Semester Project

Advanced NEMS Group

CHARACTERIZATION OF MECHANICAL PROPERTIES OF MICRO/NANO BEAMS

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Abstract

In this project, characterization of a series of previously fabricated Aluminium clamped-clamped micro beams and cantilevers by electrical measurements in order to investigate the effects of scaling down on mechanical properties of micro/nano structures is presented. The Young's modulus that depends on the thickness of the microstructure, residual stress and surface properties as opposed to a constant Young's modulus in macro scale theories was investigated. Due to insufficient sample space and high stresses during fabrication, a correlation was not obtained.

Keywords- Aluminium beams, Coupled Stress theory, Residual Stress theory, Surface Elasticity theory, Combined stress model, electrical characterization.

1. Introduction

1.1 Theoretical Model

Experimental work [1,2] show that at the microstructures do not follow classical Euler Bernoulli beam theory and the Young's modulus does not remain constant with dimensions, in particular the thickness. The main theoretical models that explain the behaviour of mechanical properties on scaling down are Residual Stress Theory (RST), Couple Stress Theory (CST), Grain Boundaries Theory (GBT), Surface Stress Theory (SST) and Surface Elasticity Theory (SET) [3]. In order to obtain a combined model to explain the behaviour, the RST, CST and SET were considered neglecting the SST and GBT since they are considered as secondary effects and not applicable to all cases.

The GBT is applicable when the thickness of the structure is equal to few times the grain size. This is not a frequent occurrence in the micro-scale. The SST is also neglected since it cannot be applied to Cantilevers and other free structures as they have zero surface stress and the effect of SST is similar to noise that is not observed experimentally.

The CST predicts the stiffening effect on scaling down by taking into account the length scaling effect in the Euler-Bernoulli theory. The addition component arising due to this effect can be described by an 'Effective Young's Modulus' E_{eff} given by

$$E_{eff} = E + 24\left(\frac{E}{1+\nu}\right)\left(\frac{l}{h}\right)^2 \qquad (1)$$

Where E is the Young's Modulus, ν is the Poisson's ratio, l is the length scaling parameter and h is the thickness of the beam.

The SET also accounts for stiffening or softening but it is a surface effect theory as opposed to CST that accounts only for Bulk stiffening. This arises to due to the surface being of a more amorphous nature due to defects and having different interatomic interactions with respect to those of bulk atoms. The equation of E_{eff} is derived from composite beam theory, where the microstructure consists of a bulk volume with bulk material properties and is surrounded by a thin shell with surface material properties [4]. This relation is given by Equation (3), where δ is the thickness of the shell, the Surface Elasticity is C_s is

$$C_S = \delta\left(E_{bulk} - E_{surf}\right) \qquad (2)$$

$$E_{eff} = E_{bulk} - 6C_S\left(\frac{1}{h}\right) \qquad (3)$$

Finally the RST should be taken into account to model the intrinsic stresses (particularly in clamped-clamped beams) developed to micro fabrication of the structure. The E_{eff} for a clamped-clamped beam is derived to be [3]:

$$E_{eff} = E + \frac{3}{10}\sigma_0\left(\frac{L}{h}\right)^2 \qquad (4)$$

Where $\sigma_0$the intrinsic stress and L is the length of the beam. Taking these three theories into account, a combined model for a clamped-clamped beam has been proposed by [3] given by:

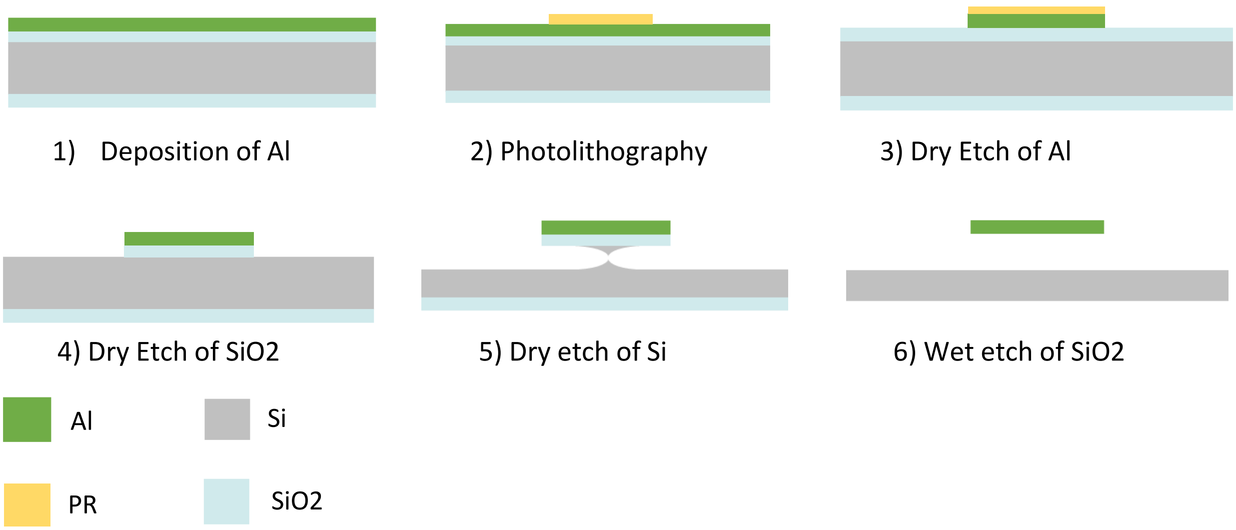
$$E_{eff} = E_{bulk} - 6C_s\left(\frac{1}{h}\right) + \frac{24E}{1+\nu}l^2\left(\frac{1}{h}\right)^2 + \frac{3}{10}\sigma_0\left(\frac{L}{h}\right)^2 \qquad (5)$$

Applying a combined model of the Coupled Stress Theory, Surface Elasticity Theory and Residual Stress theory to existing experimental data has shown promising results [3]. In order to further develop this combined model, the Advanced NEMS group has fabricated Aluminium clamped-clamped micro beams and cantilevers to study in detail by characterization and observing the trend of the E_{eff} as a function of dimensions, surface elasticity and residual stress. This is done by finding the pull in voltage (V_P) of each beam which is a function of E_{eff} as describe in Equation (5) for a clamped-clamped beam [5].

$$V_P = 3.08 * \sqrt{\frac{g^3h^3E_{eff}}{\epsilon_0 L^4}} \qquad (6)$$

1.2 Fabrication

The aluminium microstructures were fabricating on Si wafer with a layer of 200nm wet oxide. The process flow consists of six steps briefly described below and the process flow and run card is attached in the appendix. These structures were fabricated by Kaitlin Howell of the ANMES group.


1) Deposition of Al
2) Photolithography
3) Dry Etch of Al
4) Dry Etch of SiO2
5) Dry etch of Si
6) Wet etch of SiO2

Step 1: Aluminium of 2μm thickness was deposited by sputtering on the Si wafer with a 200nm thick SiO2 layer on top.

Step 2: Photolithography was carried out with the patterns of the beams and electrodes required using AZ ECI positive photoresist of thickness 3μm. The photolithography has a CD of 1μm.

Step 3: Using the post baked mask as protection, the rest of the Aluminium was removed by Dry Etching.

Step 4: Dry etching of 200nm thick SiO2 was carried out, still keeping the AZ ECI mask. Since the next step was isotropic etching, the PR was stripped.

Step 5: Isotropic dry etching of Si was carried out to release the structures..

Step 6: Isotropic wet etching of SiO2 was carried out to completely release the Al beams.

It can be noticed from the SEM images after fabrication that the longer beams with small widths and cantilevers are deformed and the portions of the beam close to the anchors seem fragile as shown in Figure 1.

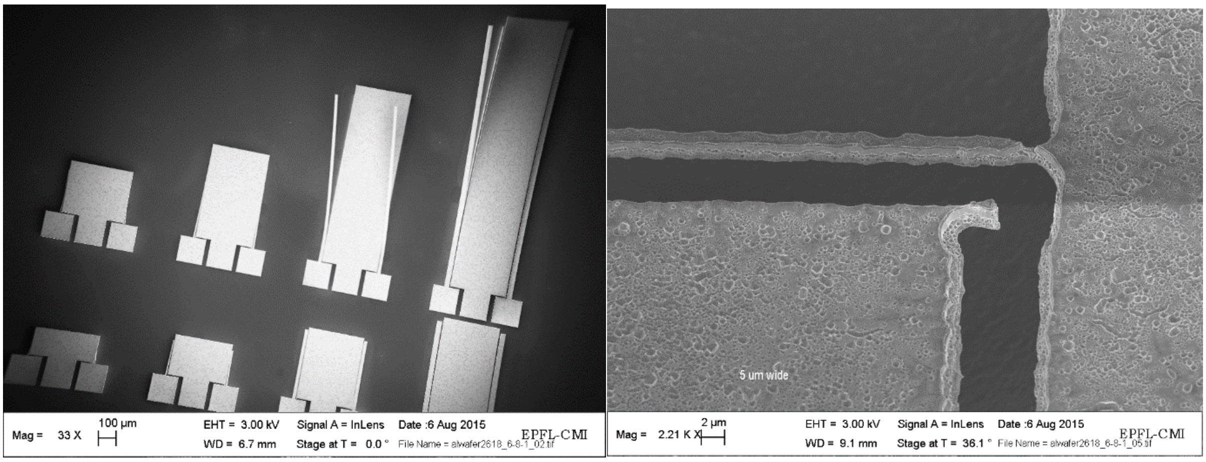


Figure 1: SEM images post SiO2 etching

2. Experimental Setup

The characterization was conducted on an electrical probe station using two probe measurements. The input of the two probes were connected to a high voltage supply with a 10MΩ resistance in series in order limit the output current to the range of μA as per specifications of the current readout of the readout and to avoid damage of the HV supply unit. The wafer with the Aluminium beams was placed on the vacuum chuck with one probe connected to the beam pad (A) and the other to the 2nd electrode (B) as shown in Figure 2.1.

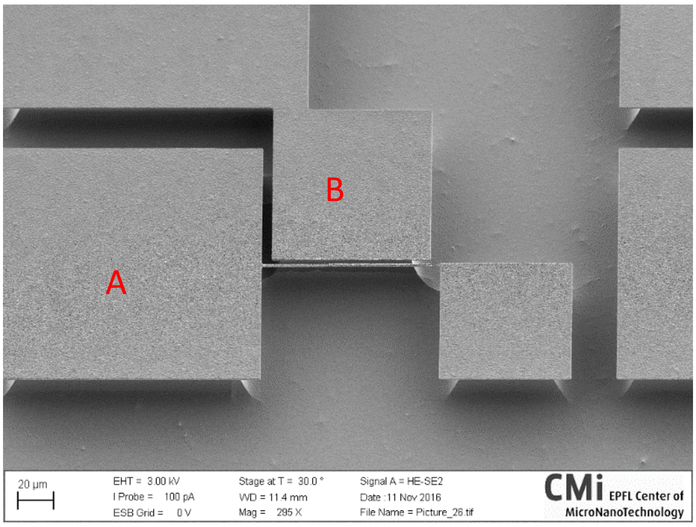


Figure 2.1: SEM image showing the two actuating electrodes of the Al Beam

2.1 Control of the Source meter

Taking into account the SRS source meter, for a sweep of 0 to 300V in steps of 2.5V and a required stabilization of 3 seconds for each applied voltage, the measurement of each beam would take approximately 6minutes. In order to reduce the measurement time, an NI GPIB bus was used to connect to the source meter and desktop to automatically sweep the required voltages using Matlab from the desktop. The two HV supplies used were Keithley 2400 and SRS source meters were used, whose Matlab control codes are as shown in Appendix 1 and 2.

The Keitley 2400 has a faster measurement time of 1 second however it can supply only upto 210 V as opposed to a measurement time of 3 seconds by the SRS supply with can supply upto 1kV. Hence both were alternately used based on the pull in voltages required for a particular rom of beams.

3. Results

The beams were arranged in units of varying air gaps ranging from 1, 2, 2.5 and 3μm. Each of these units contained rows of groups of 2 beams with widths varying from 1, 1.5, 2, 2.5, 3, 4, 5 and 10μm and columns of lengths varying from 100, 200, 400, 600 and 800μm.

Due to assumed high stress in the deposition step, all the cantilevers on the wafer were pre-deformed or collapsed. Few of these examples are as shown in Figure 2.2. Hence the cantilevers on the wafer were not characterized for pull-in voltage.

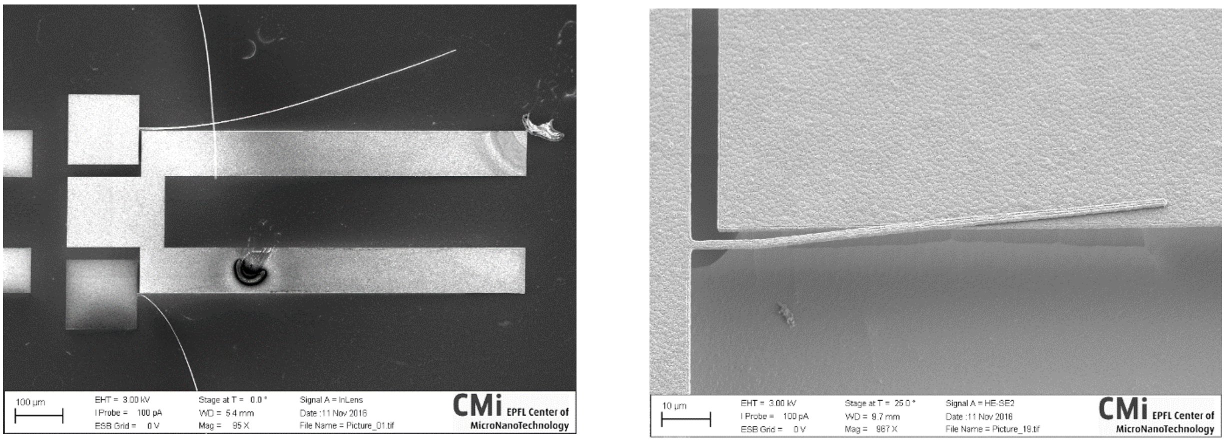


Figure 2.2: SEM images showing defects of the cantilevers

Furthermore on testing that many clamped-clamped beams did not pull in even over 500V, SEM imaging was carried out and it was observed that only the clamped-clamped beams of lengths 100μm and 200μm were intact due to their high stiffness and the rest of the columns of lengths 400, 600 and 800μm had missing beams or beams broken from one of their anchors as shown in Figure 2.3. Some examples of intact beams of lengths 100 and 200μm are as shown in Figure 2.4. However not all beams of 100μm and 200μm were released or intact. Few of these examples are as shown in Figure 2.5.

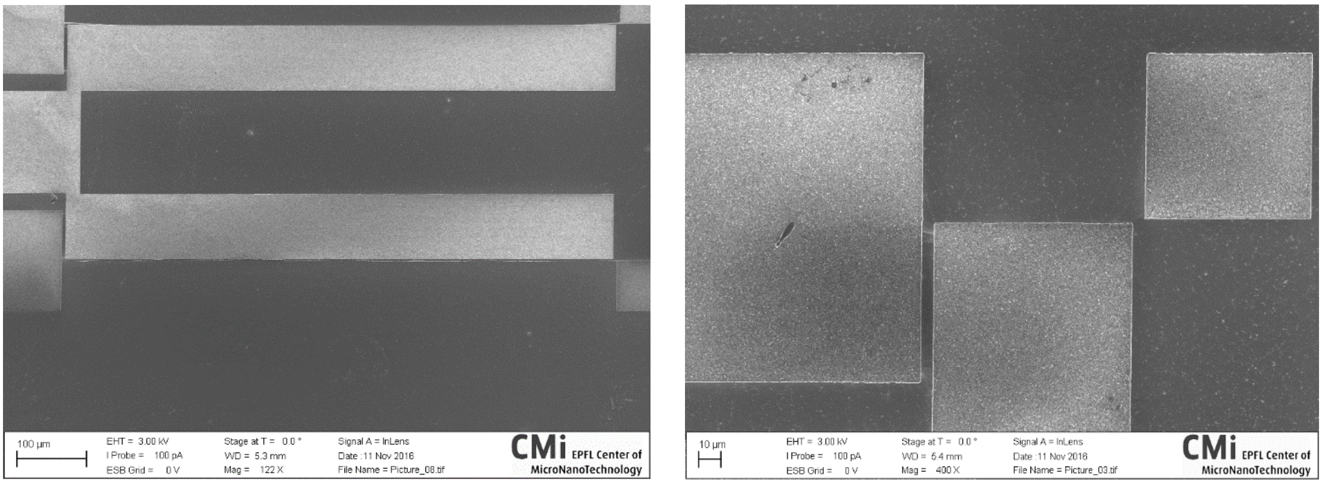


Figure 2.3: SEM images of collapsed and missing double clamped beams

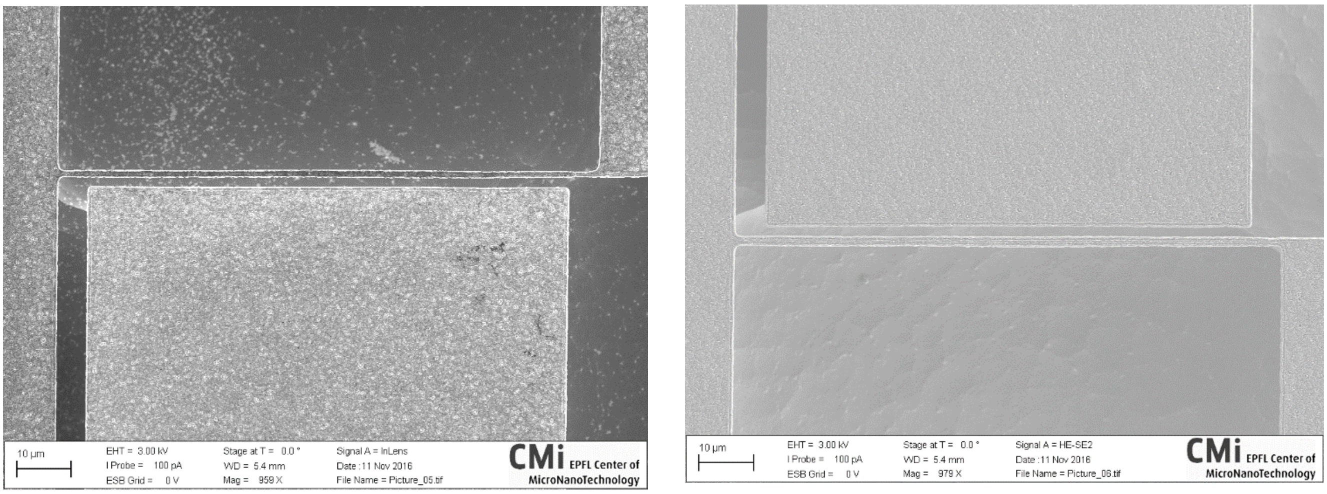


Figure 2.4: SEM images of intact double-clamped beams of lengths 200 and 100μm

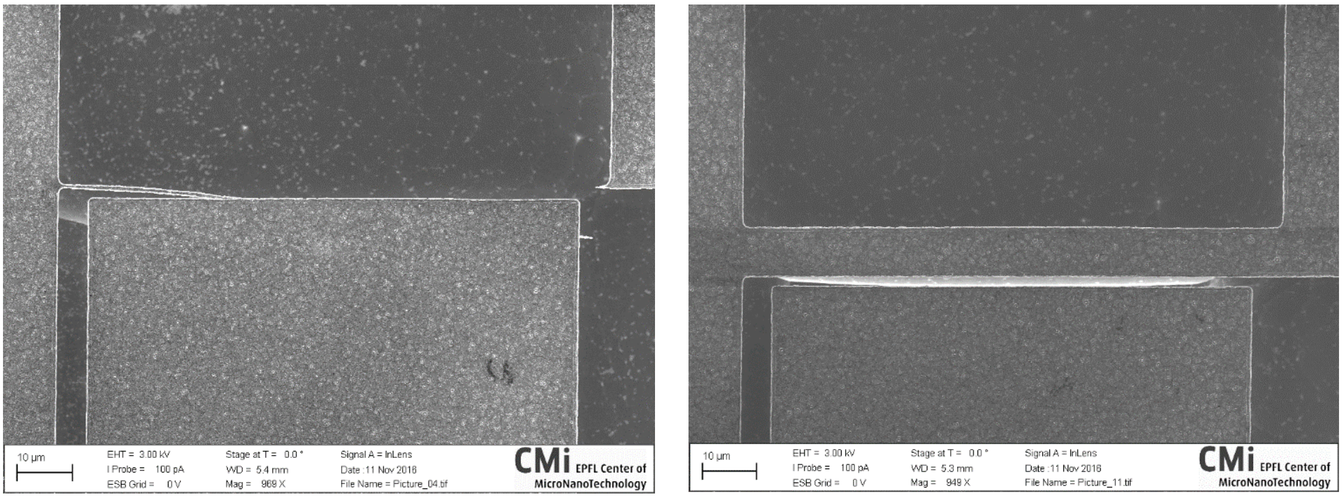


Figure 2.5: SEM images of broken and partially unleased double clamped beams.

3.1 Results based on I-V Characteristics

The extracted I-V curves from the source meters in most of the tested clamped-clamed beams shows the behaviour of a capacitor connected in series with a resistor. When the beam comes in contact with its adjacent actuation electrode, the air-gap capacitor is shorted and the inverse of slope of the I-V curve from the pull in point is equal to 10MΩ which is the external resistance connected in series with the source meter and the probes. Some I-V curves obtained are as shown in Figure 2.6

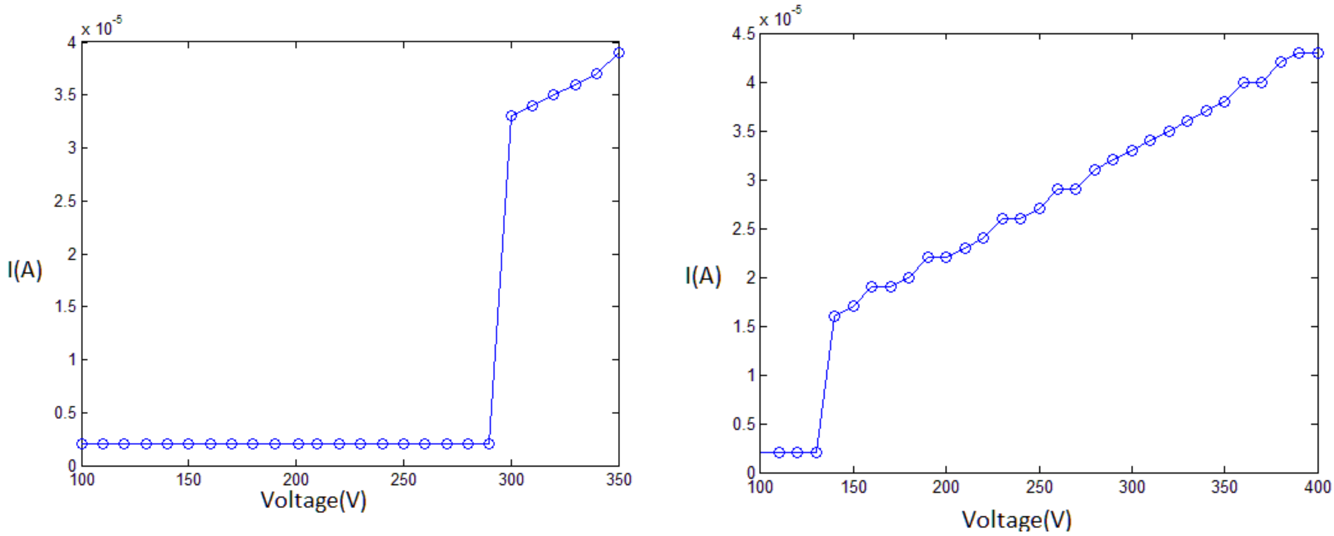


Figure 2.6: IV curves obtained from voltage sweep using SRS Sourcemeter and GPIB interface with Matlab

A compilation of the pull in voltages as a function of widths, airgaps and lengths of the clamped-clamped beams is as shown in Figure 2.7. The pull-in voltage is expected to decrease with an increase in length, increase with increase in width and increase with increase in gap. However as seen from Figure 2.7 there is no correlation between the dimensions of the beams and the pull in voltage. This is attributed to:

1) Due to stresses in the deposition step as seen from the deformed beams in Figure 2.8 a, the stiffness and mechanical behaviour cannot be predicted.
2) Due to insufficient control in etching process or material defects as seen in section 1.2, the beam does not snap in symmetrically due to weak anchors as can be seen in Figure 2.8 b. Leading to reduced correlation between pull-in voltage and dimensions.
3) Since high voltages ranging from 150-500V are being applied, charge crowding at the anchors leading to breaking and snapping of the beam at the anchor with 'burnt marks' as in Figure 2.8 c.
4) Many data points are missing as in Figure 2.7 g and h due to no pull-in voltage (including 1kV) as shown in Figure 2.8 d. This could be attributed to high stiffness or unreleased beams. Particularly in the beams of larger width (5 and 10μm) and shorter length$100\mu m$.

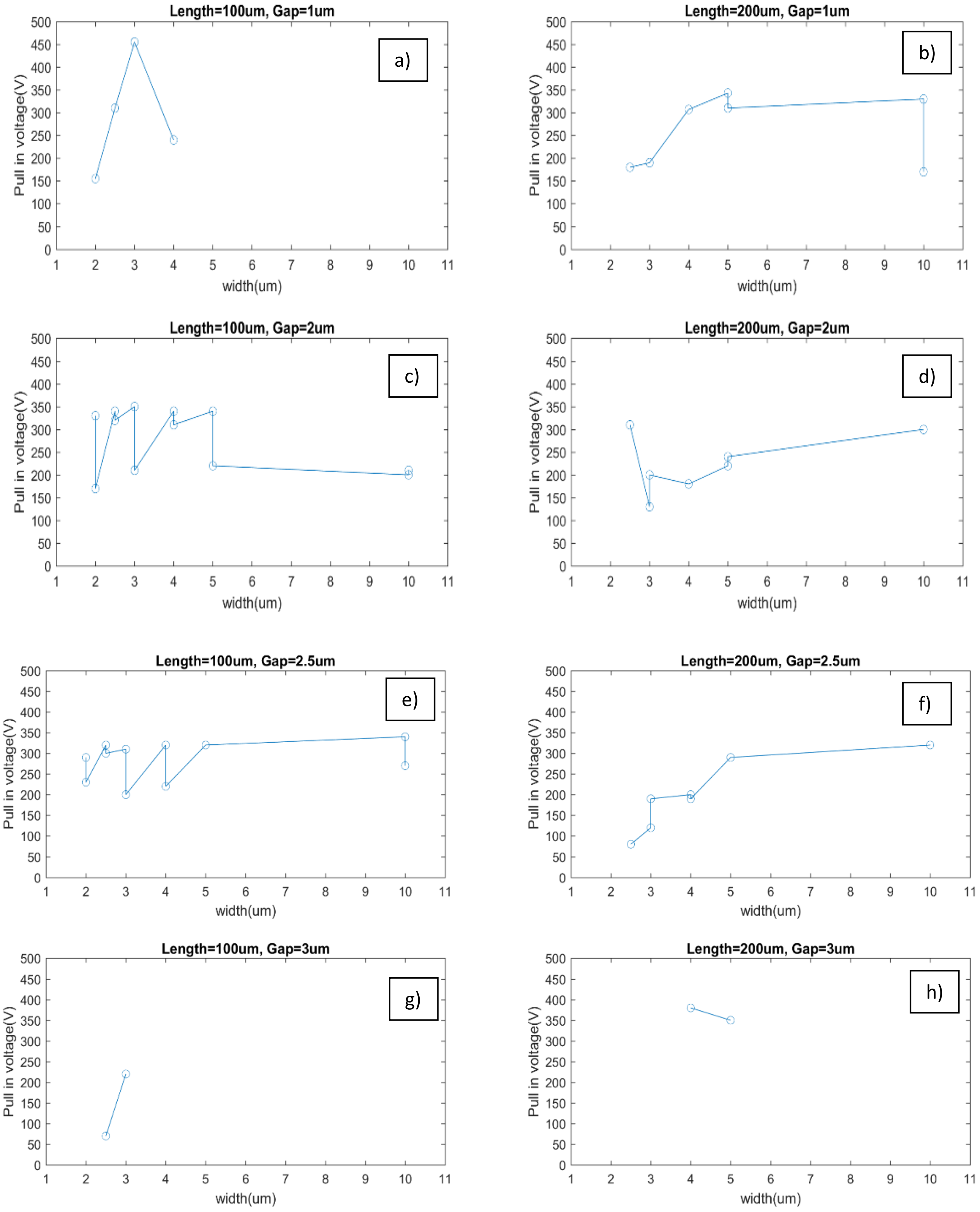


Figure 2.7: Compiled pull in voltage vs. width curves from varying lengths and gaps. The missing points represent beams which were already collapsed (short), missing beams (open) and beams which did not pull in even at very high voltages

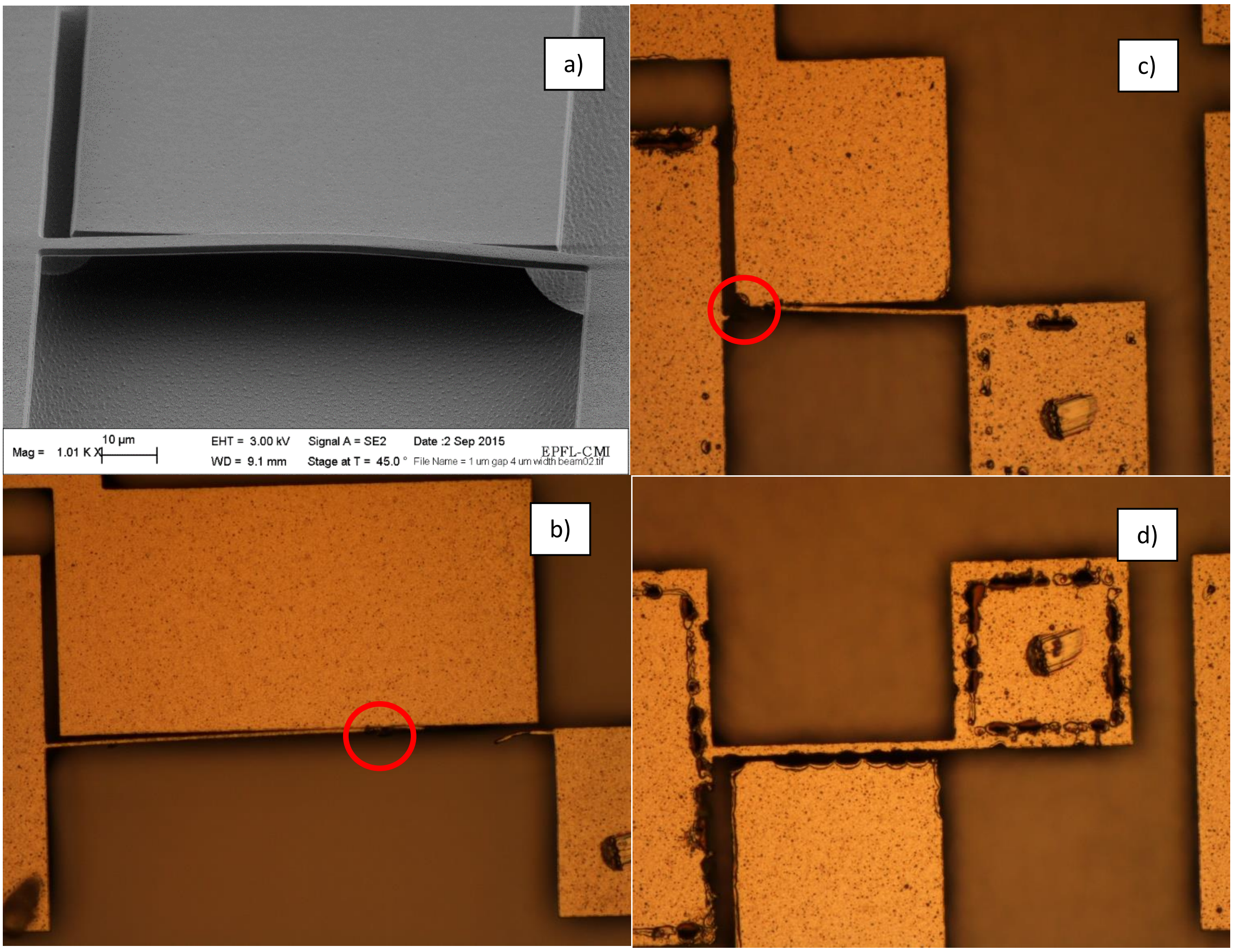


Figure 2.8 a) SEM image of deformed beam. b) Asymmetric Pull in c)Pull In at the corner d) No pull in for applied voltage of 1kV

4. Conclusion

During this semester project, electrical characterization of fabricated Al clamped-clamped beams for investigating mechanical properties of micro structures was carried out. Matlab codes were implemented to control the Keithley 2400 and SRS source meters using GPIB interface.

Unfortunately due to lack of large sample space, i.e. missing cantilevers and only the shortest two lengths (100, 200μm) of clamped-clamped beams with highest stiffnesses being present out of 5 lengths as well as lack of correlation between the dimensions of the beams with pull-in voltage, the obtained data could not be analysed further. Future work would include optimizing the etching process to obtain higher yield and investigating the reasons for occurrence of the pre-stressed beams.

I would like to thank Dr Tom Larsen for directly me throughout this project to help me setup the measurement system and his insightful suggestions to come to the right conclusions for the behaviour we were observing. I would like to thank Prof. Guillermo Villanueva for this opportunity to work with the ANEMS group and his guidance and suggestions with progressing in this project smoothly and in the right direction.

References

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Appendix 1: Matlab code for SRS Source Meter

```
function IV(filename,Vstart,Vstep,Vstop)


% Find a GPIB object.
obj1 = instrfind('Type', 'gpib', 'BoardIndex', 0, 'PrimaryAddress', 13,
'Tag', '');

% Create the GPIB object if it does not exist
% otherwise use the object that was found.
if isempty(obj1)
    obj1 = gpib('NI', 0, 13);
else
    fclose(obj1);
    obj1 = obj1(1)
end

% Connect to instrument object, obj1.
fopen(obj1);

% Communicating with instrument object, obj1.
fprintf(obj1, '*RST');    %Reset
fprintf(obj1,'HVON');    %High Voltage setting on: Doesn't work without
this
fprintf(obj1,'ILIM0.001');  % Setting current limit
fprintf(obj1,'VLIM600');   %Setting voltage limit
ii=0;
timewait=((Vstop-Vstart)/Vstep)*4
for i=Vstart:Vstep:Vstop   %Loop to sweep voltage
volt=i
ii=ii+1;
str1=num2str(volt);   % converting voltage value to string
voltset=strcat('VSET',str1);  % Concatenating string
fprintf(obj1,voltset);   % Setting voltage
pause(4)

data3 = query(obj1, 'VSET?');  % Reading/query set voltage
data4 = query(obj1, 'VOUT?');   % Reading/query output voltage
vout(ii)=str2double(data4);   % converting string to double
vset(ii)=str2double(data3);

if volt~=vset(ii)   % Break the loop if voltage is not set correctly
    break
end

data2 = query(obj1, 'IOUT?');  % Reading/query set current
iout(ii)=str2double(data2);

end
fprintf(obj1, '*RST');
figure
plot(vset,iout,'b-o')
xlabel('V (volt)')
ylabel('I (A)')

end
```

Appendix 2: Matab code for Keitley's Series 2400 Source Meter Unit

```
function out=IV_Curve_New(filename,V_Start,V_Stop,V_Step)
%Slightly modified code from Keithley
http://www.keithley.com/matlab/instruments
%Makes an I-V sweep using a 4-wire configuration - Keithley 2400
%sourcemeter
%1: Connect current wires to the input/output.
%2: Connect voltage wires to 4-wire sense.
%3: Set min and max current and step size
%4: Run the code

% Find a GPIB object.
obj1 = instrfind('Type', 'gpib', 'BoardIndex', 0, 'PrimaryAddress', 24,
'Tag', '');

% Create the GPIB object if it does not exist
% otherwise use the object that was found.
if isempty(obj1)
    obj1 = gpib('NI', 0, 24);
else
    fclose(obj1);
    obj1 = obj1(1)
end


% Create the instrument object.

g.InputBufferSize = 1000; %Make sure that the buffer size is larget enough

% Set the property values.
set(obj1, 'BoardIndex', 0);
set(obj1, 'ByteOrder', 'littleEndian');
set(obj1, 'BytesAvailableFcn', '');
set(obj1, 'BytesAvailableFcnCount', 48);
set(obj1, 'BytesAvailableFcnMode', 'eosCharCode');
set(obj1, 'CompareBits', 8);
set(obj1, 'EOIMode', 'on');
set(obj1, 'EOSCharCode', 'LF');
set(obj1, 'EOSMode', 'read&write');
set(obj1, 'ErrorFcn', '');
set(obj1, 'InputBufferSize', 2000);
set(obj1, 'Name', 'GPIB0-24');
set(obj1, 'OutputBufferSize', 2000);
set(obj1, 'OutputEmptyFcn', '');
set(obj1, 'PrimaryAddress', 24);
set(obj1, 'RecordDetail', 'compact');
set(obj1, 'RecordMode', 'overwrite');
set(obj1, 'RecordName', 'record.txt');
set(obj1, 'SecondaryAddress', 0);
set(obj1, 'Tag', '');
set(obj1, 'Timeout', 10);
set(obj1, 'TimerFcn', '');
set(obj1, 'TimerPeriod', 1);
set(obj1, 'UserData', []);

if nargout > 0
    out = obj1;
```

```
end
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%Find buffer size
BufferSize = (V_Stop-V_Start)/V_Step


% Model 2400 Specific Functions
% Sweep current and measure back voltage
fopen(obj1)
fprintf(obj1,':*RST')
% setup the 2400 to generate an SRQ on buffer full
fprintf(obj1,':*ESE 0')
fprintf(obj1,':*CLS')
fprintf(obj1,':STAT:MEAS:ENAB 1024')
fprintf(obj1,':*SRE 1')
% buffer set up
fprintf(obj1,':TRAC:CLE')
fprintf(obj1,':TRAC:POIN %d', BufferSize)    % buffer size
% Set up the Sweep
fprintf(obj1,':SOUR:FUNC:MODE VOLT')
fprintf(obj1,':SOUR:VOLT:STAR %f',V_Start)  %Voltage start [V]
fprintf(obj1,':SOUR:VOLT:STOP %f', V_Stop)  %Voltage stop [V]
fprintf(obj1,':SOUR:VOLT:STEP %f', V_Step)  %Voltage step size [V]

fprintf(obj1,':SOUR:CLE:AUTO ON')
fprintf(obj1,':SOUR:VOLT:MODE SWE')
fprintf(obj1,':SOUR:SWE:SPAC LIN')
fprintf(obj1,':SOUR:DEL:AUTO OFF')
fprintf(obj1,':SOUR:DEL 0.5')

fprintf(obj1,':SENS:FUNC "CURR"')
fprintf(obj1,':SENS:FUNC:CONC ON')
fprintf(obj1,':SENS:VOLT:RANG:AUTO OFF')

%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
% IMPORTANT: if the unit goes into compliance,
% adjust the compliance or the range value
fprintf(obj1,':SENS:CURR:PROT 5E-3')
%fprintf(obj1,':SENS:CURR:PROT:LEV ') % voltage compliance
%fprintf(obj1,':SENS:VOLT:RANG 7')   % volt measurement range
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
fprintf(obj1,':SENS:CURR:NPLC 1')
fprintf(obj1,':FORM:ELEM:SENS CURR,VOLT')
fprintf(obj1,':TRIG:COUN %d', BufferSize)
fprintf(obj1,':TRIG:DEL 2') %Set source delay to 500 ms
fprintf(obj1,':SYST:AZER:STAT OFF')
fprintf(obj1,':SYST:TIME:RES:AUTO ON')
fprintf(obj1,':TRAC:TST:FORM ABS')
fprintf(obj1,':TRAC:FEED:CONT NEXT')
fprintf(obj1,':OUTP ON')
fprintf(obj1,':INIT')

for T = 1:BufferSize*5
    T
    BufferSize*5
    pause(1)
end

% Used the serial poll function to wait for SRQ
val = [1];          % 1st instrument in the gpib object, not the gpib add
```

```
spoll(obj1,val);    % keep control until SRQ
fprintf(obj1,':TRAC:DATA?')

A = scanstr(obj1,',','%f');

%parse the data & plot
Curr=A(4:2:length(A),1); %Take out the first point as it always looks wired
Volts=A(3:2:length(A)-1,1); %Take out the first point as it always looks
wired

figure(1);
plot(Volts,Curr,':bo','LineWidth',0.5,...
                'MarkerEdgeColor','k',...
                'MarkerFaceColor','r',...
                'MarkerSize',5)
xlabel('Source-volts (V)'),ylabel('Measured-current(A)')
title('Keithley 2400: Sweeps V & Measure I');

figure(2)
plot(Volts, Volts./Curr,':bo','LineWidth',0.5,...
                'MarkerEdgeColor','k',...
                'MarkerFaceColor','r',...
                'MarkerSize',5)
xlabel('Source-volts (V)'),ylabel('Measured-Resistance(Ohm)')


save(filename)

% reset all the registers & clean up
% if the registers are not properly reset,
% subsequent runs will not work!
fprintf(obj1,':*RST')
fprintf(obj1,':*CLS ')
fprintf(obj1,':*SRE 0')
% make sure STB bit is 0
STB = query(obj1, '*STB?');
fclose(obj1)
delete(obj1)
clear obj1


end

%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
```

Appendix 3: Process Flow

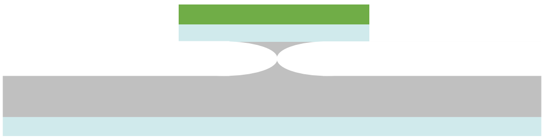
Technologies used
Sputtering, positive resist, dry etching,

Photolith masks			
Mask #	**Critical Dimension**	**Critical Alignment**	**Remarks**
1	**1 um**	First Mask	Structure patterning

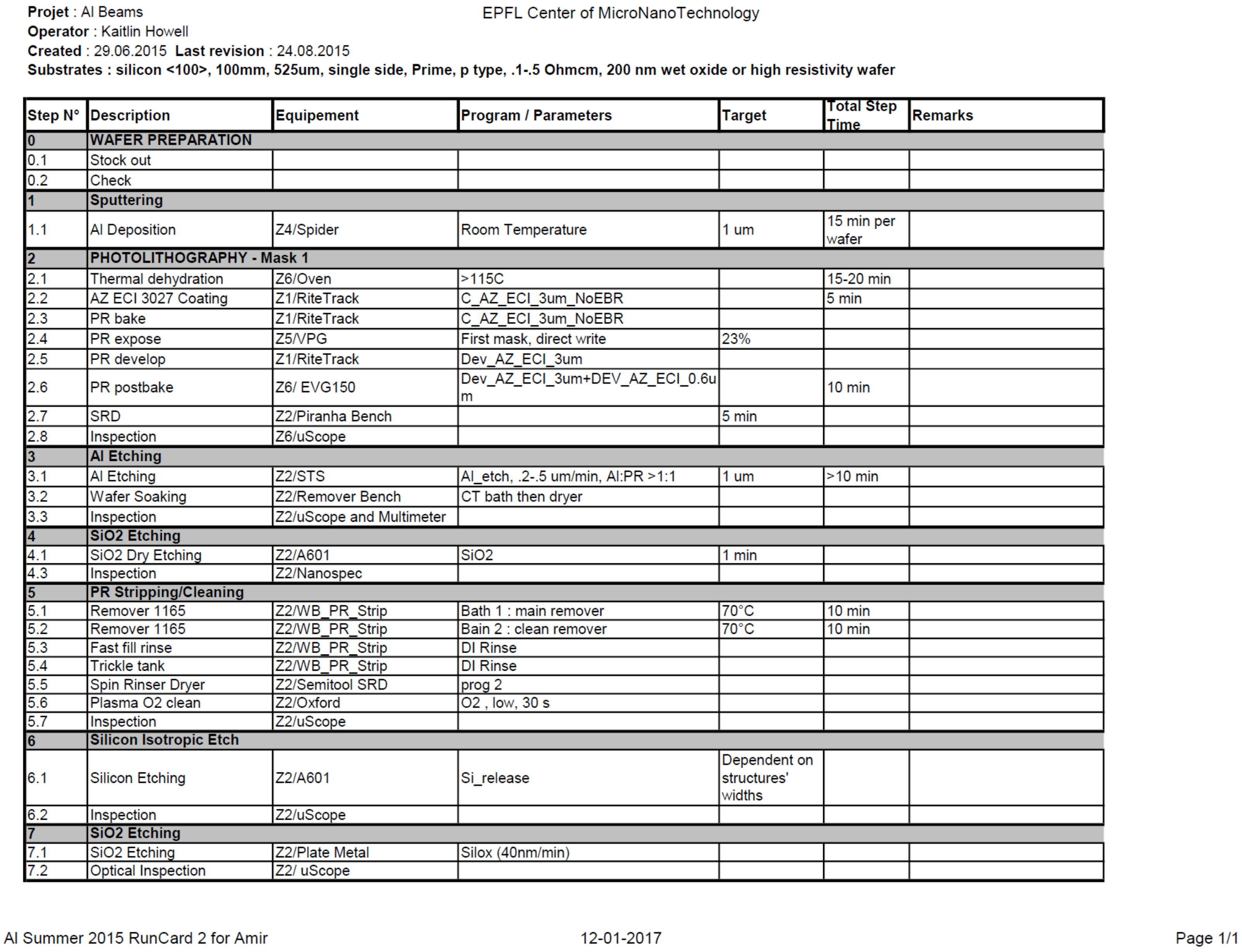
Substrate Type
Silicon <100>, Ø100mm, 525um thick, Single Side polished, Prime, p type, 15-25 Ohm.cm, 200 nm wet SiO_2

Process outline

Step	**Process description**	**Cross-section after process**
01	*Metal Evaporation + Anneal 400°C* Machine: Pfeiffer *SPIDER 600 + JETFIRST 200* Metal :*Al* Thickness : *2 um*	
02	*Photolith* Machine: *Rite Track + VPG200* PR : AZ *ECI 3007 – 3 μm* Mask : *CD = 1 um*	
03	*Dry Etch* Material : *Al* Machine: *STS* Depth : *2 μm*	
04	*Dry Etch* Material : *Si02* Machine: *A601* Depth : *0.2 μm* + Resist strip	

05	*Isotropic Dry Etch* Material : *Si* Machine: *Alcatel 601E*	
06	*Wet Etch* Material : *SiO_2* Machine: Plate Metal, Silox Depth : *$0.2\ \mu m$*	

Appendix 4: Run Card



Projet : Al Beams
Operator : Kaitlin Howell
Created : 29.06.2015 **Last revision** : 24.08.2015
Substrates : silicon <100>, 100mm, 525um, single side, Prime, p type, .1-.5 Ohmcm, 200 nm wet oxide or high resistivity wafer

EPFL Center of MicroNanoTechnology

Step N°	**Description**	**Equipement**	**Program / Parameters**	**Target**	**Total Step Time**	**Remarks**
0	**WAFER PREPARATION**					
0.1	Stock out					
0.2	Check					
1	**Sputtering**					
1.1	Al Deposition	Z4/Spider	Room Temperature	1 um	15 min per wafer	
2	**PHOTOLITHOGRAPHY - Mask 1**					
2.1	Thermal dehydration	Z6/Oven	>115C		15-20 min	
2.2	AZ ECI 3027 Coating	Z1/RiteTrack	C_AZ_ECI_3um_NoEBR		5 min	
2.3	PR bake	Z1/RiteTrack	C_AZ_ECI_3um_NoEBR			
2.4	PR expose	Z5/VPG	First mask, direct write	23%		
2.5	PR develop	Z1/RiteTrack	Dev_AZ_ECI_3um			
2.6	PR postbake	Z6/ EVG150	Dev_AZ_ECI_3um+DEV_AZ_ECI_0.6um		10 min	
2.7	SRD	Z2/Piranha Bench		5 min		
2.8	Inspection	Z6/uScope				
3	**Al Etching**					
3.1	Al Etching	Z2/STS	Al_etch, .2-.5 um/min, Al:PR >1:1	1 um	>10 min	
3.2	Wafer Soaking	Z2/Remover Bench	CT bath then dryer			
3.3	Inspection	Z2/uScope and Multimeter				
4	**SiO2 Etching**					
4.1	SiO2 Dry Etching	Z2/A601	SiO2	1 min		
4.3	Inspection	Z2/Nanospec				
5	**PR Stripping/Cleaning**					
5.1	Remover 1165	Z2/WB_PR_Strip	Bath 1 : main remover	70°C	10 min	
5.2	Remover 1165	Z2/WB_PR_Strip	Bain 2 : clean remover	70°C	10 min	
5.3	Fast fill rinse	Z2/WB_PR_Strip	DI Rinse			
5.4	Trickle tank	Z2/WB_PR_Strip	DI Rinse			
5.5	Spin Rinser Dryer	Z2/Semitool SRD	prog 2			
5.6	Plasma O2 clean	Z2/Oxford	O2 , low, 30 s			
5.7	Inspection	Z2/uScope				
6	**Silicon Isotropic Etch**					
6.1	Silicon Etching	Z2/A601	Si_release	Dependent on structures' widths		
6.2	Inspection	Z2/uScope				
7	**SiO2 Etching**					
7.1	SiO2 Etching	Z2/Plate Metal	Silox (40nm/min)			
7.2	Optical Inspection	Z2/ uScope				

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