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Fracture Toughness of Electrospun Fibrous Mats

Annabella Mascot

Washington University in St. Louis

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Washington University in St. Louis

JAMES MCKELVEY SCHOOL OF ENGINEERING

Fall 2022 MEMS 400 Independent Study

Topic: Fracture Toughness of Electrospun Fibrous Mats

Researcher: Annie Mascot

PI: Dr. Michelle Oyen

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Abstract

Fiber reinforced composite hydrogels have become a popular material used in tissue engineering in recent years due to their robust nature under tension and biocompatibility qualities [1]. However, little is known of the fracture mechanics of the fiber reinforcements themselves (which are typically created of Electrospun materials, a process in which a high voltage differential is applied to a polymer solution, creating a jet of nanofibers that collect into a mat-like material) [2]. This study provides an analysis of the fracture toughness of two Electrospun materials: one created from a 100 bloom strength gelatin solution, and one created from a 300 bloom strength gelatin solution. It is found that the fracture toughness increases as the rate of extension increases during standard tear testing in both materials, and the fracture toughness of the 300 bloom strength material is greater than that of the 100 bloom strength material. Further experimentation is needed to confirm the results of this preliminary data.

Introduction

Chorioamnion (CA) membrane within the human uterus requires significant mechanical forces to rupture during human birth, which is a physiologically necessary outcome [3]. However, if rupture of these membranes happens prior to full-term gestation, it is associated with significant fetal and maternal morbidity [4]. Physiological rupture at birth is a process where the toughness of the material is vital, however poorly quantified and understood. The amnion layer of the CA is composed primarily of fibrillar collagen-I, and this fibril structure is associated with the significant toughness of the tissue and provides a significant amount of the soft biological tissue's mechanical integrity [5], [6].

This paper focuses on the linear elastic fracture mechanics of biomimetic nanofibrous materials, which describes the toughness of a material in terms of a critical strain energy release

rate (G_c) where crack propagation begins under the influence of applied stress [7]. Furthermore, the value G_c refers to the balance of strain energy release from the specimen and the energy required to create new fracture surfaces as the crack begins to propagate. It has been shown that a tear test can be used to measure the critical strain energy release rate of materials such as rubbers (which are non-linear elastic materials) [8]. The specimen geometry of a tear test is shown in Figure 1.

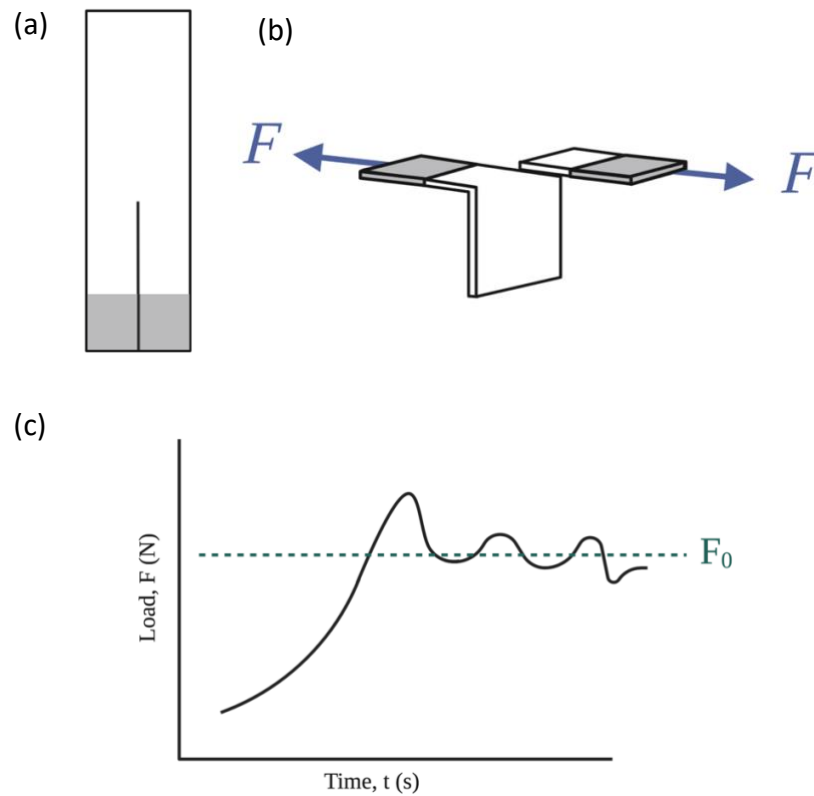


Figure 1: (a) Unloaded specimen geometry (b) Tear test under load (c) Example time-load curve

Using a tear test, the critical strain energy release rate for a non-linear elastic material can be calculated by eqn 1.

$$T_{ch} = \frac{W_0 A_0 - 2\lambda F_0}{t_h} \quad (1)$$

This equation is explained in terms of the variable t_{ch} to distinguish it from G_c which is calculated by other means. In eqn 1, t_h is the specimen thickness, F_0 is the plateau tearing force, λ is the extension ratio of the specimen at F_0 , W_0 is the strain energy density of the specimen at F_0 , and A_0 is the cross-sectional area of the specimen. The stress in the specimen is considered low, meaning the total deformation in the specimen is approximately equivalent to the total length of crack propagation, which allows for the approximation of $\lambda \cong 1$ and $W_0 \cong 0$, thus a reasonable approximation of eqn 1 is shown in eqn 2.

$$|T_{ch}| = \frac{2F_0}{t_h} \quad (2)$$

Methods

Material Preparation

Two polymer solutions were created using 12% wt gelatin of 80-120 and 300 bloom strength (Sigma-Aldrich, St. Louis, MO) dissolved in 90% wt aqueous acetic acid (Sigma-Aldrich). These solutions were stirred at room temperature for 2-3 hours until homogenous. All solutions were used within 7 days of their creation. Electrospun fibrous mats were created using a NANON-01A (Mecc Co, Fukuda, Japan) electrospinner. All fibers were spun using a voltage differential of 10 kV and a solution feed rate of 0.3 mL/h. Fibers were left to spin for a duration of four hours before being stored in a desiccator with desiccant (Sigma-Aldrich) for 24 hours to completely dry.

Mechanical Characterization

Once dry, fibrous mats were removed from their aluminum foil backing and cut to a dimension of 25 mm x 125 mm using sharp scissors. A slit was then cut up the length of the to approximately half their length to complete trouser tear testing. Samples then underwent trouser tear testing at three rates of applied displacement using an ElectroForce 3200 (TA Instruments,

New Castle, DE), standard manufactured tensile grips, and 45 N load cell to determine the effect extension rate has on the critical strain energy release rate of the material. Figure 1a shows the dimensions of the samples, with the indicated region showing where on the specimen the grips are placed. Specimens underwent tensile load according to the mode of fracture depicted in Figure 1b.

The rates of displacement used were 0.005 mm/min, 0.158 mm/min, and 0.5 mm/min. Four samples of each type at each rate of displacement were used. Samples were left to tear until displacement had reached 6 mm, or until sufficient time in the tearing region was reached (at least 20 minutes). The dimensions of the thickness of the sample (t_h), length of slit before tearing (L_0), and length of slit after tearing (L_f), were collected in this process. After tearing occurred, the subsequential load was averaged and was classified as F_0 . The test was repeated with standard sticky-note paper (without tacky backing) to compare samples to a known material.

The critical strain energy release rate of each sample was calculated, and simple statistics were performed to determine the mean and standard deviation of critical strain energy release rate of each sample at each rate of extension.

Results/Discussion

Figure 2 below shows the relationship between the rate of extension and calculated toughness of 300 bloom strength Electrospun fibrous mats.

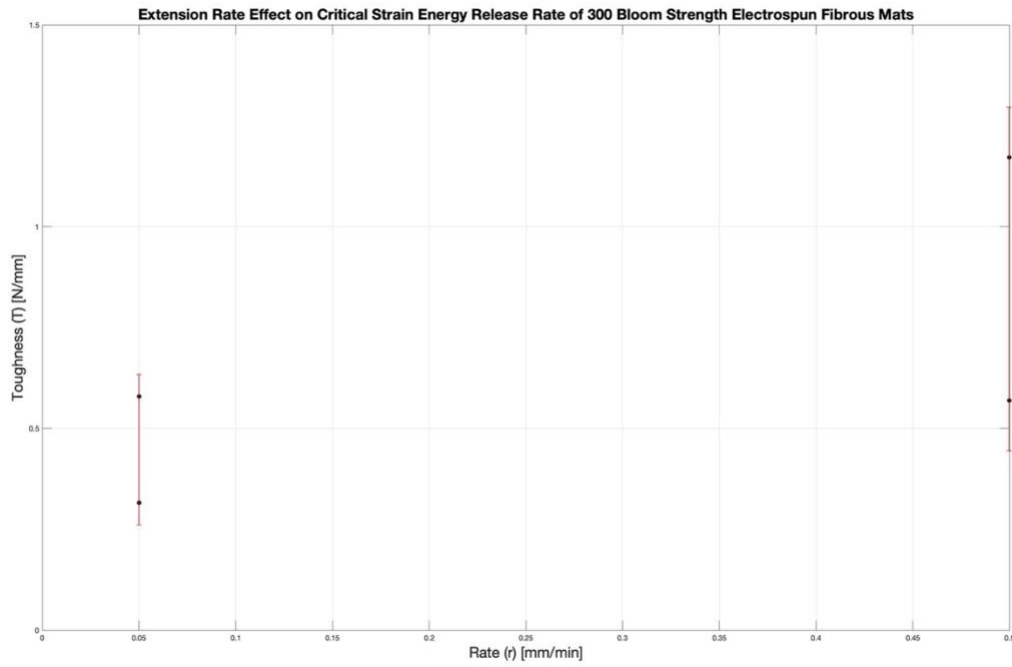


Figure 2: Extension are and toughness of 300 bloom electrospun fibrous mats

As shown in Figure 2, the material displays greater toughness at higher rates of displacement than it does at lower rates of displacement.

Figure 3 shows the relationship between the rate and toughness of 100 bloom strength Electrospun fibrous mats.

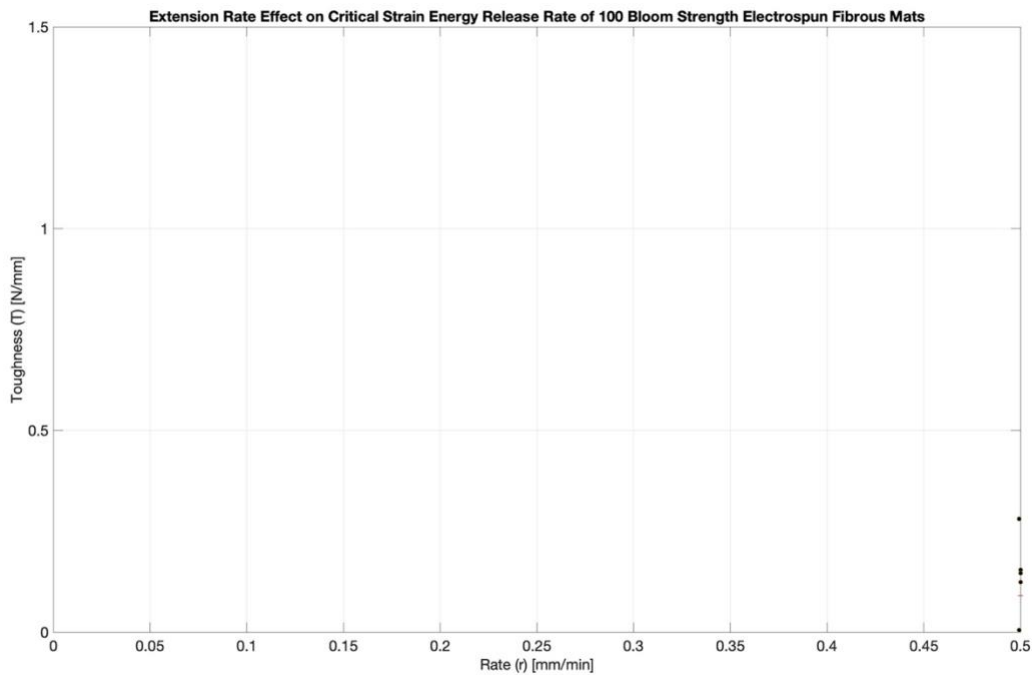


Figure 3: Extension rate and toughness of 100 bloom strength Electrospun fibrous mats

At the same rate of displacement of 0.5 mm/min, the 100 bloom strength fibrous mats show significantly lower toughness than their 300 bloom strength counterparts. This is expected based on preliminary experimentation by Ludwick [9].

While this shows preliminary results of experimentation, there are not enough data points to confirm this conclusion. In mid-October the Oyen Lab moved locations where the NANON-01A electrospinner was also moved. However, upon inspection by the Environmental Health and Safety administration on the WashU campus, there were safety precautions not met by the electrospinner in its new location. Thus, new samples could not be made for the remainder of the semester, as these protocols had to be met. Thus, this research will continue into next semester, and complete all necessary data collection to confirm the preliminary results of this experimentation.

Figure 4 shows the effect of extension rate on the toughness of manufactured sticky-note paper (without the tacky backing).

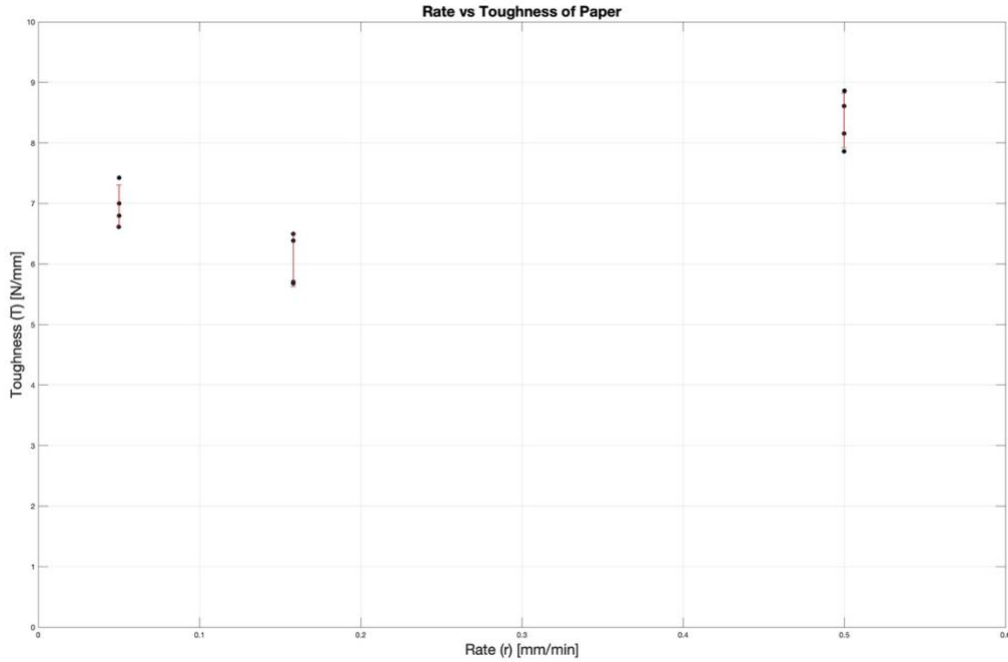


Figure 4: Extension rate and toughness of paper

There were no limitations in curating samples for this experimentation, so the three rates of displacement: 0.05 mm/min, 0.158 mm/min, and 0.5 mm/min are represented. There is no significant difference shown between the rates of displacement and the calculated toughness of paper, which is expected, as paper is not a material that undergoes time-dependent relaxation under tension. The results of this experimentation provide a control that is useful in characterizing the calculated toughness of electrospun fibrous mats.

Appendix A shows sample raw data of the load-time curves of each sample reported in this study, and Appendix B shows the MATLAB code used for analysis of this experimentation.

Conclusion

It is preliminarily shown that there is a time-dependent factor in the toughness of 100 bloom strength and 300 bloom strength Electrospun fibrous mats when undergoing tension, as there is a discrete increase in toughness values of each material as its extension rate of tension increases. Furthermore, the critical strain energy release rate (our toughness) of 300 bloom strength electrospun material is greater than that of 100 bloom strength electrospun material when undergoing extension at the same rate. However, more experimentation is needed with a higher sample volume to confirm the results of this experimentation. Furthermore, there is no significant difference in the fracture toughness of paper as its extension rate increases when undergoing tear testing. Thus, confirming that paper does not display rate-dependent properties when undergoing tension.

References

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- [3] Oyen, Michelle and S.E., Calvin, “Thickness effects on fracture properties of the placental membranes,” *J. Biomech.*, 2006, doi: 10.1016/S0021-9290(06)84358-7.
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- [5] Koh, Won-Gun, “Recent Developments in Nanofiber Fabrication and Modification for Bone Tissue Engineering,” *Int. J. Mol. Sci.*, 2019, doi: 10.3390/ijms21010099.
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- [8] Rivlin and Thomas, “Rupture of rubber. I. Characteristic energy for tearing,” *J. Polym. Sci.*, 1953, doi: 10.1002/pol.1953.120100303.
- [9] Ludwick, Jacob, “Fabrication and Characterization of a Hydrogel-Nanofiber Composite for Cartilage Replacement,” East Carolina University, 2020.

Appendix A: Sample Load-Time Curves

Figures 5, 6, and 7 show samples of raw load-time curves of 100 bloom strength samples, 300 bloom strength samples, and paper samples, respectively.

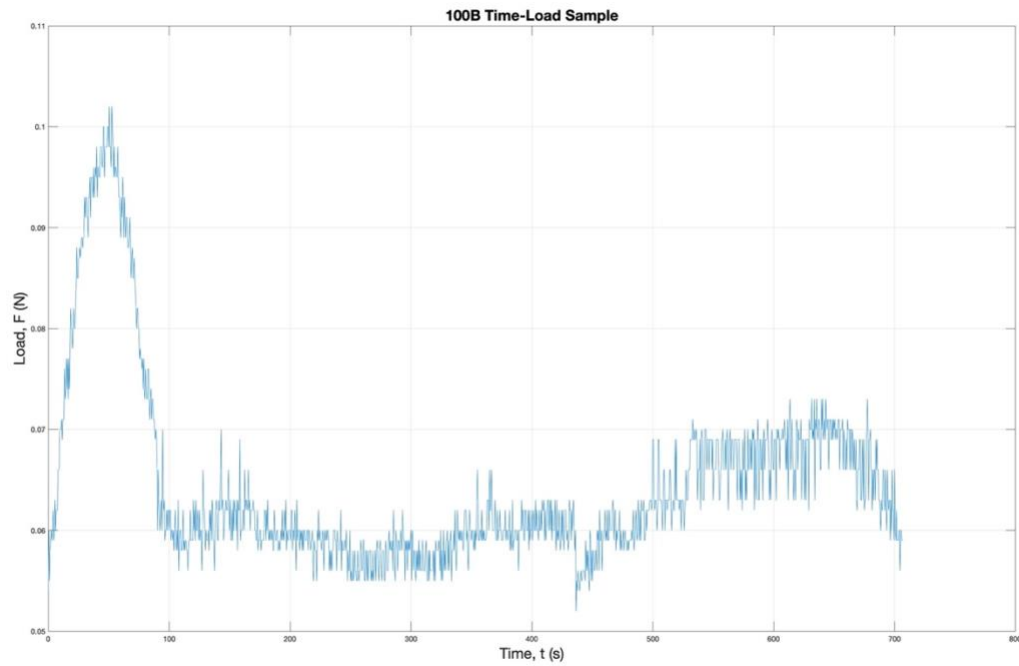


Figure 5: Sample load-time curve of 100 bloom strength Electrospun fibrous mat undergoing tear test

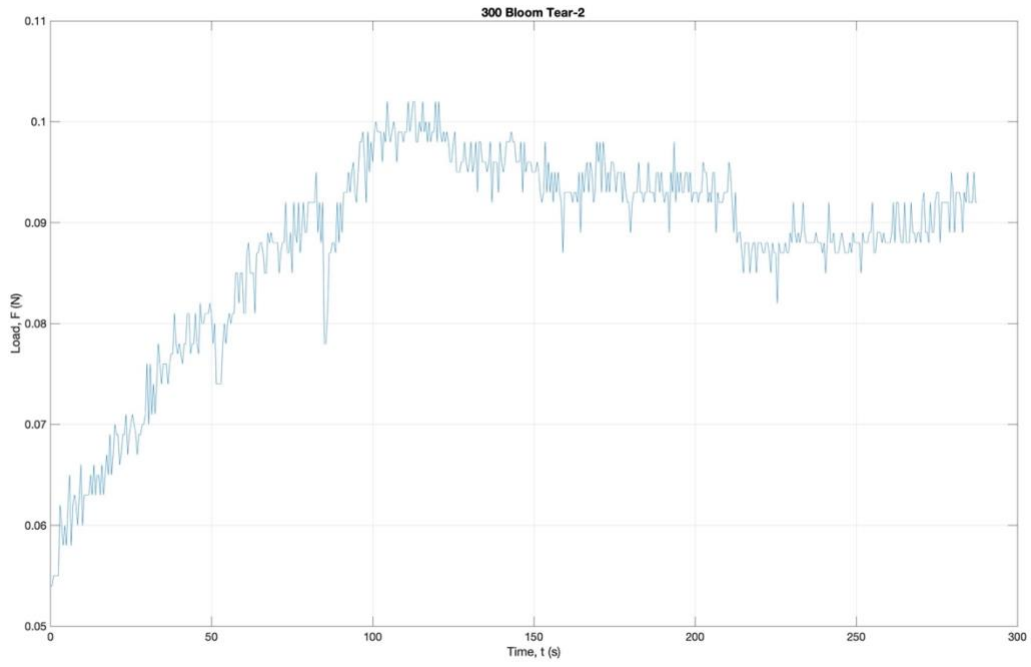


Figure 5: Sample load-time curve of 300 bloom strength Electrospun fibrous mat undergoing tear test

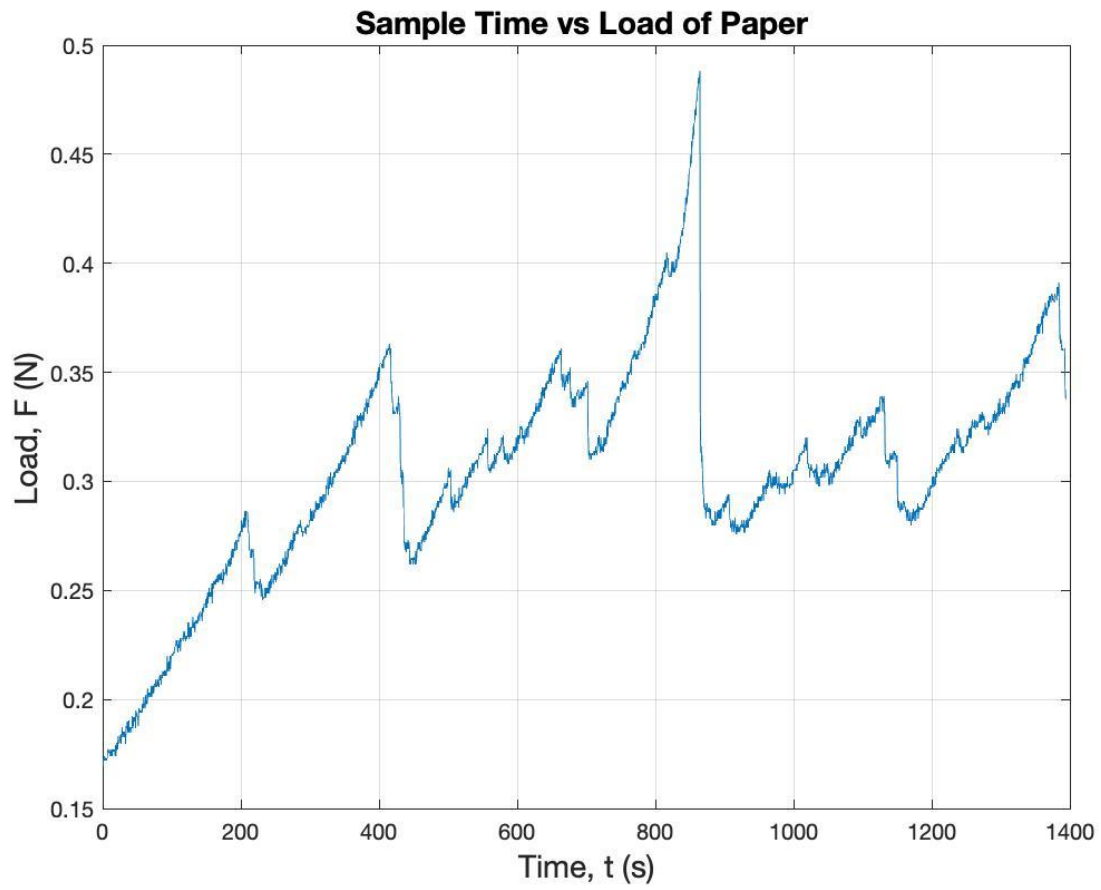


Figure 5: Sample load-time curve of paper undergoing tear test

Appendix B: MATLAB Code

```
% Tear Testing Analysis
% Created by Annie Mascot, WashU (July 2022)

% insert file directory

clear all
clc

fnam01= '/Users/anniemascot/Desktop/100B';

cd(fnam01);
d = dir;
datadir = fnam01;
k = length(dir);
klen = k-3;
leg = strings(klen,1);
% insert measured thickness of sample in mm
% thick_measure = [1.24, 1.19, 0.95]/1000; %[mm]
% thickness = mean(thick_measure);
% Lo = 23.72; %[mm]
% Lf = 28.35; %[mm]

% LoMap = containers.Map({'300B1-1.CSV', '300B1-2.CSV', '300B2-1.CSV',
'300B2-2.CSV'},...
%     {40, 35, 20, 20});
%
% LfMap = containers.Map({'300B1-1.CSV', '300B1-2.CSV', '300B2-1.CSV',
'300B2-2.CSV'},...
%     {42, 37, 21, 22});
%
% thickMap = containers.Map({'300B1-1.CSV', '300B1-2.CSV', '300B2-1.CSV',
'300B2-2.CSV'},...
%     {.15, .27, .19, .19});

LoMap = containers.Map({'100B1-1.CSV', '100B1-2.CSV', '100B1-3.CSV', '100B2-
1.CSV'...
'100B2-2.CSV', '100B2-3.CSV'},...
{33, 17, 21, 30, 28, 19});

LfMap = containers.Map({'100B1-1.CSV', '100B1-2.CSV', '100B1-3.CSV', '100B2-
1.CSV'...
'100B2-2.CSV', '100B2-3.CSV'},...
{35, 21, 39, 35, 31, 22});

thickMap = containers.Map({'100B1-1.CSV', '100B1-2.CSV', '100B1-
3.CSV', '100B2-1.CSV'...
'100B2-2.CSV', '100B2-3.CSV'},...
{.46, .34, .29, .33, .28, .16});

for i = 1:klen
    ii = i+3;
```



```

testname = d(ii).name;
num2str = string(i);
Lo = LoMap(testname);
Lf = LfMap(testname);
thick = thickMap(testname);
leg(i) = num2str + ': ' + testname; % create legend of all tests
% read in datafile
DT = readtable(testname);
% place table variables into a single array called DT
A = DT.Var2; % time
B = DT.Var3; % displacement
C = DT.Var4; % load
DT = [A, B, C];
% Need just first three column data
DT=[DT(:,1) DT(:,2) DT(:,3)]; % FORMAT- Time(s) Load(N) Displacement(mm)
% label what columns of data represent
time = DT(:,1);
time = time(1:50:end);
load = DT(:,3);
load = load(1:50:end);
disp = DT(:,2);
disp = disp(1:50:end);
r = (disp(end)-disp(1))/(time(end)-time(1));
% initial displacement
do = disp(1);

% find strain
%   for j=1:length(load)
%       extension(j) = disp(j)-do;
%       strain(j) = (extension(j)-do)/do;
%   end

% find significant drop in force. This is variable depending on sample
% type
for j=1:length(time)-1
    slope(j) = (load(j+1)-load(j))/(time(j+1)-time(j));
    tear = find(slope <= 0);
end

% create an array of the tear loading thresholds then average them to
find
% average plateau load
tearload = load(tear(1):end);
tearloadavg(i) = mean(tearload);
teartime = time(tear(1):end);

[maxload(i), indexmax] = max(tearload);
maxtime(i) = teartime(indexmax);

teartime_tot = teartime(end)-teartime(1);
crackvel = (Lf-Lo)/teartime_tot;

T(i) = (2*tearloadavg(i))/thick;

```

```

toughness(i) = T(i);
rate(i) = r;

%     figure(i)
%     plot(time, load)
%     title(testname)
%     xlabel('Time, t (s)')
%     ylabel('Load, P (N)')
%     dim = [.2 .5 .3 .3];
%     str = string(T);
%     str = strcat('Tear Toughness, T = ', ' ', str, ' N/m');
%     annotation('textbox',dim,'String',str,'FitBoxToText','on');

figure(i)
plot(time, load)
xlabel('Time, t (s)', 'FontSize', 14)
ylabel('Load, P (N)', 'FontSize', 14)
title(testname, 'FontSize', 14)
grid on

%     figure(100)
%     plot(time, load)
%     title('Comparison of Tear Behavior of Paper, 300 Bloom Strength, and
100 Bloom Strength Nanofibers', 'FontSize', 28)
%     xlabel('Time, t (s)', 'FontSize', 14)
%     ylabel('Load, P (N)', 'FontSize', 14)
%     hold on
%     grid on
%     legend('300 Bloom', 'Paper', '100 Bloom', 'FontSize', 14)
%     dim = [.2 .5 .3 .3];
%     str = string(T);
%     str = strcat('Tear Toughness, T = ', ' ', str, ' N/m');
%     annotation('textbox',dim,'String',str,'FitBoxToText','on');
%
%
%     figure(2)
%     plot(extension, load);
%     title(testname)
%     xlabel('Extension, (mm)')
%     ylabel('Load, P (N)')

%     if ~exist(fullfile(datadir,'Tear_Plots'))
%         mkdir (fullfile(datadir,'Tear_Plots'))
%     end
%     h=findobj('type','figure'); % find the handles of the opened
figures
%     plotfolder=[datadir '/Tear_Plots']; % Desination folder
%     for k=1:numel(h)
%         plotfilename=sprintf(strcat(testname,'plot.pdf'),k);
%         plotfile=fullfile(plotfolder,plotfilename);
%         saveas(h(k),plotfile)
%     end

```

```

        %     saveas(h(1),fullfile(plotfolder, 'All.fig'))
        %     cd(fnam01)
%     load_table(:,i) = load;
%     time_table(:,1) = time;
end

% simple statistics
k = find(rate>0.008);
for i=1:length(k)
    rate1(i)=rate(k(i));
    tough1(i)=toughness(k(i));
end
%
% k = find(rate > 0.002 & rate < 0.003);
% for i=1:length(k)
%     rate2(i)=rate(k(i));
%     tough2(i)=toughness(k(i));
% end
%
k = find(rate<0.0009);
for i=1:length(k)
    rate3(i)=rate(k(i));
    tough3(i)=toughness(k(i));
end
% %
mean1 = mean(tough1);
% mean2 = mean(tough2);
mean3 = mean(tough3);
rate1 = rate1(1)*60;
% rate2 = rate2(1)*60;
rate3 = rate3(1)*60;
std1 = std(tough1);
% std2 = std(tough2);
std3 = std(tough3);
%
% avgs = [mean1, mean2, mean3];
% stds = [std1, std2, std3];
% rates = [rate1, rate2, rate3];

% figure(100)
% hold on
% plot(maxtime, maxload, 'ko', 'MarkerFaceColor', 'k')
% legend('300 Bloom', 'Paper', '100 Bloom', 'Maximum Load After Tear',
'FontSize', 28)
% tough_100 = [toughness(1),toughness(2),toughness(3)];
% tough_300 = [toughness(4),toughness(5)];
% rate_100 = [rate(1), rate(2), rate(3)];
% rate_300 = [rate(4), rate(5)];
%

hold off
figure(100)
set(gca, 'FontSize', 20)
lograte = logspace(-1, 2, length(toughness));
logtough = logspace(-1, 2, length(toughness));

```

```

plot(rate.*60, toughness, 'o','MarkerFaceColor','k')
hold on
% errorbar(rate1, mean1, std1,'LineWidth',1, 'Color', 'r');
% errorbar(rate2, mean2, std2,'LineWidth',1, 'Color', 'r');
errorbar(0.5, mean3, std3,'LineWidth',1, 'Color', 'r');
grid on
title('Extension Rate Effect on Critical Strain Energy Release Rate of
Paper','FontSize', 20)
xlabel('Rate (r) [mm/min]','FontSize', 20)
ylabel('Toughness (Tch) [N/mm]','FontSize', 20)
ylim([0,1.5])
xlim([0,0.5])
% figure(i+2)
% plot(crackvel, toughness, 'o','MarkerFaceColor',[0 0.447 0.741])
% grid on
% title('Crack Velocity vs Toughness')
% xlabel('Crack Velocity (V) [mm/s]')
% ylabel('Toughness (T) [N/mm]')

% if ~exist(fullfile(datadir,'Tear_Plots'))
%     mkdir (fullfile(datadir,'Tear_Plots'))
% end

% h=findobj('type','figure'); % find the handles of the opened figures
% plotfolder=[datadir '/Tear_Plots']; % Desination folder
% plotfilename=sprintf(strcat('RatevsToughness','plot.pdf'),k);
% plotfile=fullfile(plotfolder,plotfilename);
% saveas(gcf,plotfile)

```