INL/CON-20-60273-Revision-0

In situ observation of short- and long-timescale material property evolution under extreme conditions

Cody Andrew Dennett

November 2020

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Idaho National Laboratory
Idaho Falls, Idaho 83415

http://www.inl.gov

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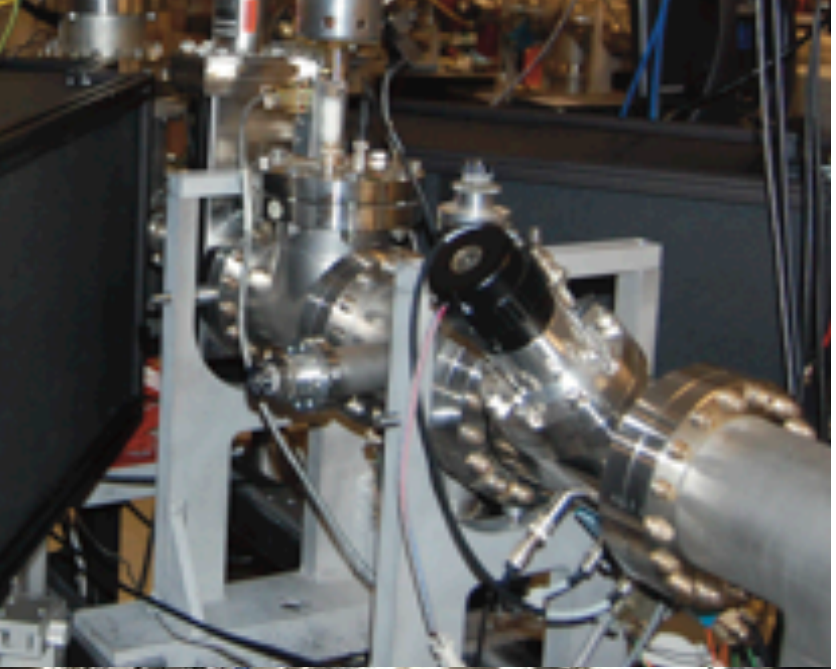

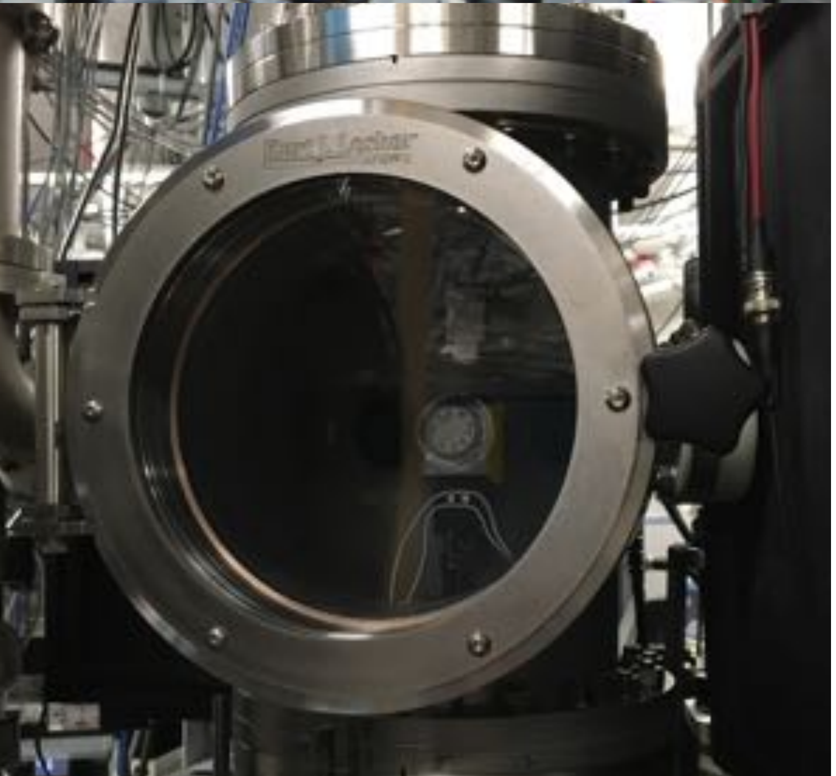

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MS&T 2020 – November, 2020

Cody A. Dennett
Materials Science and Engineering Department
Idaho National Laboratory
cody.dennett@inl.gov

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Collaborators
Benjamin R. Dacus (MIT)
Trevor Clark (SNL)
Christopher M. Barr (SNL)
Yanwen Zhang (ORNL)
Khalid Hattar (SNL)
Michael P. Short (MIT)

material performance is challenged under extreme environments

temperature
corrosives
radiation
pressure
fatigue

material performance is challenged under extreme environments

temperature

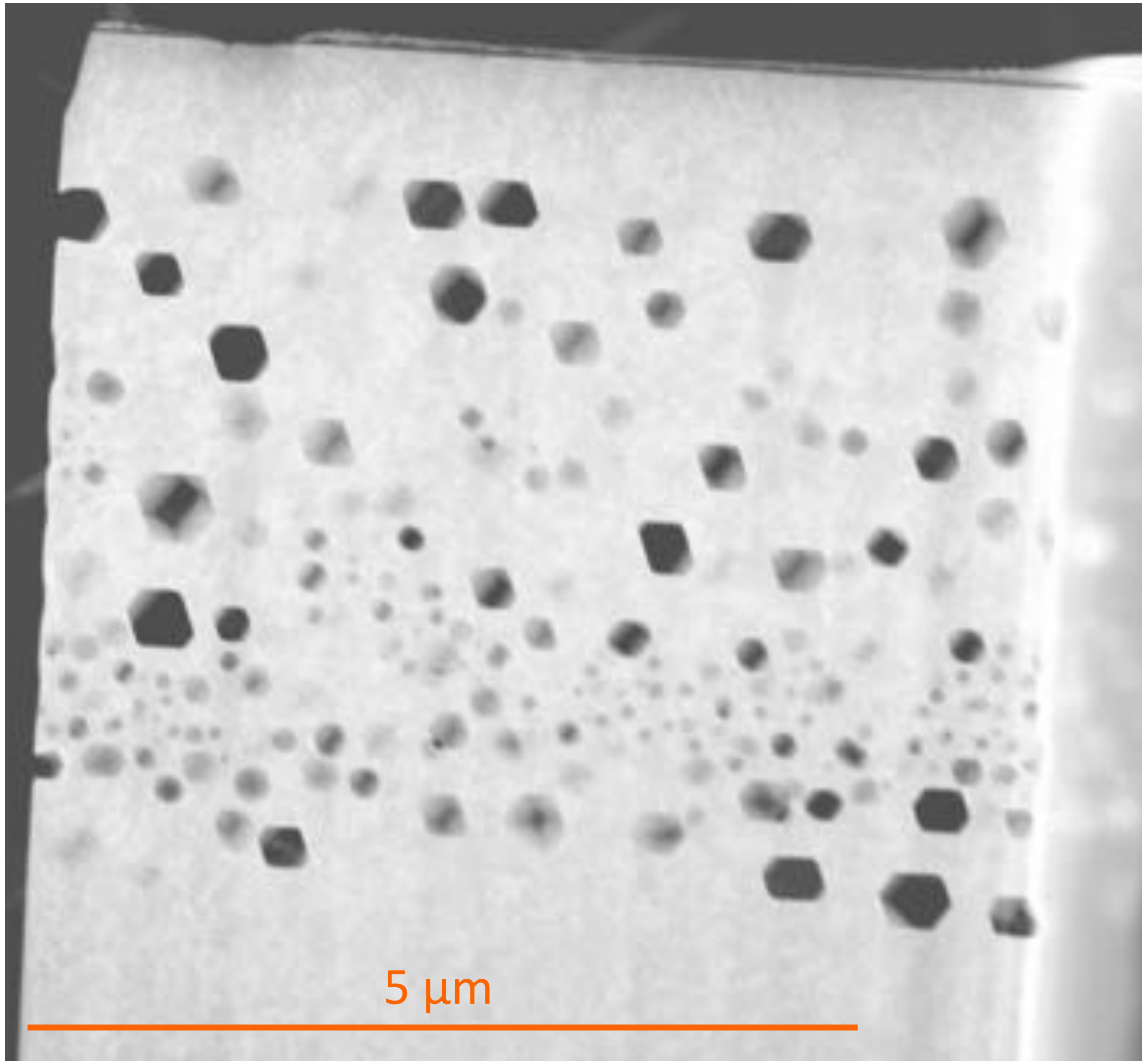
corrosives

radiation

pressure

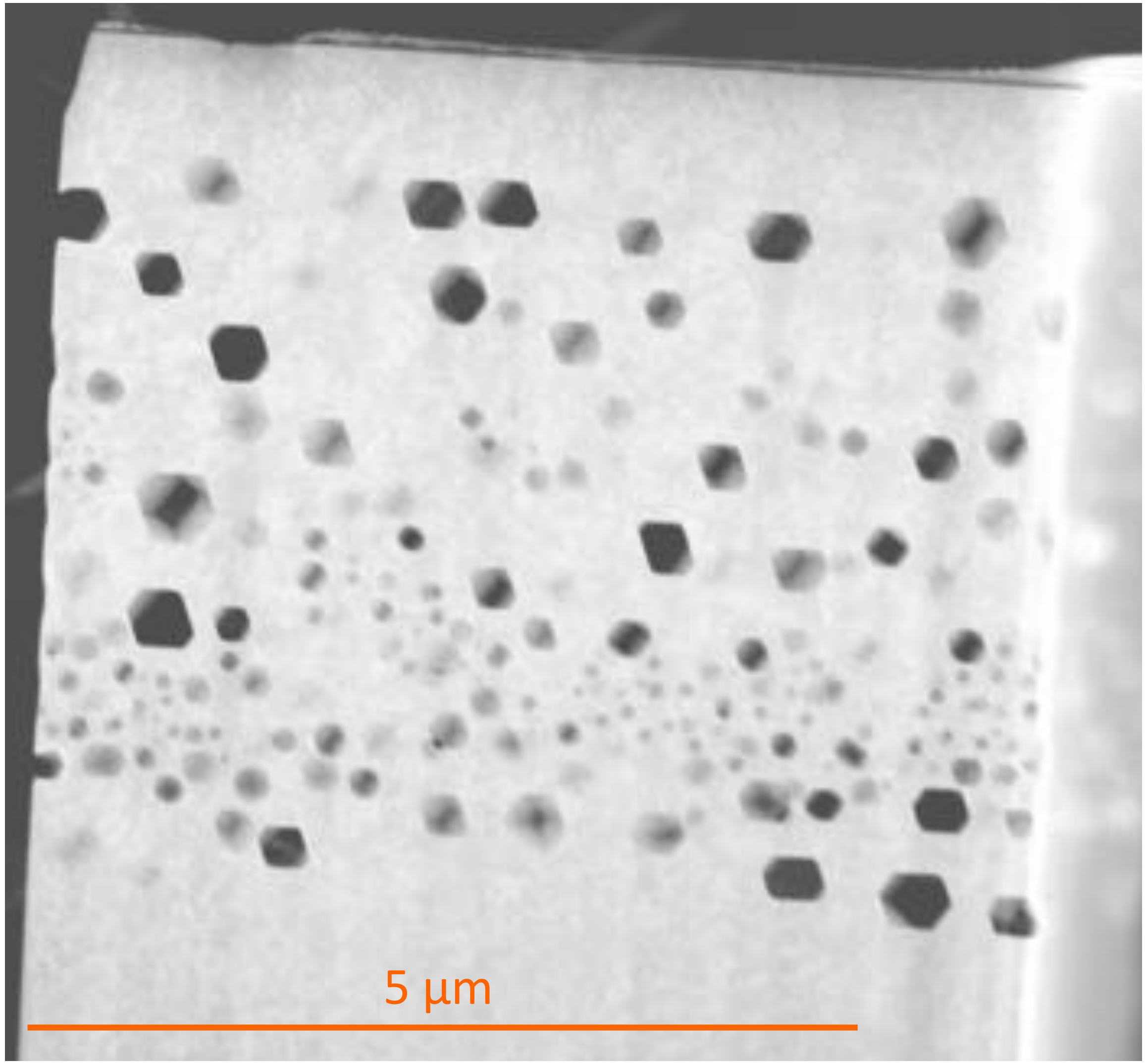
fatigue

micro-scale



Pure Cu
35 MeV Cu^{6+}
400°C

micro-scale



Pure Cu
35 MeV Cu^{6+}
400°C

macro-scale

Mansur, *J. Nucl. Mater.* **216** (1994)

in situ characterization

material structure

material properties

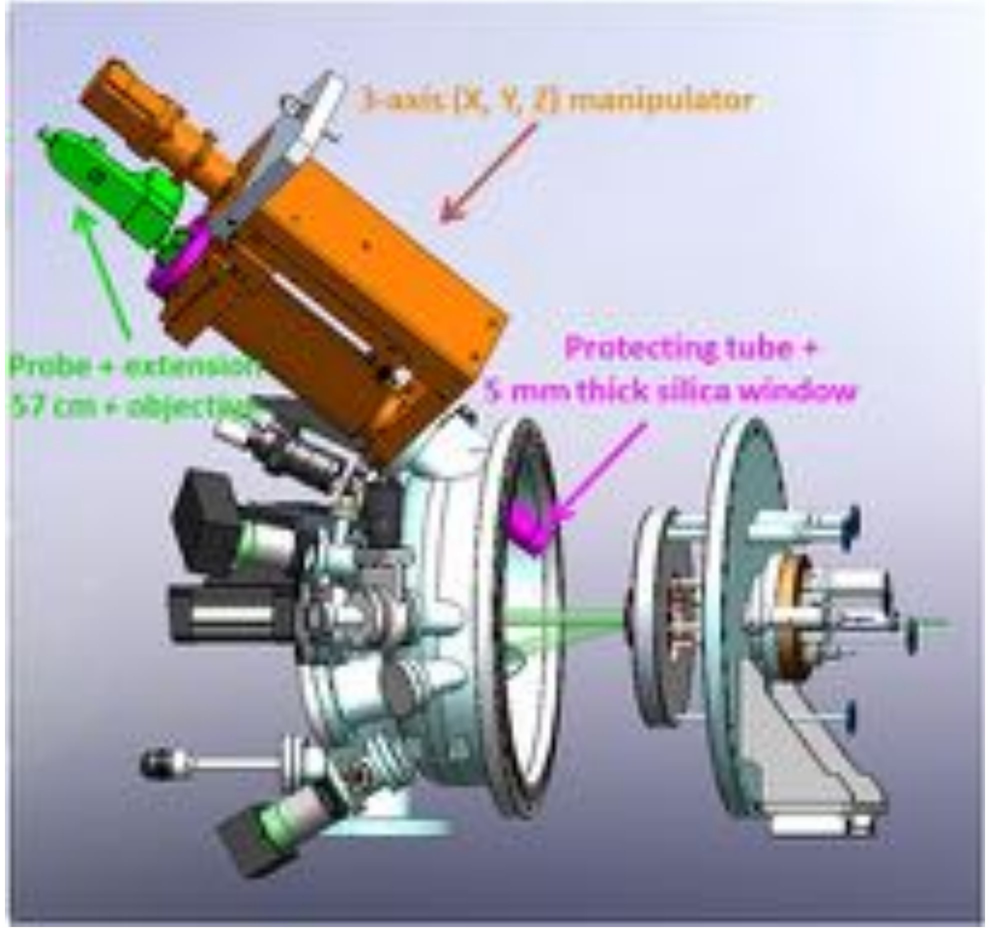
in situ characterization

material structure

material properties

in situ TEM
Sandia Ion Beam Lab

in situ Raman spectroscopy
JANUUS-Saclay



K. Hattar et al., *Nucl. Instrum. Meth. Phys. Res. B* **338** (2014)

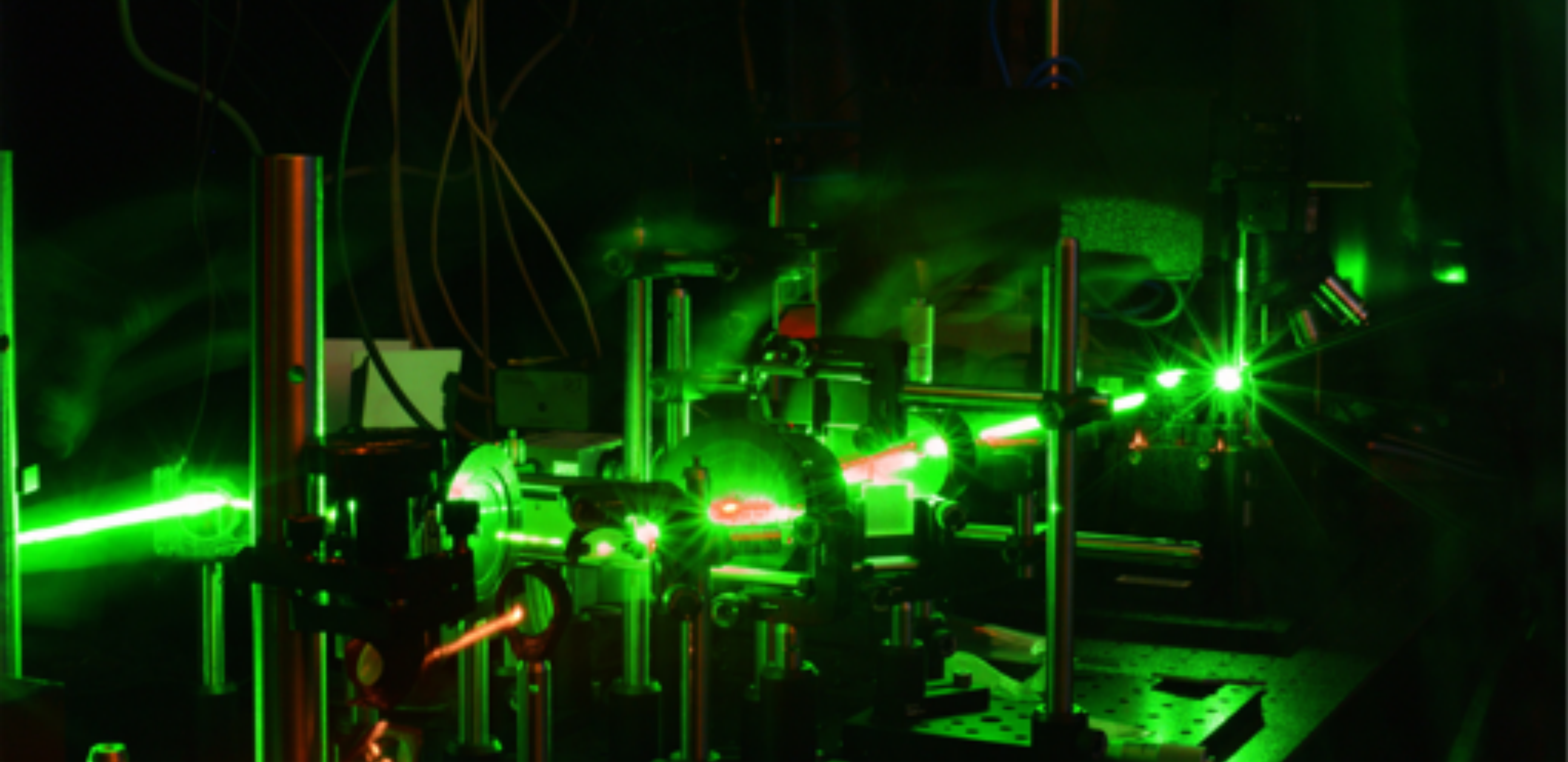
S. Miro et al., *J. Raman Spectrosc.* **47** (2016)

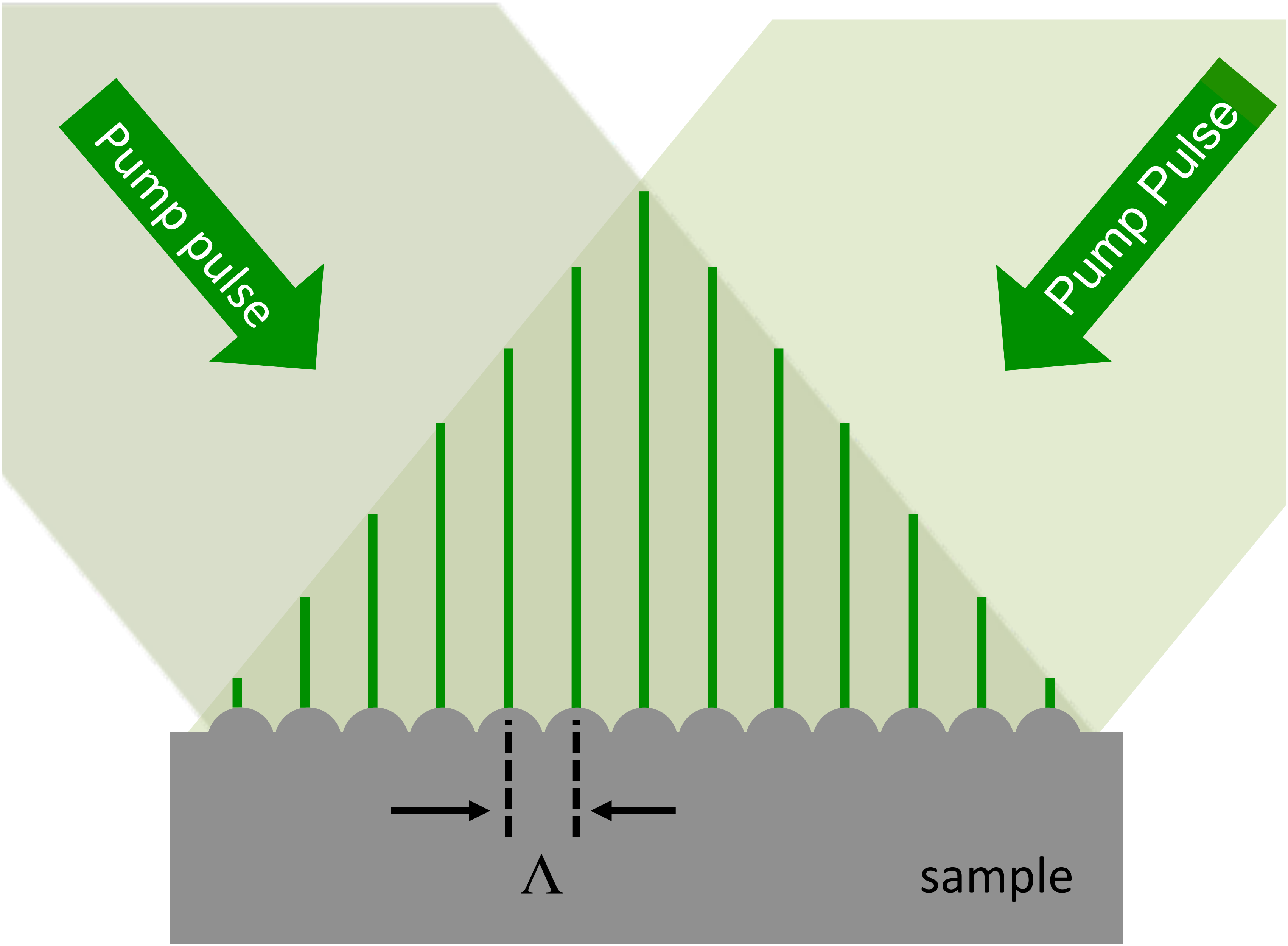
in situ characterization

material structure

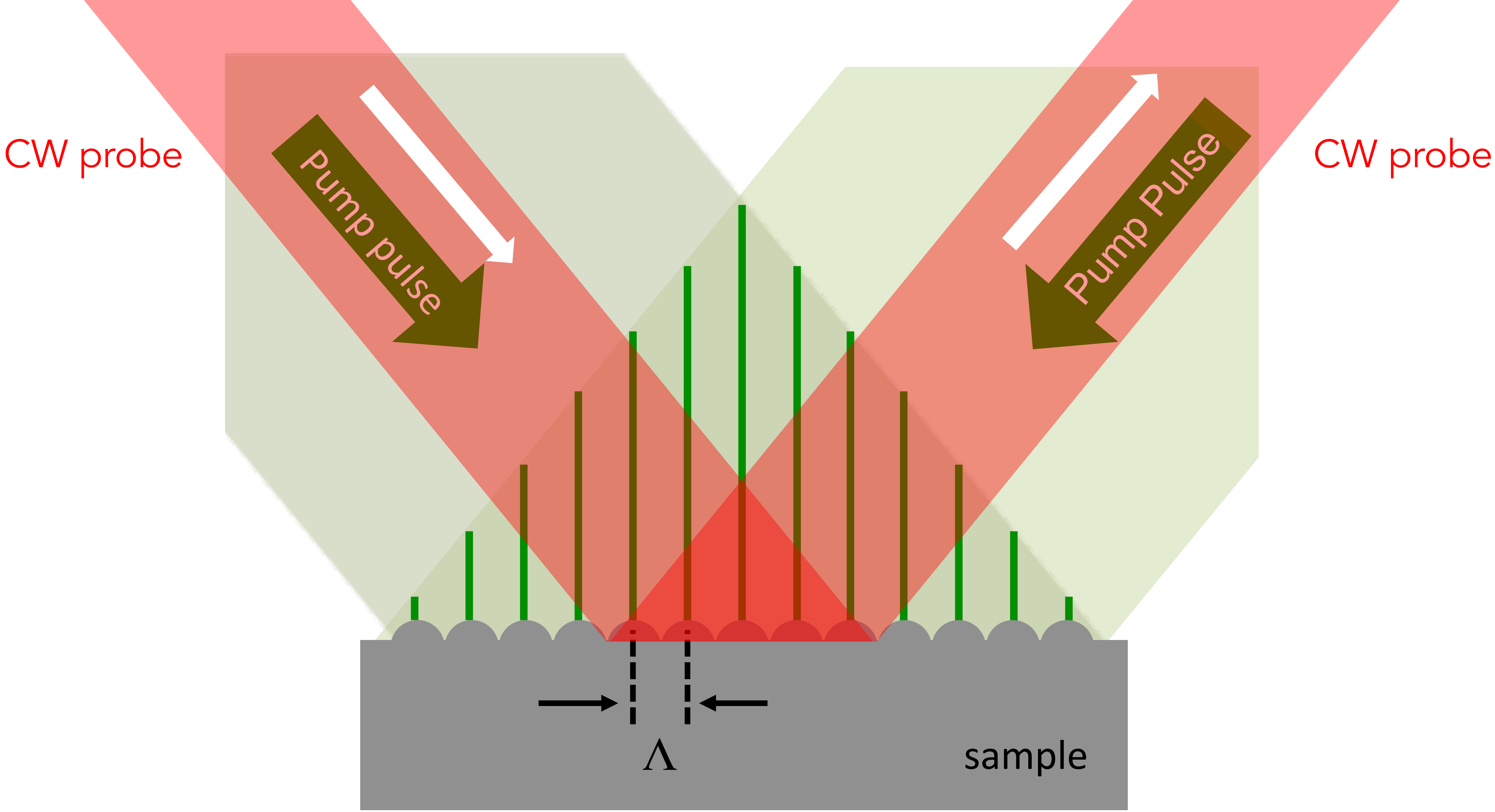
material properties

Transient Grating Spectroscopy (TGS)

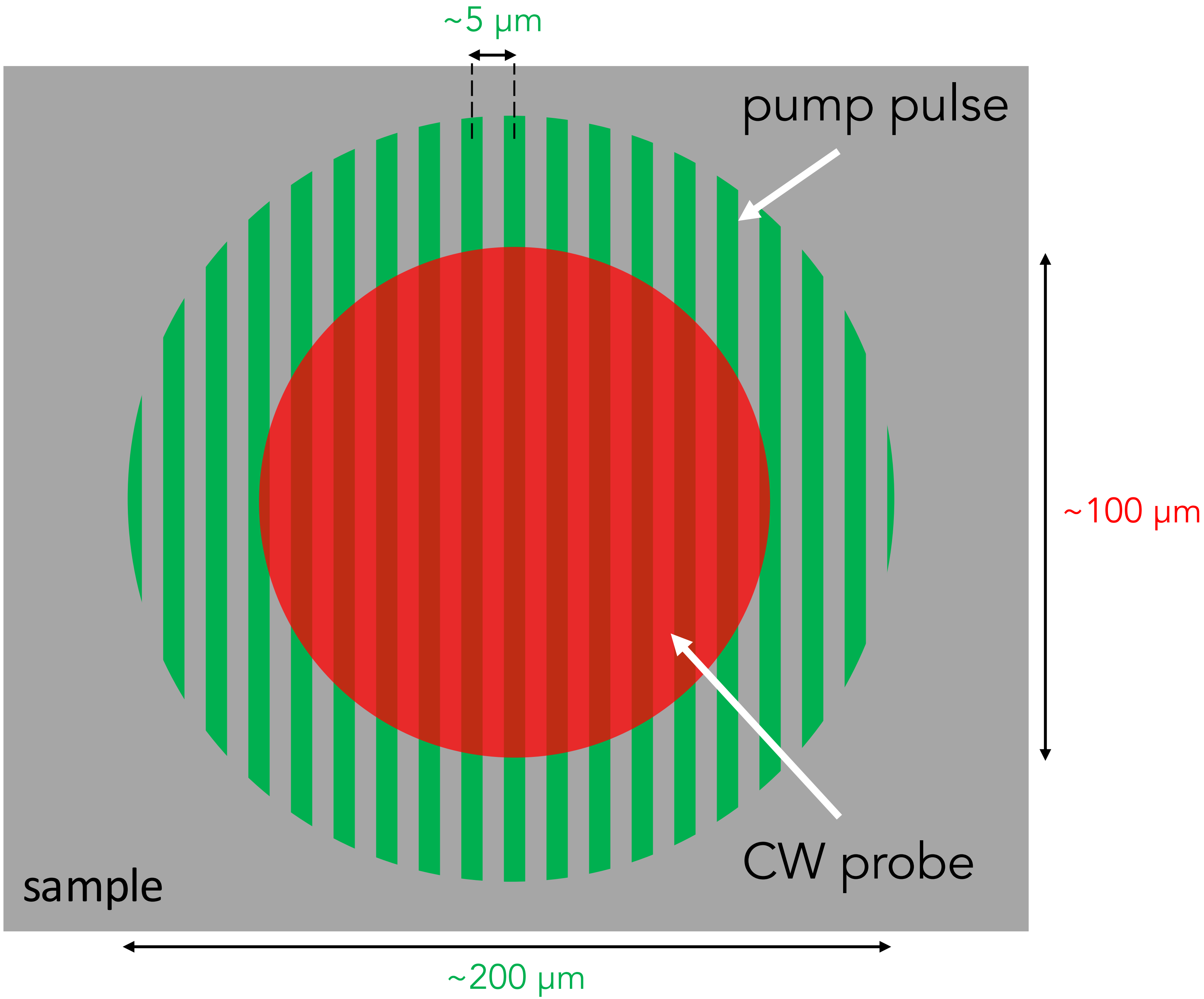


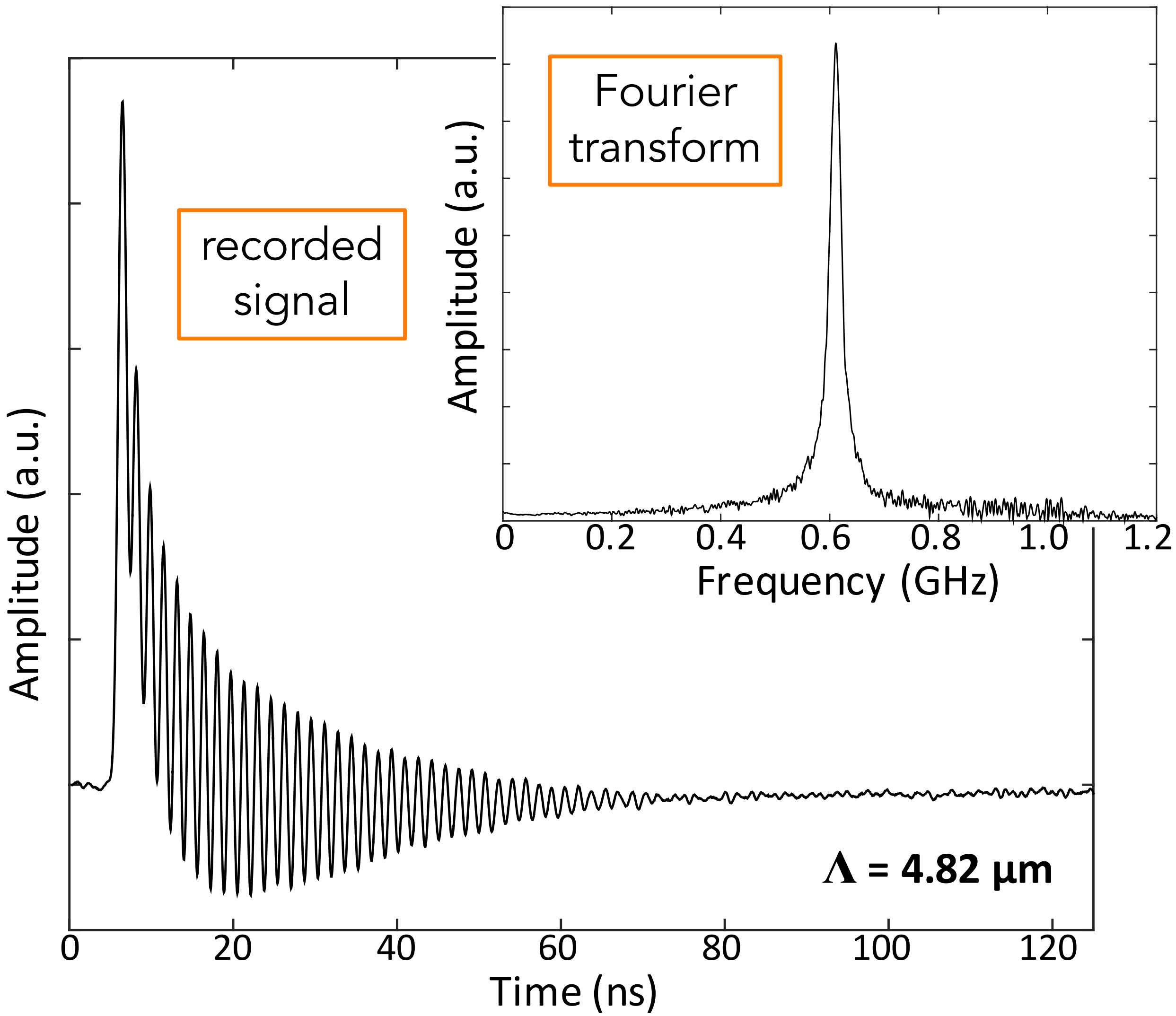


Dennett et al., *Phys. Rev. B* **94** (2016)

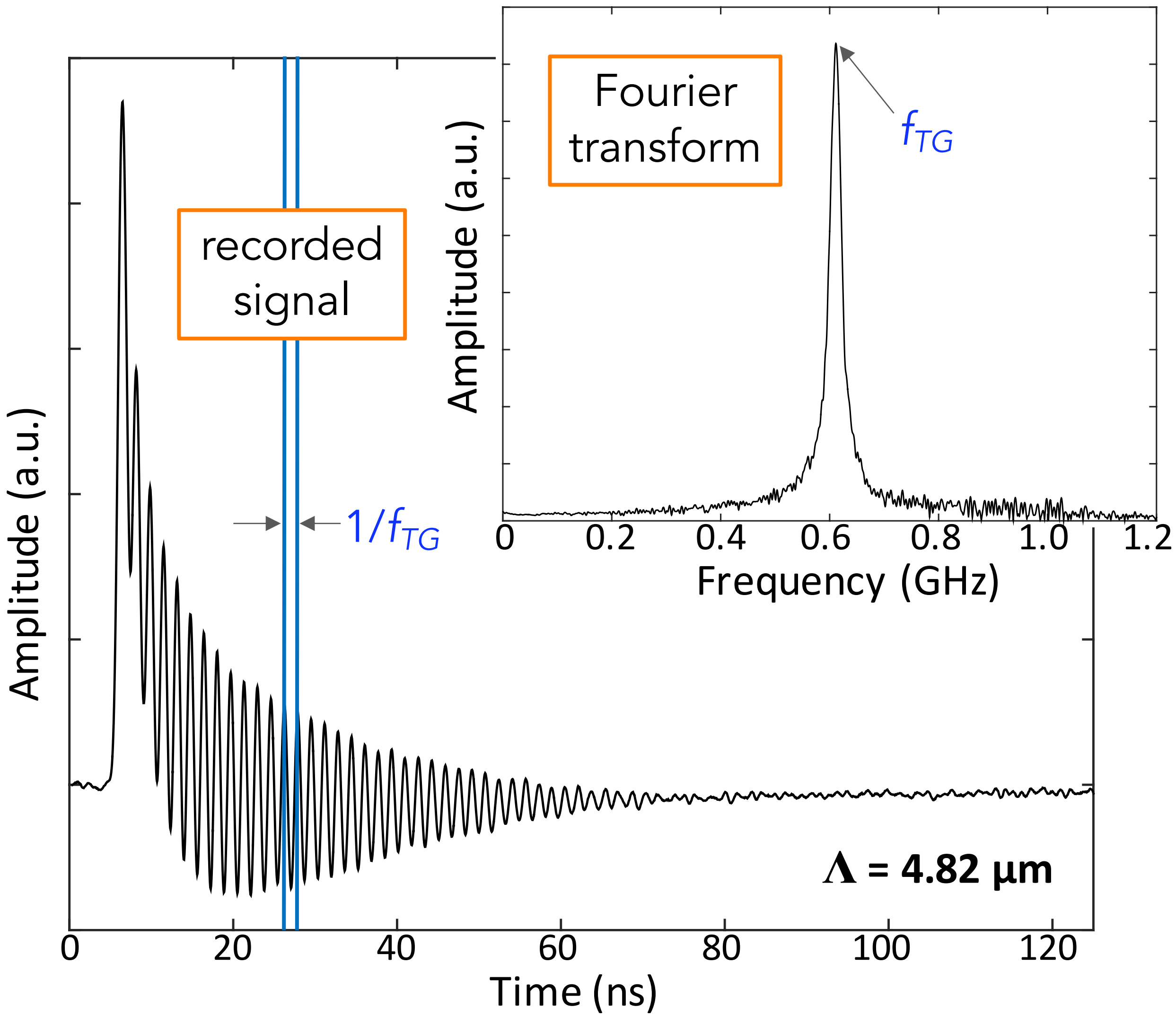


Dennett et al., *Phys. Rev. B* **94** (2016)


~5 μm
pump pulse
~100 μm
CW probe
sample
~200 μm

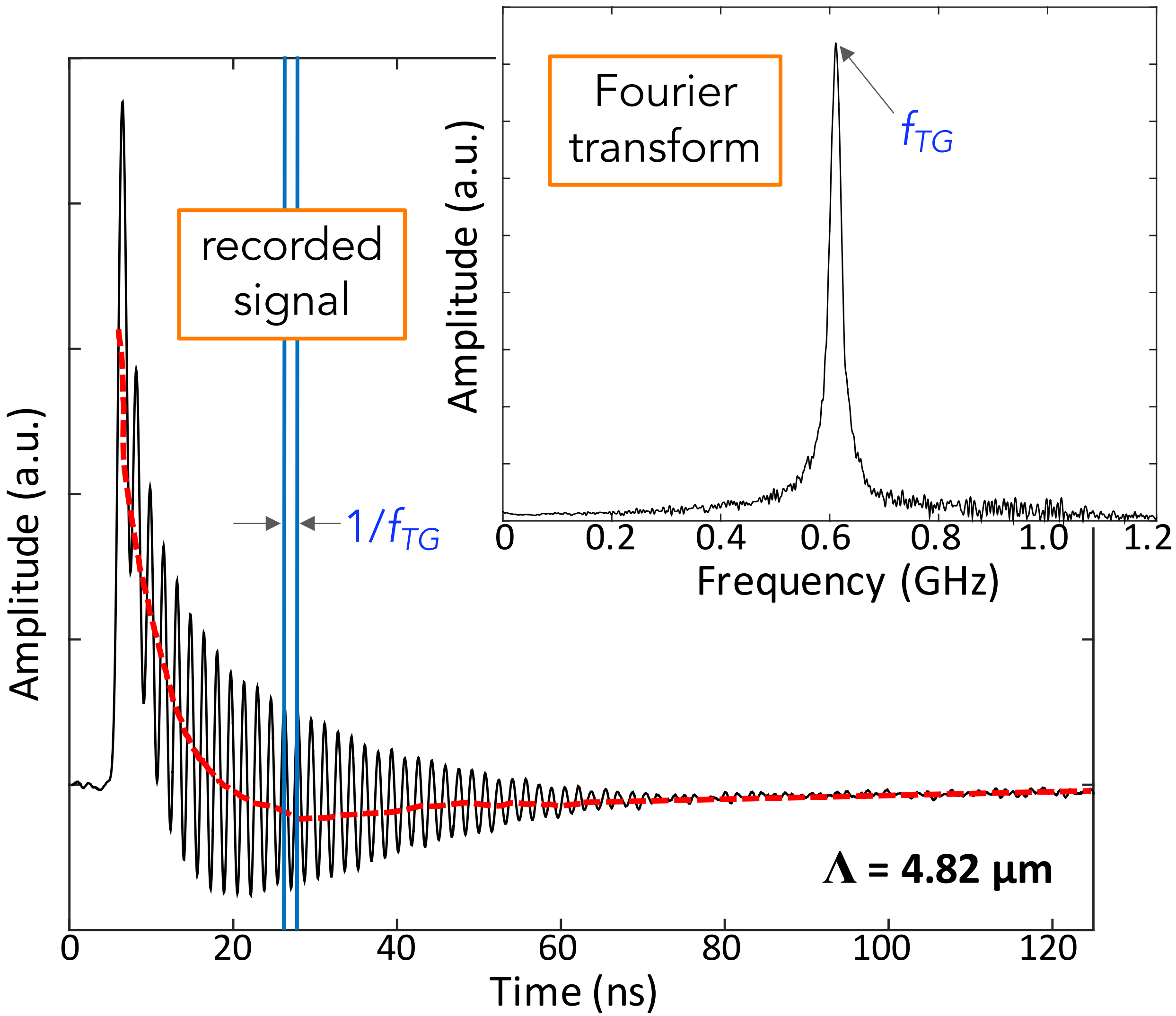


Dennett et al., *Phys. Rev. B* **94** (2016)



acoustic oscillations return **elastic mechanical** properties

Dennett et al., *Phys. Rev. B* **94** (2016)



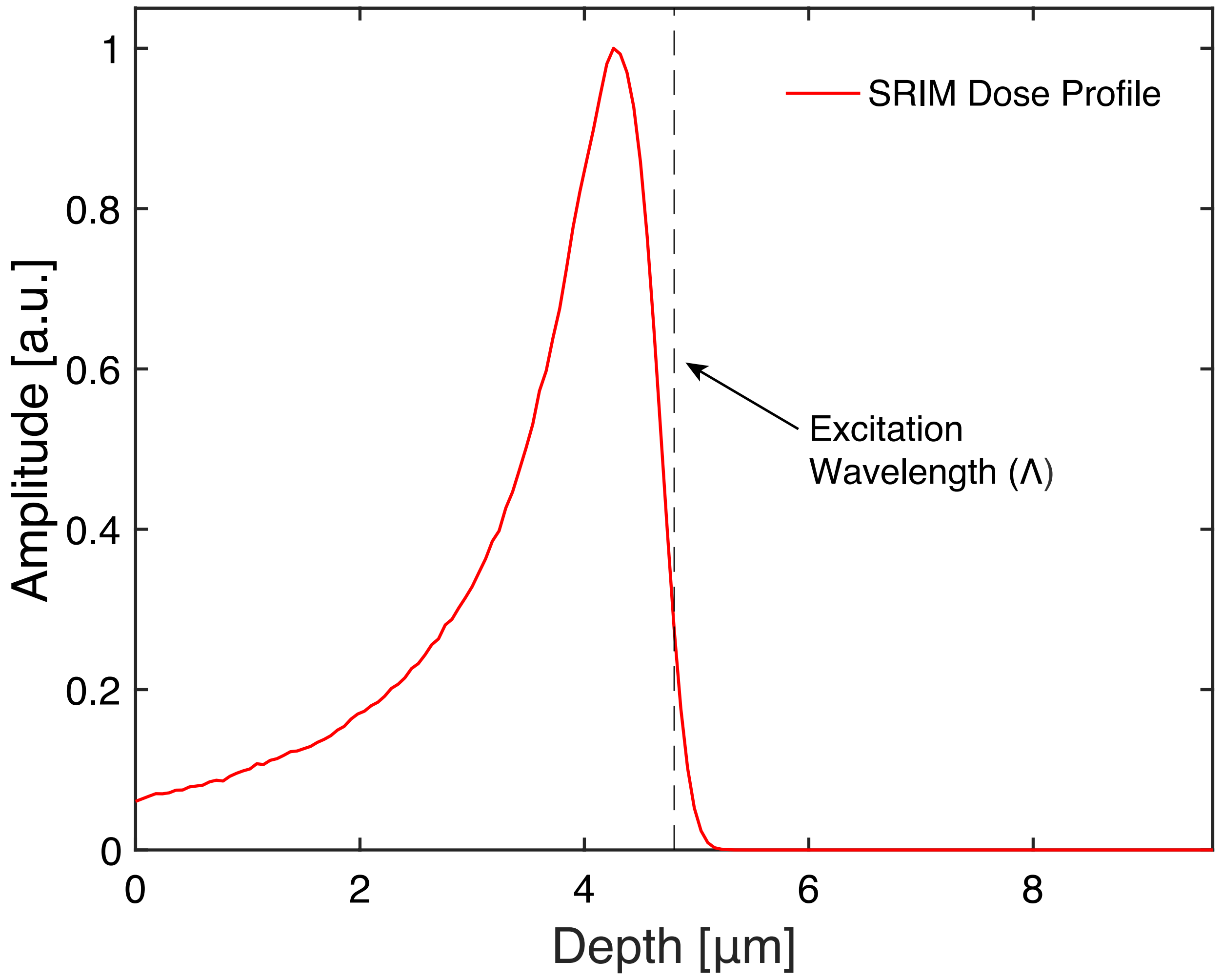
acoustic oscillations return **elastic mechanical** properties

grating decay returns **thermal transport** properties

Dennett et al., *Phys. Rev. B* **94** (2016)

material	single crystal {111} Cu
ion species	Cu^{6+}
ion energy	35 MeV
temperature	400˚C
spot size	0.19 cm^2
beam current	100-250 nA
target doses (peak)	0, 5, 10, 30, 50, 100 dpa

35 MeV Cu^{6+} in Cu at 400˚C



Dennett et al., *Acta Mater.* **145** (2018)

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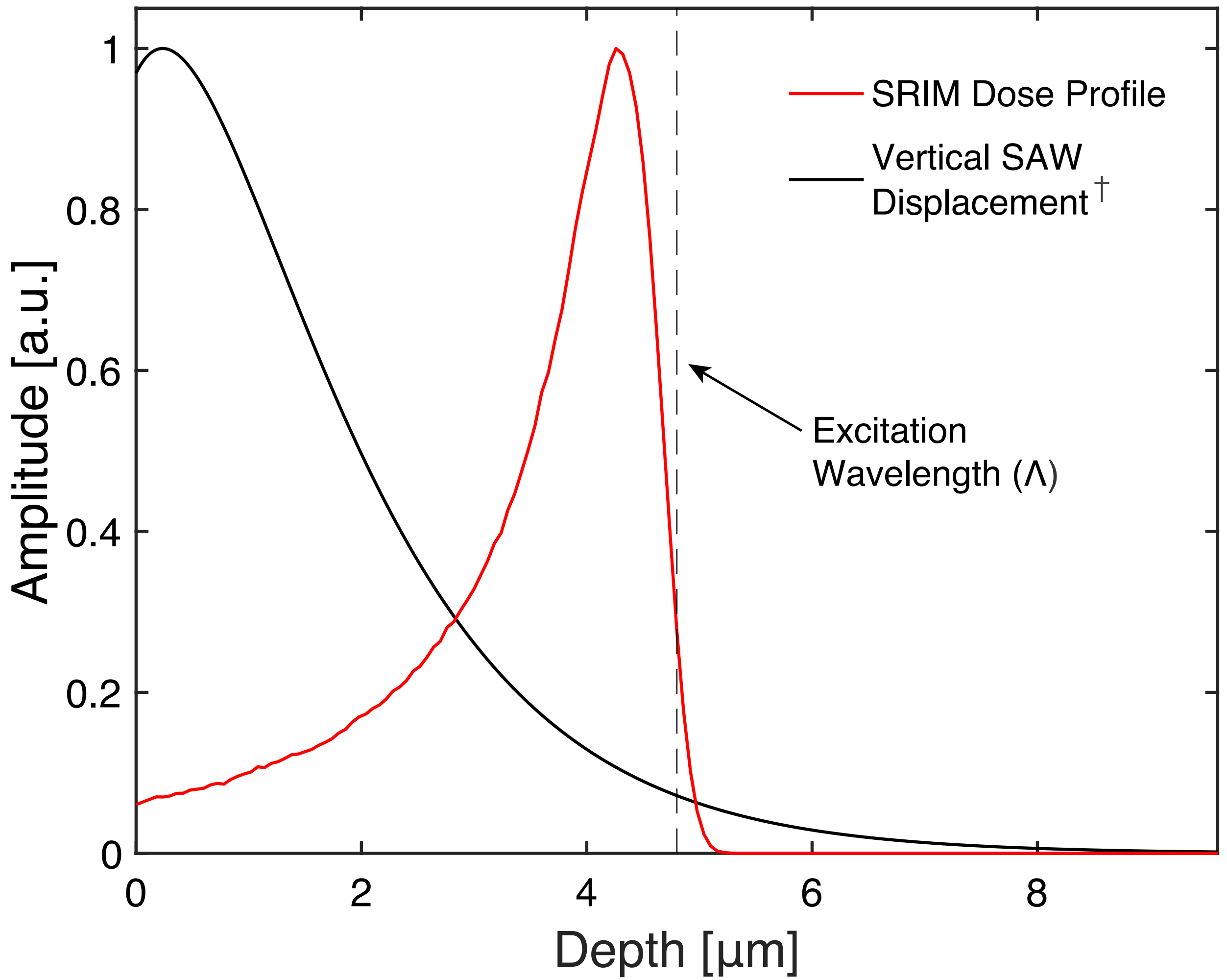
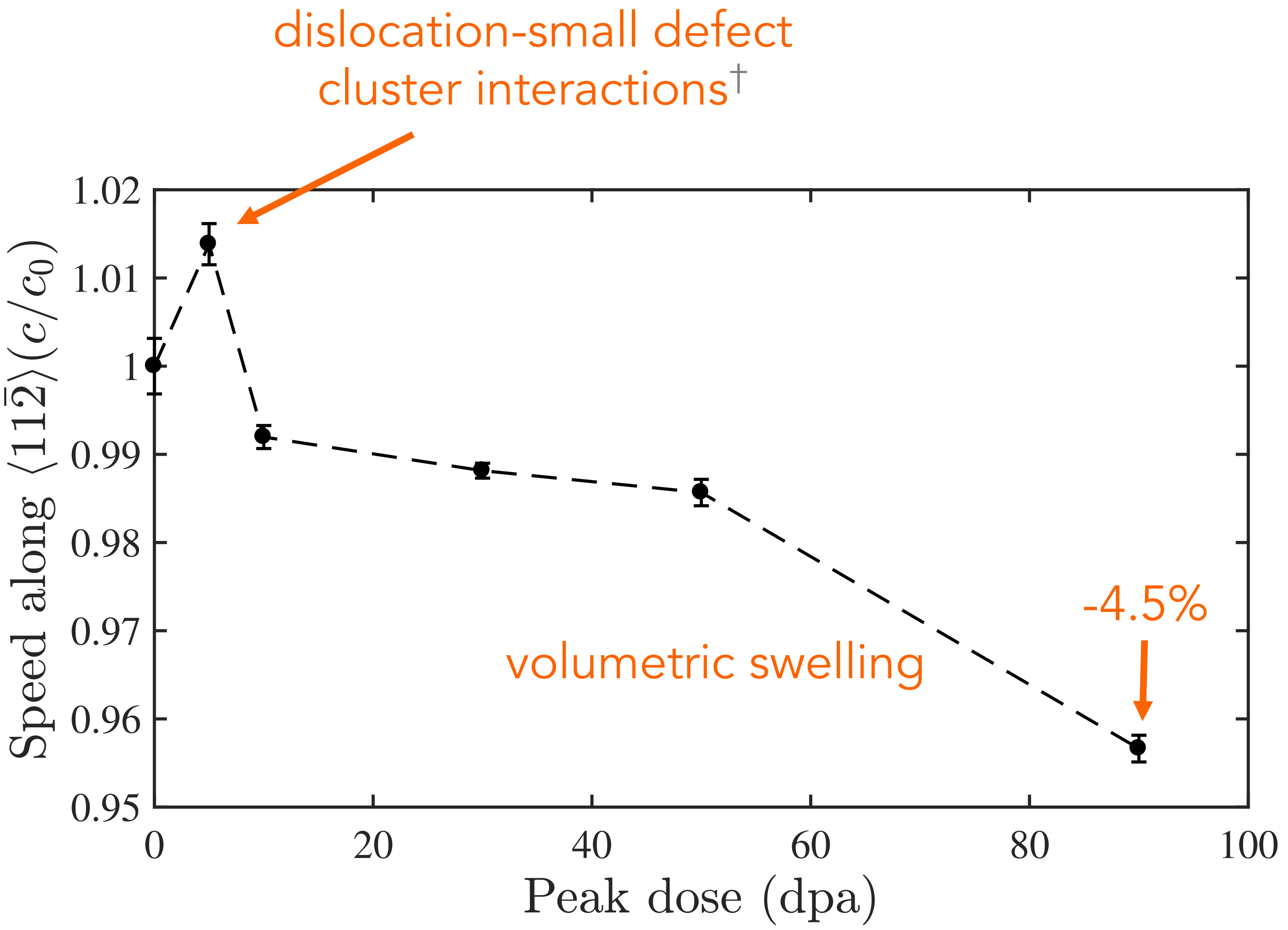


Dennett et al., *Acta Mater.* **145** (2018)

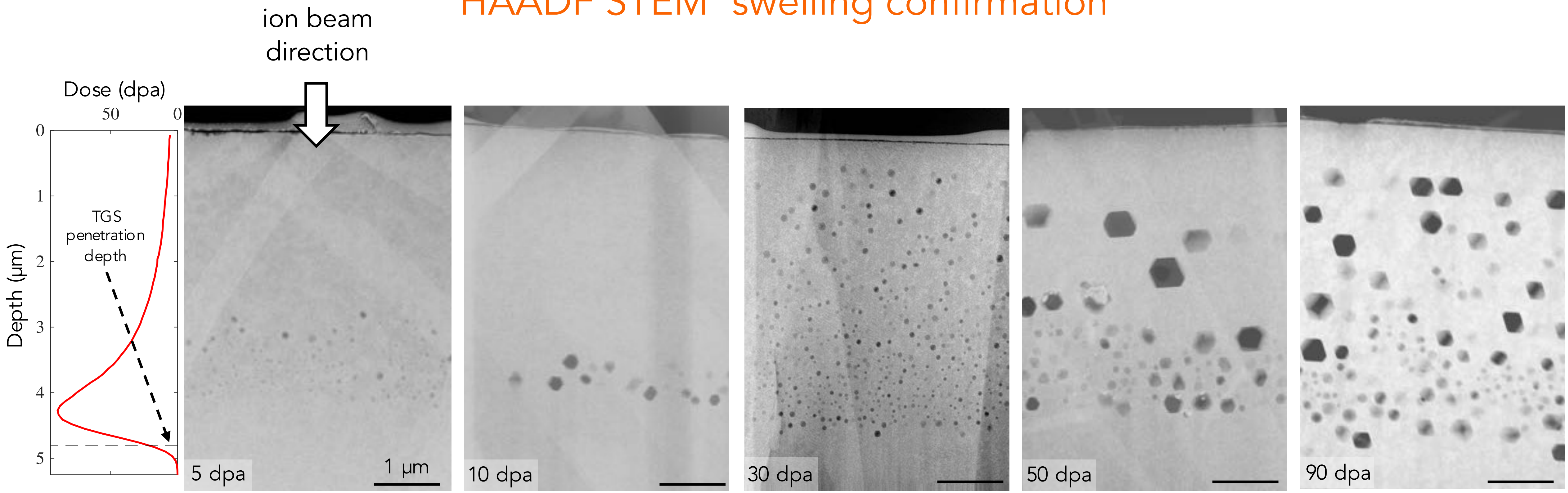
†Royer et al., *J. Acoust. Soc. Am.* **76** (1984)



Dennett et al., *Acta Mater.* **145** (2018)

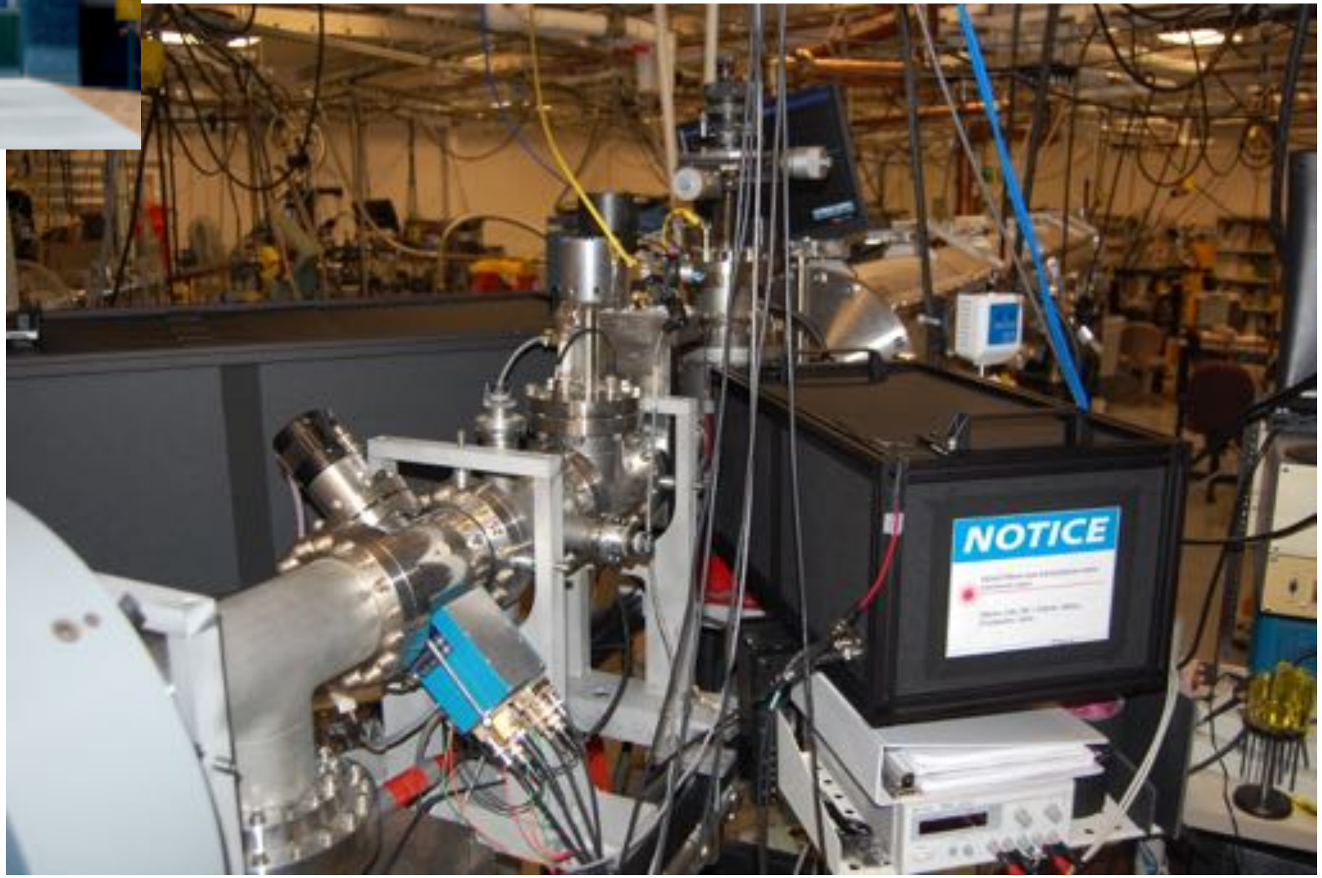
†Parkin et al., *J. Phys. F: Met. Phys.* **17** (1987)

HAADF STEM* swelling confirmation

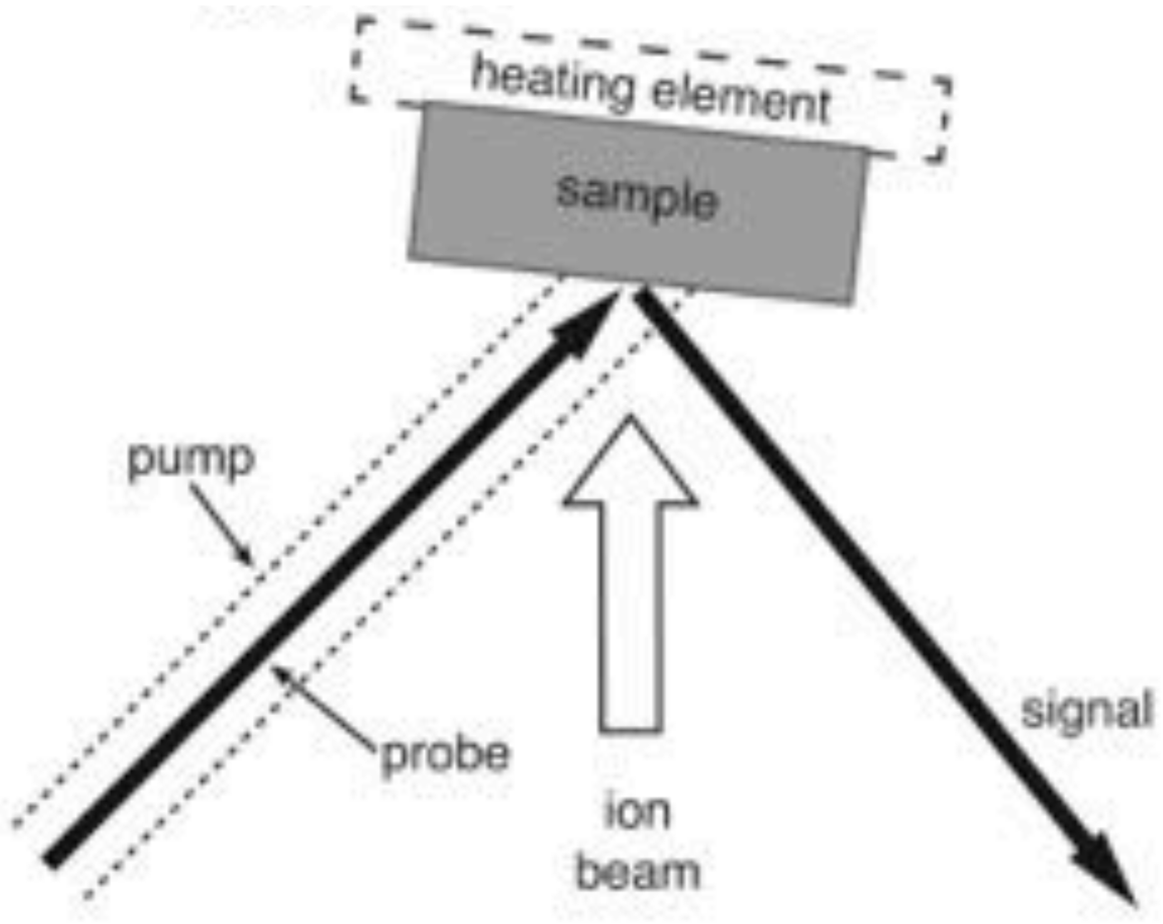


*high angle annular dark field scanning transmission electron microscopy

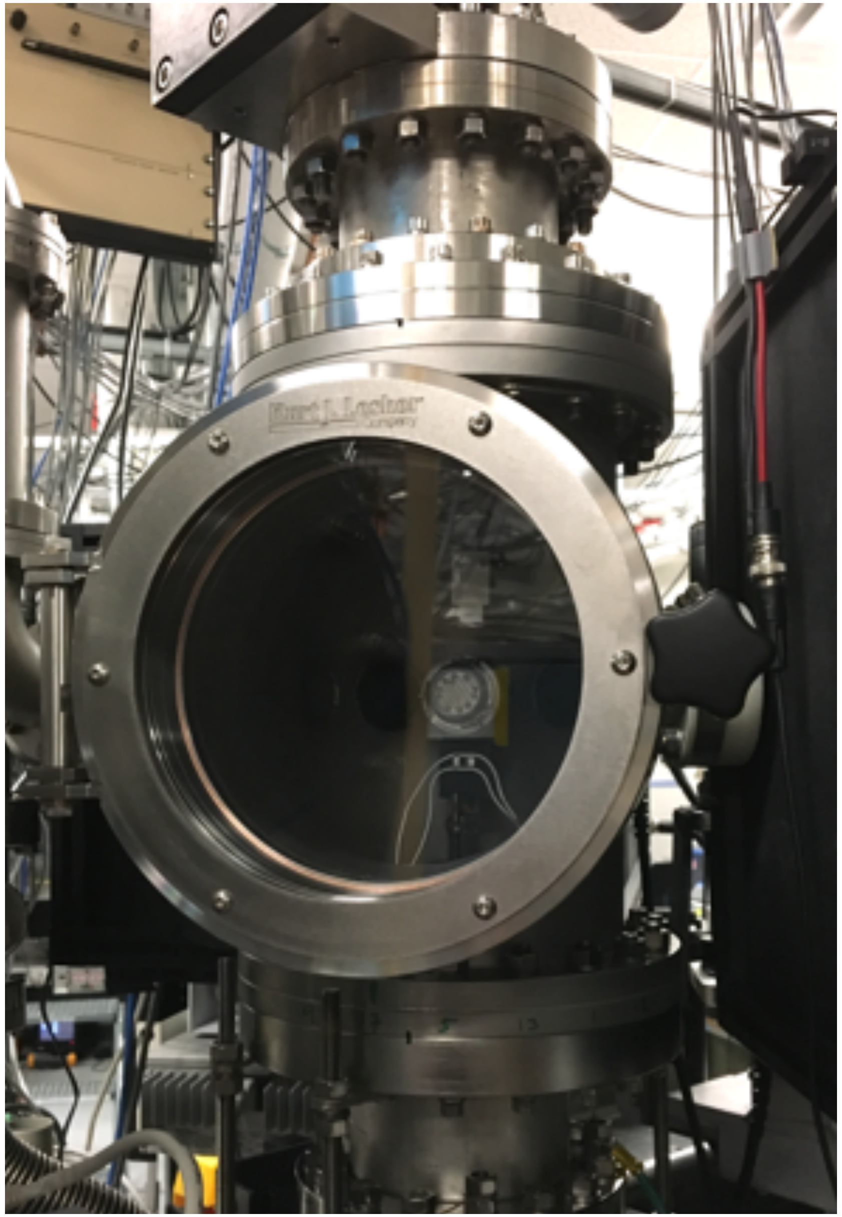
Dennett et al., *Acta Mater.* **145** (2018)



in situ ion irradiation TGS (I^3TGS) beamline at the Sandia Ion Beam Lab



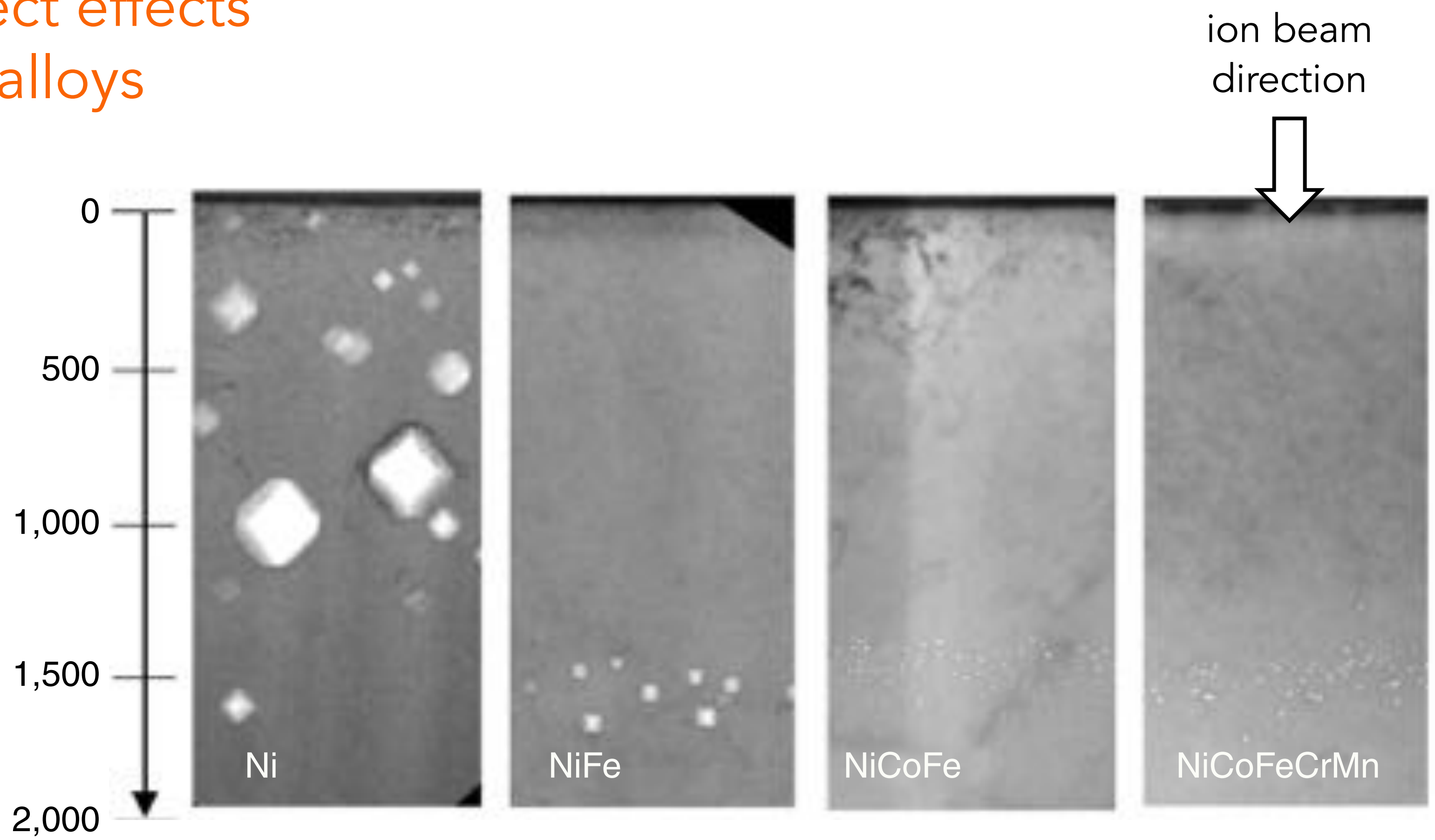
Dennett et al., *NIMB* **440** (2019) 126-138
doi: 10.1016/j.nimb.2018.10.025



I^3TGS target chamber

long- and short-timescale defect effects in Ni-based solid solution alloys

- high strength
- thermal stability
- wear resistance
- corrosion resistance
- irradiation tolerance



bright-field TEM of alloys irradiated with 1.5 MeV Ni^{+} to 60 dpa peak at 500°C

Lu et al., *Nat. Commun.* **7** (2016)

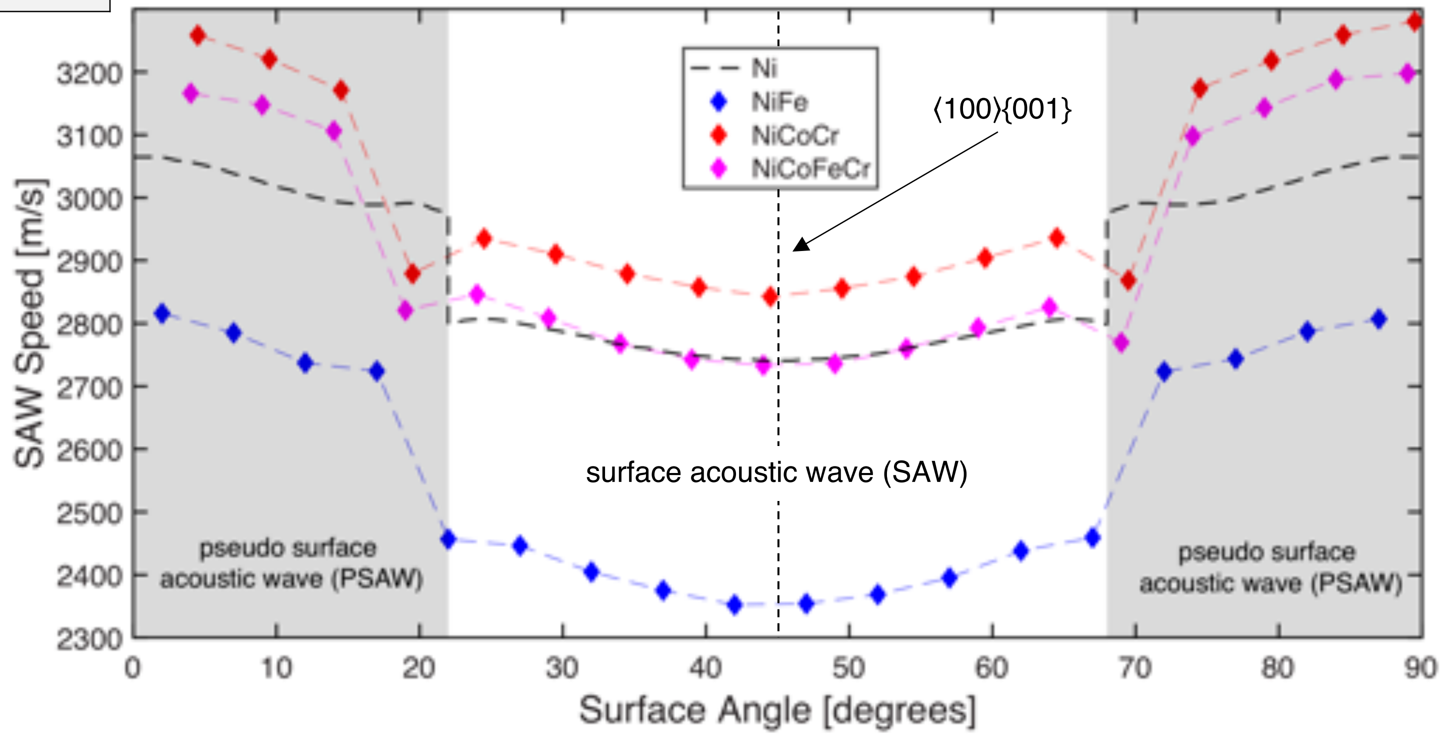
equiatomic composition	single/ polycrystal	source
Ni	SC {001}	commercial
NiFe	SC {001}	ORNL
NiCoCr	SC {001}	ORNL
NiFeCoCr	SC {001}	ORNL
NiFeCoCrMn	polycrystal	ORNL

in situ long-timescale
irradiation test matrix

equiatomic composition	single/ polycrystal	source
Ni	SC {001}	commercial
NiFe	SC {001}	ORNL
NiCoCr	SC {001}	ORNL
NiFeCoCr	SC {001}	ORNL
NiFeCoCrMn	polycrystal	ORNL

in situ long-timescale irradiation test matrix

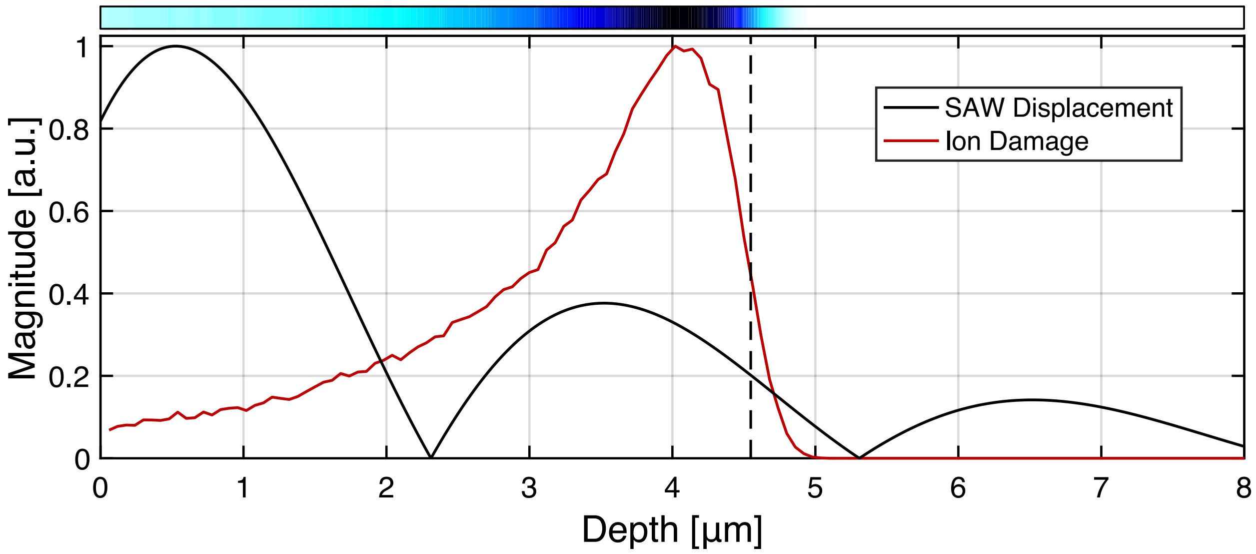
pre-irradiation TGS characterization used to identify acoustic polarization for *in situ* testing



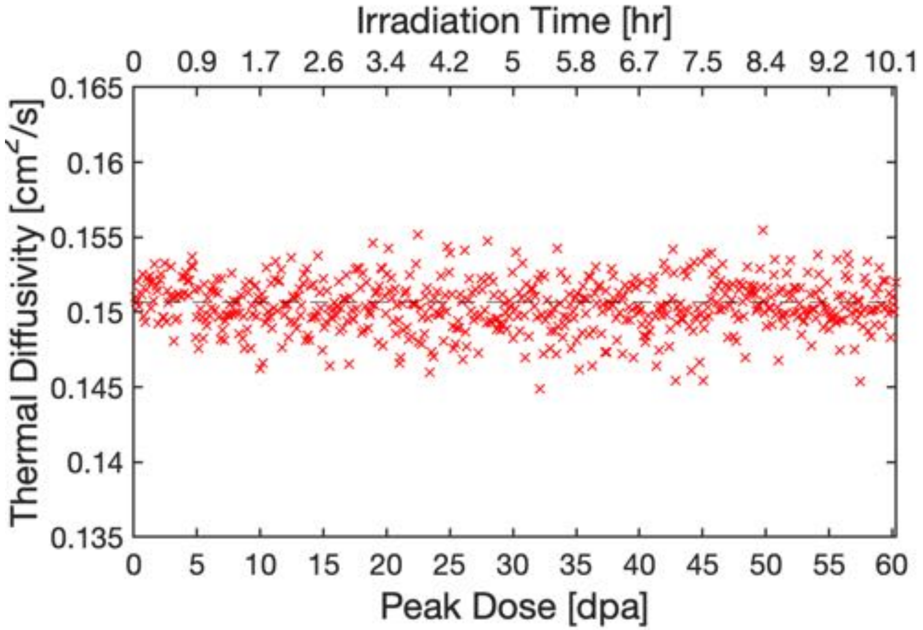
surface angle	<100>{001}
ion species	Ni^{5+}
ion energy	31 MeV
temperature	550˚C
TGS wavelength	4.55 µm
peak dose	60 dpa
peak dose rate	1.6-1.8 x 10^{-3} dpa/s
measurement time	35 sec
measurement interval	60 sec
exposure time per sample	9.5-10.5 hours

void swelling at high temperatures and high doses

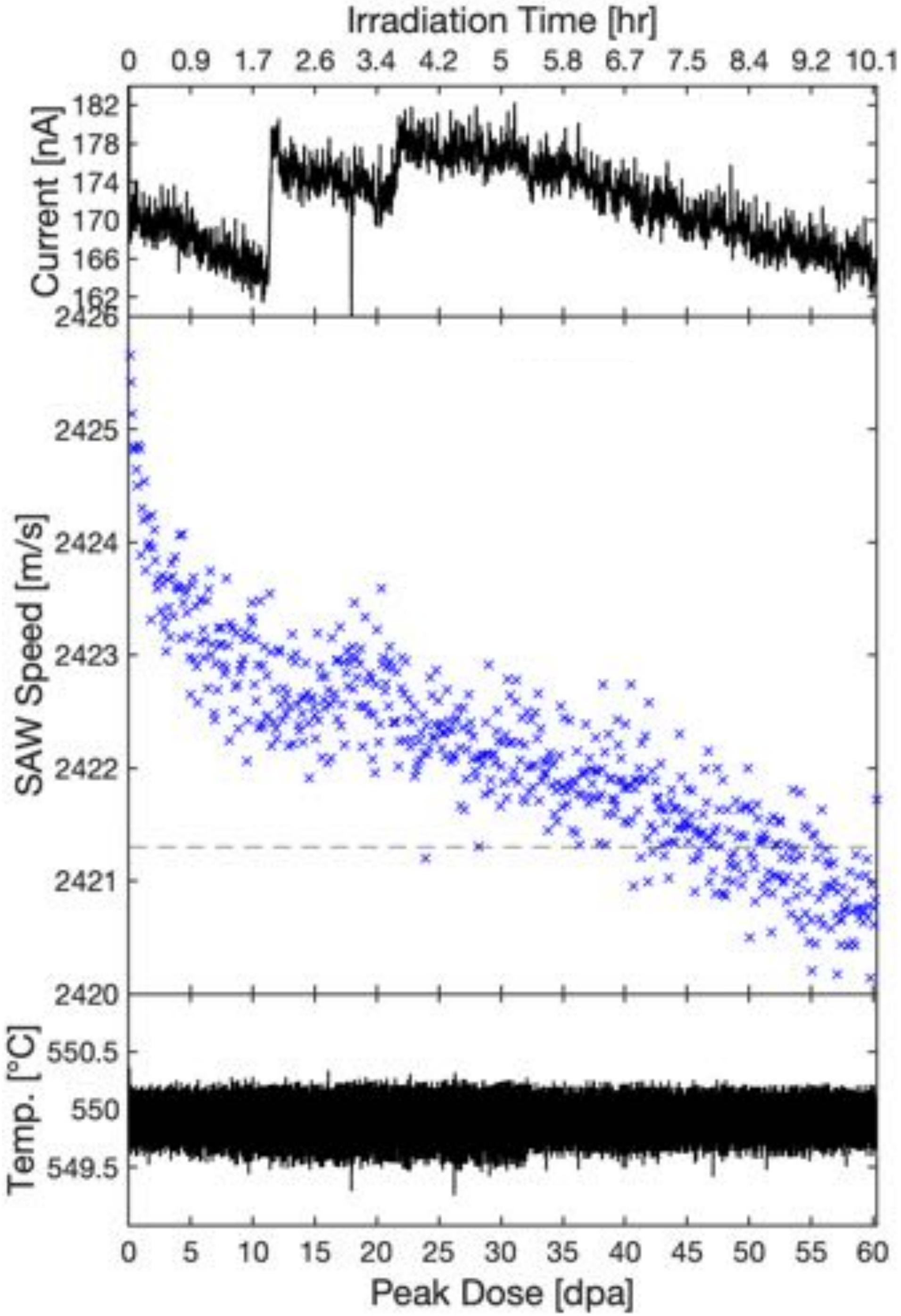
31 MeV Ni^{5+} in Ni at 550˚C



Pure Ni

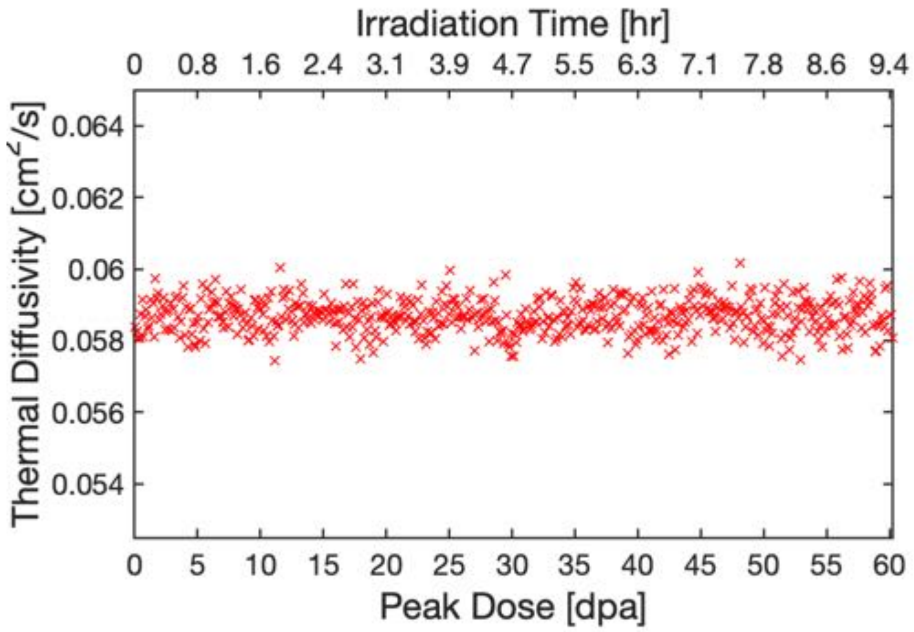


Irradiation Time [hr]
0 0.9 1.7 2.6 3.4 4.2 5 5.8 6.7 7.5 8.4 9.2 10.1
Thermal Diffusivity [cm²/s]
0.165 0.16 0.155 0.15 0.145 0.14 0.135
0 5 10 15 20 25 30 35 40 45 50 55 60
Peak Dose [dpa]

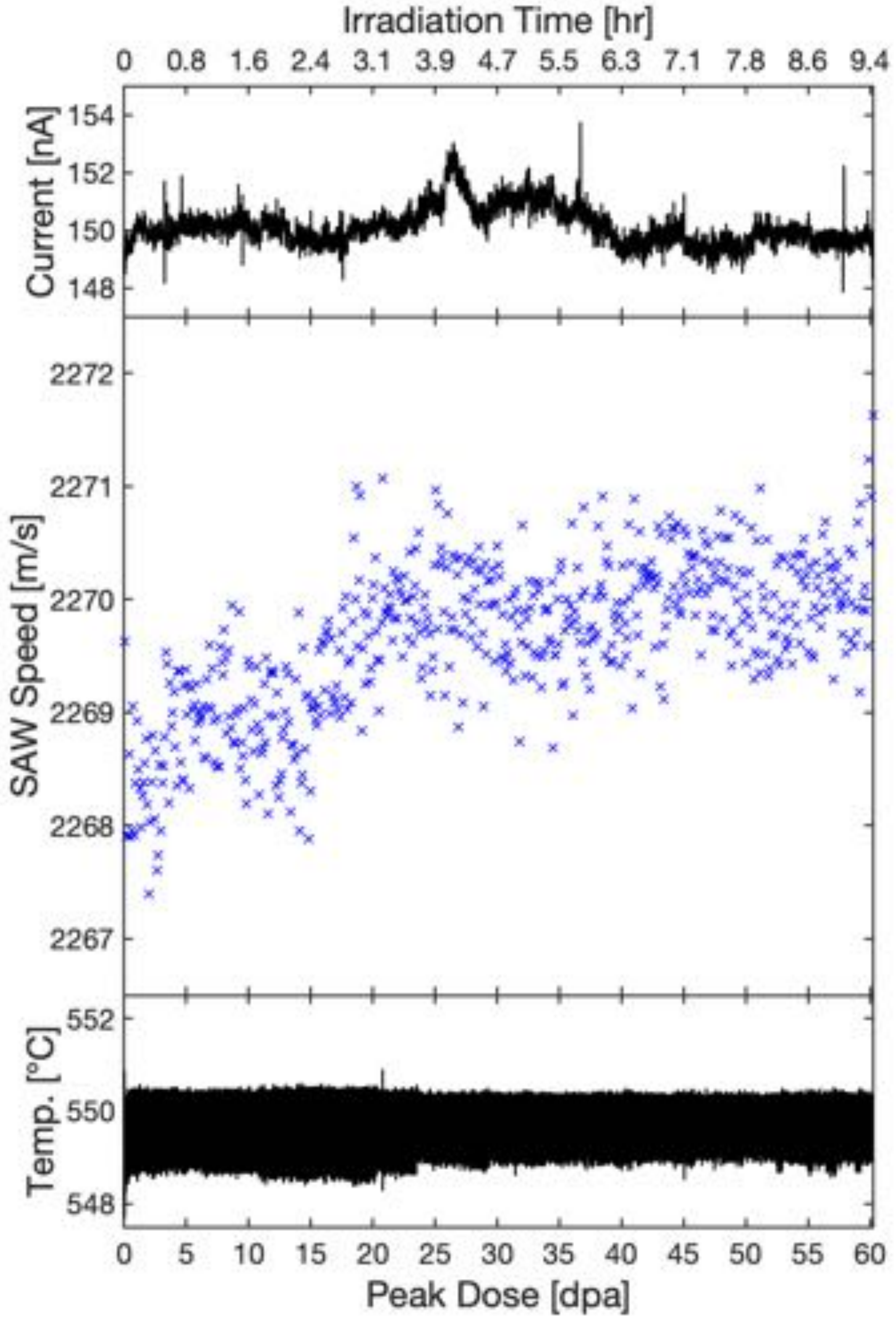


Irradiation Time [hr]
0 0.9 1.7 2.6 3.4 4.2 5 5.8 6.7 7.5 8.4 9.2 10.1
Current [nA]
182 178 174 170 166 162
SAW Speed [m/s]
2426 2425 2424 2423 2422 2421 2420
Temp. [°C]
550.5 550 549.5
0 5 10 15 20 25 30 35 40 45 50 55 60
Peak Dose [dpa]

NiFe

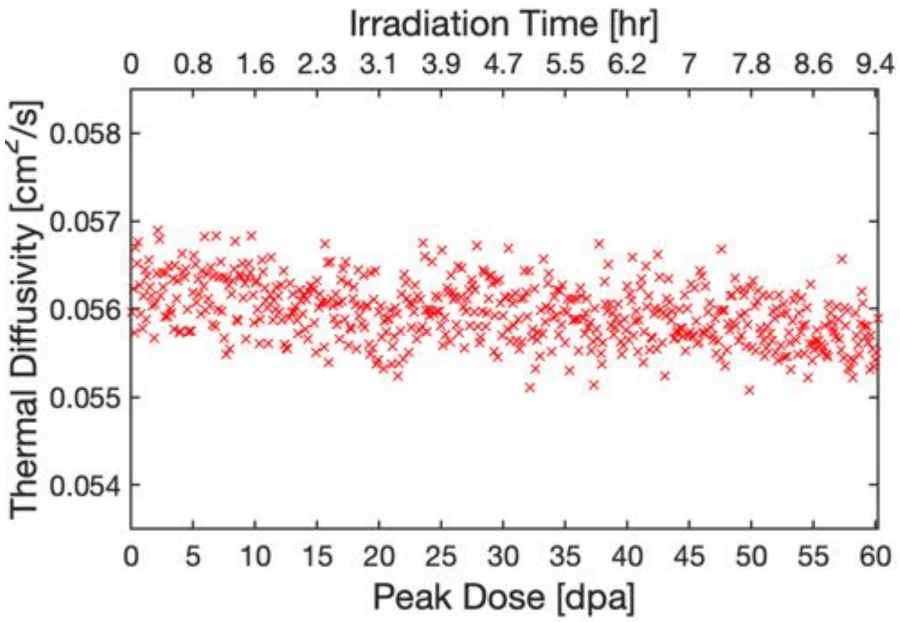


Irradiation Time [hr]
0
0.8
1.6
2.4
3.1
3.9
4.7
5.5
6.3
7.1
7.8
8.6
9.4
Thermal Diffusivity [cm²/s]
0.064
0.062
0.06
0.058
0.056
0.054
0
5
10
15
20
25
30
35
40
45
50
55
60
Peak Dose [dpa]

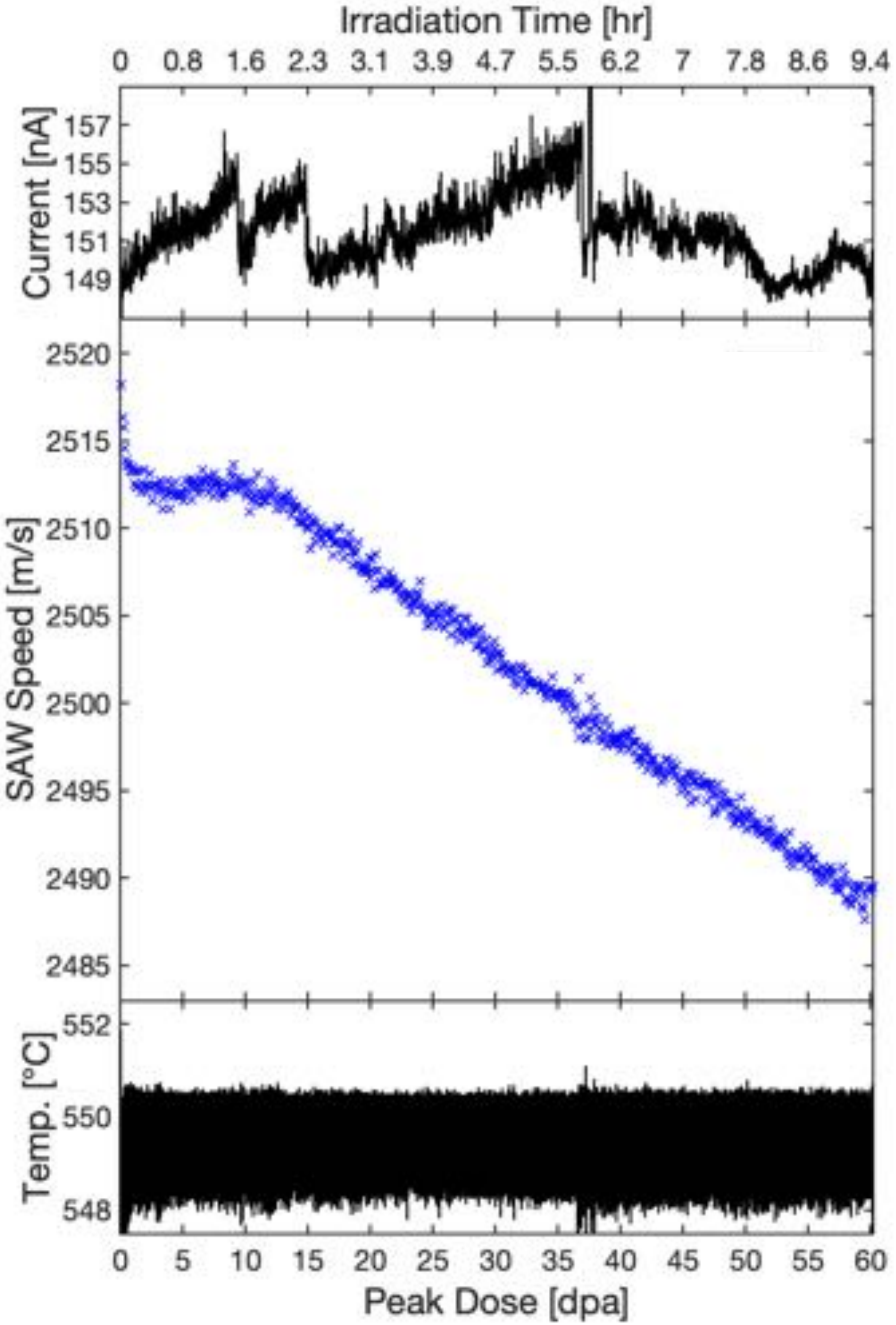


Irradiation Time [hr]
0
0.8
1.6
2.4
3.1
3.9
4.7
5.5
6.3
7.1
7.8
8.6
9.4
Current [nA]
154
152
150
148
SAW Speed [m/s]
2272
2271
2270
2269
2268
2267
Temp. [°C]
552
550
548
0
5
10
15
20
25
30
35
40
45
50
55
60
Peak Dose [dpa]

NiCoCr

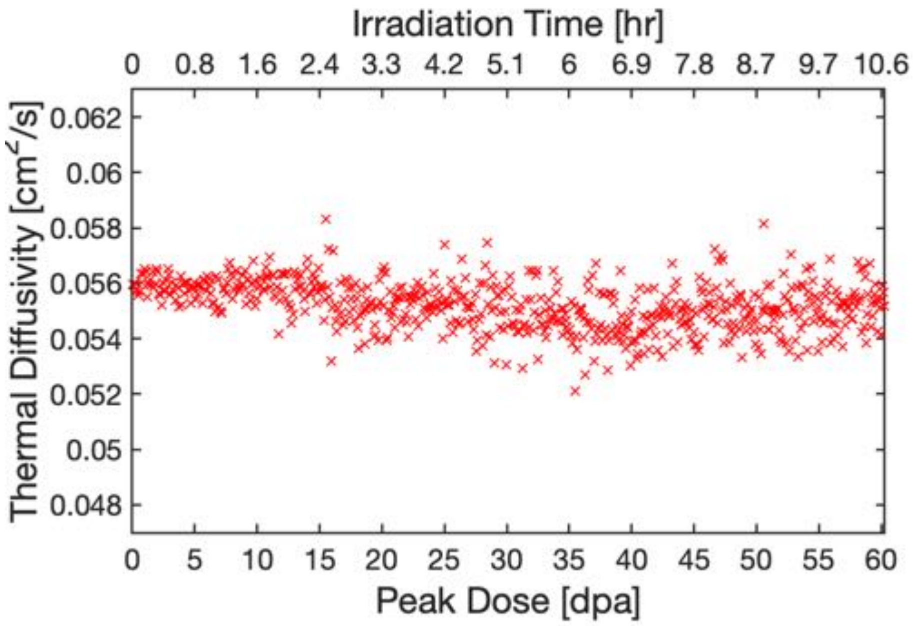


Irradiation Time [hr]
0 0.8 1.6 2.3 3.1 3.9 4.7 5.5 6.2 7 7.8 8.6 9.4
Thermal Diffusivity [cm²/s]
0.058 0.057 0.056 0.055 0.054
0 5 10 15 20 25 30 35 40 45 50 55 60
Peak Dose [dpa]

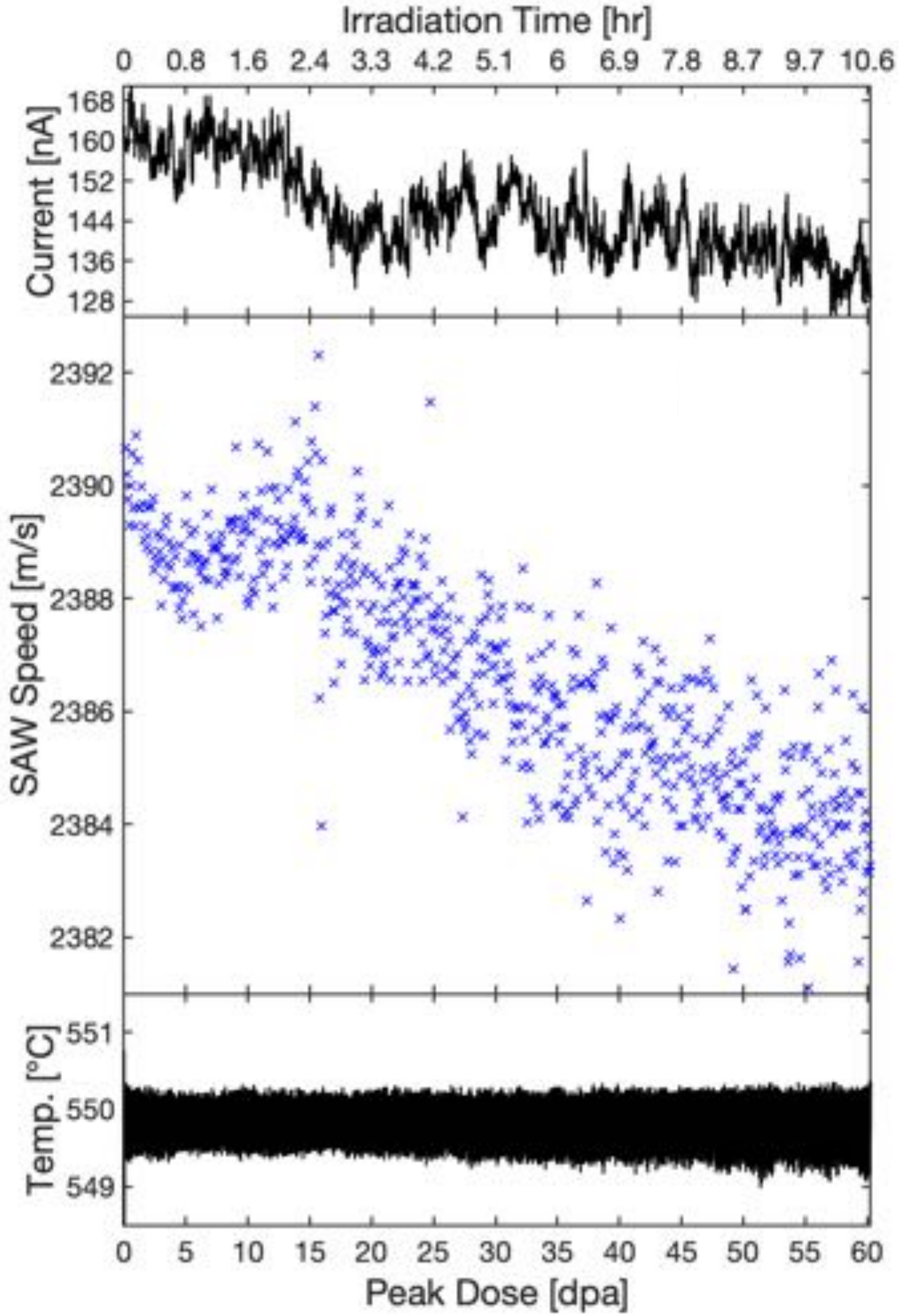


Irradiation Time [hr]
0 0.8 1.6 2.3 3.1 3.9 4.7 5.5 6.2 7 7.8 8.6 9.4
Current [nA]
157 155 153 151 149
SAW Speed [m/s]
2520 2515 2510 2505 2500 2495 2490 2485
Temp. [°C]
552 550 548
0 5 10 15 20 25 30 35 40 45 50 55 60
Peak Dose [dpa]

NiFeCoCr

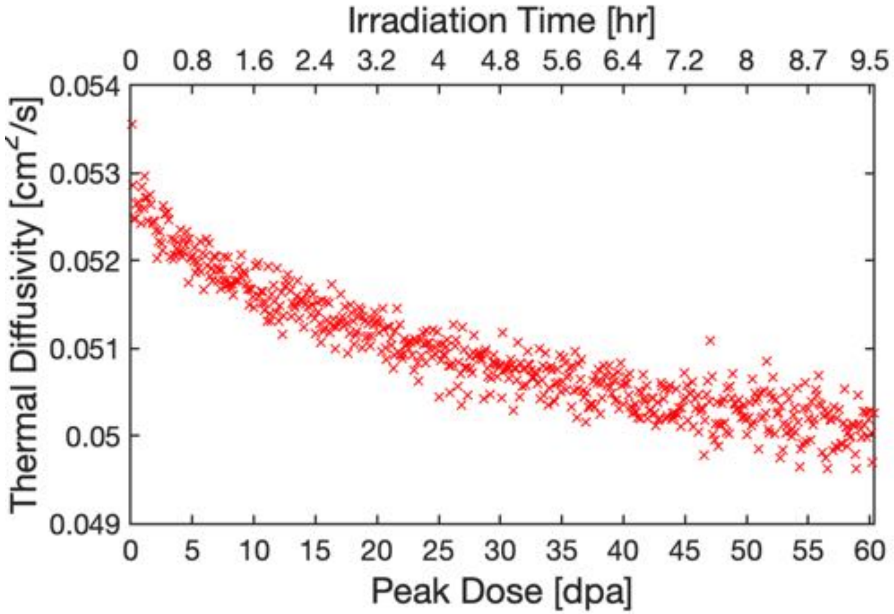


Irradiation Time [hr]
0 0.8 1.6 2.4 3.3 4.2 5.1 6 6.9 7.8 8.7 9.7 10.6
Thermal Diffusivity [cm²/s]
0.062
0.06
0.058
0.056
0.054
0.052
0.05
0.048
0 5 10 15 20 25 30 35 40 45 50 55 60
Peak Dose [dpa]

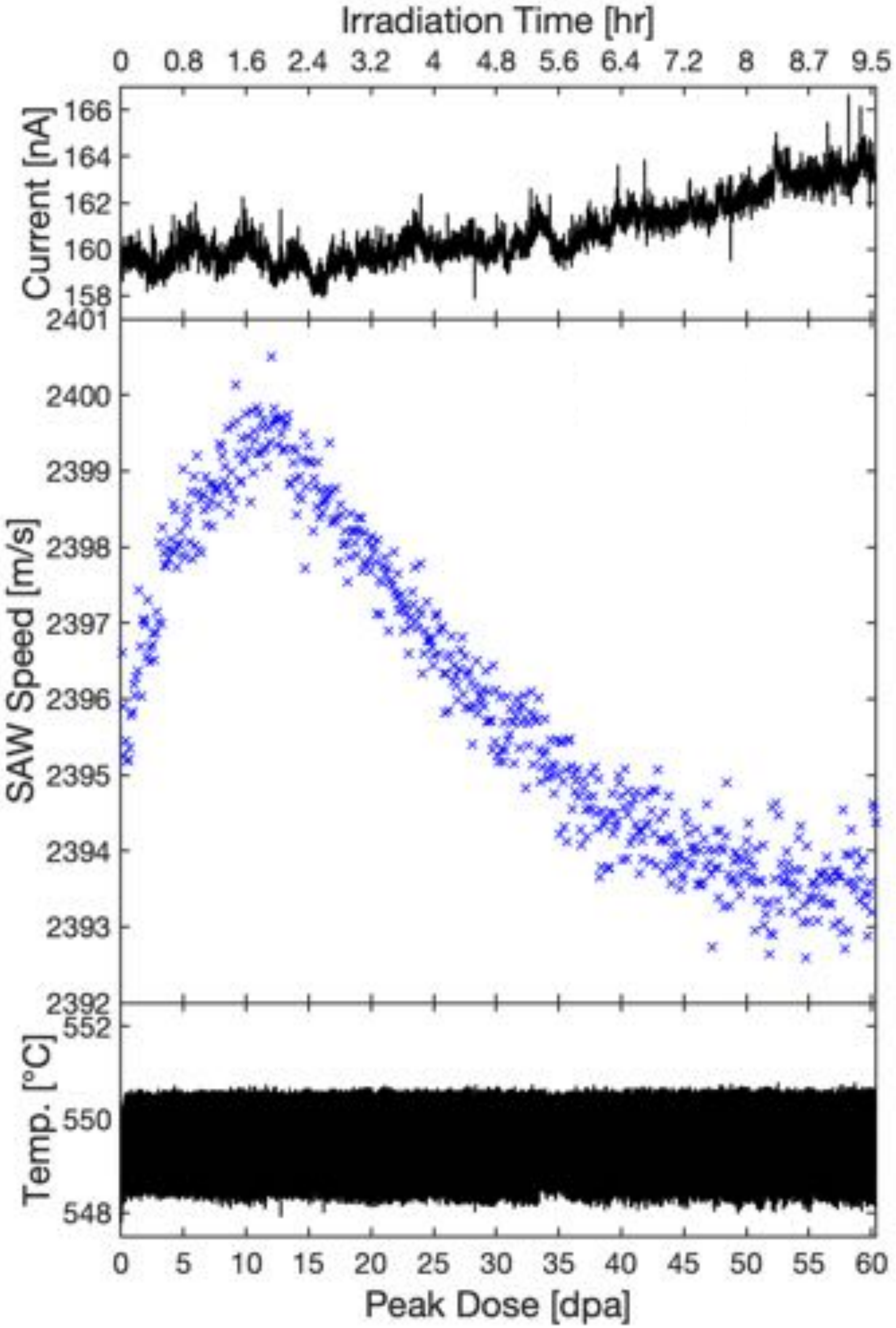


Irradiation Time [hr]
0 0.8 1.6 2.4 3.3 4.2 5.1 6 6.9 7.8 8.7 9.7 10.6
Current [nA]
168
160
152
144
136
128
SAW Speed [m/s]
2392
2390
2388
2386
2384
2382
Temp. [°C]
551
550
549
0 5 10 15 20 25 30 35 40 45 50 55 60
Peak Dose [dpa]

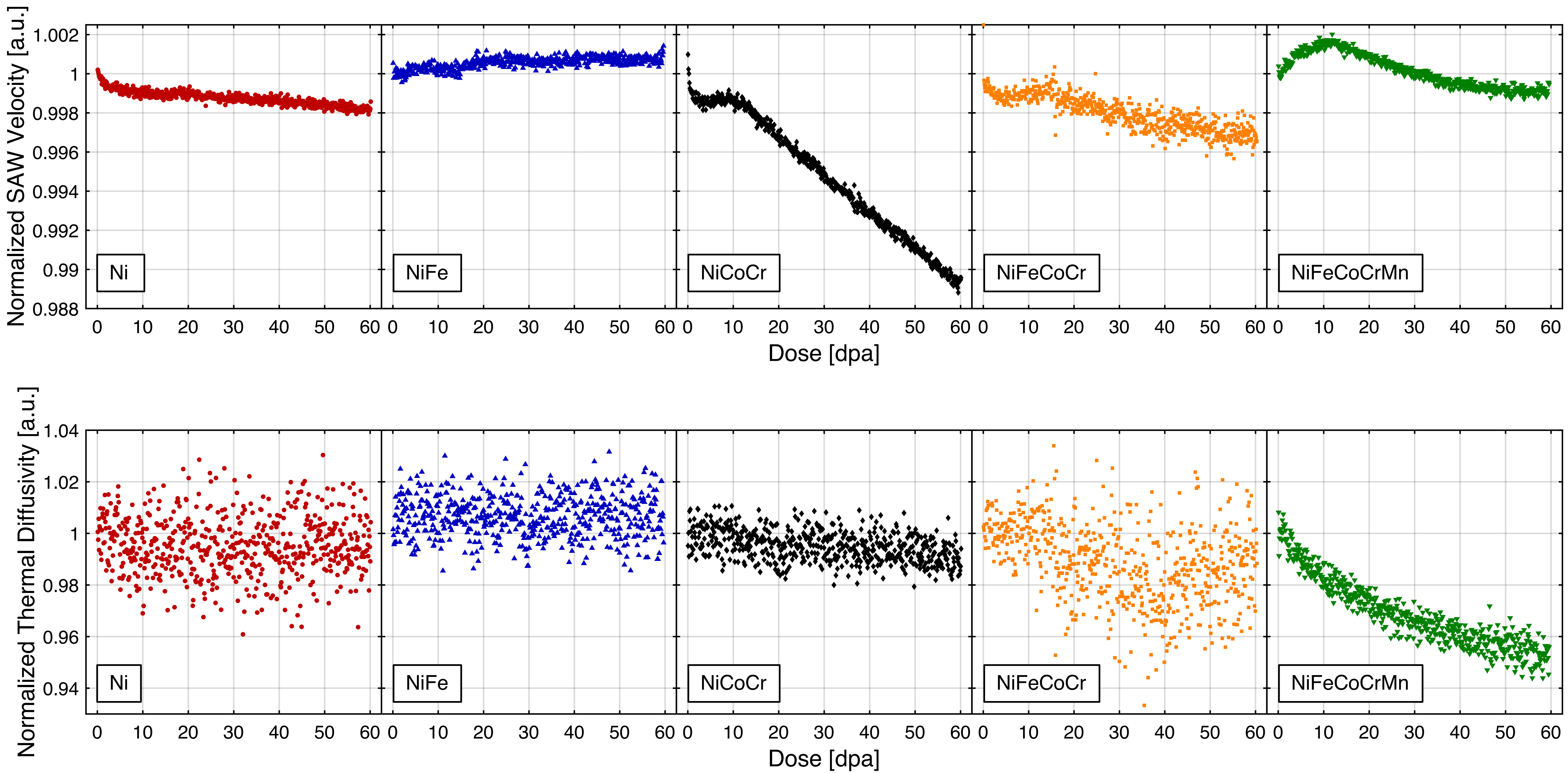
NiFeCoCrMn



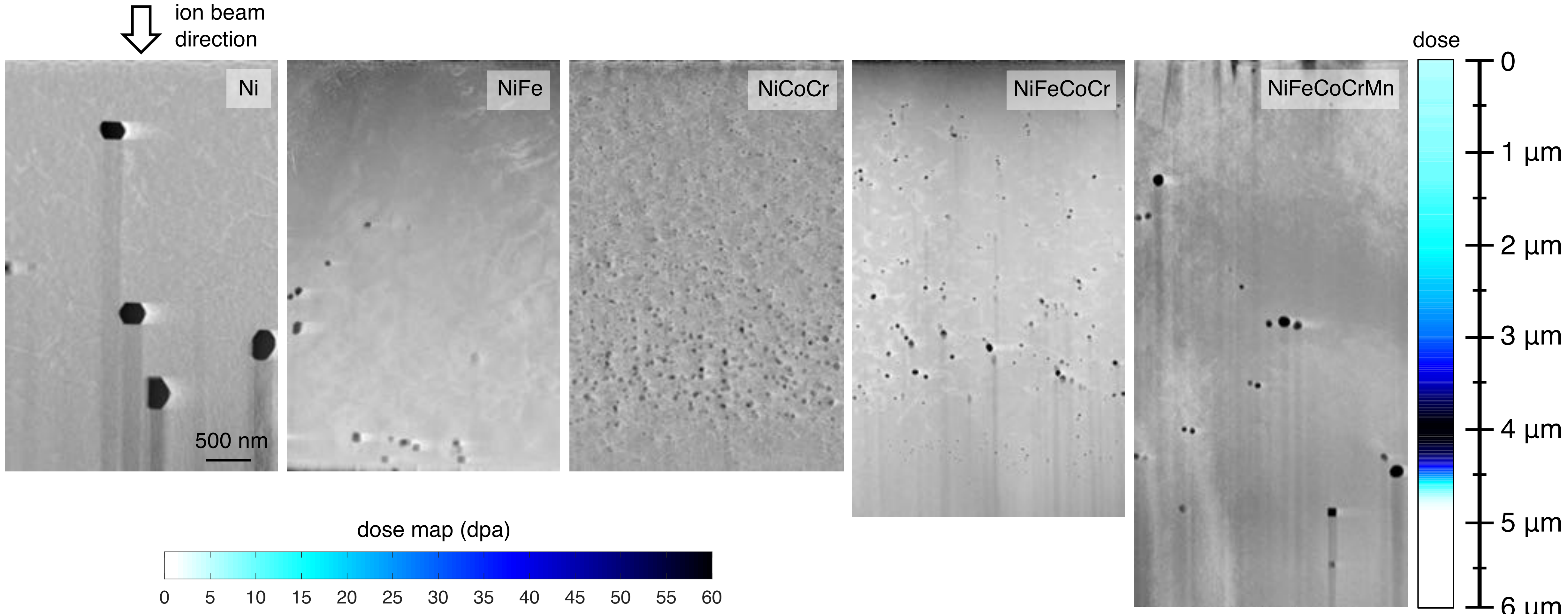
Irradiation Time [hr]
0 0.8 1.6 2.4 3.2 4 4.8 5.6 6.4 7.2 8 8.7 9.5
Thermal Diffusivity [cm²/s]
0.054 0.053 0.052 0.051 0.05 0.049
0 5 10 15 20 25 30 35 40 45 50 55 60
Peak Dose [dpa]



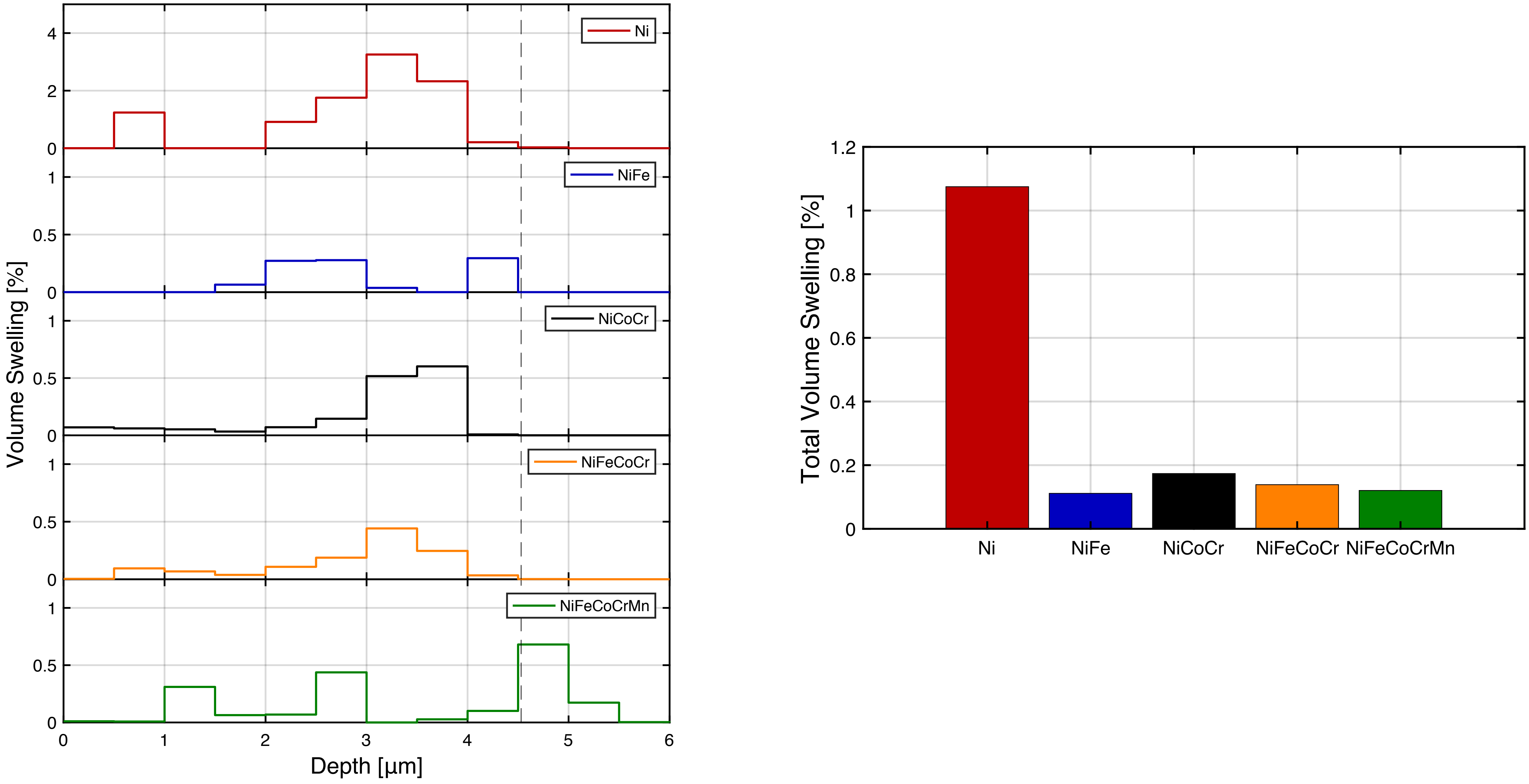
Irradiation Time [hr]
0 0.8 1.6 2.4 3.2 4 4.8 5.6 6.4 7.2 8 8.7 9.5
Current [nA]
166 164 162 160 158
SAW Speed [m/s]
2401 2400 2399 2398 2397 2396 2395 2394 2393 2392
Temp. [°C]
552 550 548
0 5 10 15 20 25 30 35 40 45 50 55 60
Peak Dose [dpa]



Normalized SAW Velocity [a.u.]
1.002
1
0.998
0.996
0.994
0.992
0.99
0.988
Ni
NiFe
NiCoCr
NiFeCoCr
NiFeCoCrMn
0 10 20 30 40 50 60
Dose [dpa]
Normalized Thermal Diffusivity [a.u.]
1.04
1.02
1
0.98
0.96
0.94
Ni
NiFe
NiCoCr
NiFeCoCr
NiFeCoCrMn
0 10 20 30 40 50 60
Dose [dpa]

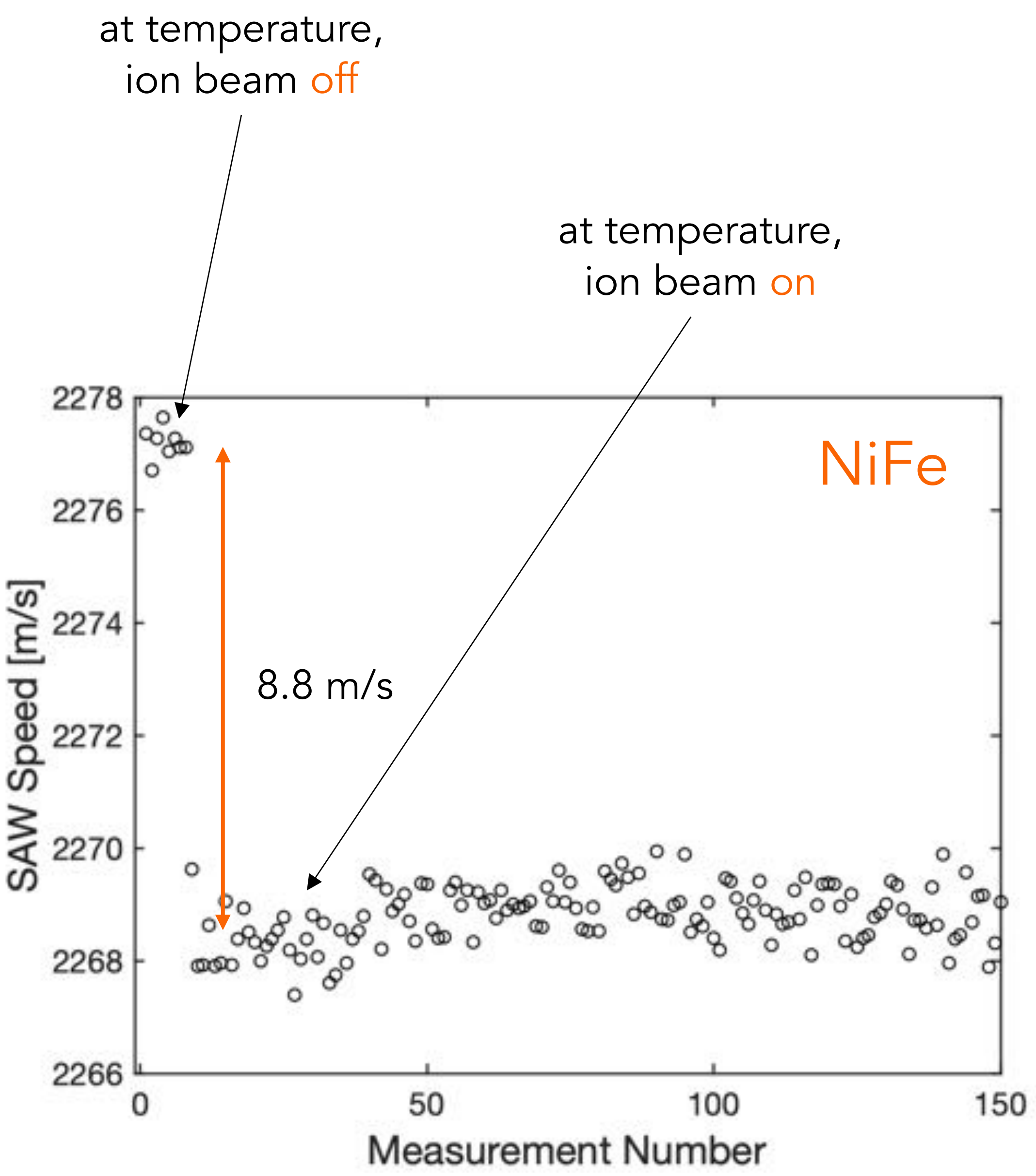


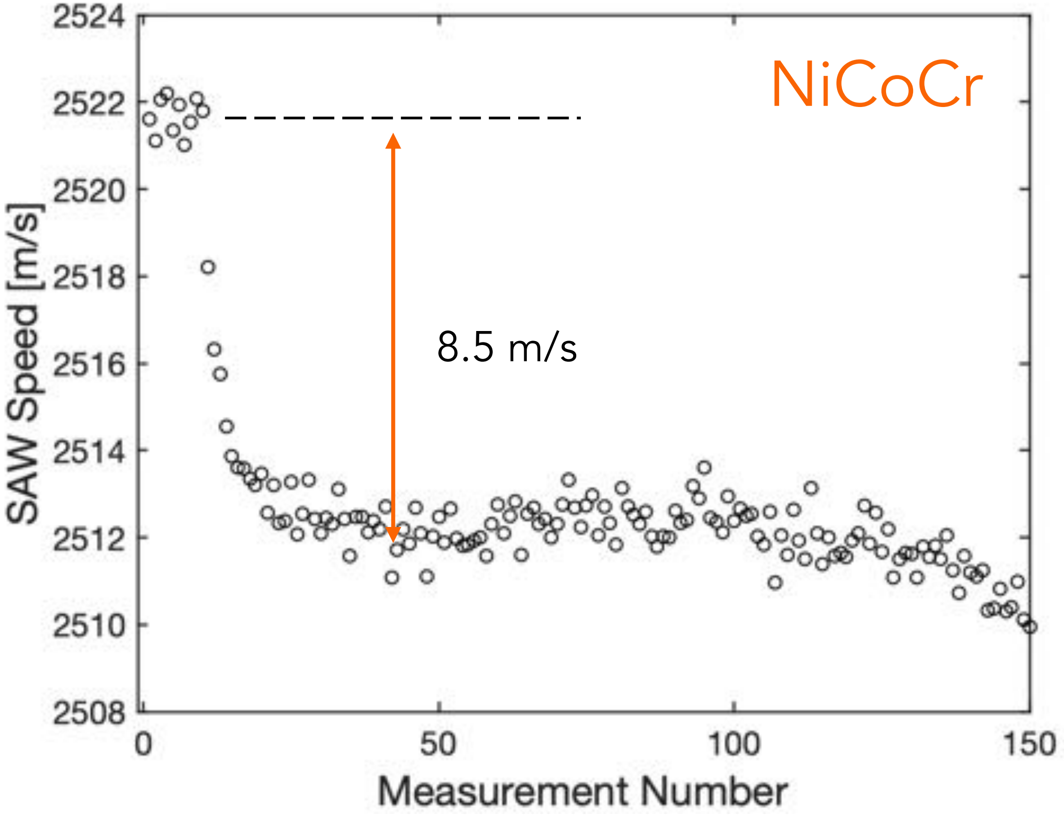
ion beam
direction
Ni
NiFe
NiCoCr
NiFeCoCr
NiFeCoCrMn
500 nm
dose
0
1 μm
2 μm
3 μm
4 μm
5 μm
6 μm
dose map (dpa)
0
5
10
15
20
25
30
35
40
45
50
55
60



Volume Swelling [%]
Depth [µm]
Ni
NiFe
NiCoCr
NiFeCoCr
NiFeCoCrMn
0
1
2
3
4
5
6
0.5
Total Volume Swelling [%]
1.2
0.8
0.6
0.4
0.2
Ni
NiFe
NiCoCr
NiFeCoCr
NiFeCoCrMn

`instantaneous' defect generation affects mechanical properties

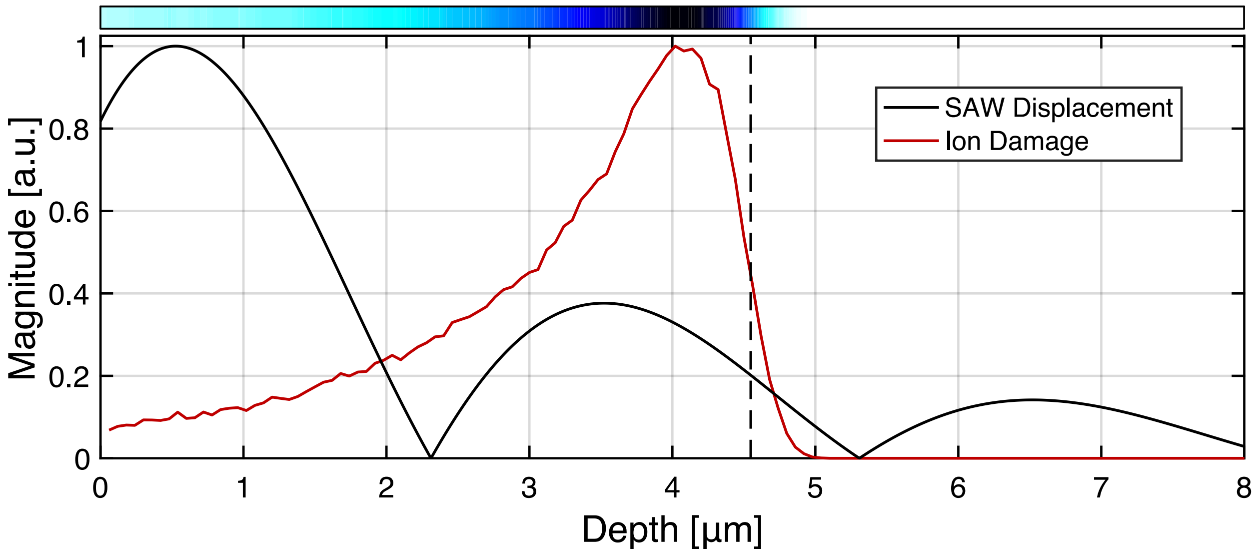




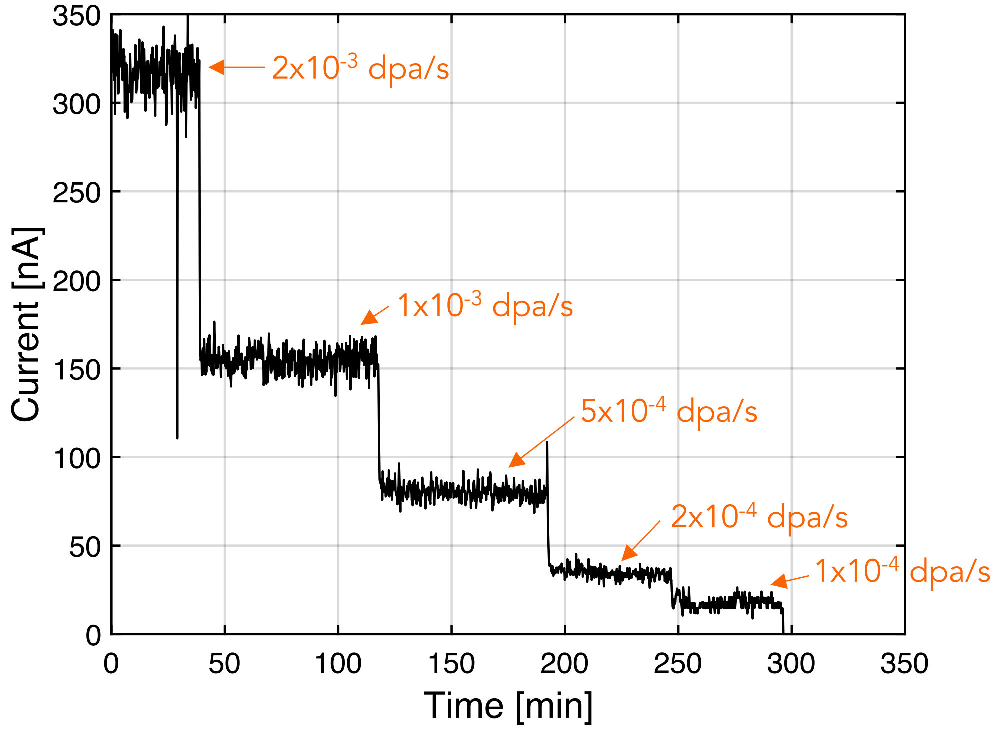
instantaneous defect effects at varying dose rates

surface angle	<100>{001}
ion species	Ni^{6+}
ion energy	31 MeV
temperature	500˚C
TGS wavelength	4.55 μm
dose rates	[1, 2, 5, 10, 20] x 10^{-4} dpa/s
measurement interval	~30 sec
annealing time between impulses	~30 min

31 MeV Ni^{5+} in Ni at 550˚C

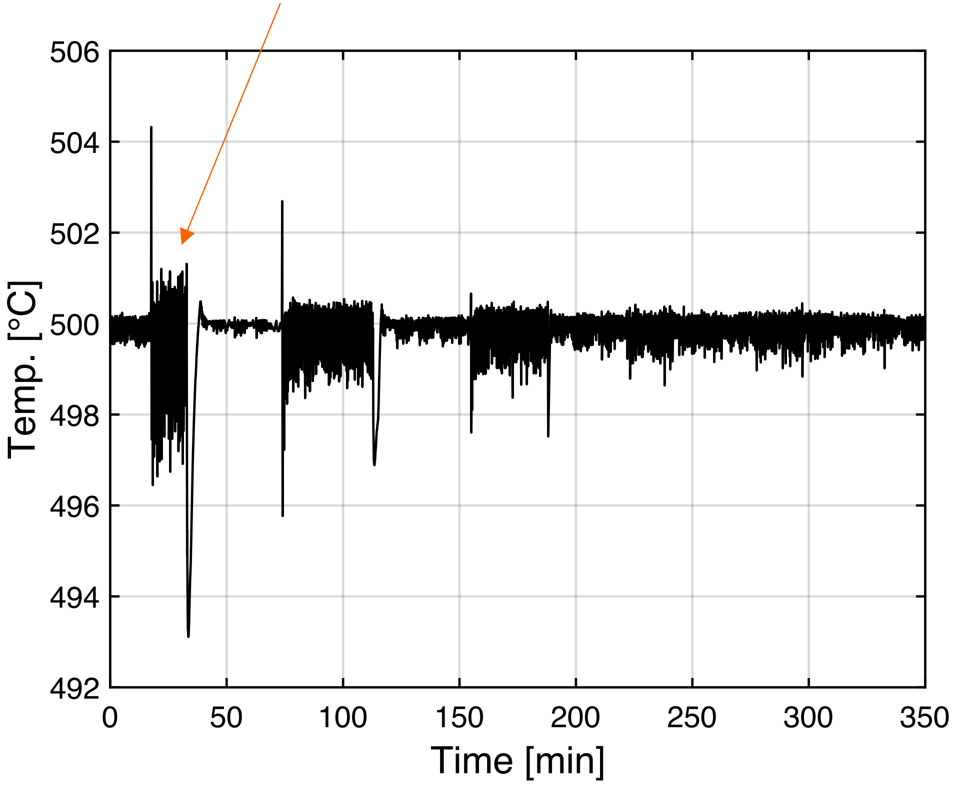


example current and temperature records for NiCoCr

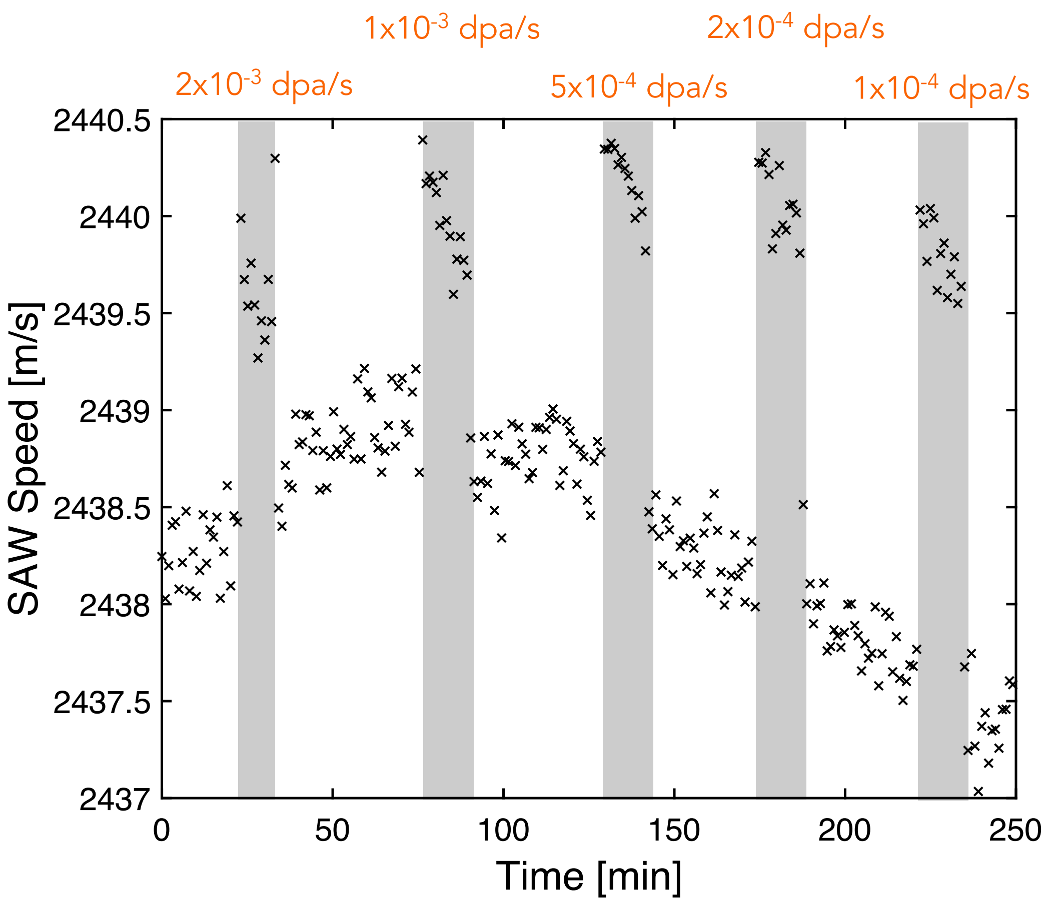


beam current lowered in steps between impulse experiments

higher beam heating and larger temperature fluctuations during high flux experiments

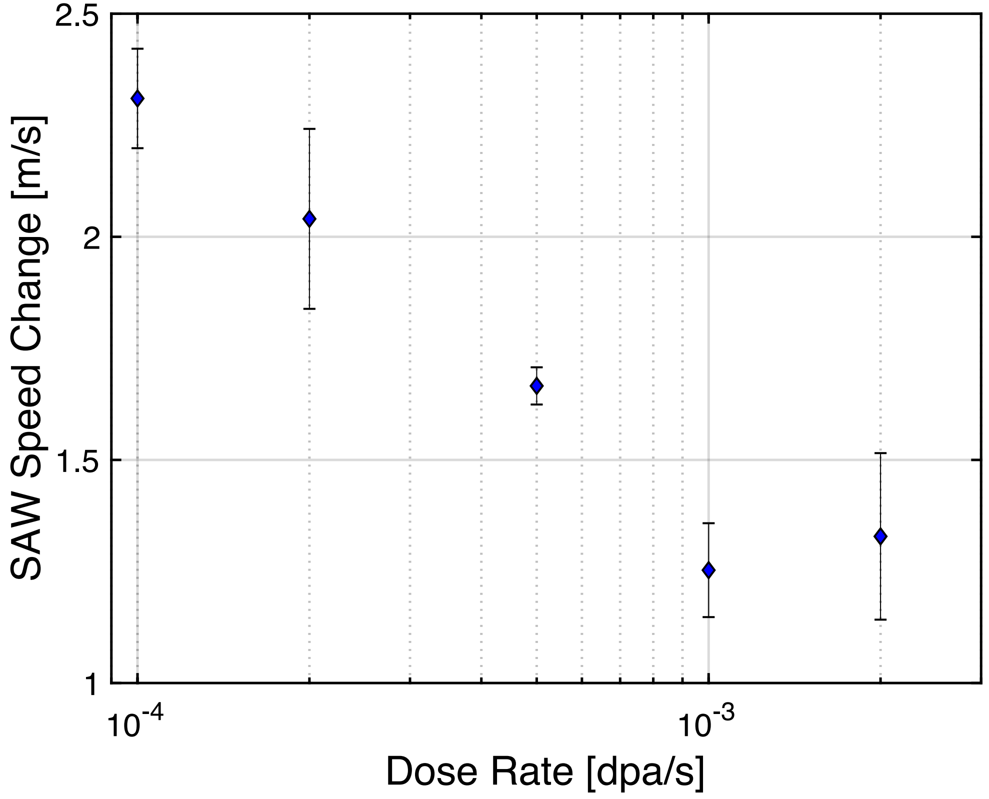


Pure Ni

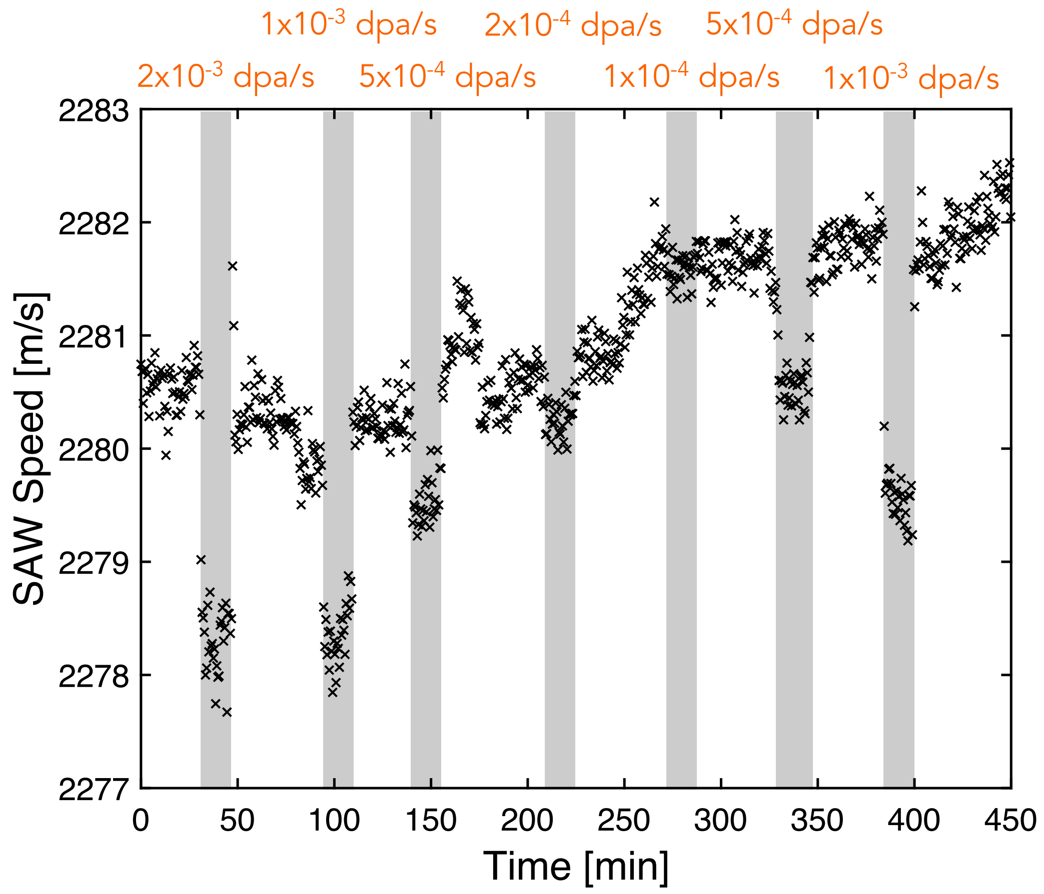


background defect accumulation continually evolving and not fully annealing

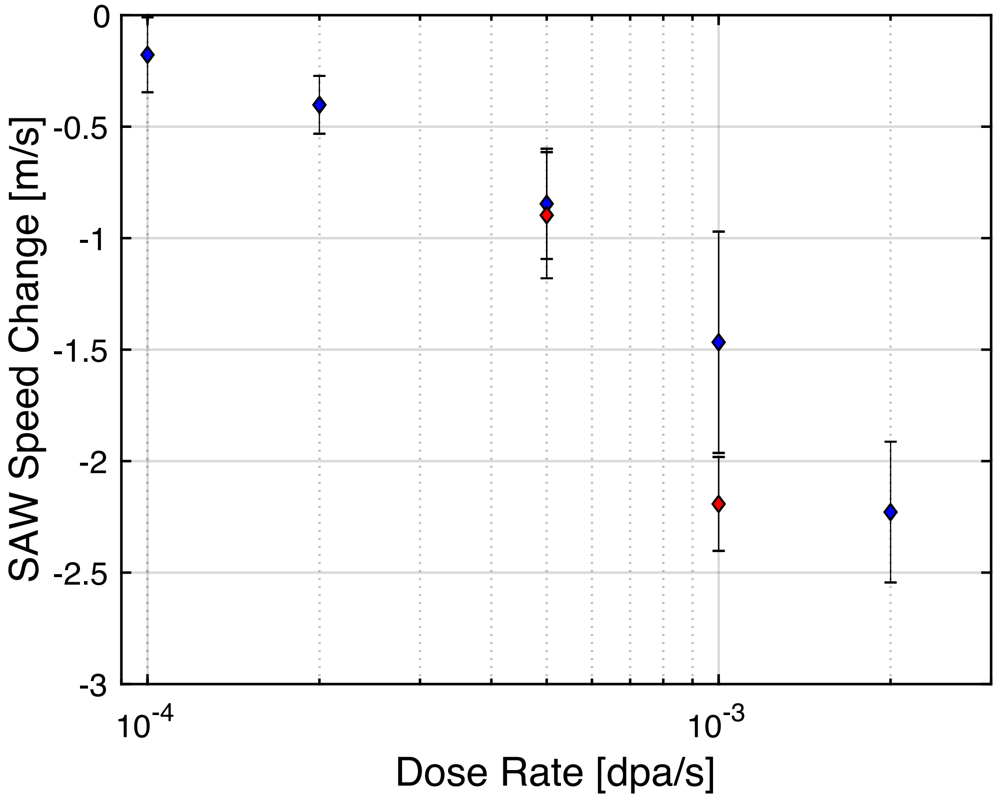
transient defect populations stiffen the elastic modulus, inversely correlated with dose rate



NiFe

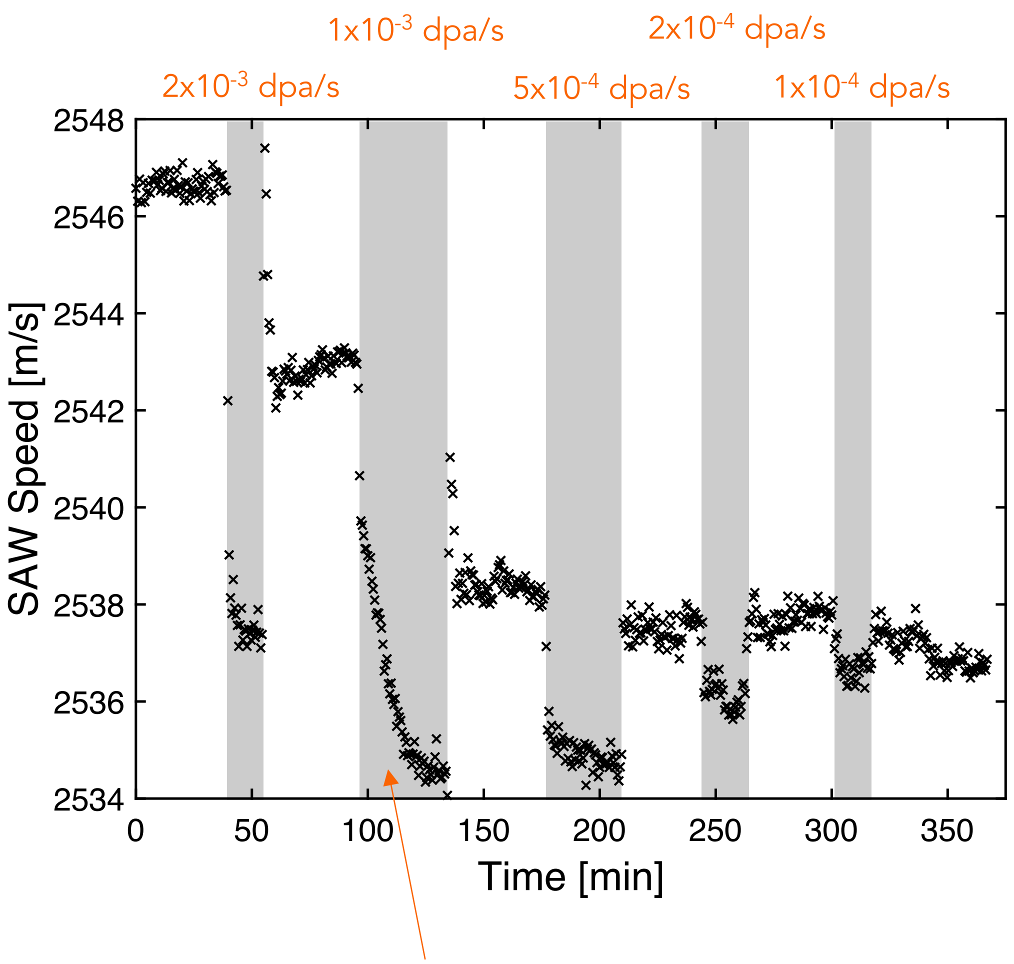


blue → dose rate high to low
red → dose rate low to high



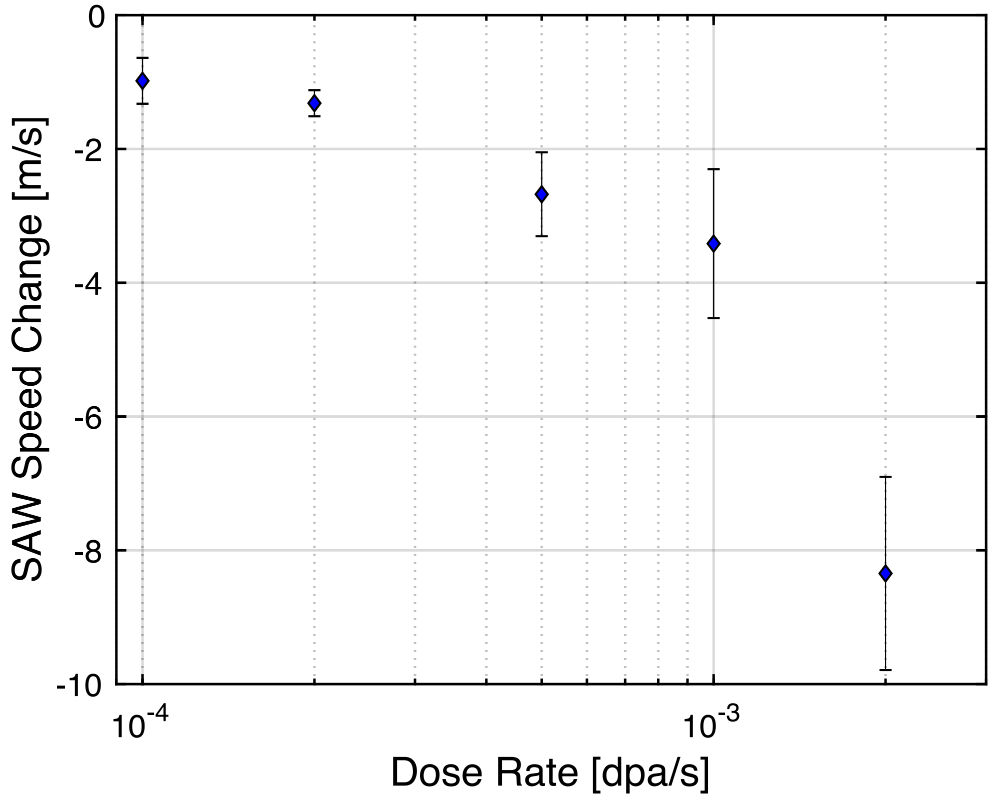
transient defect populations soften/reduce the elastic modulus, magnitude directly correlated with dose rate

NiCoCr

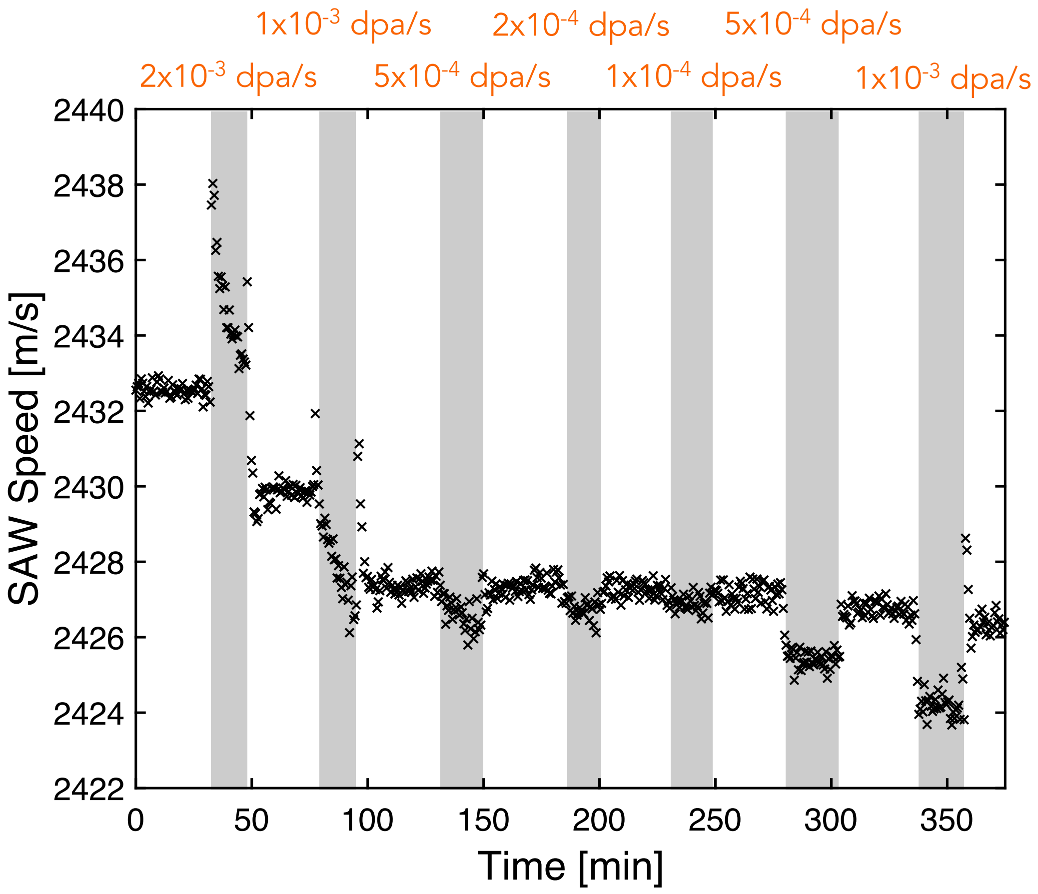


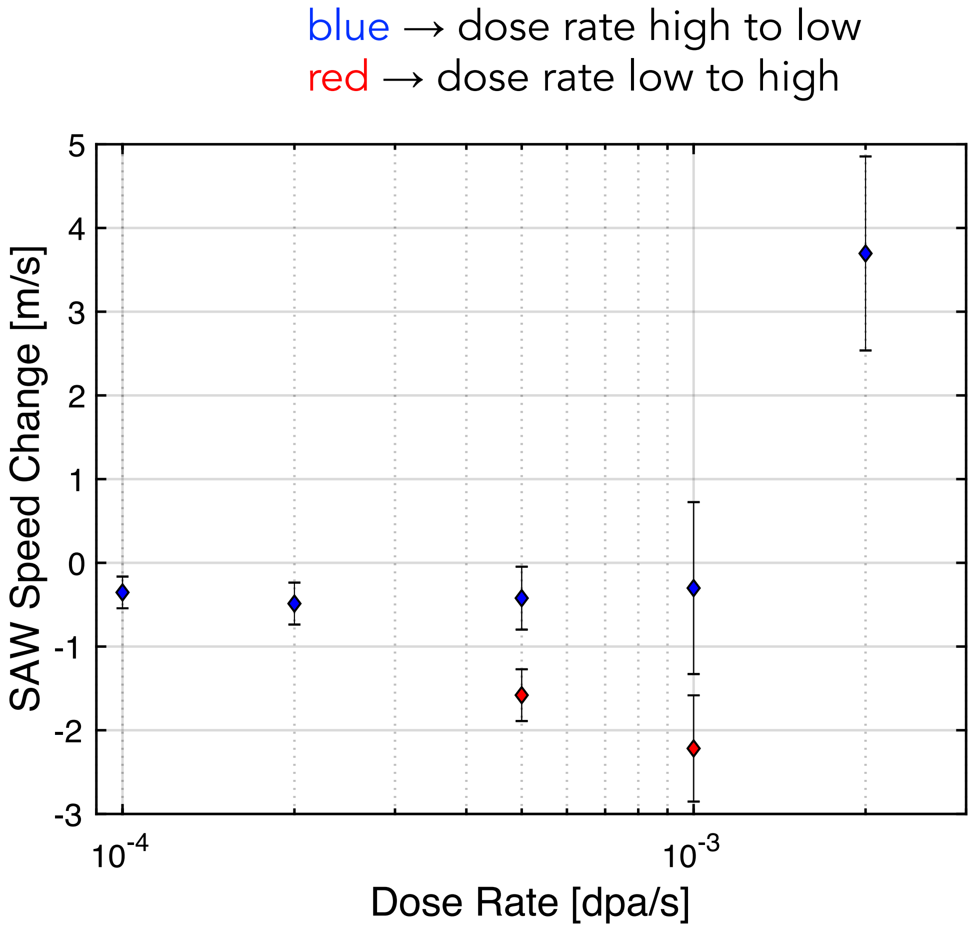
transient accumulation time longer than for other alloys

elastic constant reduction greatest observed of any alloy chemistry studied



NiFeCoCr





initial lattice stiffening is irreversible, softening
observed at identical dose rates following saturation

short-timescale defect accumulation observations and question

stiffening vs. softening in different alloys

defect type variation?

limitations of subsequent irradiation impulses

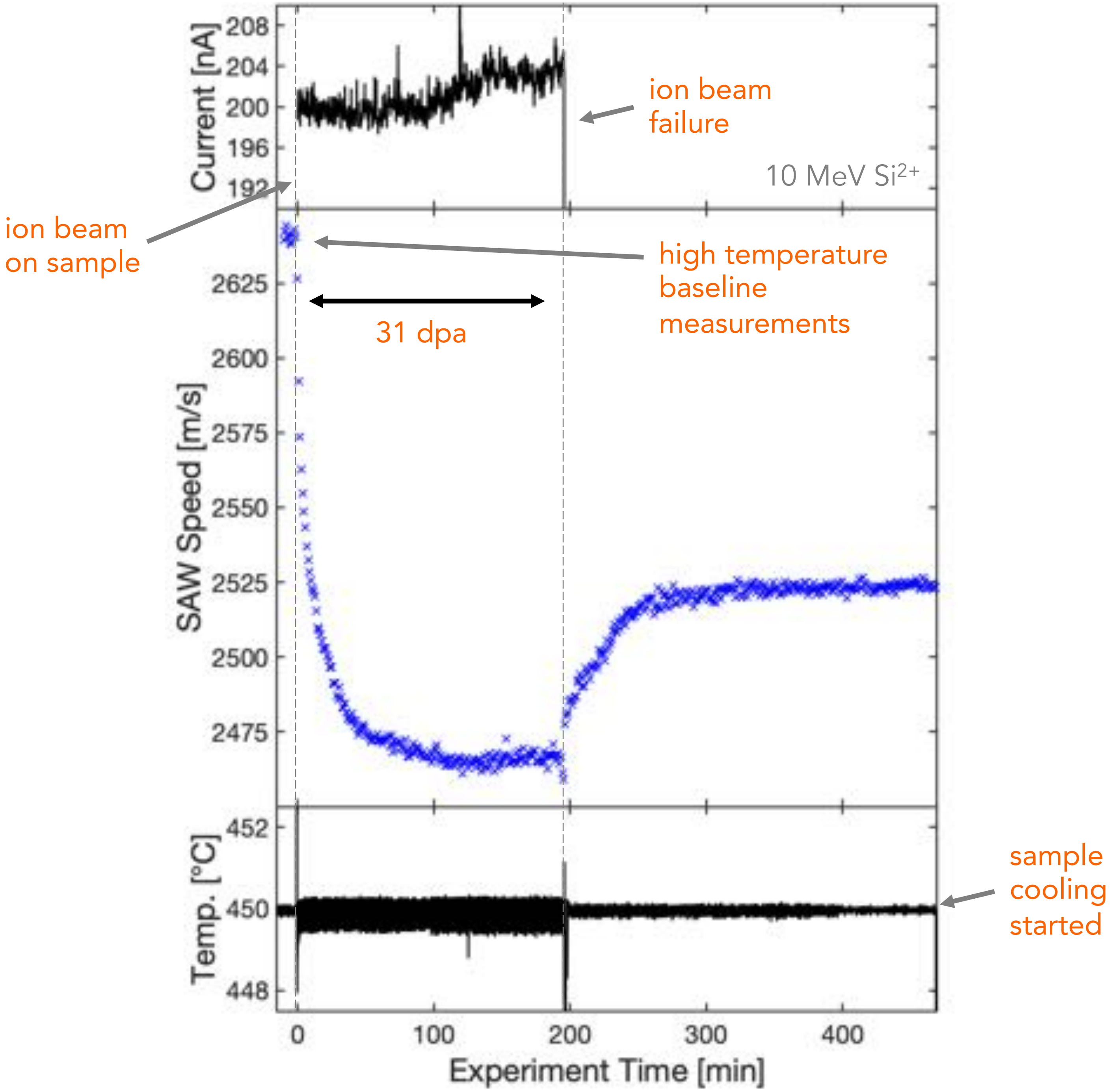
underlying background evolution?

higher temperatures needed for annealing?

in situ means interesting failure

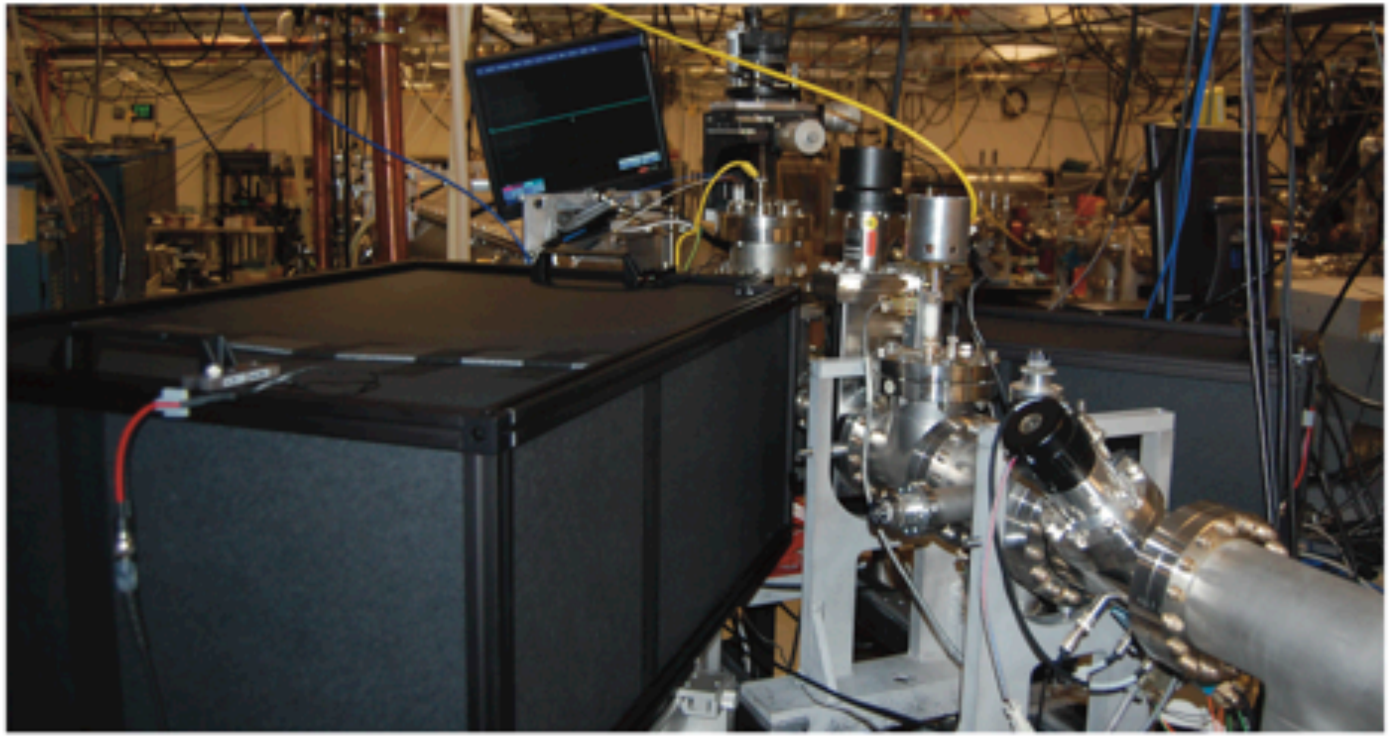
13Cr-7Al-23Zr-30Mo-24Nb-4Ta

refractory multiple principle element alloy



- *in situ* ion irradiation TGS beamline available for use at Sandia National Labs

- thermoelastic material properties explored directly *in situ* under extreme conditions at both short and long timescales



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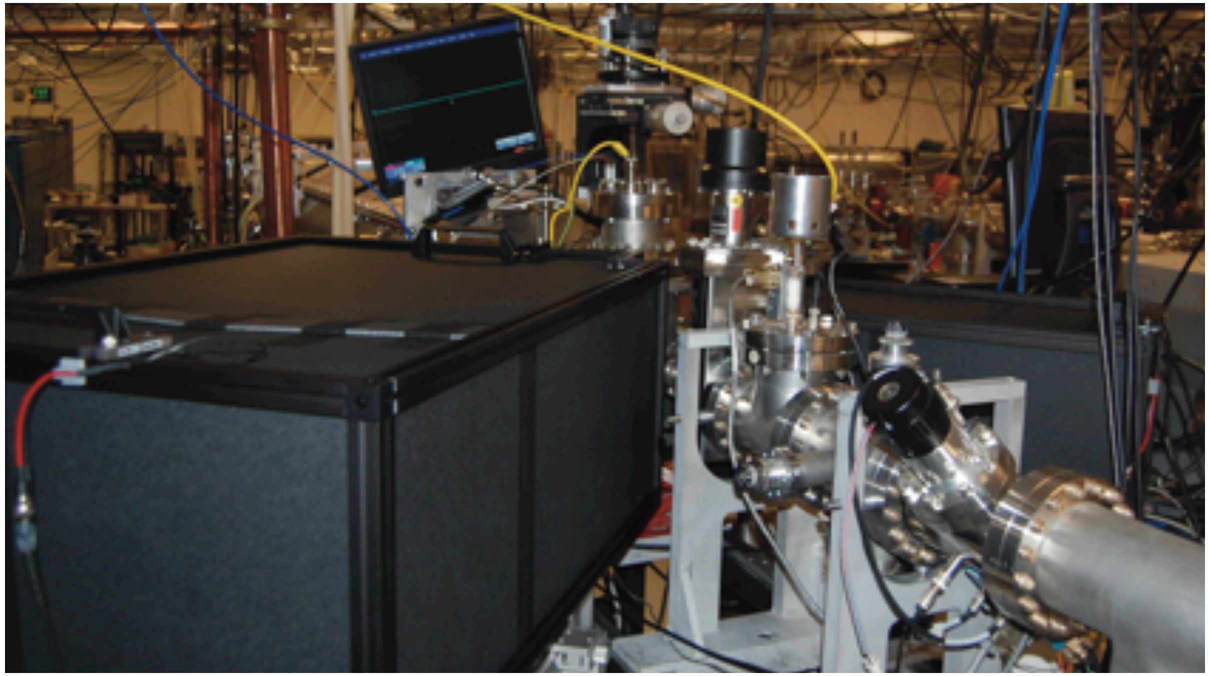
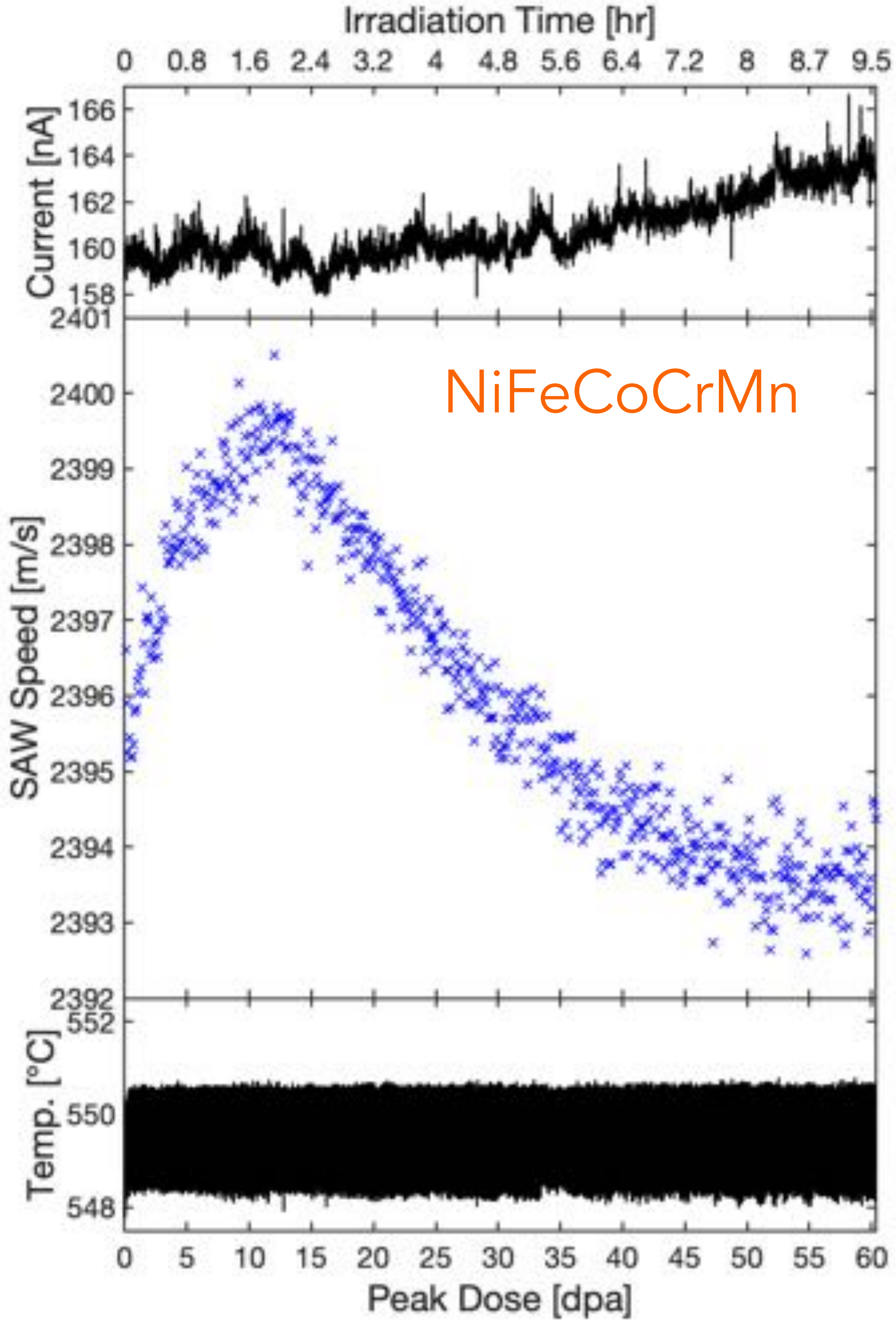
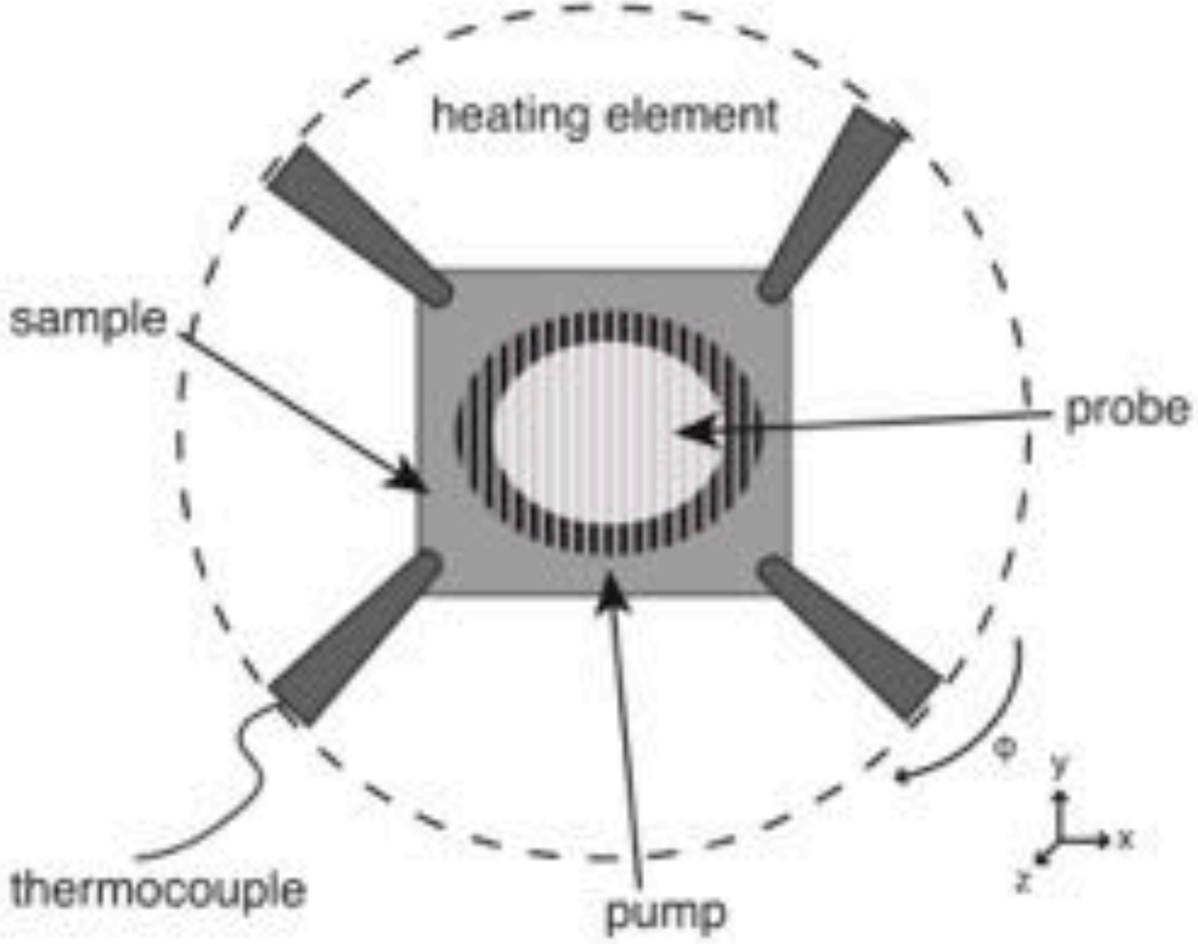
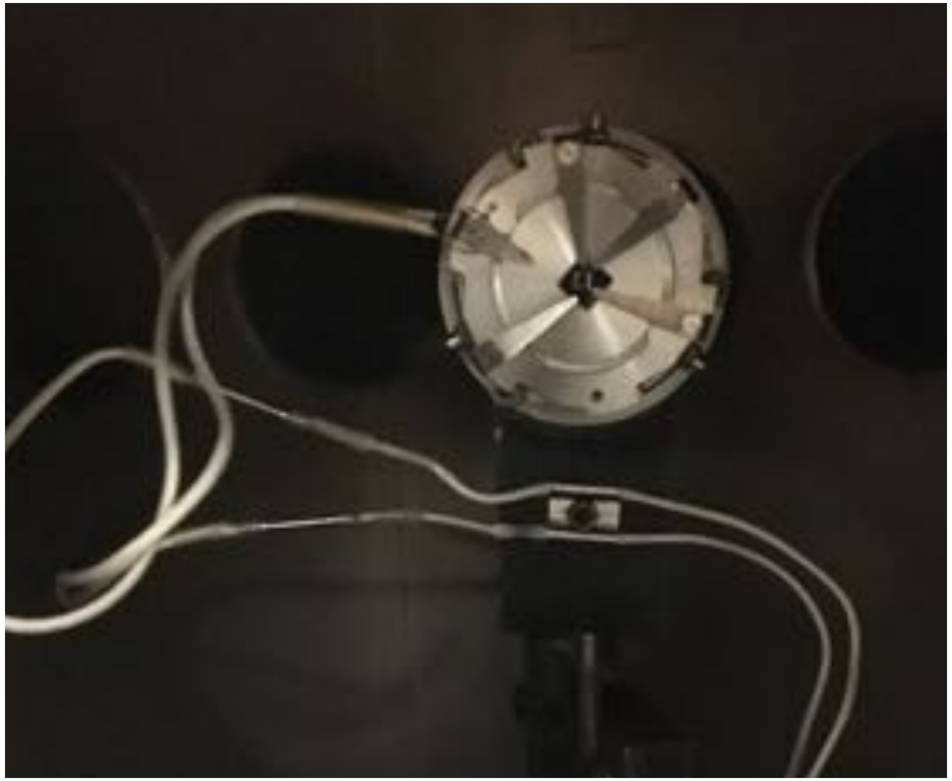




contact: **cody.dennett@inl.gov**



sample
in place



high temp.
operation