple measurements of indentation cracks.

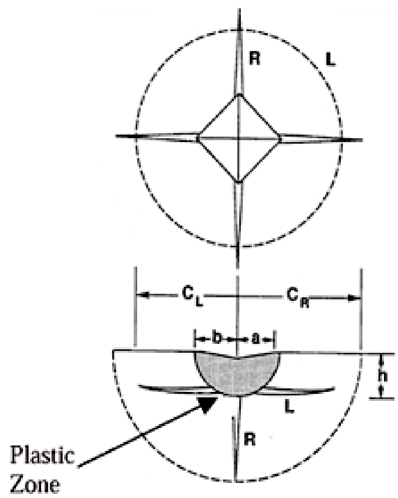


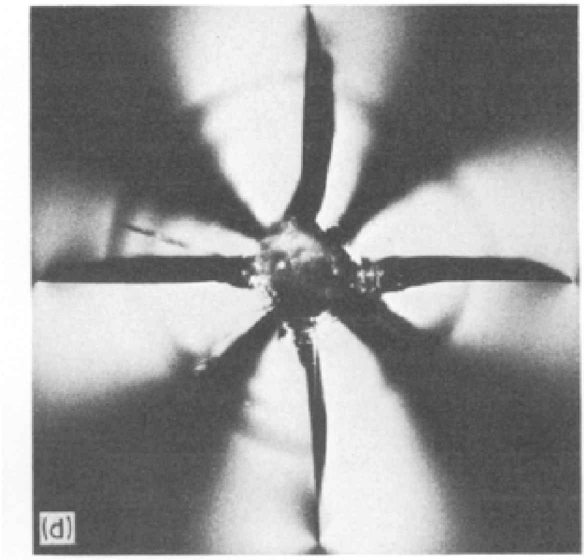
Figure 2. Schematic representation (in upper view and axial section) of indenture in vitro, caused by a Vickers indentor, where C_R represents the radial crack R dimension and C_L represents the lateral crack L one. The limited region is the plastic deformation zone, with h depth. (Adapted from Marshall (1983), apud Malkin and Ritter (1989).

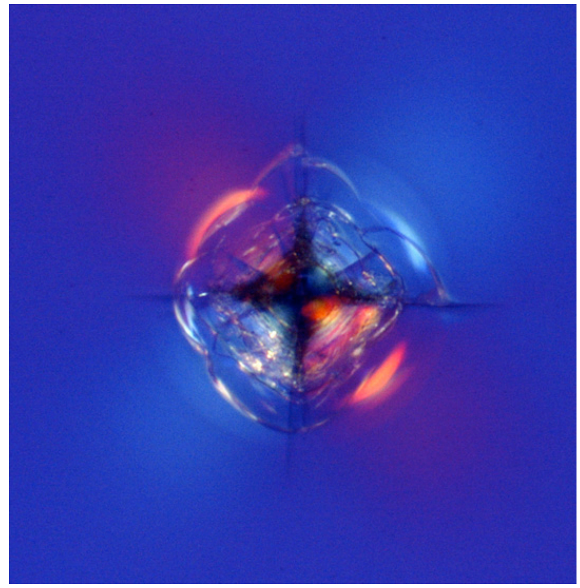
http://www.scielo.br/img/fbpe/jbsms/v23n1/a02fig02.gif

The laboratory instructor will discuss the recommended procedures for estimating the fracture toughness by the indentation method. The sample will be a soda-lime glass slide. Use the Clark microhardness tester with a Vickers diamond indentor. Produce 5 sound indents each at 4 different loads between 10 N and 100 N [*Newtons, not kilograms!*]

Using the the Nikon Optiphot, for each indent, measure the size of the Vickers indent and the length of all four crack traces. The position of the crack tip is rather subjective, so have several individuals measure some of the cracks, and discuss any variation in interpretation. Capture a few nice images for the report.

Examine the residual stress fields around the idents using one of the Nikons in polarized transmitted light. You may see a pattern such as the one below from Marshall and Lawn.





Left: Residual Stresses in glass under Vickers indent, as revealed by transmitted polarized light From Figure 3 of D.B. Marshall and B.R. Lawn, “Residual stress effects in sharp contact cracking” *Journal of Materials Science* **14**(1979) 2001-2012

Right: A rather more decorative version of the same, entered in the Princeton “Art of Science” competition as *Blue Indentation,* by Elizabeth Allaway ‘07, Ryan Rimmele ‘07, Richard Li ‘07, Nate Beck ‘06, and George Scherer of the Princeton Department of Civil and Environmental Engineering “This is an indentation in glass made with a pyramidal diamond tip (called a Vickers indenter) as part of a lab in CEE 364 Materials in Civil Engineering. The goal was to make a flaw of controlled size and measure its effect on the strength of the glass—but the result was a thing of beauty. The color results from crossed polarizers that indicate the shape of the stress distribution.”

REPORT-- Vickers indentation Activity 3

1) Plot the crack length $C^{3/2}$ vs. the indentation load and determine the slope with linear regression

2) Use the slope of this curve, with the Anstis et al. equation to calculate the fracture toughness of the glass.

3) Calculate a toughness estimate for every individual indent, and compare these values with the one obtained in (2)

4) Discuss the residual stress patterns seem in polarized light.

Activity 4: Residual Stresses

For this activity, examine a number of photoelastic samples, using a variety of methods. We will make this up at the time of the lab.

Fracture of Glass – Background

Flexural Strength Testing

The flexural strength technique we use is based upon the standard ASTM C1161 and MIL-STD 1942 for fixture dimensions, loading rates, and other pertinent variables. Typically brittle materials are tested in bending as machined bars. The strength results are quite sensitive to the quality of the machining, and the literature has guidelines for recommended grinding techniques. The edges on the tensile side of the bar should be ground flat, or chamfered, to eliminate artifacts from stress concentrations. However, for this lab, we adopt rod-shaped samples in an as-manufactured condition. This is primarily for convenience and low cost, since we avoid the bother of machining.

Recall what you learned in ME 211 and MSE 420 about the mechanics of a beam in bending. The normal stress varies from compressive on one surface, to zero at the neutral axis, and tensile at the opposite surface. Shear stress varies from zero at the outer fibers to a maximum at the neutral axis.[5] For a rod, the maximum tensile stress exists only along a narrow line in the "outer fiber" of the rod. Typically we *assume* that the fracture originates at the point of maximum tensile stress[6] and estimate the flexural strength from the maximum tensile stress at the point of fracture. For any beam, the tensile stress at a distance 'c' from the neutral axis is:

$$\sigma = {}^{Mc}\!/_{I}$$

where I is the moment of inertia and M is the bending moment applied to the beam. For a rod, c=R, the radius. For a rod with circular cross-section, the moment of inertia is:

$$I = {}^{\pi R^4}\!/_{4}$$

For a beam loaded in four-point flexure, with characteristic lengths l_{inner} and l_{outer} (both with respect to the center of mass of the beam), bearing a load F, the moment is:

$$M = \frac{F\left(l_{outer} - l_{inner}\right)}{2}$$

For our particular fixture, $l_{inner} = L$ and $l_{outer} = 2L$.

In a flexural test, we increase the deflection of the beam at a constant rate by moving the crosshead while measuring the load with a load cell. The peak load at the point of fracture is related to the maximum stress at the outer fiber and gives the fracture strength as:

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[5]Bending beams are useful despite their complex stress state only for quite brittle materials. In very brittle materials, cracks grow in response to the tensile stress, typically from pre-existing flaw which are much smaller than the specimen size.

[6]Sometimes there is a more severe flaw in another location. With fractographic analysis, one can determine whether the fracture did indeed originate in the 'outer fiber'. If it did not, it is possible to calculate the stress at the location of the originating flaw, although this is rarely done.

$$\sigma_f = \frac{2L}{\pi R^3} F$$

For a proper test, one must assure that the only stresses on the sample come from the bending moment. If the sample is not straight and flat (for bars), the sample will twist in the fixture. This torsion can cause major errors. The fixture must apply only normal loads. Take the time to examine the Instron 2810-400 5kN fixture we are using, and ask your lab instructor to explain its features.

Fracture Statistics

If the strength variability of a set of specimens[7] fit a Weibull distribution, the probability that a sample will fail at a particular strength (σ_i) is P_i

$$P_i = 1 - \exp\left[-\left(\frac{\sigma_i - \sigma_u}{\sigma_o} \right)^m \right]$$

where σ_o is the characteristic strength, σ_u is the threshold stress (below which there is no failure) and 'm' is the Weibull modulus. This three-parameter Weibull expression is actually rarely used, because there does not seem to be a threshold stress for brittle materials[8], and because it is mathematically inconvenient. A more practical form is the two-parameter Weibull expression:

$$P_i = 1 - \exp\left[-\left(\frac{\sigma_i}{\sigma_o} \right)^m \right]$$

Thus, we characterize the central tendency of strength with the characteristic strength σ_o (strength at 63.2 % probability of failure) and the scatter in the strength data with the Weibull modulus. For many brittle ceramics, 'm' falls in the range 5-20.

To fit a set of strength data to the Weibull distribution, one first rank-orders the samples in order of increasing strength and assigns an index: i=1 for the weakest with strength σ_1, i=2 for the second weakest with strength σ_2, and so on, up to i=N for the strongest sample. For each sample, assign an estimate for the probability of failure by

$$P_i = (i - 0.5)/N.$$

Plot the data in the following linearized form of the Weibull distribution:

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[7]This form is strictly for samples of a standard volume, subject to uniform tensile stress. This is not strictly applicable for flexural tests, but this formula is commonly applied. Data for one set of samples (of a certain size and test type) can be related to another set (different size and test type) are reviewed by Geoge Quinn in "Strength and Proof Testing" and Curt Johnson "Advanced Statistical Concepts" in *ASM Engineered Materials Handbook, Vol. 4: Ceramics and Glasses, 1987.*

[8]Physically, a threshold strength implies a maximum possible flaw size. Why should there be a limit to flaw size, other than the trivial limit set by the size of the sample itself?

$$\ln\left[\ln\left(\frac{1}{1-P_i}\right)\right] = m\ln(\sigma_i) - m\ln(\sigma_o)$$

The Weibull modulus is obtained from the slope of a plot of lnln(1/1-P) vs. lnσ, and the characteristic strength obtained from the intercept. These are often determined by least squares, although this might exaggerate the importance of the low-strength data points.

The error in the estimate of the Weibull parameters, strictly from mathematical factors (not experimental), can be appreciable, particularly for small sample sizes. The figures below show confidence interval bands for the slope and characteristic value versus the number of samples. Notice that 40-60 samples are required for reasonably small errors.
(Figure 7 from A.F. McLean and D.L. Hartstock, "An Overview of the Ceramic Design Process", *ASM Engineered Materials Handbook, Vol. 4: Ceramics and Glasses* , ASM International, 1987, p. 681)

Handling the data

One can extract the Weibull parameters from a set of data only if it fits the Weibull distribution. Maybe your data will not fit. Be skeptical -- avoid a foolish force-fit. Plot all of your data in the linear form. Look for the "outliers"-- atypically strong or weak samples. Treating them properly requires your judgment. Outliers may be telling you something important (such as hinting at the existence of a second flaw population, which is rare but severe when it happens) or they may be telling you nothing (maybe a bad measurement or an error in data logging). Some people strictly include every data point, which is honest but can distort the "true" data with a false data point. Others cull their data of "suspicious" outliers[9] before fitting the Weibull. This can be done legitimately if there is some reason to suspect that the outlier is not a reliable data point. The danger of this approach is obvious. It is your data, so you have to decide[10].

If you have a good linear regression line, you can get the slope 'm' value, from least squares. Most regression packages also report the "correlation coefficient", R^2, which is a goodness-of-fit parameter. It reports if the data are well-correlated, but does not give an estimate of the error of the slope. Johnson and Tucker discuss "maximum likelihood estimators"[11], but these involve rather complicated analysis.

[9]Manufacturers sometimes do this. Often it is valid, since occasional errors exist. However, it IS rare to see someone reject a specimen because it was too strong!

[10]Another way to assess the sensitivity of Weibull parameters is by calculating them from the complete data set, and then re-calculating them after rejecting a few of the weaker and stronger samples. If the parameters change little, then once might have more confidence in them. If they vary substantially, then one might have much less confidence in them.

[11]C.A. Johnson and W.T. Tucker, "Advanced Statistical Concepts" in *ASM Engineered Materials Handbook, Vol. 4: Ceramics and Glasses, 1987.*

You can get an estimate of the standard deviation of the *slope* from the residual sum of squares, following a procedure from Rektorys[12]**.** To fit a set of N data points (y_i,x_i) with a linear equation of the form $y = a + bx$, one can estimate the intercept by:

$$a = \frac{1}{N}\left(\sum_{i=1}^{N} y_i\right) - b\frac{1}{N}\left(\sum_{i=1}^{N} x_i\right)$$

and the slope by:

$$b = \frac{\sum_{i=1}^{N} x_i y_i - \frac{\left(\sum_{i=1}^{N} x_i\right)\left(\sum_{i=1}^{N} y_i\right)}{N}}{\sum_{i=1}^{N} x_i^2 - \frac{1}{N}\left(\sum_{i=1}^{N} x_i\right)^2}$$

An estimate of the standard deviation of the intercept (s_a) and the standard deviation of the slope (s_b) is obtained from the "residual sum of squares" Σ_o, with:

$$s_a = \sqrt{S_o/(N-2)}\sqrt{\frac{\frac{1}{N}\left(\sum_{i=1}^{N} x_i\right)^2}{\sum_{i=1}^{N} x_i^2 - \frac{1}{N}\left(\sum_{i=1}^{N} x_i\right)^2}}$$

$$s_b = \frac{\sqrt{S_o/(N-2)}}{\sqrt{\sum_{i=1}^{N} x_i^2 - \frac{1}{N}\left(\sum_{i=1}^{N} x_i\right)^2}}$$

with the residual sum of squares from:

$$S_o = \sum_{i=1}^{N} y_i^2 - \frac{\left(\sum_{i=1}^{N} y_i\right)^2}{N} - b\left\{\sum_{i=1}^{N} x_i y_i - \frac{\left(\sum_{i=1}^{N} x_i\right)\left(\sum_{i=1}^{N} y_i\right)}{N}\right\}$$

The probable error on the slope is 0.674 s_b. It is useful to calculate the probable error on your slope (Weibull 'm'), and report the Weibull moduli as "m +/- the probable error".

Indentation Fracture Toughness

Refer to Amin's article "Toughness, Hardness, and Wear" from the *ASM Engineered Materials Handbook, Vol. 4: Ceramics and Glasses, 1987.* This article reviews many fracture

[12]K. Rektorys, *Survey of Applicable Mathematics*, MIT press 1969, page 1291. This is in the Media Union Library QA 37.R373 1969.

toughness techniques, including the widely used indentation fracture technique pioneered by investigators such as Anthony Evans, Brian Lawn, and David Marshall. Lawn's text, *"Fracture of Brittle Materials"* (second edition), Cambridge University Press, 1993 [TA409.L.37 1993] is an excellent reference for these methods and their analysis.

Fractography

Refer to Michalske's article "Quantitative Fracture Surface Analysis" from the *ASM Engineered Materials Handbook, Vol. 4: Ceramics and Glasses, 1987*. There is also a brief discussion in the article by Amin entitled "Toughness, Hardness, and Wear".