Electric field stimulated growth of Zn and Sn whiskers

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Motivation

- Trying electric field as a common whisker driver
- Comparing the effects of various electric field sources
- Trying the external field as a factor accelerating whisker growth rates ➔ accelerated life testing
- Verifying the electrostatic theory[1] where the effects of random local fields of imperfect metal surfaces explain:

– Why whiskers grow exceptionally on metals

– Why they can have abnormally high aspect ratios (up to 10^4)

– The nature of their randomness and poor predictability

– Sensitivity to stresses, humidity, and contaminations

– Kinetic features (incubation, constant rate, 'stop and go'...)

– Lack of quantitative theory of metal whiskers so far

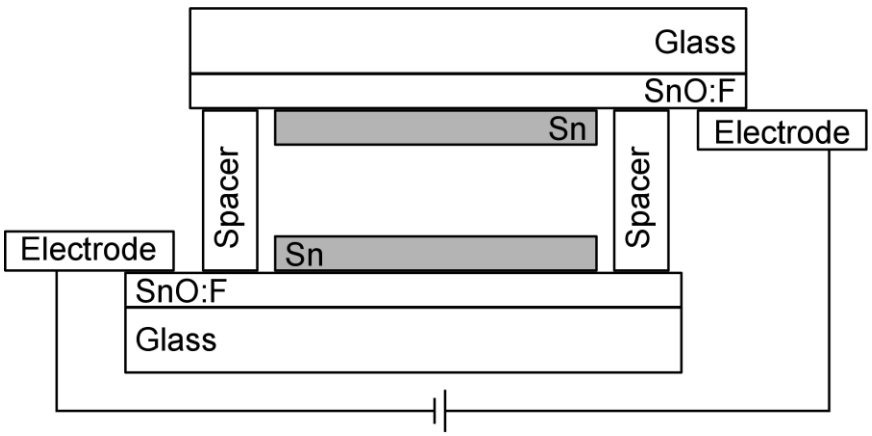
[1] V. G. Karpov, Phys. Rev. Applied, **3**, 044004 (2015)

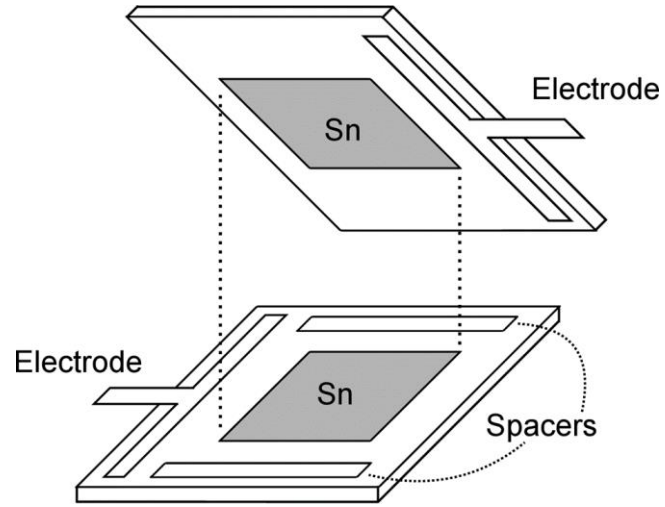
Outline

- Capacitive setup experiments (Sn)
- SEM e-beam charging (Zn)
- Medical e-beam accelerator charging (Sn, Zn)
- Van der Graaff generator field experiments (Zn)
- Surface plasmon polariton (SPP) field experiments (Sn, Zn)
- Ion beam charging experiments (Zn)
- Discussion
  - Observations by others
  - Masking factors
  - Future directions

Capacitive setup

Spacer height: 50±5µm





- Bias held at 14.5 V, (BK Precision 1696 regulated power supply), average field between the plates 2900 V/cm
- Contact with conductive tape applied to the SnO:F layer
- Films were held under bias for 1 week

Evaporated tin sample

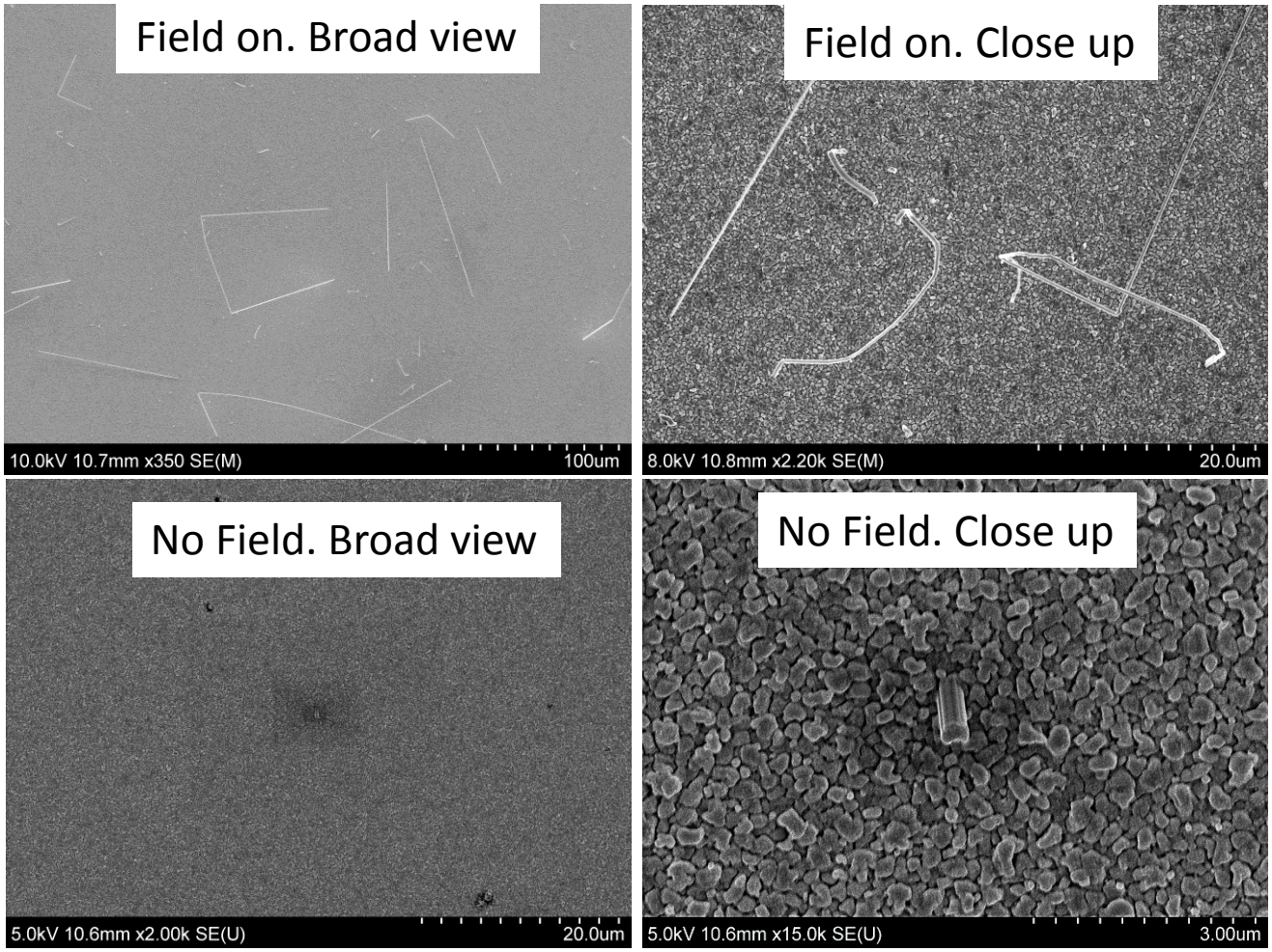
- 10 cm^2 tin films were thermally evaporated on Pilkington TEC15 substrates (soda lime glass with nominal 15 Ω/□ sheet resistance SnO_2:F coating)

- Tin film thickness of 0.15 µm far below the typical micron range of efficient whisker growing films

Sputtered tin sample

- 10 cm^2 tin films sputter coated on Pilkington TEC15 substrates (soda lime glass with nominal 15 Ω/□ sheet resistance SnO_2:F coating)
- Sputtering at room temperature from a pure 0.25” tin target, 20 mTorr working pressure atmosphere of argon gas, using 50W RF power
- Film thickness of 0.15 μm far below the typical micron range of efficient whisker growing film thickness
- Never polished, no surface modifications

Summer (2014) observations
(hot and humid: 27º C, 80%RH, evaporated Sn)



More quantitative measures: from Astronomy to Physics

define whisker creation rate

$$R = \frac{\#\text{ of whiskers}}{\text{area} \cdot \text{time}}, \text{cm}^{-2}\text{s}^{-1}$$

$R_{\text{spontaneous}}$ (no external field)

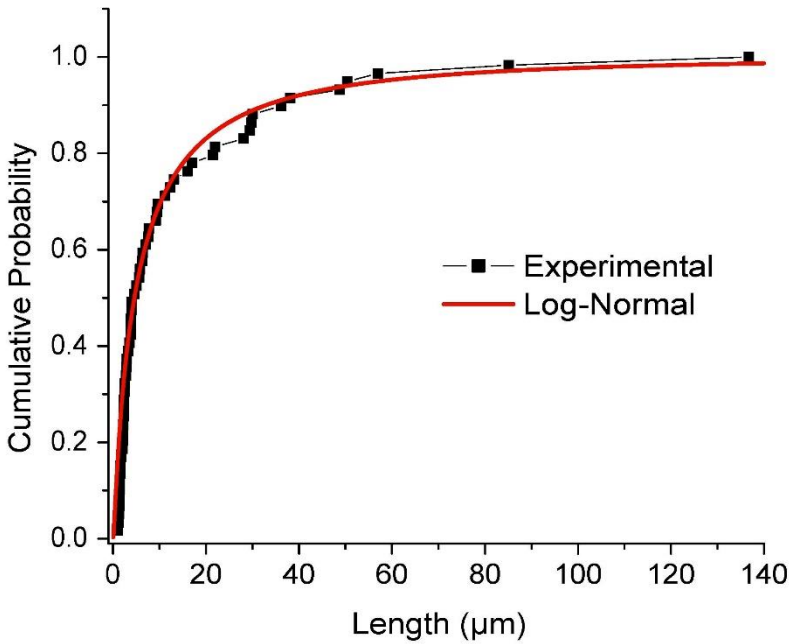
$R_{\text{stimulated}}$ (under external fgield)

Acceleration ratio

$$a = \frac{R_{\text{stimulated}}}{R_{\text{spontaneous}}}$$

We expect and seek $a > 1$, better yet $a >> 1$

Summer 2014 results one week experiment



Whisker parameters	Field on	No field
Whiskers/cm^2	29,000	<700
Average length (μm)	14	6

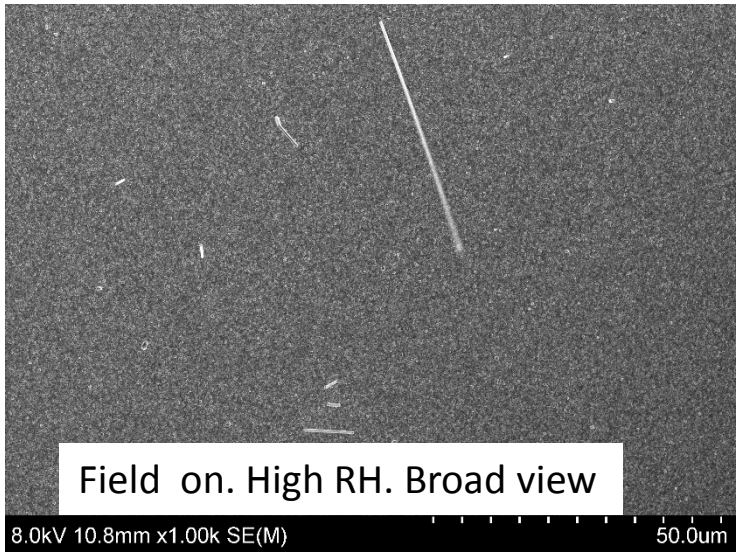
Comparison: Erika R. Crandall, Ph.D. Thesis, Auburn 2012

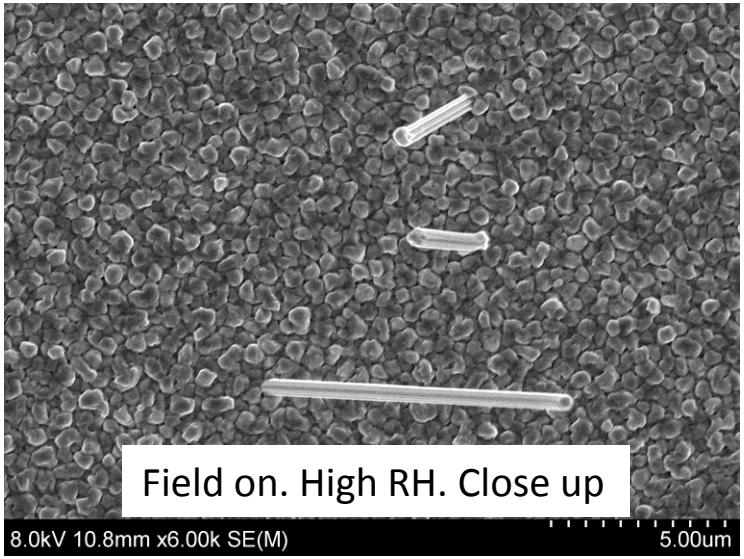
Table 2: Whisker Statistics on Ultra-Thin Sn Films after 140 Days of Incubation

Sn Film Thickness (Å)	Polished (P) Unpolished (U)	Whisker Density (cm^{-2})	Average Whisker Length (μm)	Standard Deviation (μm)	Mode
375	P	5,868	73.5	112.4	14
	U	0	0	0	0
750	P	6,758	36.8	74.5	5
	U	1,048	16.5	16.4	5
1125	P	6,339	18.7	20.5	5
	U	1,938	7.6	4.4	7
1500	P	1,729	14.2	23.7	5
	U	0	0	0	0

Acceleration ratio ***a* = 300**
For the same whisker size
IF we compare to polished,
Much Higher for unpolished

November 2014 field experiments: colder & drier

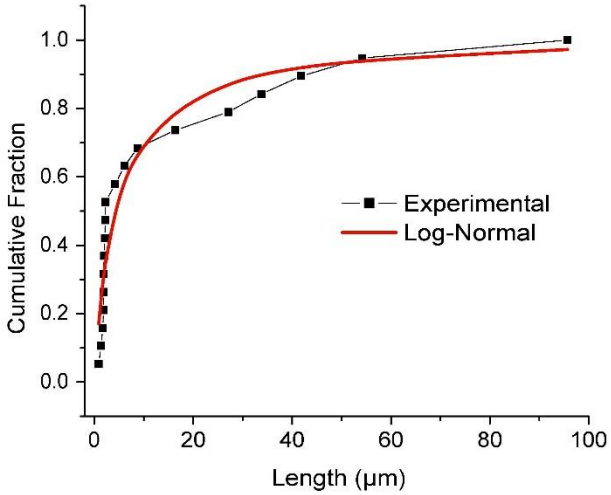




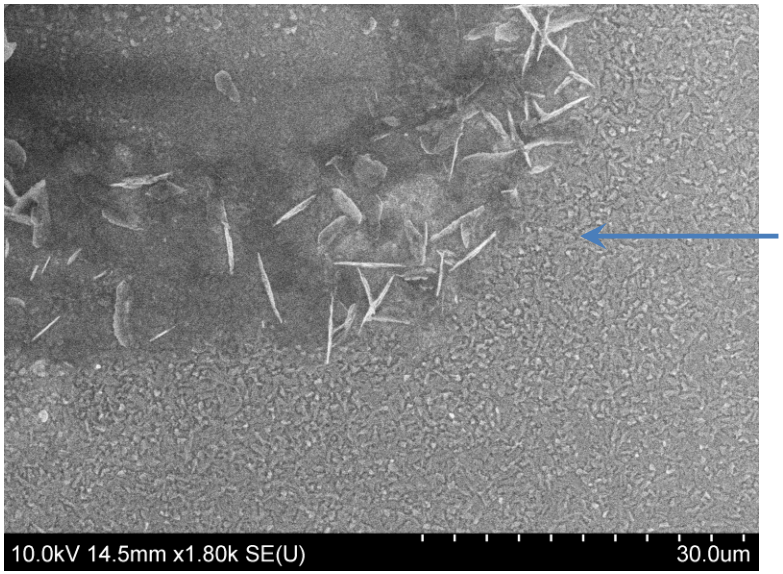
Field induced whisker growth required controlled humidity:
High RH vs. Low RH

Humidity	Whiskers/cm2	Whiskers/cm2
High RH=75%	43,000	<300
Low RH=40%	0	0

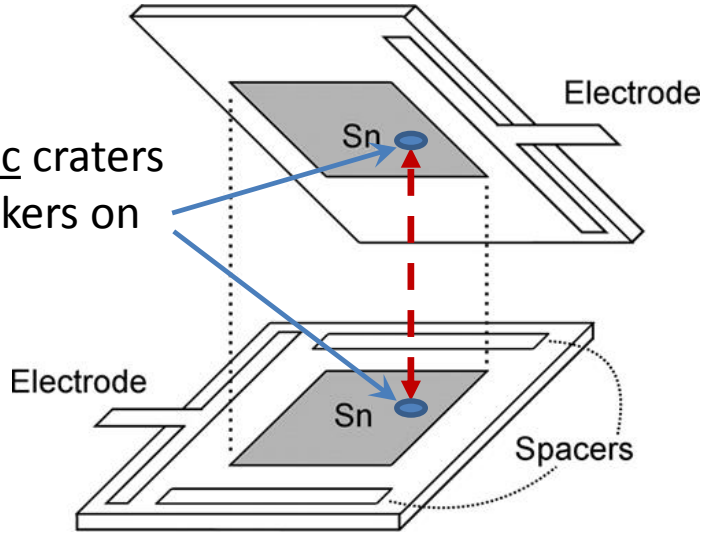
Acceleration ratio α = 400



Evidence of explosive whisker growth in sputtered Sn films



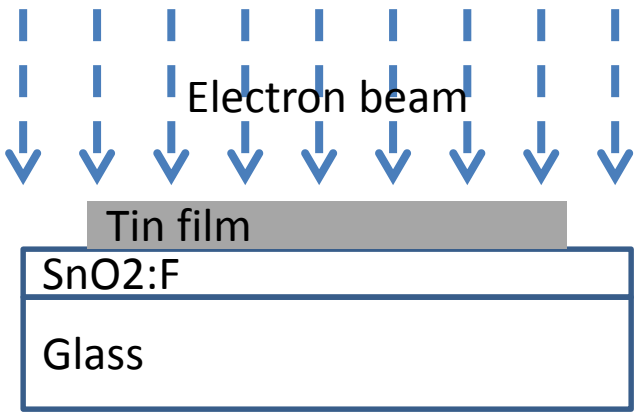
Symmetric craters with whiskers on the rim



Interpretation:

- Fast growing whisker shorts between up and down films leading to strong electric discharge with enough energy to create craters with smaller whiskers around.
- Can be quantitatively described by the electrostatic theory of metal whiskers (see back-up slide).

Linear Medical Accelerator: e-beam, sputtered Sn 0.15 μm thick

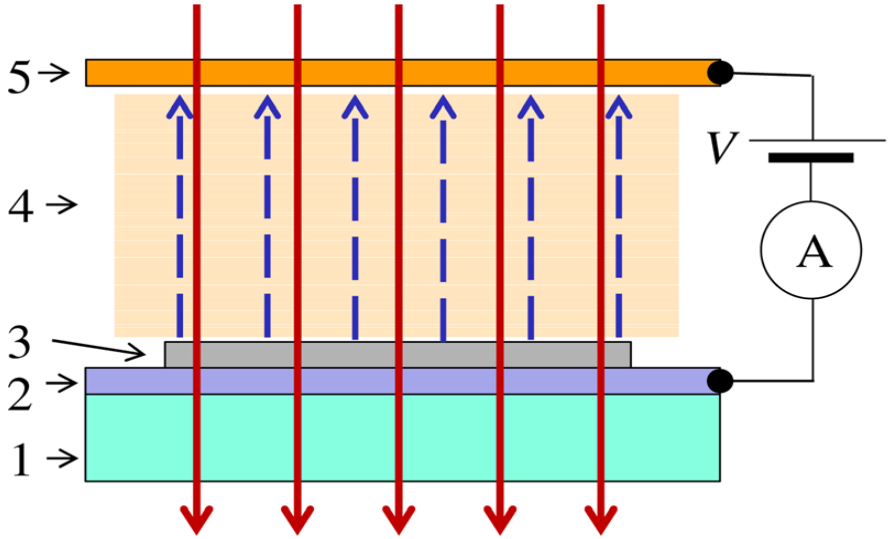


- Glass substrate thickness 3mm
- Additional plastic insulator beneath
- 6 MeV electron range in glass is ~2cm
- Electric field is due to the trapped charges in a glass created under the beam

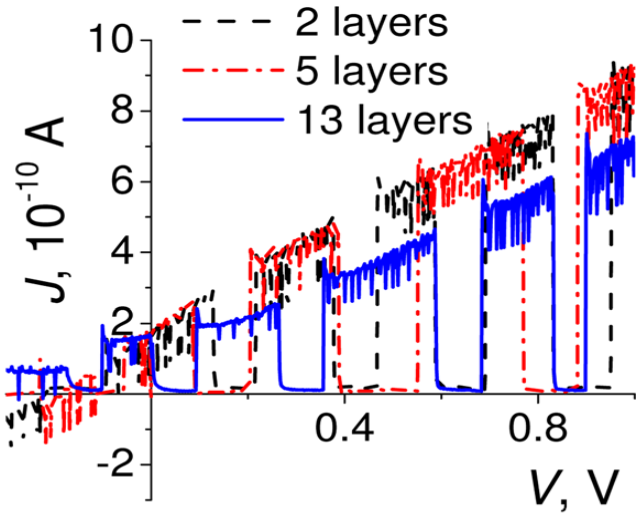
Accelerator setup

- Sample mounted on a 3mm thick piece of glass
- Irradiated for the total time of 20 hours in multiple sessions
  - Evaluated after 10 and 20 hours
- Beam current 1 nA, electron energy 6 MeV
- Electrons penetrate through the sample and glass support

In situ characterization



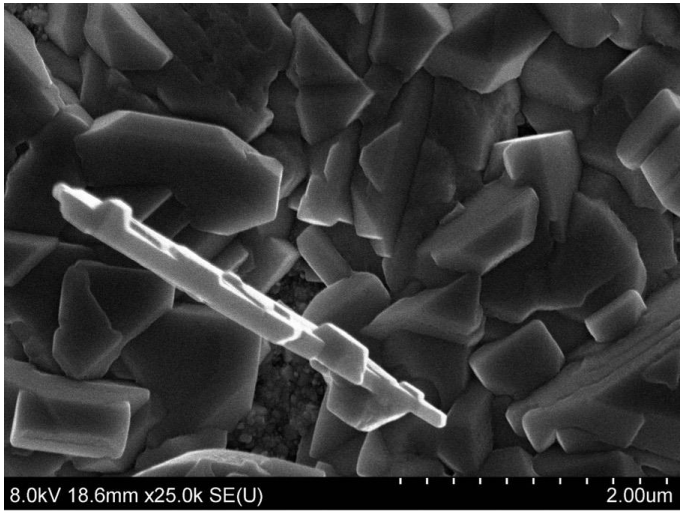
(a)

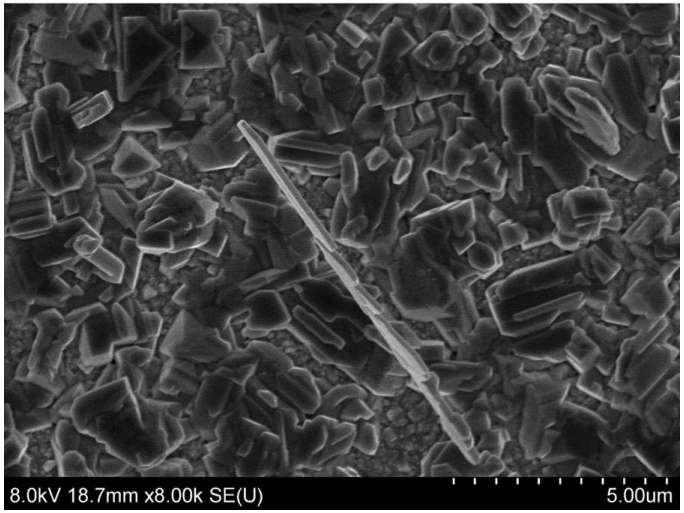


(b)

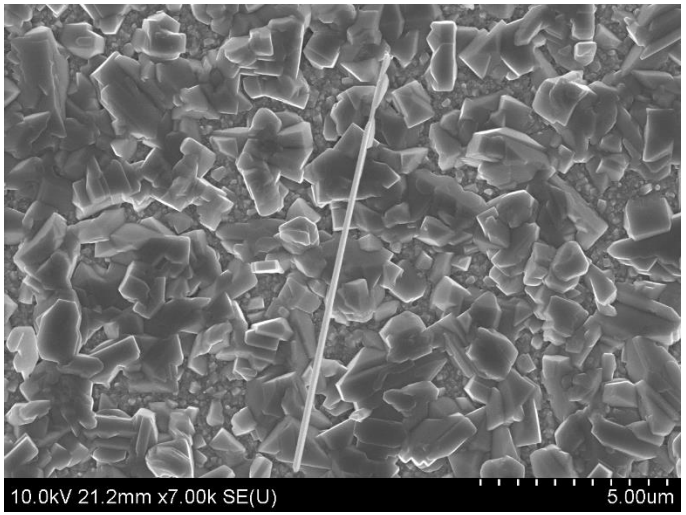
- 1-glass substrate, 2-conductive oxide, 3-tin, 4-spacer with plastic sheets, and 5-foil electrode
- Current-voltage characteristic under e-beam showed:
  - field is present only during the beam on time;
  - electric current effects negligibly small
  - substrate charge is negative

Electron beam stimulated whiskers

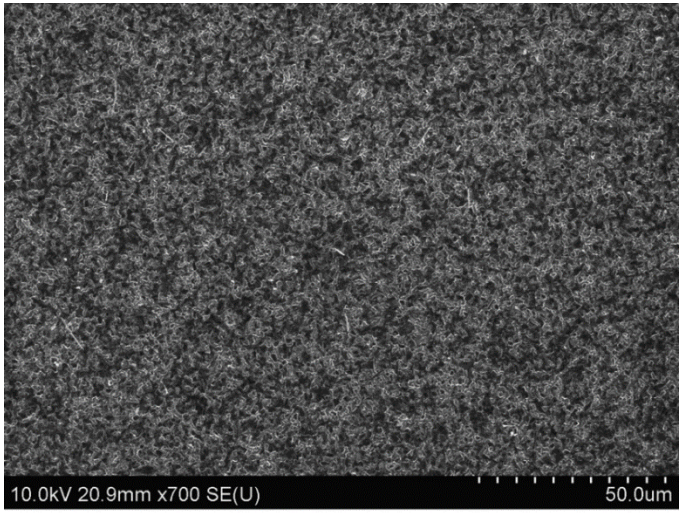


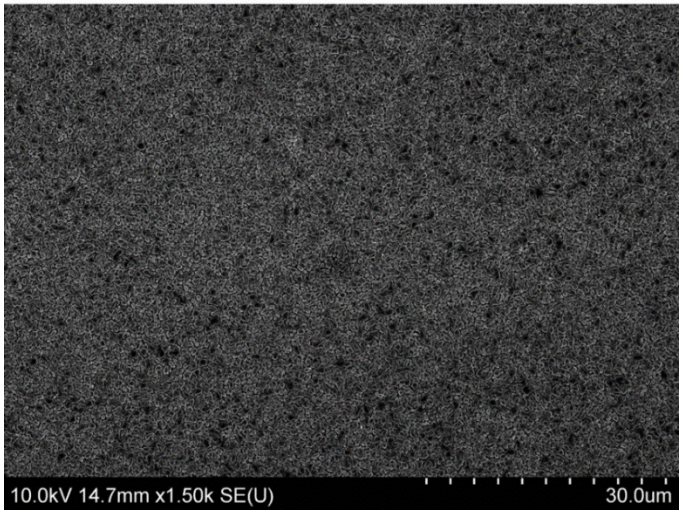


- Representative whiskers grown after 10 hrs under 6 MeV e-beam
- No whiskers observed on the shelf sample



E-beam stimulated whisker observations





- Top: irradiated sample, 10 whiskers of lengths below 4.7 μm counted:

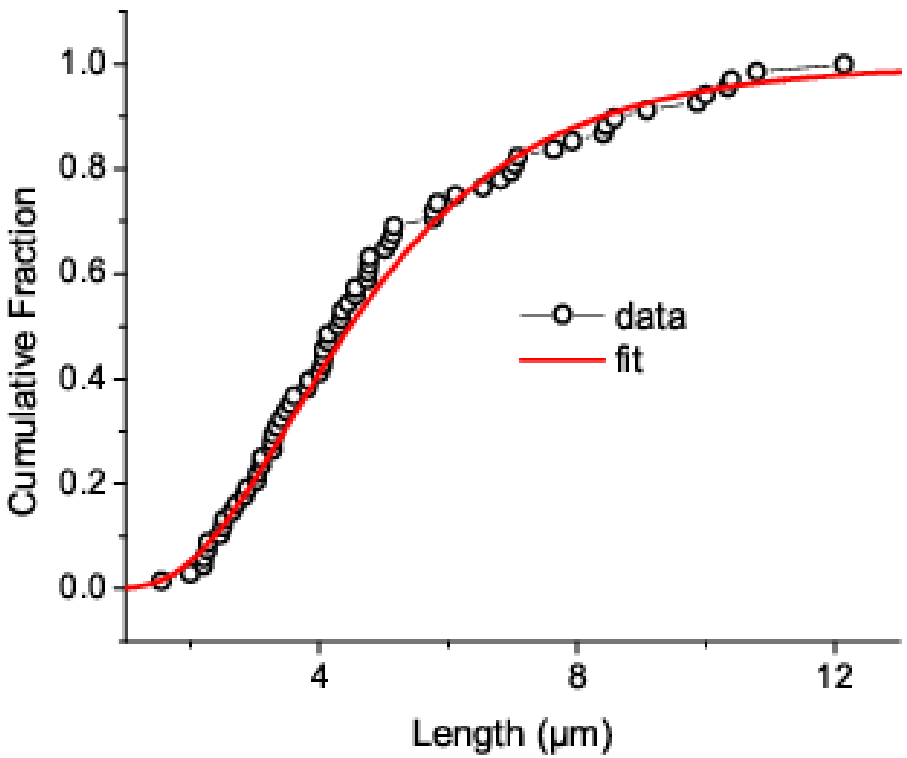
  400 whiskers/mm^2

  Acceleration ratio, a= 5,000

- Whiskers much shorter and more slender than the spontaneously grown

- Bottom: a comparable area in the control sample showing no whiskers

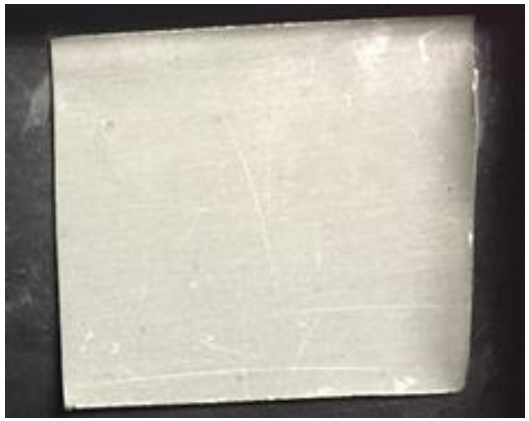
Log-normal distribution holds



- Slender faceted morphology consistent with the strongly accelerated growth rate. Rapid growth in strong electric fields: no time for surface smoothing
- High acceleration ~ 5,000 for whisker concentration
- Modest growth rates of ~ 1A/s

Zn-plated floor samples
(courtesy Jay Brusse, NASA NEPP program)

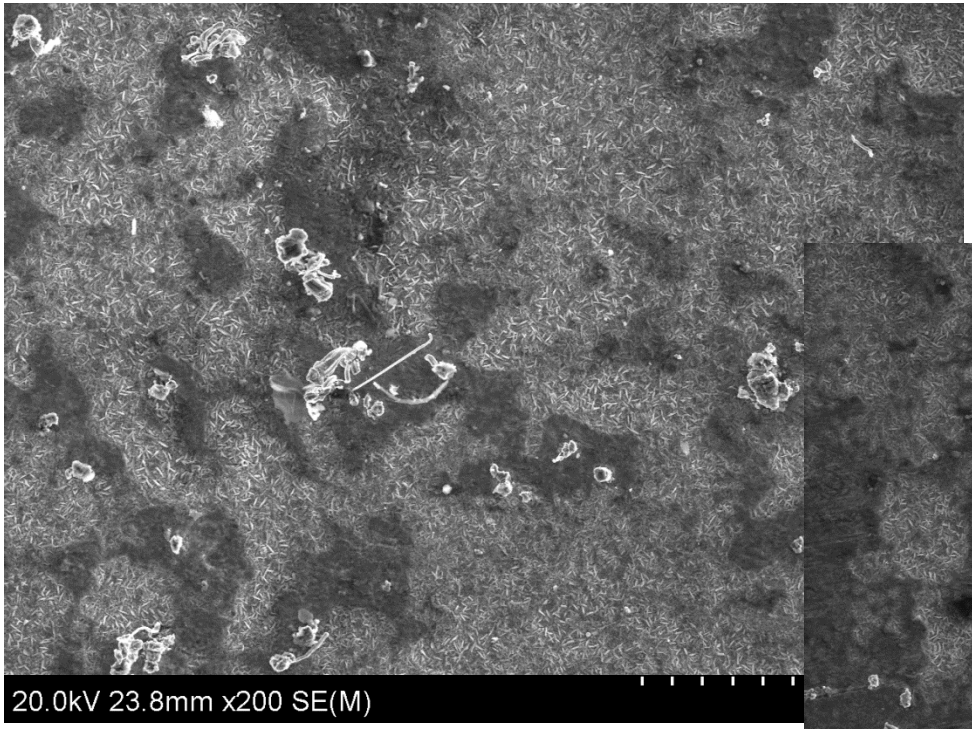
- A 1 mm thick piece of Zn-plated SS raised floor
- As-received sample shows numerous whisker
- Sample cut into multiple cells (~0.5”x0.5”)
- Control cell reserved
- Others subjected to various field sources:
  - Accelerator e-beam 6 MeV
  - SEM e-beam 10 keV
  - Van der Graaff generator



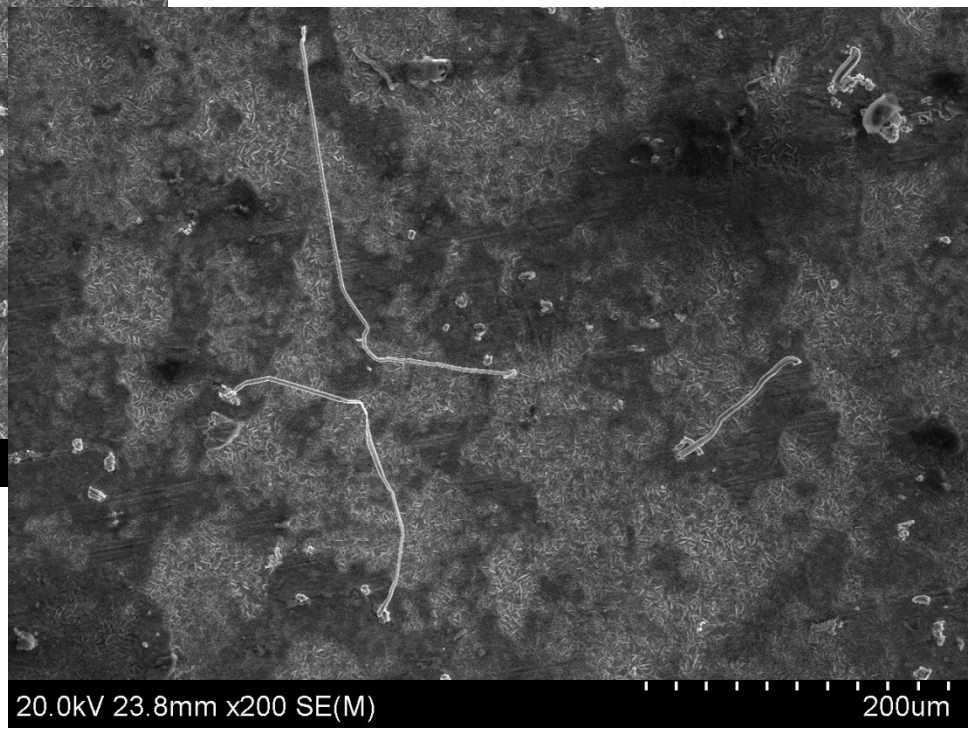
SEM evaluation details

- All samples evaluated under Hitachi S-4800 SEM
- Imaging area 0.28mm^2, 20kV electron energy, x200-220 magnification
- Evaluated 40 areas per sample
- Total duration of project ~ 2 months
- Stimulation times varying between 8 and 20 hours

Control sample

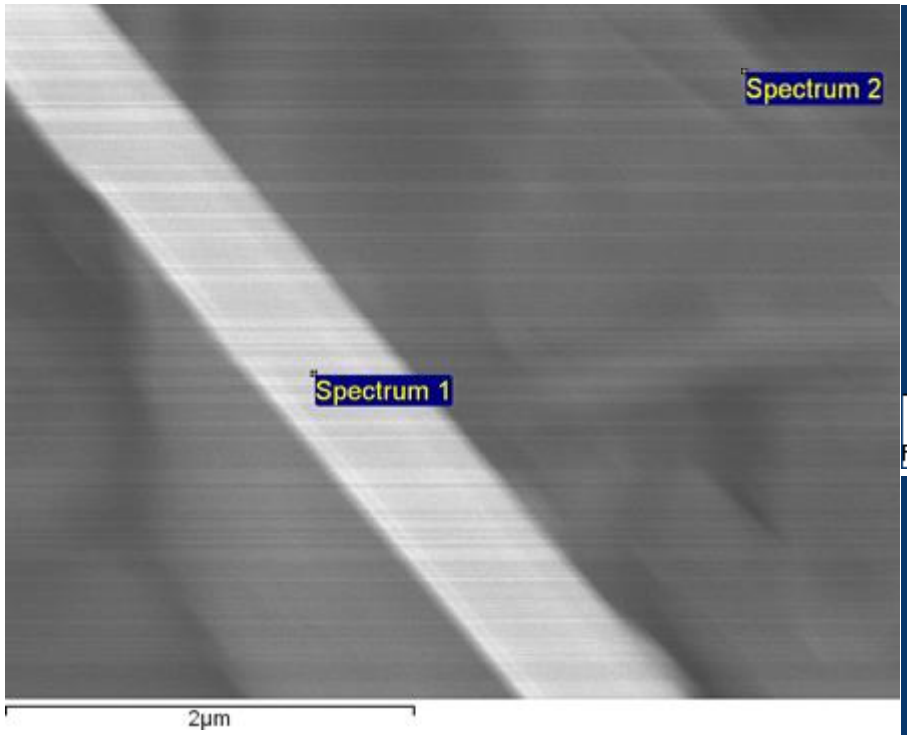


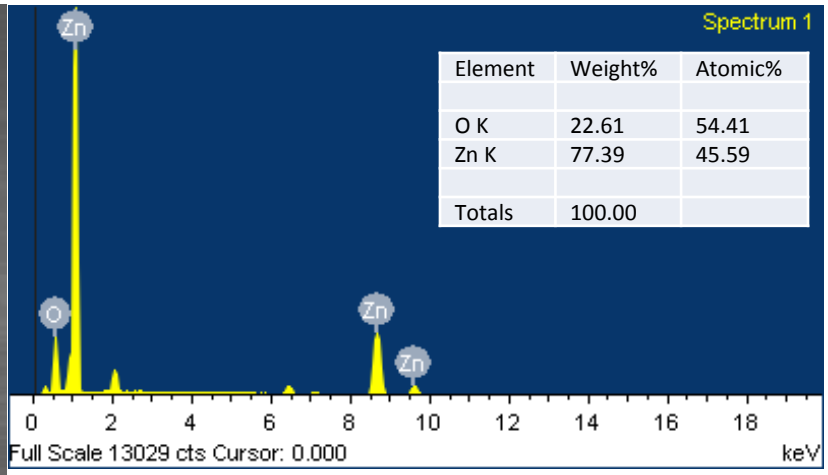
Typical image



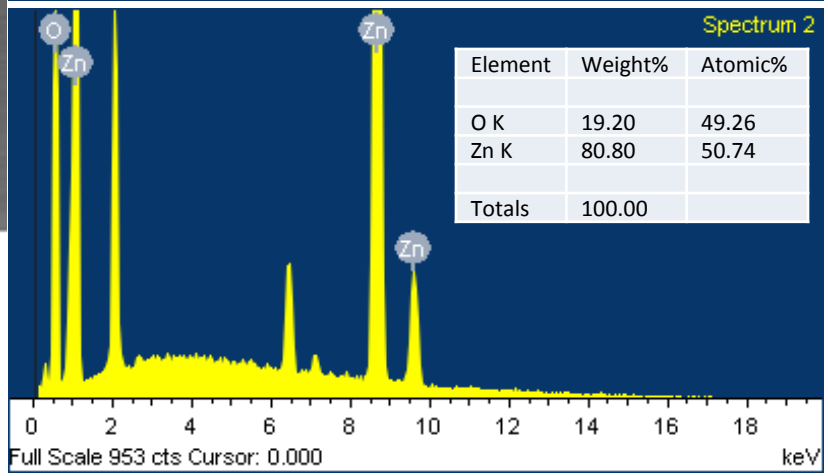
Area with multiple long whiskers

Compositional analysis





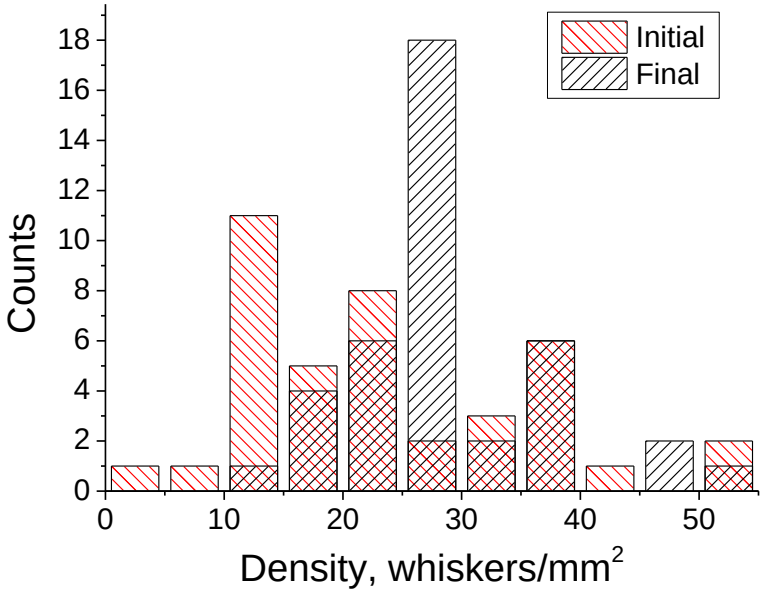
Element	Weight%	Atomic%
O K	22.61	54.41
Zn K	77.39	45.59
Totals	100.00	



Element	Weight%	Atomic%
O K	19.20	49.26
Zn K	80.80	50.74
Totals	100.00	

ZnO
Whisker: 46%Zn, 54%O
Base metal: 51%Zn, 49%O

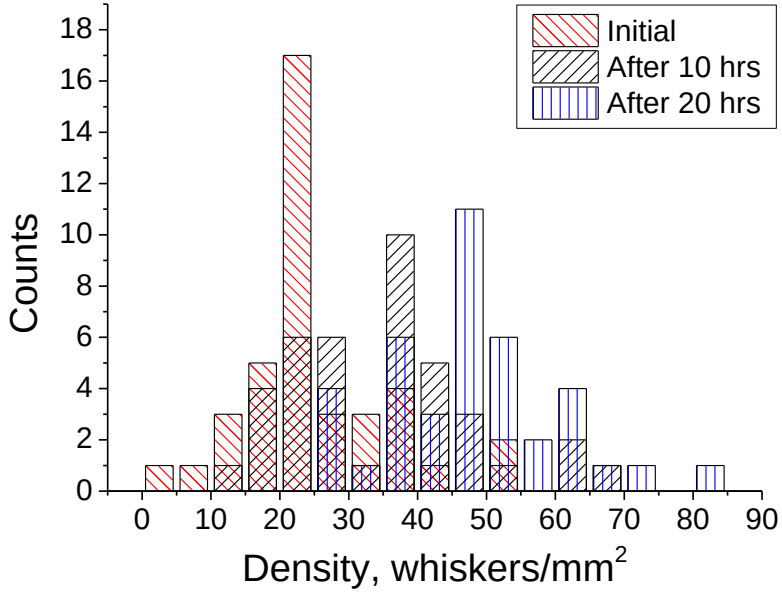
Control sample average concentration of whiskers increased during 2 months of hot humid summer



- Control sample whisker density
  - Initial
  - Final - after 40 days on a shelf

Initial density (Whiskers/mm^2)	Final density (Whiskers/mm^2)	Av. percent increase
23.5 ± 11.9	28.8 ± 8.8	22.6%

Medical accelerator e-beam whisker counts

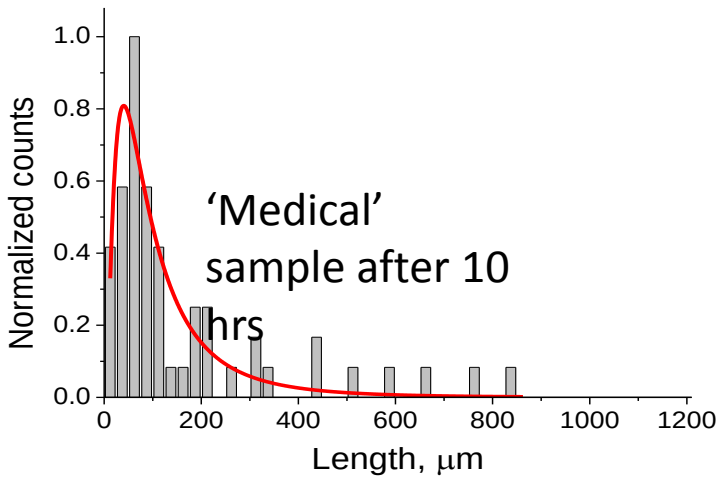


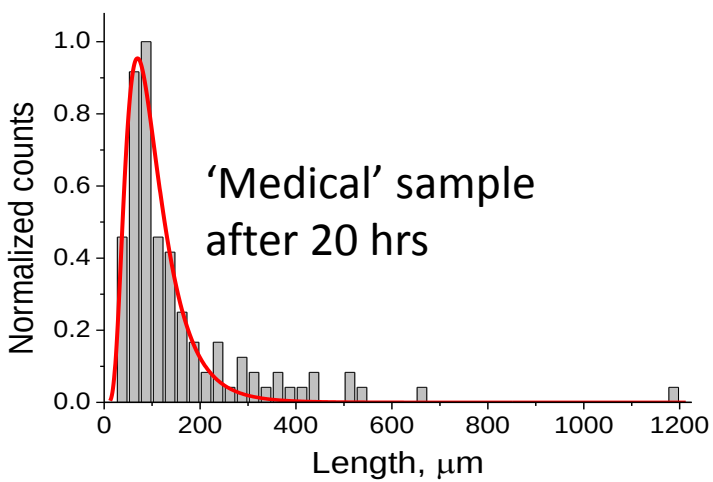
Initial density (Whiskers/mm^2)	After 10hrs (Whiskers/mm^2)	Av. percent increase
25.1 ± 10.6	34.6 ± 12.9	37.8%
Initial density (Whiskers/mm^2)	**After 20hrs (Whiskers/mm^2)**	**Av. percent increase**
25.1 ± 10.6	48.3 ± 12.3	92.4%

- Whisker counts statistics:
  - Initial
  - After 10 hours under 6MeV electron beam
  - After 20 hours under 6MeV electron beam
  - Significant increase in whisker concentration
- *Acceleration ratio*

$a \sim 3000$

Length statistics: Evolution under 6 MeV e-beam



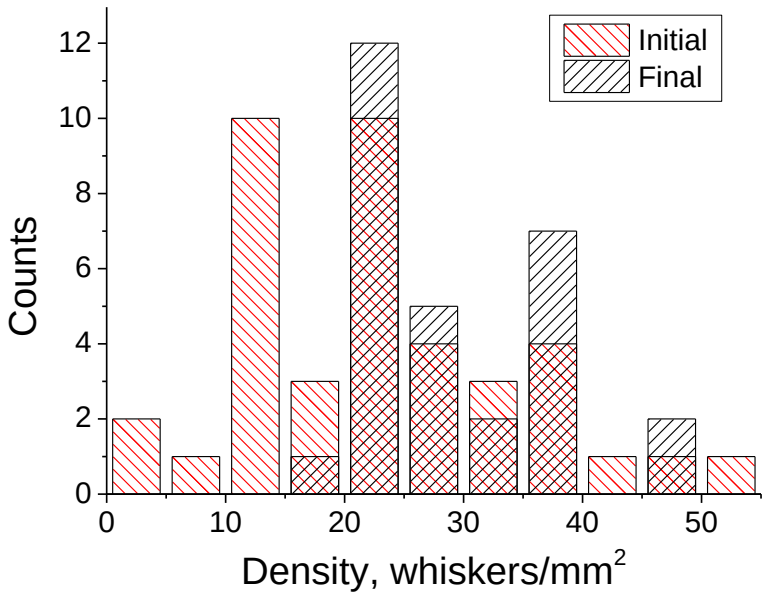


- Log-normal fits hold
- Average whisker length increased:
  - from 40 μm (after 10 hours)
  - to 74 μm (after 20 hours)

SEM sample: grounded

- Taped directly to the metal stand of SEM with conductive carbon tape
- Irradiated for total time of 10 hours
- Beam current 10 μA, e-beam energy 10 keV (~0.7 μm range in ZnO)

SEM sample: grounded



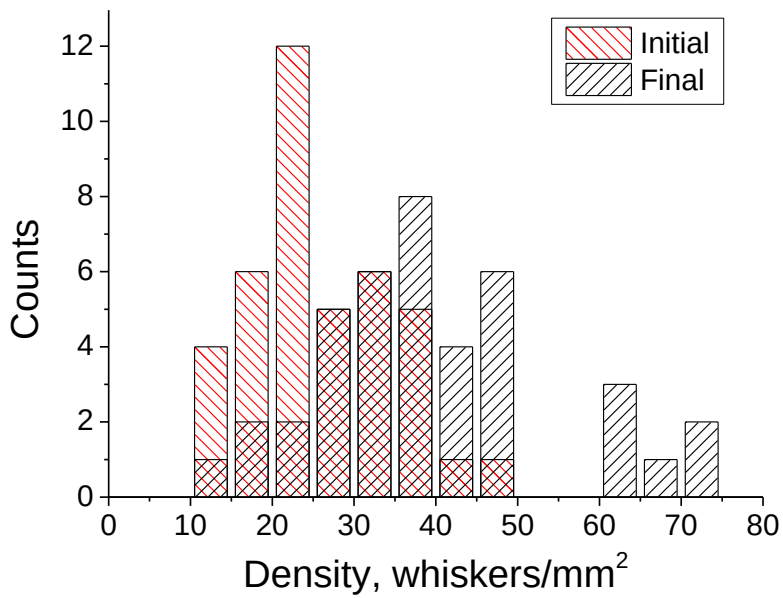
Initial density (Whiskers/mm^2)	Final density (Whiskers/mm^2)	Av. percent increase
23.1 ± 11.5	30.0 ± 7.4	29.9%

- Initial sample statistics
- Final - grounded sample after 10 hours under SEM electron beam
- Slight increase in whisker concentration (comparable to shelf sample)
- In much shorter time,
- *Acceleration ratio a~* 200

SEM sample: insulated

- Mounted on a 3mm thick piece of glass for the time of irradiation
- However taped to the metal SEM stand with carbon tape for measurements
- Irradiated for total of 10 hours
- Beam current 10 μA, electron energy 10 keV

SEM sample: insulated



Initial density (Whiskers/mm^2)	Final density (Whiskers/mm^2)	Av. percent increase
26.0 ± 8.5	39.7 ± 14.3	52.6%

- Initial sample statistics
- Insulated (ungrounded) sample after 10 hours under SEM electron beam
- Significant increase in whisker concentration, much greater than for grounded/shelf samples
- In much shorter time,
- *Acceleration ratio a*~ 500

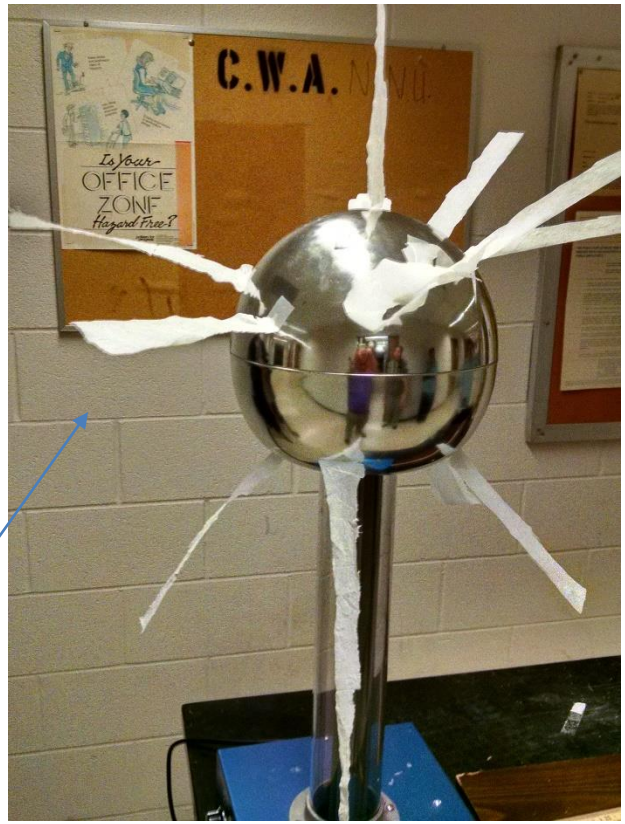
Ion beam irradiation using UT linear accelerator

- Mounted on a 3mm thick piece of glass
- Smaller size, ~0.25”x0.25”
- Bombarded with tin ion beam for 1 hour
- Beam current 100 nA, ion energy 130 keV

- **Current status: pending**. To redo the experiments avoiding mechanical sample damage incurred in earlier rounds

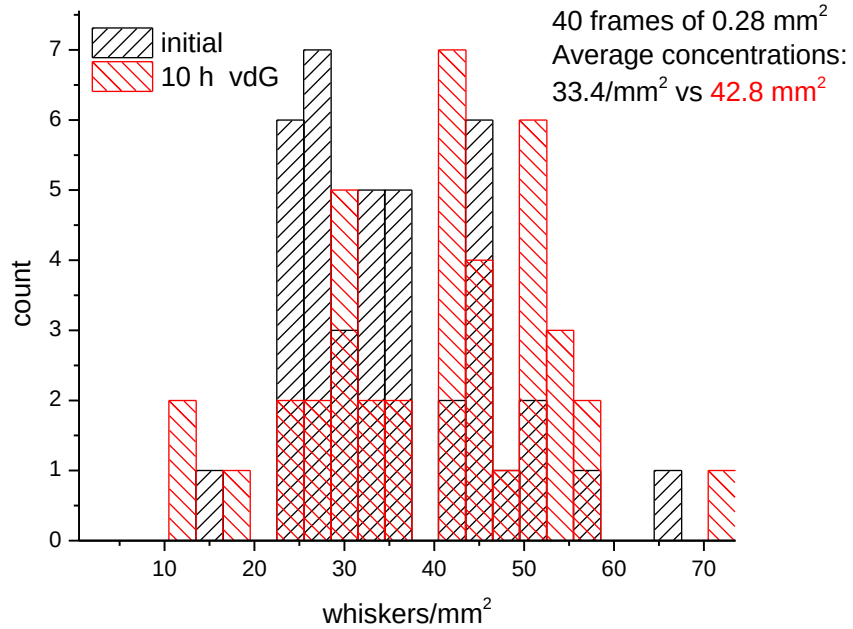
Van der Graaff generator experiment

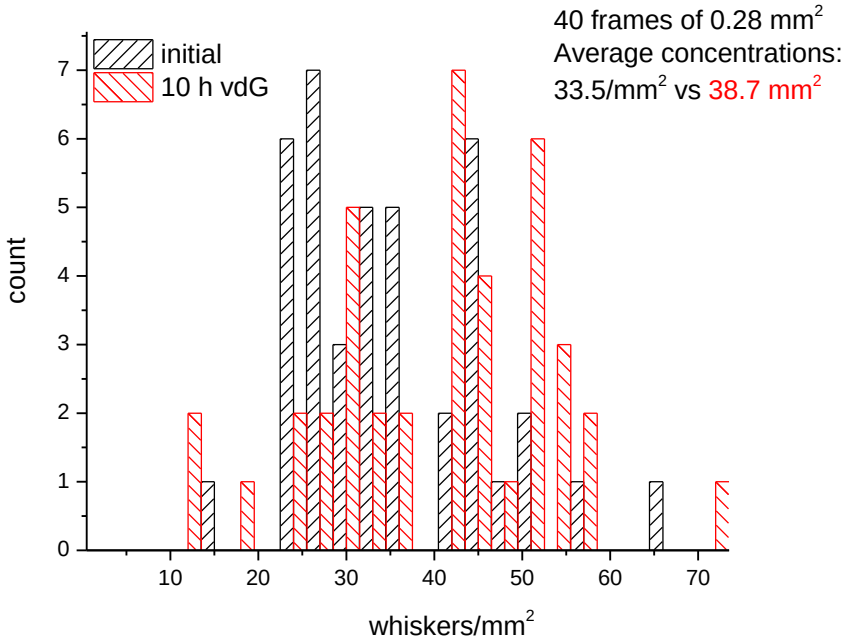
- The sample was taped directly to the ball surface
- Field up to 200,000 V/cm
- Duration: 10 hours
- Standard lab settings



Paper stripes along the field lines

Van der Graaff generator effect



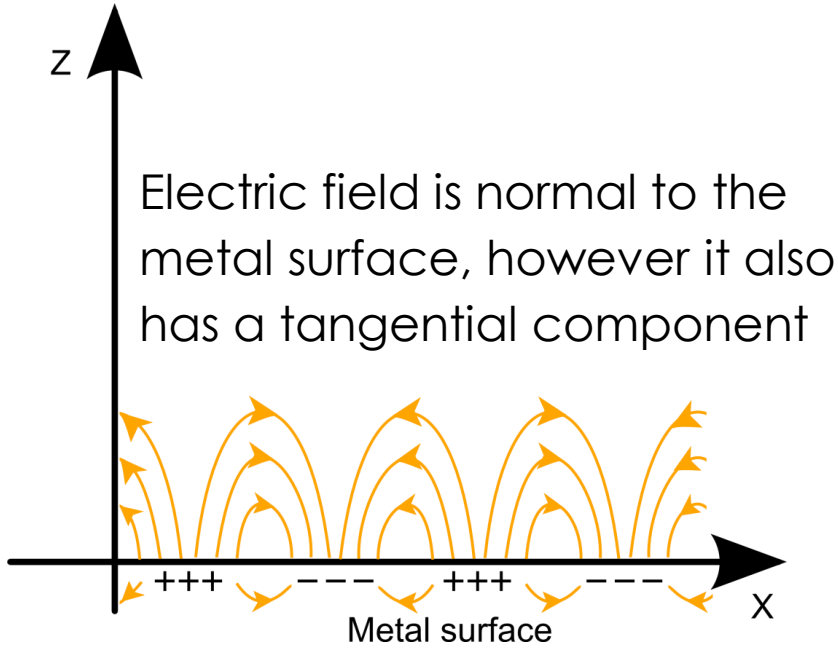


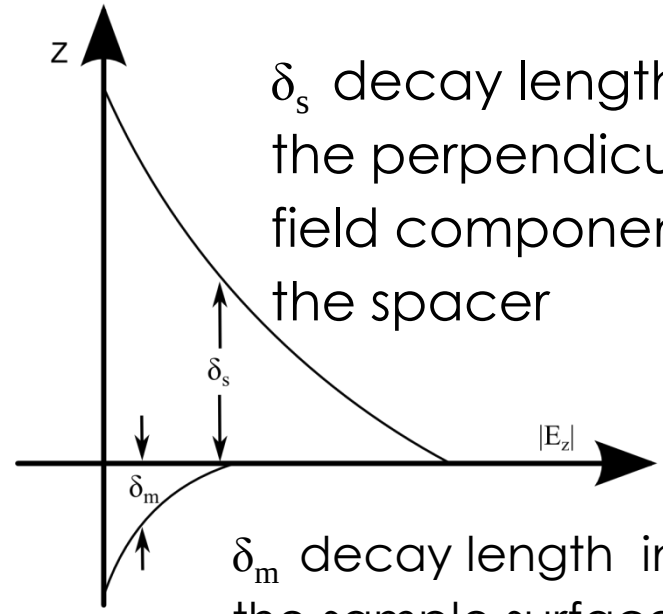
~ 30% and ~ 18% increase in average whisker concentration in two samples over 10 hours

Standard deviations practically unchanged

Acceleration ratio a~ 200

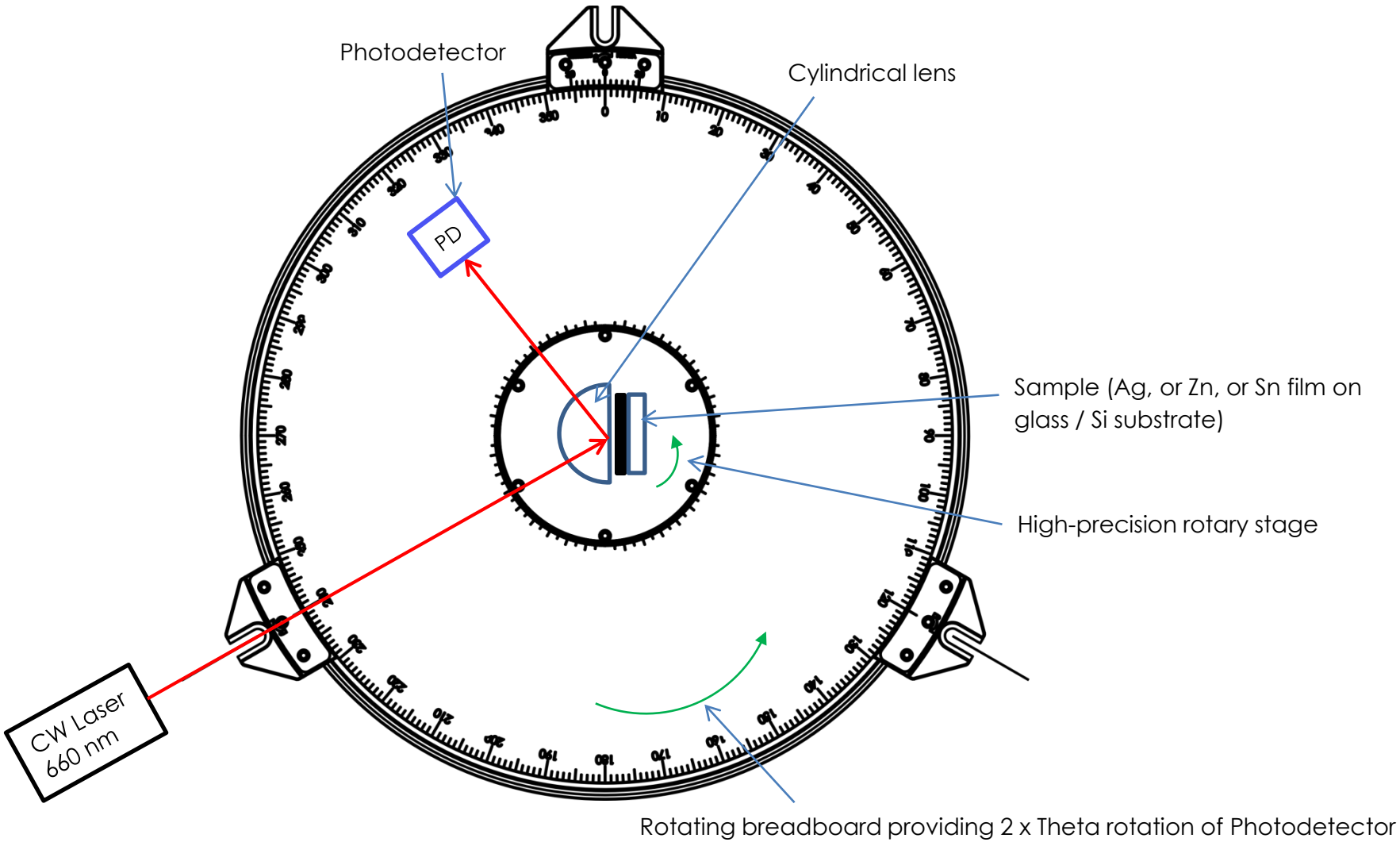
Surface plasmon polaritons (SPPs): gigantic (~1000) field enhancement



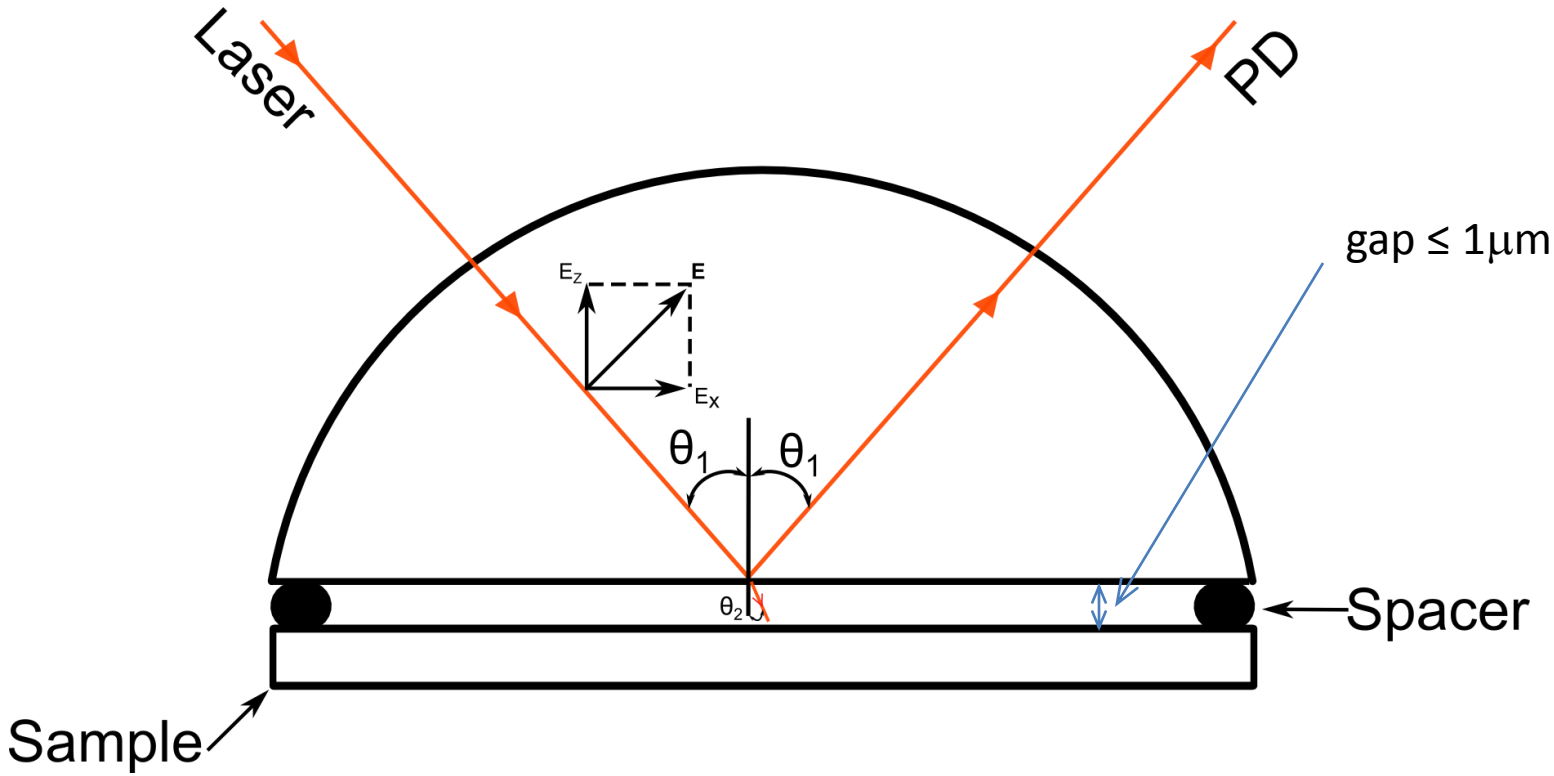


Requires special excitation geometry

SPP excitation: Experimental Setup (Otto configuration)

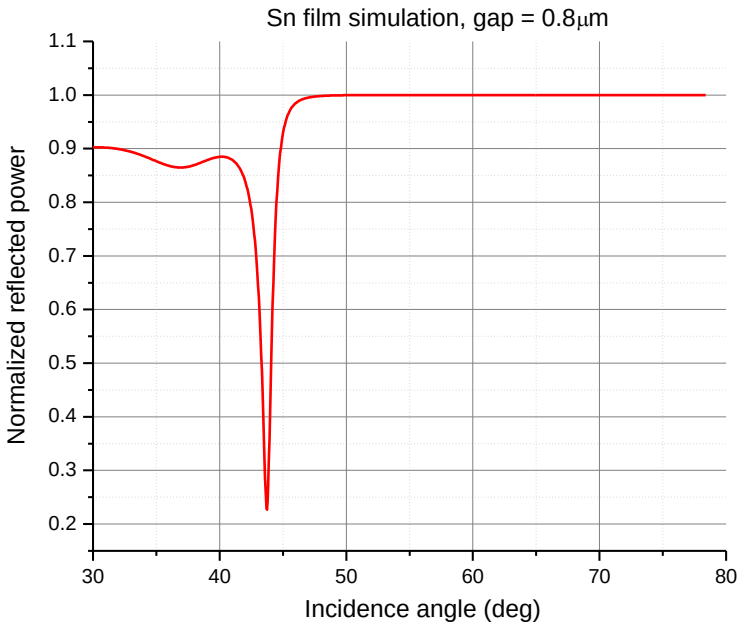


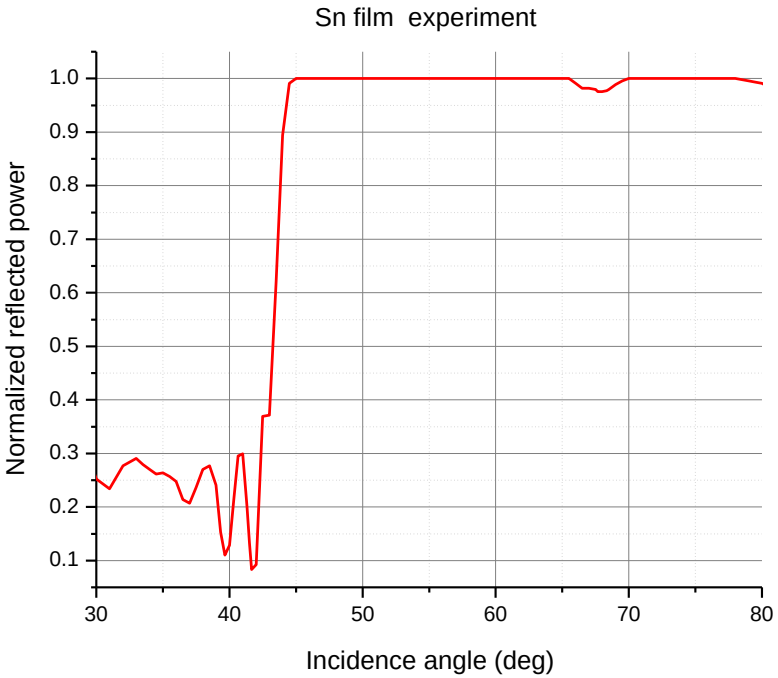
Otto configuration for SPP excitation



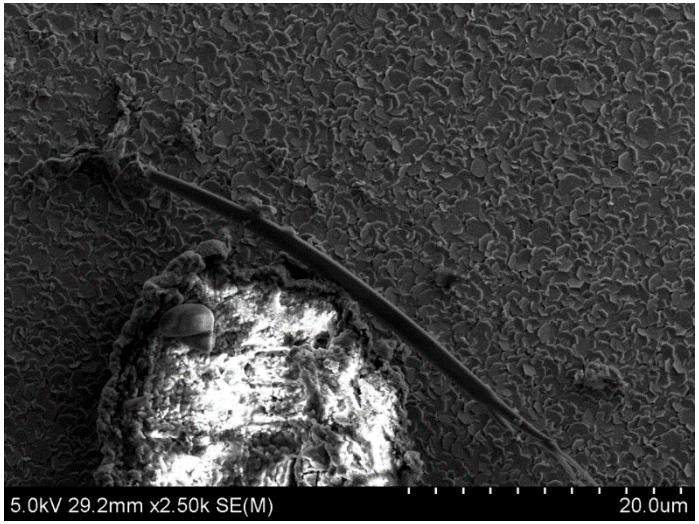
Reflectance vs. angle of incidence (Sn films)

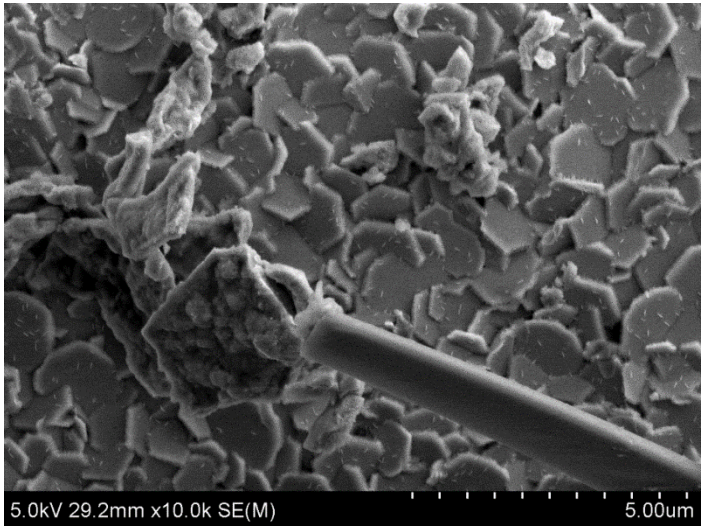
SPP dip is shifted significantly from the TIR critical angle due to the metal's low conductivity and other factors. This is also the case with Zn films.

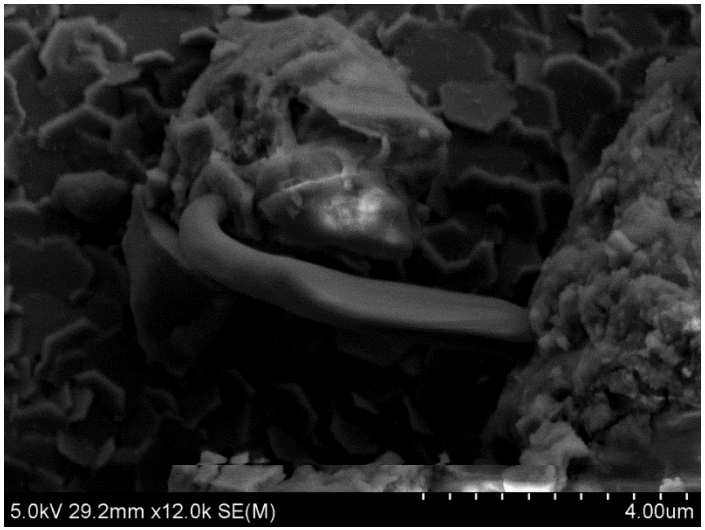




Whiskers on evaporated Zn films within the irradiation spot after several hours at the SPP angle

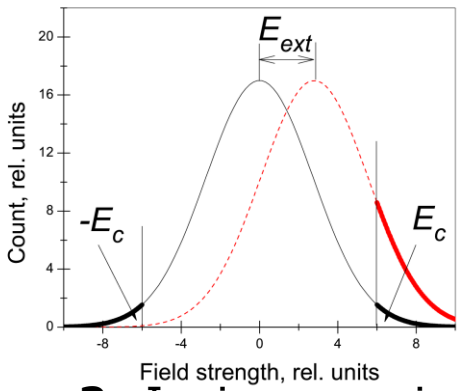






These results are not easily reproducible. Work in progress.

Discussion: masking and suppressing factors (others not seeing field effects[2])



1. In the electrostatic theory, whiskers appear just as frequently as a result of positive local random fields as they do in response to the negative ones.
The external field will enhance one random field sign while suppressing the opposite: zero integral effect is possible.

2. Ionic screening in response to the external electric bias: ions attached to the electrodes will create double electric layers limiting the electric field to nanometers.

Example: surface ion concentration ~ 10^{12} cm^{-2} will create E~ 1 MV/cm that suffices to fully balance the external bias field. Ions in electronic assemblies can mask the external field effects for pitched electrodes, etc.

3. Insufficient effort to discriminate between the effects of electric current vs. electric field.

[2] R.D. Hilty, N. Corman, and H. Herrmann, IEEE Trans. Elec. Packag. Manuf. 28, 75, (2005).
S. Meschter, P. Snugovsky et. al., 8th CALCE/IPC International Tin Whisker Symposium (2014).

Concluding remarks

- General conclusion: electric field promotes whiskering
- Acceleration factors in hundreds and thousands
- Quantitative data warrants further work
- Other suspected drivers (stress, etc.) reducible to field effects? – calls upon further analysis
- Electroplated Sn samples need to be tested as technologically most important (underway)
- Other groups' inputs would be extremely helpful
- The existing scarce data showing no electric field effect on whisker growth can be hopefully reconciled with our findings via various masking factors

Acknowledgements

- Jay Brusse (NASA) – Zn samples and discussions
- Andrew Kostic (Aerospace Corporation) – discussions and moral support
- Tin whisker teleconference (Bill Rollins et. al.) – continuous stimulating discussions
- Al Compaan (Lucintech) – deposition equipment
- D. Strickler (Pilkington) – TEC glass substrates
- Center for Materials and Sensor Characterization at the University of Toledo.
- NRC award No. NRC-HQ-12-G-38-0042 and NSF award No. 1066749 – partial support

Back-up slide: electrostatic theory on the explosive whisker growth

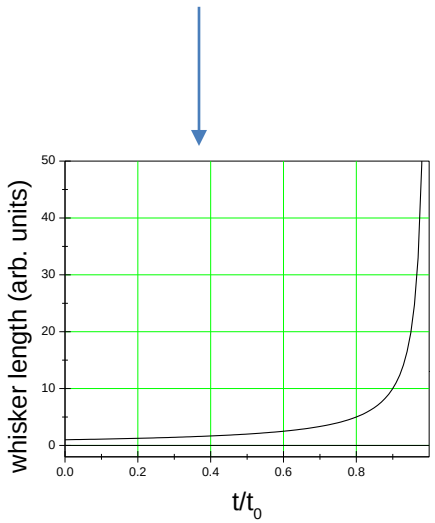
$$h = \frac{h_0}{1 - t/t_0}, \quad t_0 = \frac{3\Lambda kT}{DE^2 h_0}, \quad \Lambda = \ln\left(2h/d\right)$$

h_0 - nucleation length

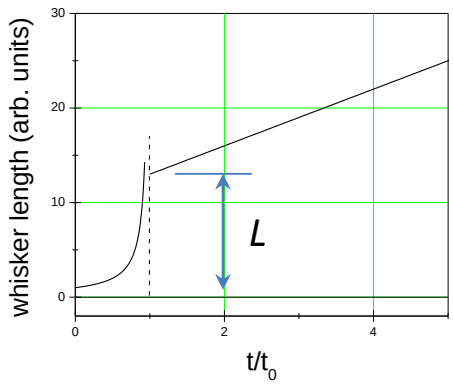
kT - thermal energy

D - diffusion coefficient

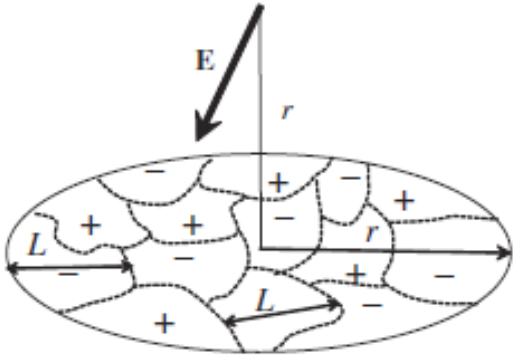
E - field strength



Explosive whisker growth in a uniform electric field [1]



Spontaneous whisker growth in the electric field of random patches [1]



Random patch (electrostatic) model[1]

[1] V. G. Karpov, Phys. Rev. Applied, **3**, 044004 (2015)