Data Wrangling with Carbon Fiber Mechanical Properties

A Computer Project
to Perform Statistical and Numerical Analysis
for Materials Properties
in Large Data Sets

Applied Statistics and Numerical Methods for Materials Scientists & Engineers
MSE 301
Department of Materials Science & Engineering
University of Tennessee
Knoxville, TN

Project Designed by
Prof. David Keffer & Prof. David Harper
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I. Objective

Engineering Objectives:

Investigate the mechanical properties of single carbon fibers using tensile testing.

Computational Objectives:

Gain experience and develop skills for analyzing large data sets where automation of analysis is essential. Perform regression, generate sample means and sample variances and confidence intervals on both for such properties as Young's modulus, ultimate tensile strength and strain at fracture.

II. Background: Tensile Testing

We provide a quick summary of tensile testing to put this data in context. Tensile testing is a materials characterization technique that can be used to quantify mechanical properties. Typical results for polymeric materials are shown in Figure 1 for three polymers: ABS, HIPS and PLA.

Acrylonitrile butadiene styrene (ABS)	High Impact Polystyrene (HIPS)	Polylactic Acid (PLA)

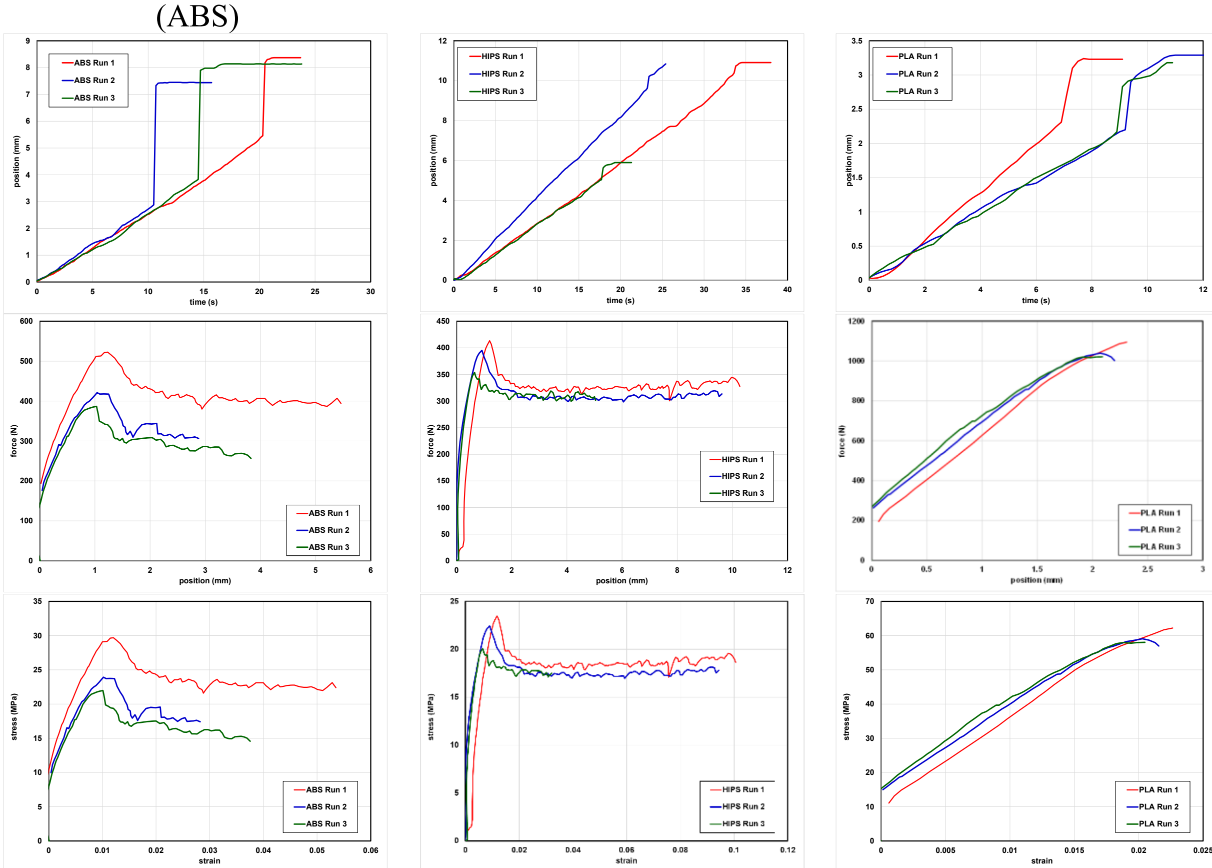

Figure 1. Tensile testing results for three different polymers. Top: position as a function of time. Middle: force as a function of position. Bottom: stress as a function of strain. For each polymer, the tensile test was run in triplicate.

In Figure 1 (top), we observe the stretching of the sample in response to an applied load in the plot of the position as a function of time. The applied force can be plotted as a function of position, as shown in Figure 1 (middle). If we divide the force by the cross-sectional area of the sample and divide the distance by the original length of the sample, we obtain a stress vs. strain curve, as shown in Figure 1 (bottom).

Two things are immediately evident from Figure 1. First, different polymers behave differently in the tensile test experiment. Second, there is variation within the same polymer from one replicate to the next.

In Figure 2, we illustrate how material properties can be extracted from the stress vs strain curve. The ultimate tensile strength corresponds to a maximum in the stress. The strain at fraction corresponds to the strain where the sample failed. The elastic modulus corresponds to the slope of the of the stress vs strain curve in the initial linear region.

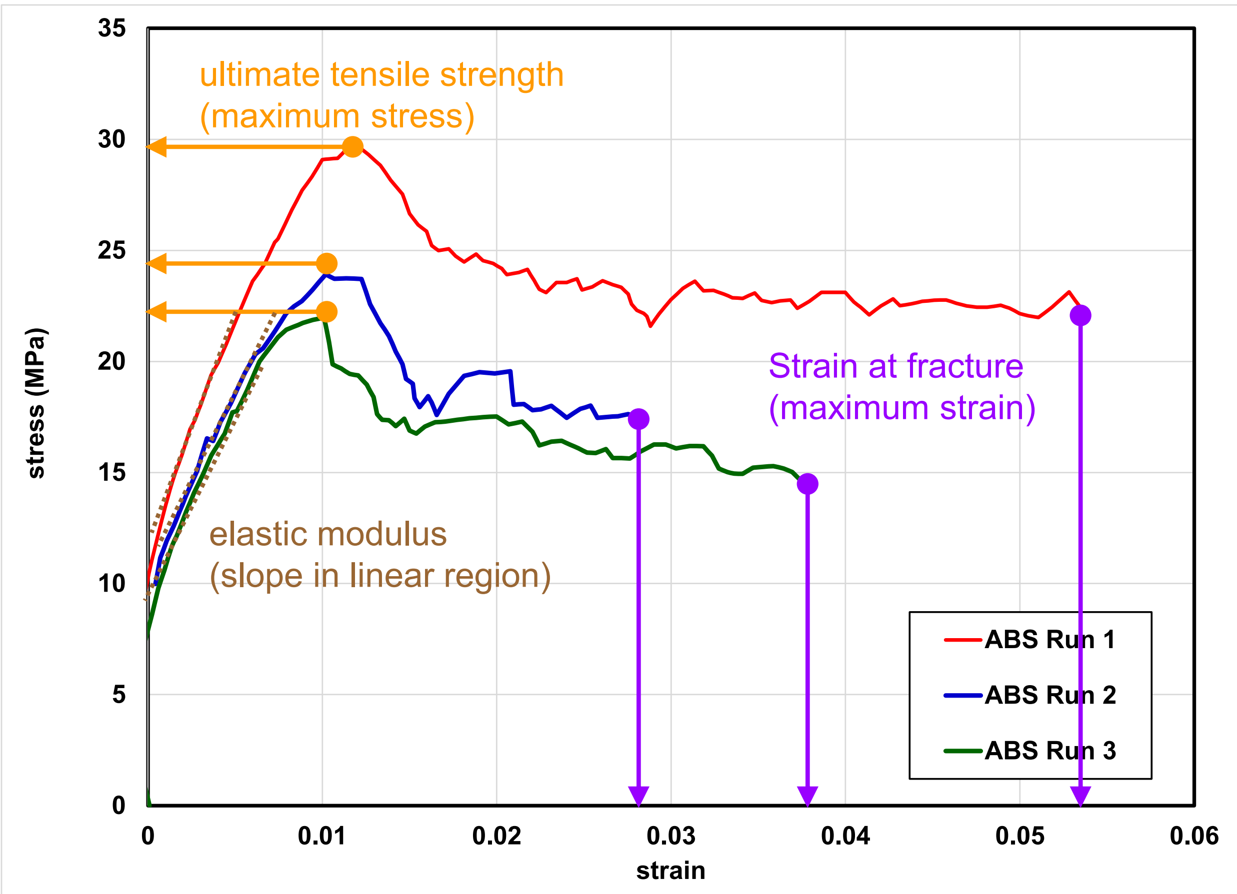

Figure 2. Mechanical properties, such as the ultimate tensile strength, maximum strain at fracture and elastic modulus, can be extracted from the tensile test experiment.

III. Tensile Testing of Single Carbon Fibers

Carbon fiber from 100% biomass lignin has been produced at the Center for Renewable Carbon at the University of Tennessee Institute of Agriculture. A method was developed where lignocellulosic feedstock goes through an organosolv fractionation process to deliver a lignin with high purity[1]. Many challenges still exist with the development of carbon fibers from lignin. One of the problems is the poor mechanical properties compared to those of from polyacrylonitrile (PAN). Conversely, many new opportunities exist for carbon materials where lignin may compete. New applications need to take advantage of the unique potential of lignin on the basis of its low-cost, large potential volume, renewable nature, partially oxidized state, amorphous structure and ease of controllable graphitization. However, the source of the lignin, extraction process, and processing conditions lead large differences in fiber properties.[2-4]

Lignin melt spinning was performed using a Haake MiniLab counter-rotating twin-screw extruder (Thermo Scientific, Watertown, WI). The extruder had been modified to have a 200 μm spinneret assembly with a heating band. The temperature of the extruder and spinneret was optimized for each lignin sample and an aluminum-rotating cylinder connected to a variable speed motor was used to collect monofilament fibers. For multifilament extrusion, a custom Alex James single screw extruder (0.5 in. 20:1) with gear pump (Zenith 0.297 cc/rev) was used to feed a 12 hole 150 μm spinneret. Fibers were collected on a Leesona winder at 100 to 500 m/min. Oxidative thermostabilization process was performed by heating the samples to 250°C under air at selected rates (0.05, 0.1, 0.2, 0.5°C/min) and holding for 30 min at 250°C using a Heratherm OGH60 oven (Thermo Scientific, Watertown, WI). The stabilized fibers were carbonized in a Lindberg/Blue M tube furnace (Thermo Scientific, Watertown, WI) by heating from room temperature to 600°C at rate of 3°C/min, holding for 5 min at 600°C, heating from 600 to 1000°C at a rate of 5°C/min and holding for 15 min at 1000°C under nitrogen.

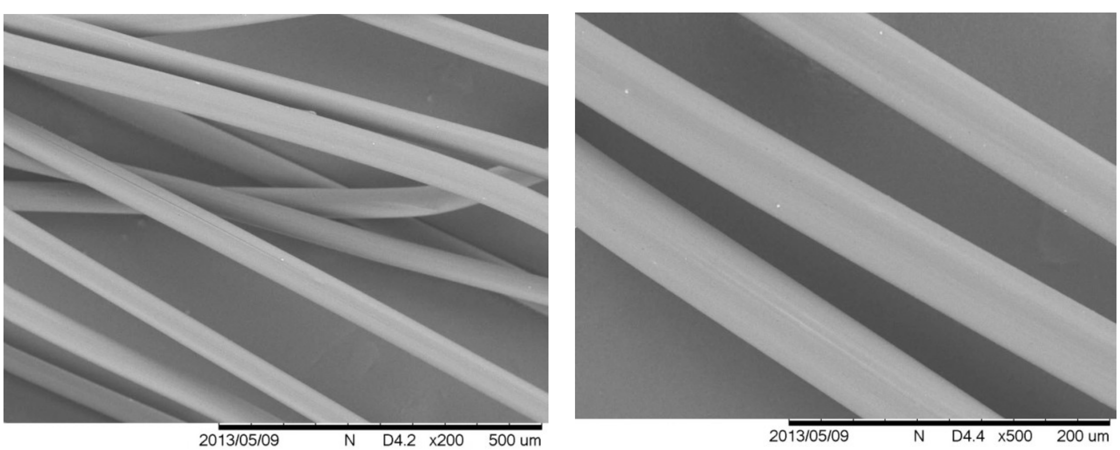

Figure 3 - Carbon fibers from extracted organosolv switchgrass.

The tensile properties of carbon fibers were measured according to the ASTM standard (ASTM C1557-03) using an Instron 5943 single column tabletop testing system. Tensile testing of single carbon fibers is subject to variability between one fiber and the next. Four examples of such variability are shown in Figure 4. In each case, there are 35 to 40 replicates. While there is an underlying average behavior apparent to the eye, there are also a number of obvious outliers.

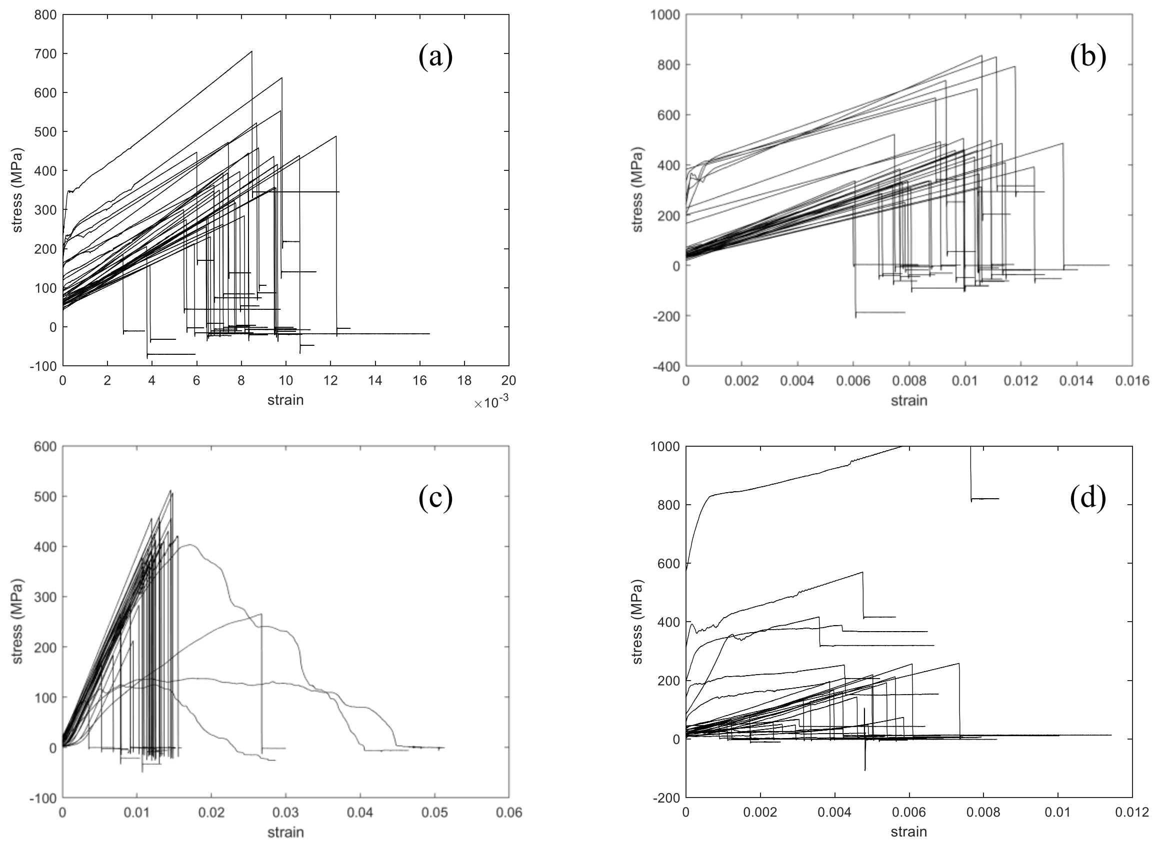

Figure 4. Four sets of tensile testing results for single carbon fibers. Each set contains 35 to 40 replicates. (a) This data set is perhaps the cleanest. It demonstrates the variation at strain at fracture. There is a significant variation in the value of stress at which the linear region begins. (b) This data set is also relatively clean although there is a separate population of sample where the y-position of the linear region is elevated. (c) This data set shows four outliers that had different mechanical behavior that allowed them to reach higher strain. (d) This data set shows outliers that experienced a different kind of initial deformation before reaching a linear regime at high stress.

IV. Statistical and Numerical Challenges

In this project, there are a number of statistical and numerical challenges.

- Computational challenge: automate a data analysis process in Matlab to investigate a large set of data files.
- Numerical Challenge: identify features in experimental data sets and extract slopes and optima.
- Statistics Challenge: determine sample mean and confidence intervals of elastic module, ultimate tensile strength and strain at fracture
- Engineering Challenge: evaluate statistical data to make engineering decisions

V. Assignment

Task 1. Design and implement a process in Matlab to take a single experimental data set and compute the elastic modulus, ultimate tensile strength and strain at fracture. Describe how you determined the start of the linear region, the end of the linear region, the maximum in stress and the strain at fracture.

Task 2. Apply this code to all of the experimental data.

- For each replicate within a data set of similar carbon fibers,
  - Identify beginning and end of linear region.
  - Perform linear regression on the linear region and obtain the elastic modulus.
  - Identify ultimate tensile strength
  - Identify strain at fracture

Task 3. Calculate statistics for each data set.

- For each set of carbon fibers,
  - Identify and exclude outliers.
  - determine sample mean of elastic module, ultimate tensile strength and strain at fracture
  - determine sample variance of elastic module, ultimate tensile strength and strain at fracture
  - determine 95% confidence interval on the sample mean of elastic module, ultimate tensile strength and strain at fracture
  - determine 95% confidence interval on the sample variance of elastic module, ultimate tensile strength and strain at fracture

Task 4. Compare across data sets.

- For each group of sets,
  - rank data sets in order of decreasing elastic modulus
  - rank data sets in order of decreasing ultimate tensile strength
  - rank data sets in order of decreasing strain at fracture
  - compute correlation coefficient between elastic modulus and ultimate tensile strength
  - compute correlation coefficient between elastic modulus and strain at fracture
  - compute correlation coefficient between ultimate tensile strength and strain at fracture

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References

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3. Meek, N.; Penumadu, D.; Hosseinaei, O.; Harper, D.; Young, S.; Rials, T., Synthesis and characterization of lignin carbon fiber and composites. *Composites Science and Technology* **2016,** *137*, 60-68.
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Appendix A. Matlab Code to Read csv data files

```
clc;
clear all;
close all;
warning('off');

% number of data sets
ncase = 6; %

% data directory base name
data_dir_base = 'data_set_';
% data file base name
data_file_base = 'Specimen_RawData_';
% top directory is where this driver script exists
topdir = fileparts(mfilename('fullpath'));
%
%colstrain = column number with strain data
colstrain = 4;
%colstress = column number with stress data
colstress = 5;

% nrep = number of replicates in each case
nrep = zeros(ncase,1);

% figure counter
figcount = 0;
%
% loop over data sets
%
for ic = 1:1:ncase
    % determine number of replicates based on number of files in directory
    dirname = strcat(data_dir_base,num2str(ic));
    d = dir([dirname, '/*.csv']);
    nrep(ic) = length(d);
%
% loop over replicates
%
    figcount = figcount + 1;
    figure(figcount);
    for ir = 1:1:nrep(ic)
%
%   open file
%
    dirfilename = strcat(data_dir_base,num2str(ic),'/',data_file_base,num2str(ir),'.csv');
    tempdata = readtable(dirfilename);
    nrow = height(tempdata);
    fprintf(1,'case %i, rep %i, file %s has %i rows of data.\n', ic, ir, dirfilename, nrow);
%
%   extract stress and strain
%
    strain = zeros(nrow,1);
    stress = zeros(nrow,1);
    strain = tempdata{1:nrow,colstrain};
    stress = tempdata{1:nrow,colstress};
    fprintf(1,'case %i, rep %i, file %s has strain from %e to %e & stress from %e to %e.\n', ...
    ic, ir, dirfilename, strain(1), strain(nrow), stress(1), stress(nrow));
    plot(strain,stress,'k-');
    hold on;
    end
    ylabel('stress (MPa)');
    xlabel('strain');
end
```