

First Progress Report for Experimental Study of Crackling Noise as Micro-mechanics of Flow

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1. Motivation and Overview of the Project

As being discussed in my proposal for the project, this project did nano-mechanical studies on monocrystalline copper for fundamentals of potential crackling noise in aLIGO suspension system¹. The method employed in this project is the energy dissipation approach. We are going to measure the elastic and loss moduli of copper nanopillars of various diameters using Dynamic Mechanical Analysis (DMA) tests. We are going to confirm former experimental result and extend to investigate the size effect to translate the microscopic observation to macroscopic applications.

Importantly, it was found in DMA tests conducted by my mentor that the loss moduli were not zero or even constant throughout the static elastic stress sweep^{1,3}. This behaviour is not expected for the perfectly elastic picture. It has been suspected that the observed anomaly is due to complex dislocation dynamics in metal pre-yield regime.

However, before DMA tests, we need to measure the Young's modulus and the yield strength as benchmark mechanical properties for certain copper nanopillar systems using uniaxial compression tests. The work done over the first three weeks has mainly been training for fabrication of copper nanopillars using a Focus Ion Beam (FIB) machine called FEI Versa and for uniaxial nano-compression tests using Hysitron.

2. Progress and Problems

So far I was trained on how to use FEI Versa for Focused Ion Beam (FIB) and Hysitron for nano-compression tests. I learned how FIB works and fabricated one batch of 500 nm Cu pillars using the templates created by my mentor. An image of one of such pillars is shown in figure 1. However for other potential pillar sizes in the future, I will need to design my own milling patterns. The original plan as proposed in the project proposal¹ is delayed by one week because FEI Versa was down for a week due to vacuum problem.

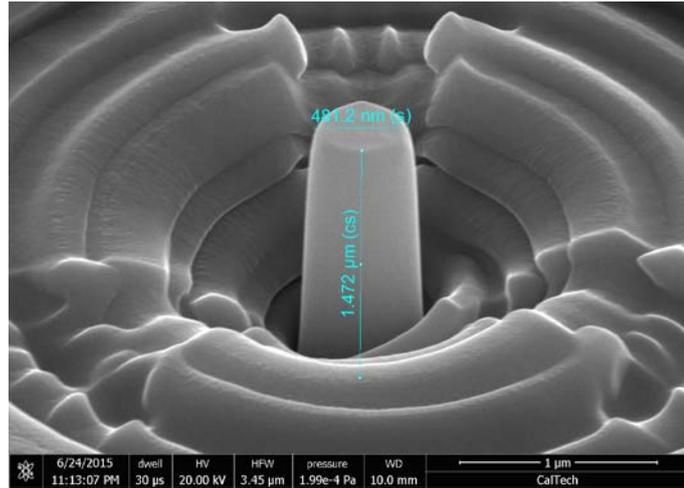


Figure 1: 500 nm copper nanopillar

For Hysitron, I learnt how to calibrate the tip to optical microscope, and do air-indentation calibration for the system. The tip used for uniaxial compression tests of copper nanopillars is an 8 μm flat diamond punch because it is substantially larger than contacting area for 500 nm diameter pillars. The machine performed nano-compression tests on the first 500 nm batch and produced satisfactory results that agreed with former literature values^{2,3}; yield strength of approximately 400 MPa and Young's modulus of approximately 140 GPa. However there was a contacting problem in the process of sample approach that led to a faulty detection of zero displacement as the sample surface. It is probably due to the dirt on the tip. We are going to try to clean the tip and solve the problem this week.

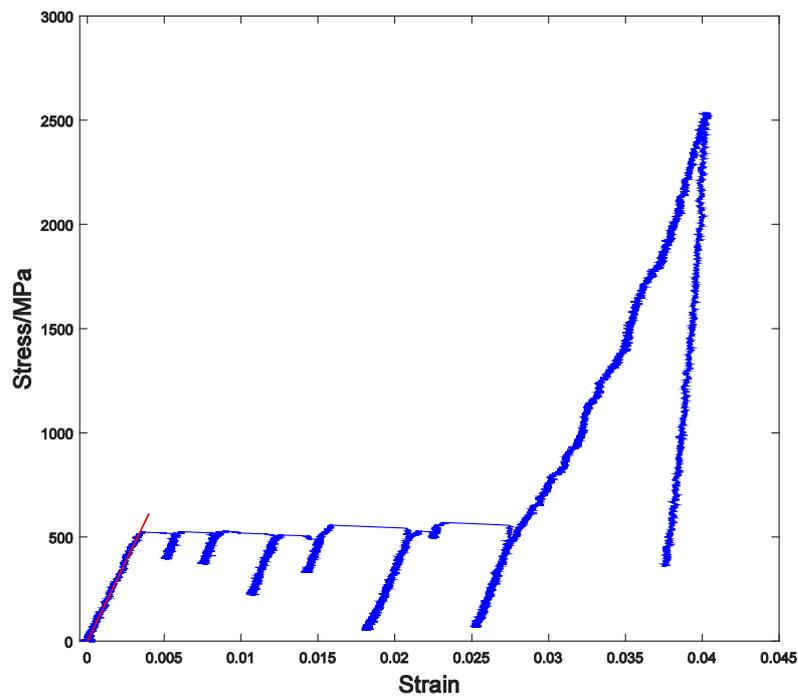


Figure 2: stress-strain curve for copper batch 1, pillar 2 (CuB1P2)

The data acquired from the compression test of each individual pillar is to be analyzed by a MatLab code to find Young's modulus and yield stress for each test. The code is given in the appendix of this report. Firstly, the raw data are in form of force and displacement. These need to be converted into stress and strain. Young's modulus can be found from the slope of the linear, elastic regime. The slope can be found by fitting a linear regression to the linear regime. Yield stress is the maximum value of stress of the whole elastic regime. The convention is 0.2% plastic strain⁴. The example stress-strain curve for batch 1, pillar 2 is shown in figure 2. The results for other pillars are given in table 1 below.

Table 1: data for copper batch 1

Pillar Code	Diameter/nm	Height/nm	Young's modulus/GPa	Yield strength/MPa
CuB1P2	481.2	1472	142.5682	316.5923
CuB1P3	467.7	1484	160.0466	369.1912

3. Future Plan

According to the schedule in the proposal¹, everything is delayed by one week due to the unexpected problem at FEI Versa. FEI Versa should be back to normal on Tuesday week 4 and I will fabricate copper nanopillars of diameters varying from 200 nm to 1 μ m according to the plan for week 5. Then we are going to apply both uniaxial compression tests and DMA tests on these pillars using Hysitron and the data are to be analysed.

Reference

1. Karava K., 2015. *Project Proposal for Experimental Study of Crackling Noise as Micro-mechanics of Flow*. LIGO
2. Greer J.R., De Hosson JTM., 2011. *Plasticity in Small-sized Metallic Systems: Intrinsic versus Extrinsic Size Effect*. Prog Mater Sci. doi:10.1016/j.pmatsci.2011.01.005
3. Ni X., et al., 2015. *What is Crackling Noise: a Study of Micro-mechanics of Flow in Metals (poster)*. LIGO
4. API SPEC 16A, *Specification for Drill-through Equipment*, Third Edition, June 2004

Appendix

MatLab code for function used for finding Young's modulus and yield strength

```
function [YoungGPa,YieldMPa] = sscon(forceuN,dispsnm,diameternm,heightnm)
% sscon converts force and displacement data into stress(MPa) and strain
% plots a stress-strain graph
% returns Young's modulus and yield strength

%Convert the raw data into stress and strain
stressori = 10^(-6).*(forceuN.*10^(-6))./(pi.*(10^(-9).*diameternm./2)^2);
strainori = dispsnm./heightnm;

%Correct the offset
%Extract only the linear regime (up to 0.15% strain)
%Locate all the points with strain <= 0.15%
indexlinstrain=find(strainori<=0.0015);
%Extract the strain in the linear regime
strainextr = strainori(indexlinstrain);
%Extract the stress in the linear regime
stressextr = stressori(indexlinstrain);
%Fit these points to a linear regression y=p1*x+p2
p = polyfit(strainextr,stressextr,1);
%y-offset is the smallest stress
yoffset = min(stressextr);
%Find the corresponding value of strain at the smallest stress using the
%linear regression
%x-offset can be found accordingly
%Point (x-offset,y-offset) defines the first point of elastic regime
xoffset = (yoffset-p(2))./p(1);

%Now correct the stress and strain data
stress = stressori - yoffset;
strain = strainori - xoffset;

%Young's modulus is the slope of the linear regime
YoungGPa = p(1).*10^(-3)

%Plot the stress-strain graph
plot(strain,stress,'b','linewidth',0.001)
xlabel('Strain')
ylabel('Stress/MPa')
xlim([-0.0005,0.045])
hold on

%Plot the linear regression
xlr = [0:0.000001:0.004];
ylr = p(1).*xlr;
plot(xlr,ylr,'r','linewidth',0.001)
hold off

%Use the convention "strain 0.2%" to determine the yield strength
val = 0.002;
err = abs(strain-val);
%Find the index of the yield strain nearest to 0.2%
[index index] = min(err);
%Locate and return the yield strength in MPa
YieldMPa = stress(index)
end
```