AMM
Advanced Methods for Manufacturing

DEC 2 – 3, 2020

# Nanodispersion Strengthened Metallic Composites with Enhanced Neutron Irradiation Tolerance

*Award Number: DE-NE0008827*

*Award Dates: 10/2018 to 09/2021*

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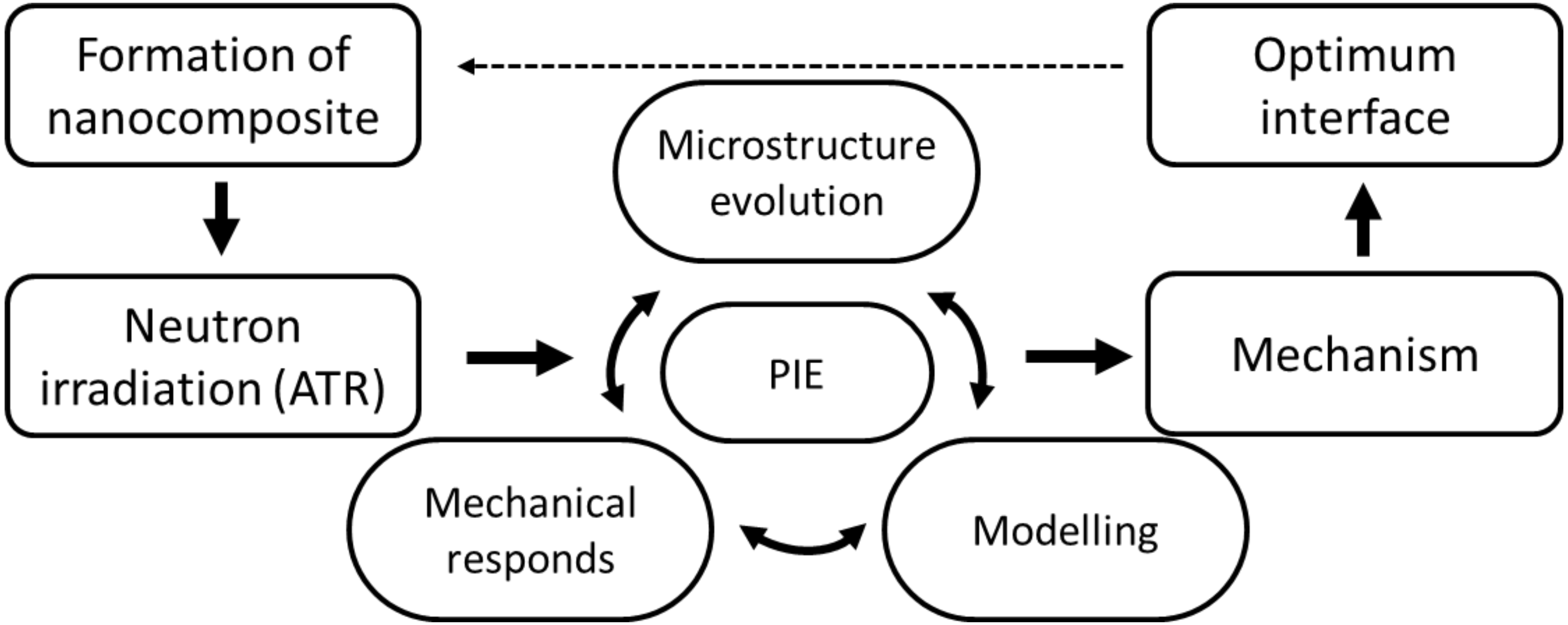
*Team Members: Mike Short, Kang Pyo So, So Yeon Kim, Alexander O'Brien, Myles Stapelberg*

# Project Objectives

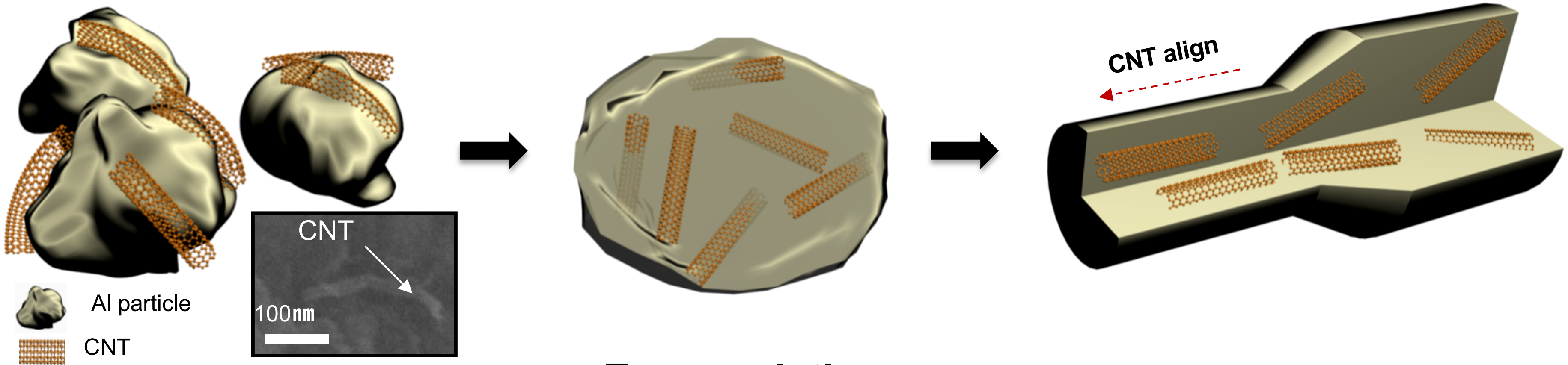
- Manufacturing and pre-characterization of nanocomposites
- Neutron irradiation of nanocomposites in a test reactor
- Microstructure characterization by SEM/FIB/TEM
- Mechanical properties characterization by uniaxial tensile testing
- Mesoscale modelling of neutron irradiation of nanocomposite materials
- Establishing a general theory of neutron-nanocomposite interactions

# Scope of work

Neutron radiation of three capsules in High Flux Isotope Reactor (HFIR).
TEM evaluation of 39 material conditions as summarized in Table.
Mechanical testing of 39 materials conditions as summarized in Table.
Atom probe tomography of 13 materials conditions as summarized in Table.



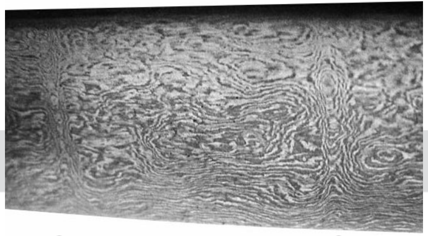
# Aluminum/Carbon Nanotube composite




**Declustered CNTs on Al** → **Encapsulation & consolidation** → **Extrusion**


ASTM E8 tensile specimen

Dr. KangPyo So
@ Li group



*"Damask"*

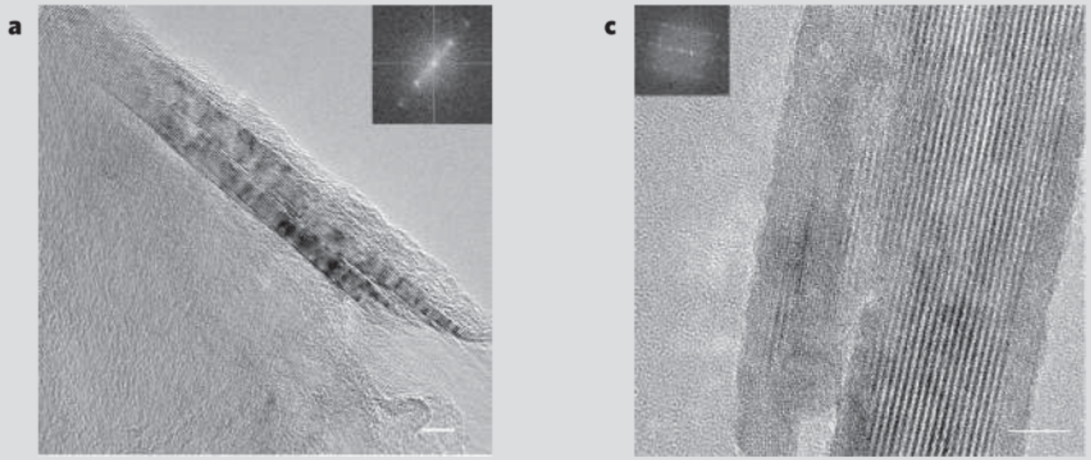


# Carbon nanotubes in an ancient Damascus sabre

The steel of Damascus blades, which were first encountered by the Crusaders when fighting against Muslims, had features not found in European steels — a characteristic wavy banding pattern known as damask, extraordinary mechanical properties, and an exceptionally sharp cutting edge. Here we use high-resolution transmission electron microscopy to examine a sample of Damascus sabre steel from the seventeenth century and find that it contains carbon nanotubes as well as cementite nanowires. This microstructure may offer insight into the beautiful banding pattern of the ultrahigh-carbon steel created from an ancient recipe that was lost long ago.

It is believed that Damascus blades were forged directly from small cakes of steel (named '*wootz*') produced in ancient India. A sophisticated thermomechanical treatment of forging and annealing was applied to these cakes to refine the steel to its exceptional quality. However, European bladesmiths were unable to replicate the process, and its secret was lost at about the end of the eighteenth century. It was unclear how medieval blacksmiths would have overcome the inherent brittleness of the plates of cementite ($Fe_3C$, a mineral known as cohenite) that form in steel with a carbon content of 1–2 wt%, as well as how the steel's

*cast iron?*



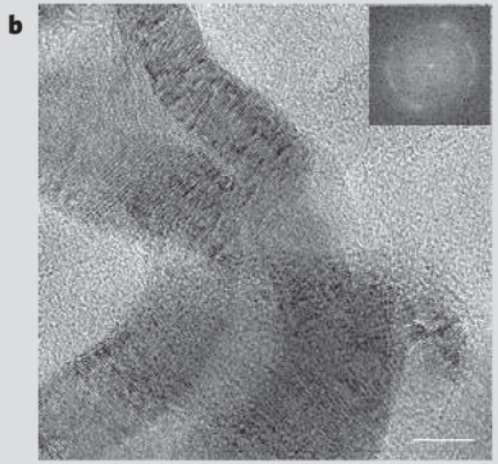
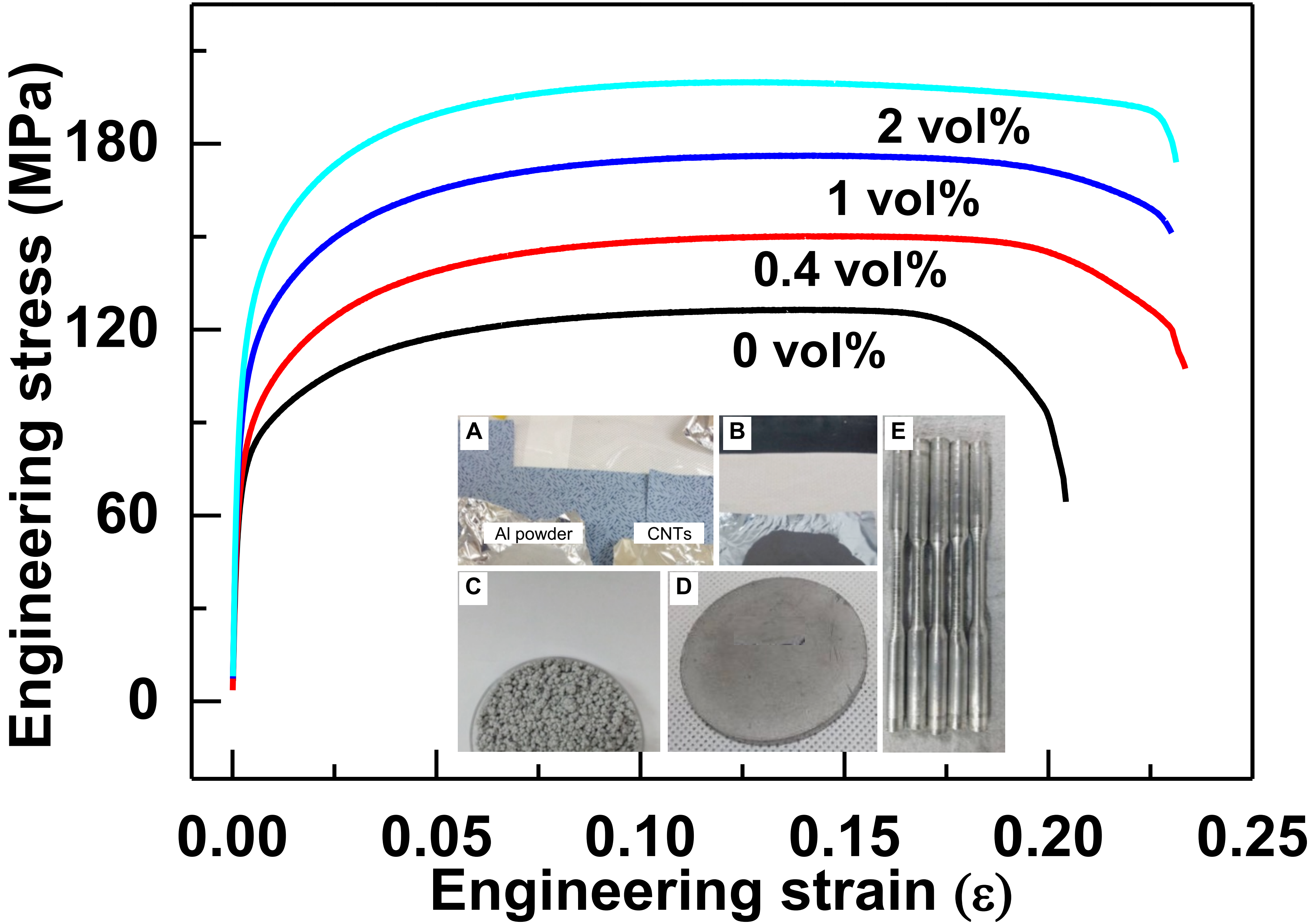




**Figure 1 | High-resolution transmission electron microscopy images of carbon nanotubes in a genuine Damascus sabre after dissolution in hydrochloric acid.** **a**, **b**, Multiwalled tubes with the characteristic layer distance $d \approx 0.34$ nm (ref. 12), as indicated by the Fourier transforms (see insets). Scale bars: 5 nm (**a**) and 10 nm (**b**). In **b**, the tubes are bent like a rope. **c**, Remnants of cementite nanowires encapsulated by carbon nanotubes, which prevent the wires from dissolving in acid. Scale bar, 5 nm. The fringe spacing of the wire is 0.635 nm, taken from the Fourier transform (inset), and is attributed to the (010) lattice planes of cementite.

# Superior Mechanical Performance



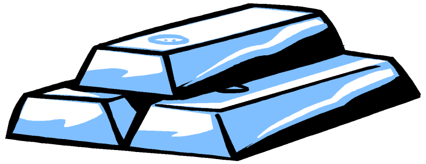

**With Prof. Young Hee Lee at Sungkyunkwan University:**

"Ton-scale metal-carbon nanotube composite: The mechanism of strengthening while retaining tensile ductility,"

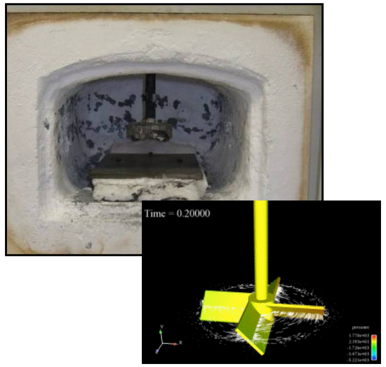
Kang Pyo So et al, *Extreme Mechanics Letters* **8** (2016) 245

# Wettability is Critical for Dispersion of CNT into Al

+

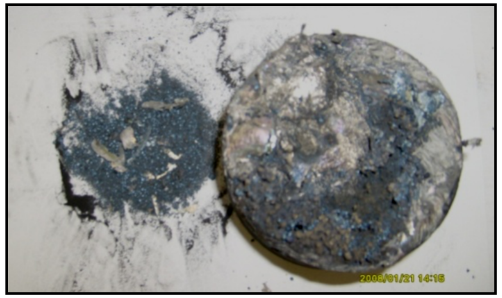


**Melt blending**



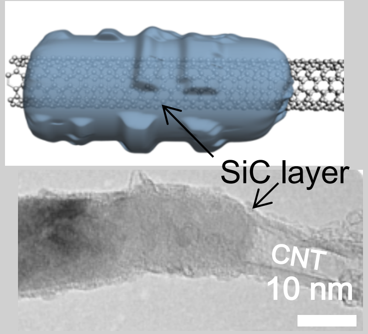
http://www.acusim.com/
images/gallery/startup.gif

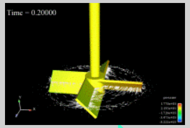
**Failed !!**



**CNT dispersed!!**

**Introduce SiC coating !!**

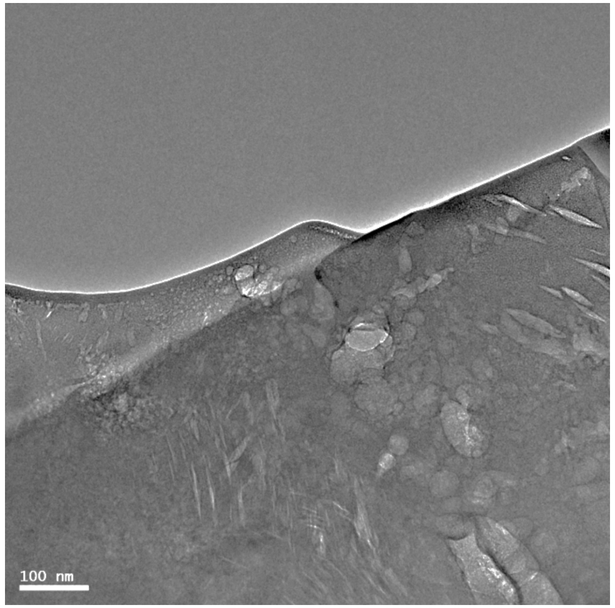


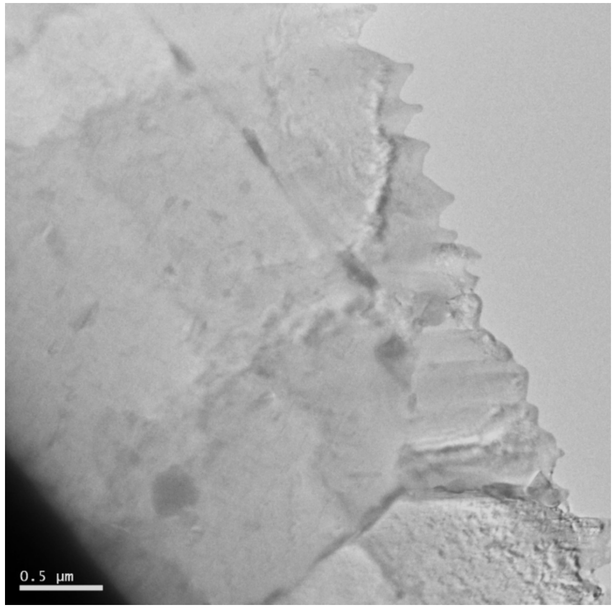



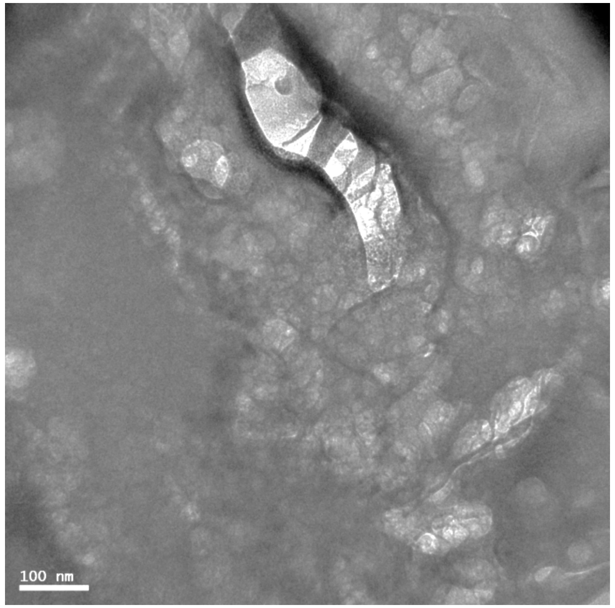
**CNT was successfully dispersed in Al melt**

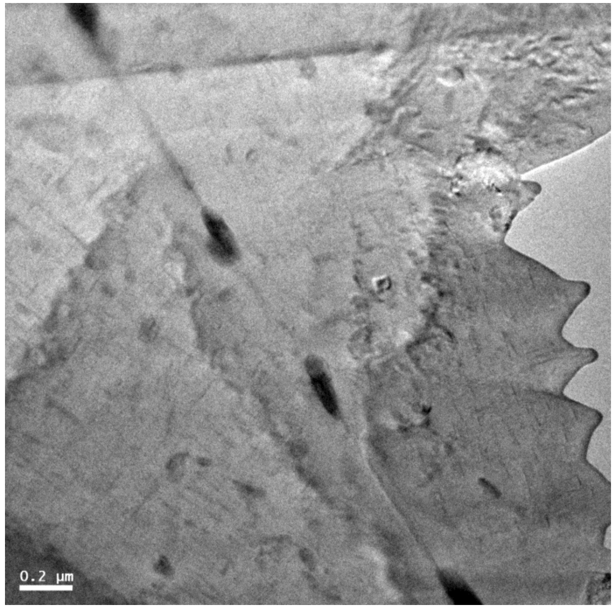

Impeller
Aluminum

# 100keV He ion, fluence $10^{17}/cm^2$, peak dose 3.5 DPA



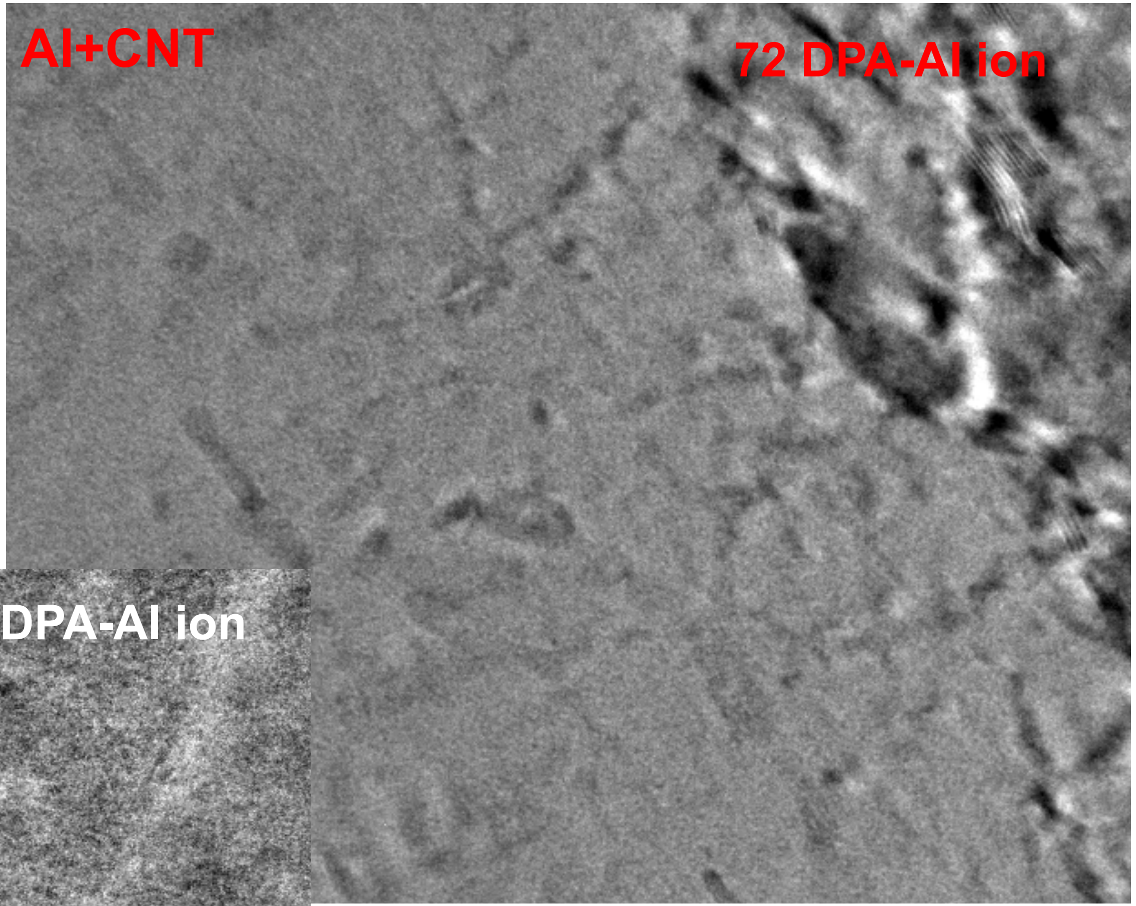



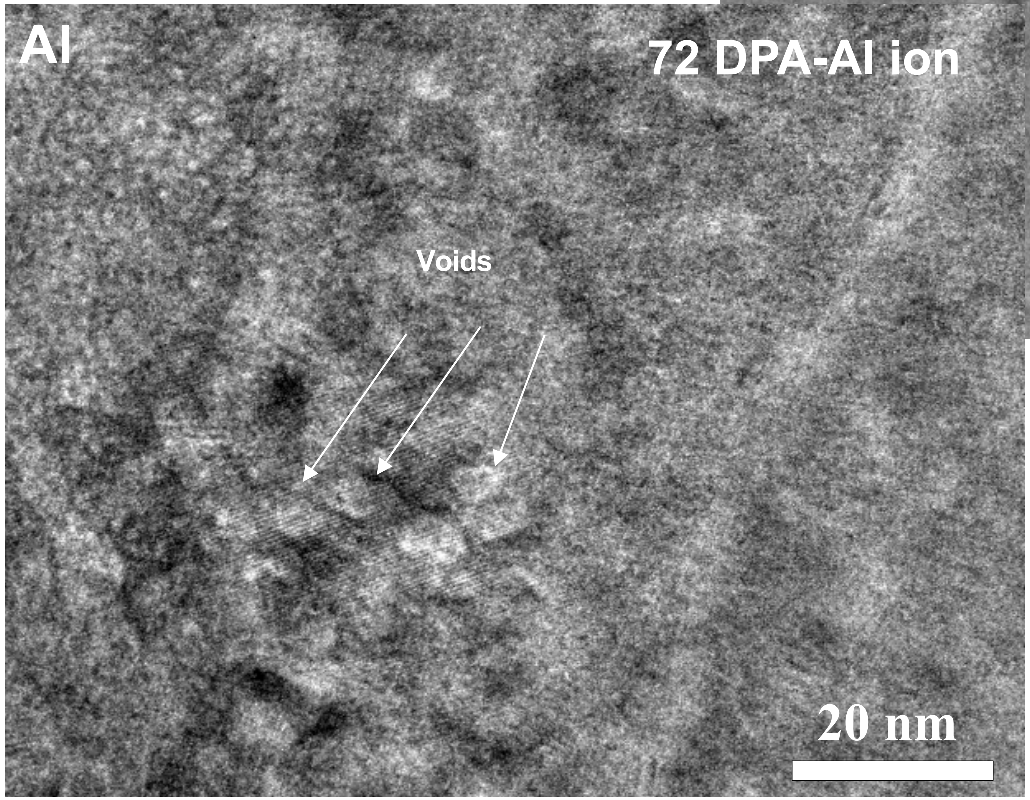




pure Al: pore size 5~50 nm

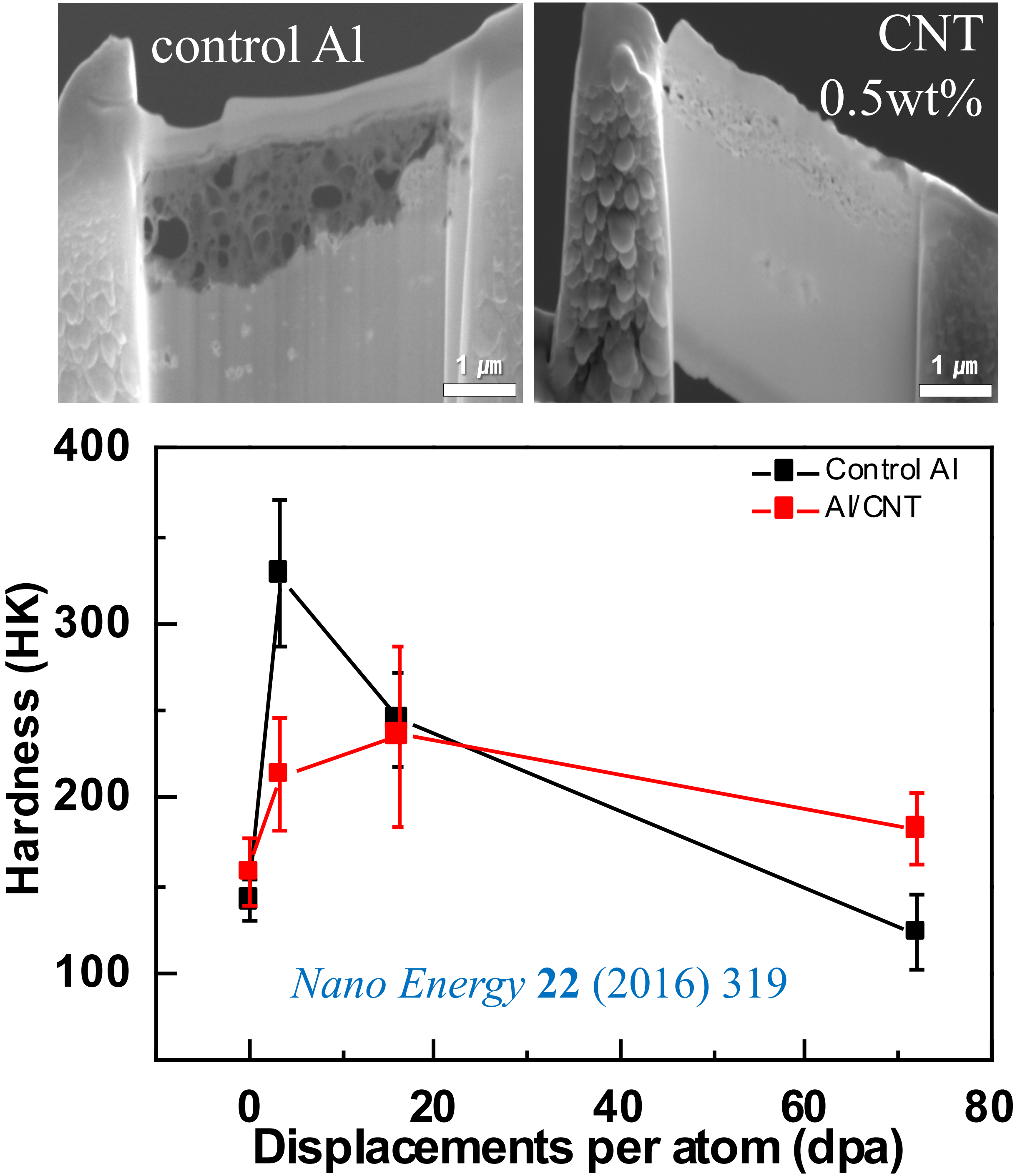
0.5wt%CNT/Al: no pore observed

# Al self-ion radiation




*Nano Energy* **22** (2016) 319

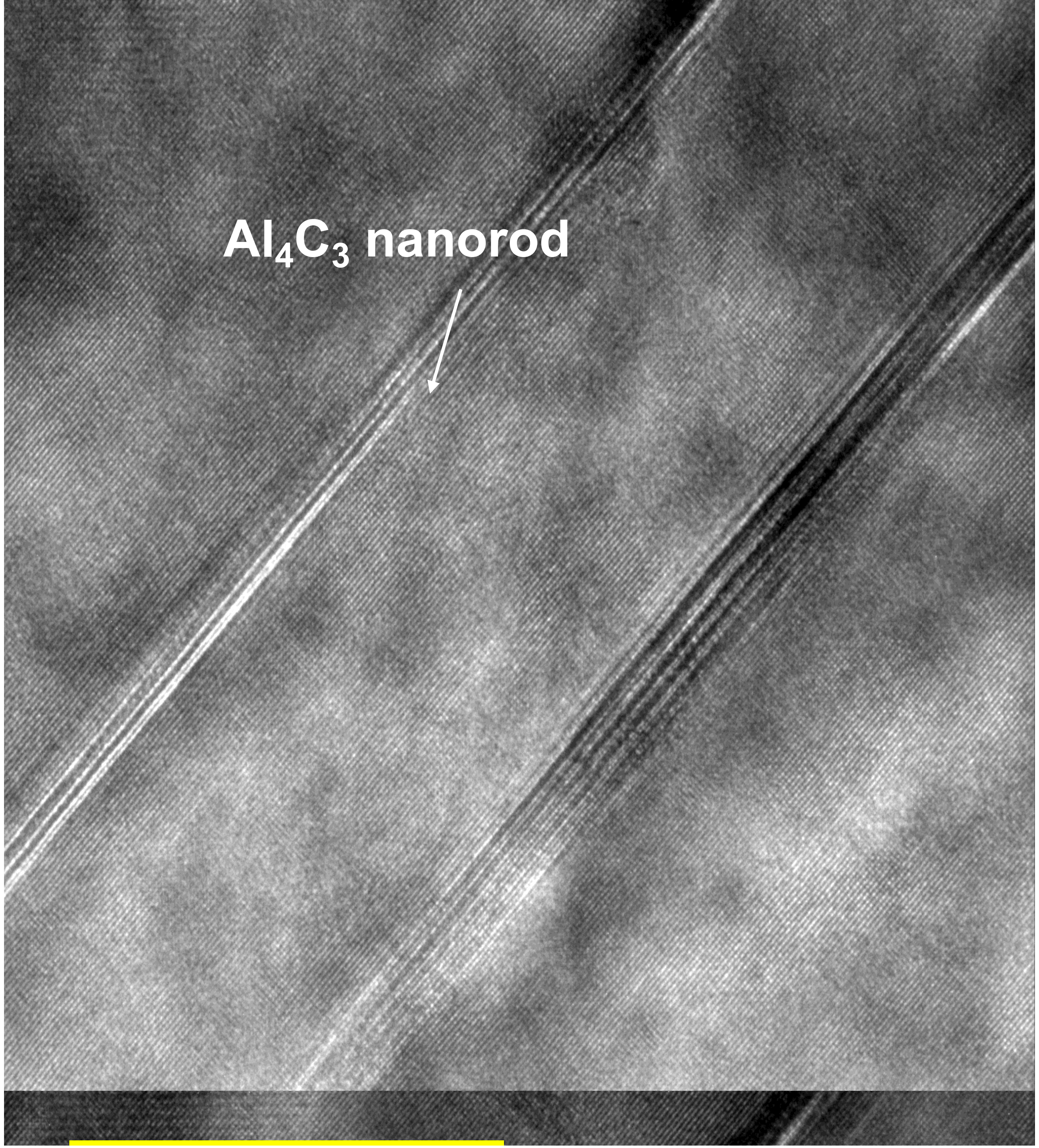



*Nano Energy* **22** (2016) 319

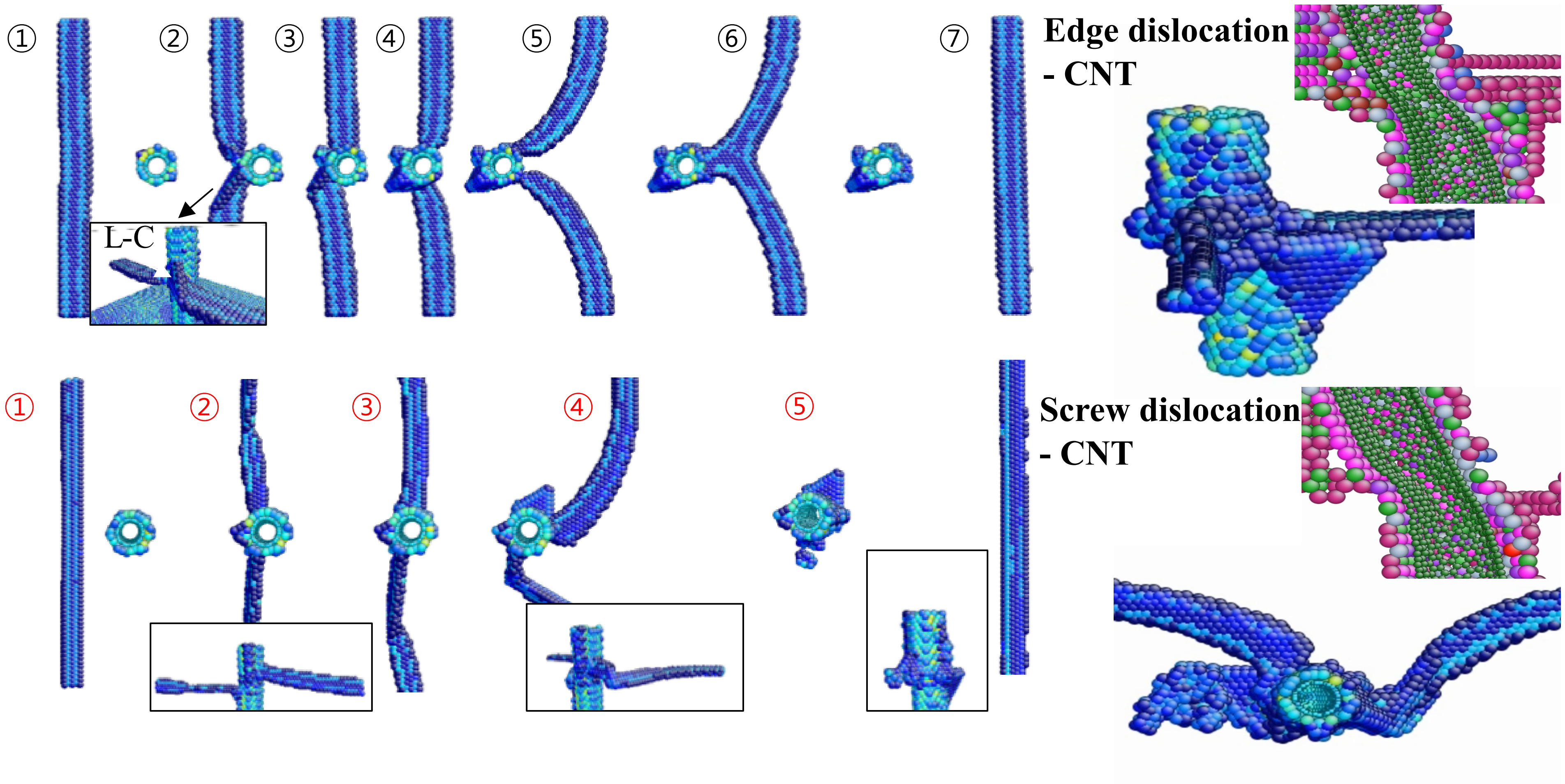
**Al self-ion radiation**

**Carbon nanotube**
**↓**
**Tubular carbide**

*Nano Energy* **22** (2016) 319

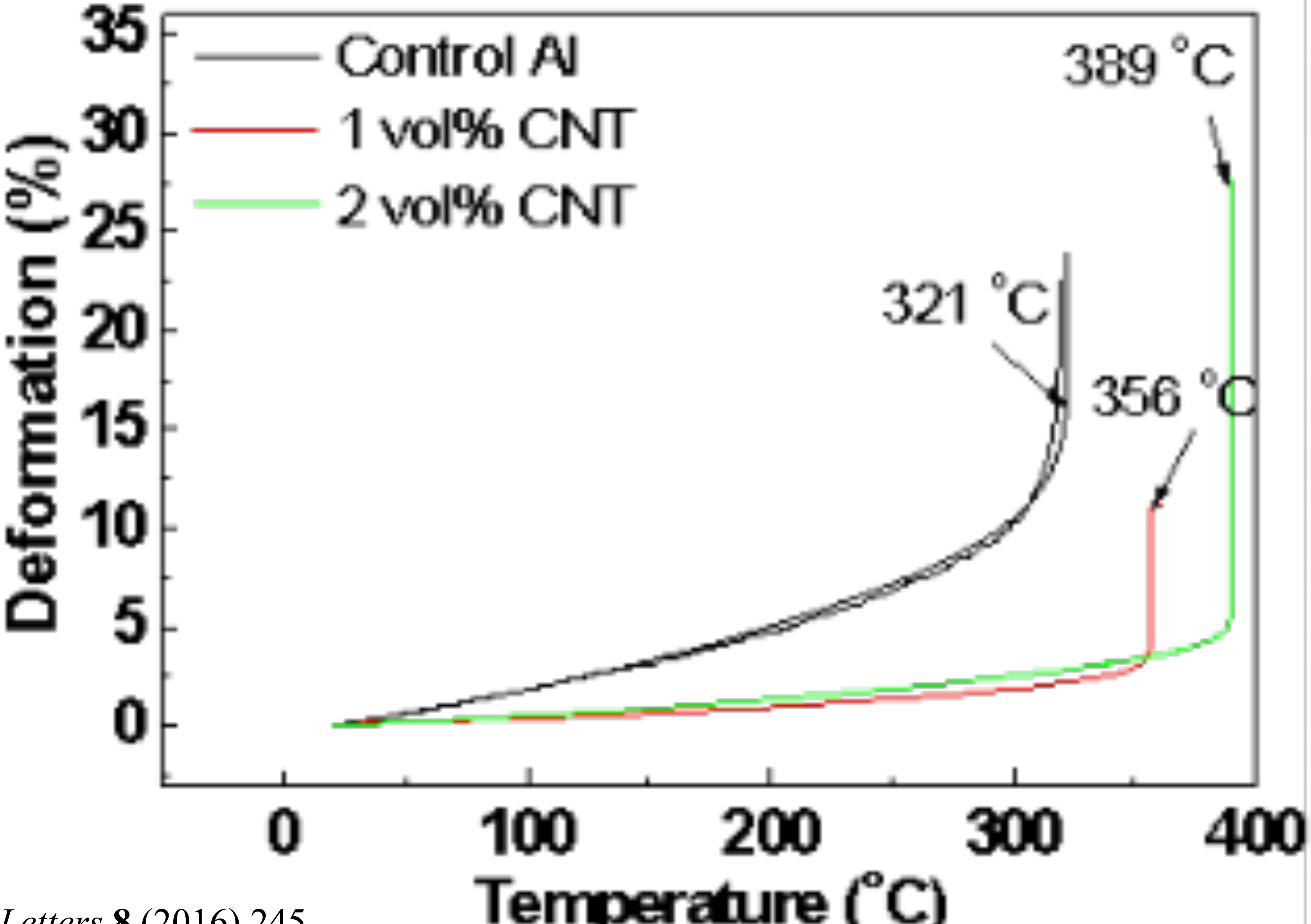


# Enhanced Creep Resistance

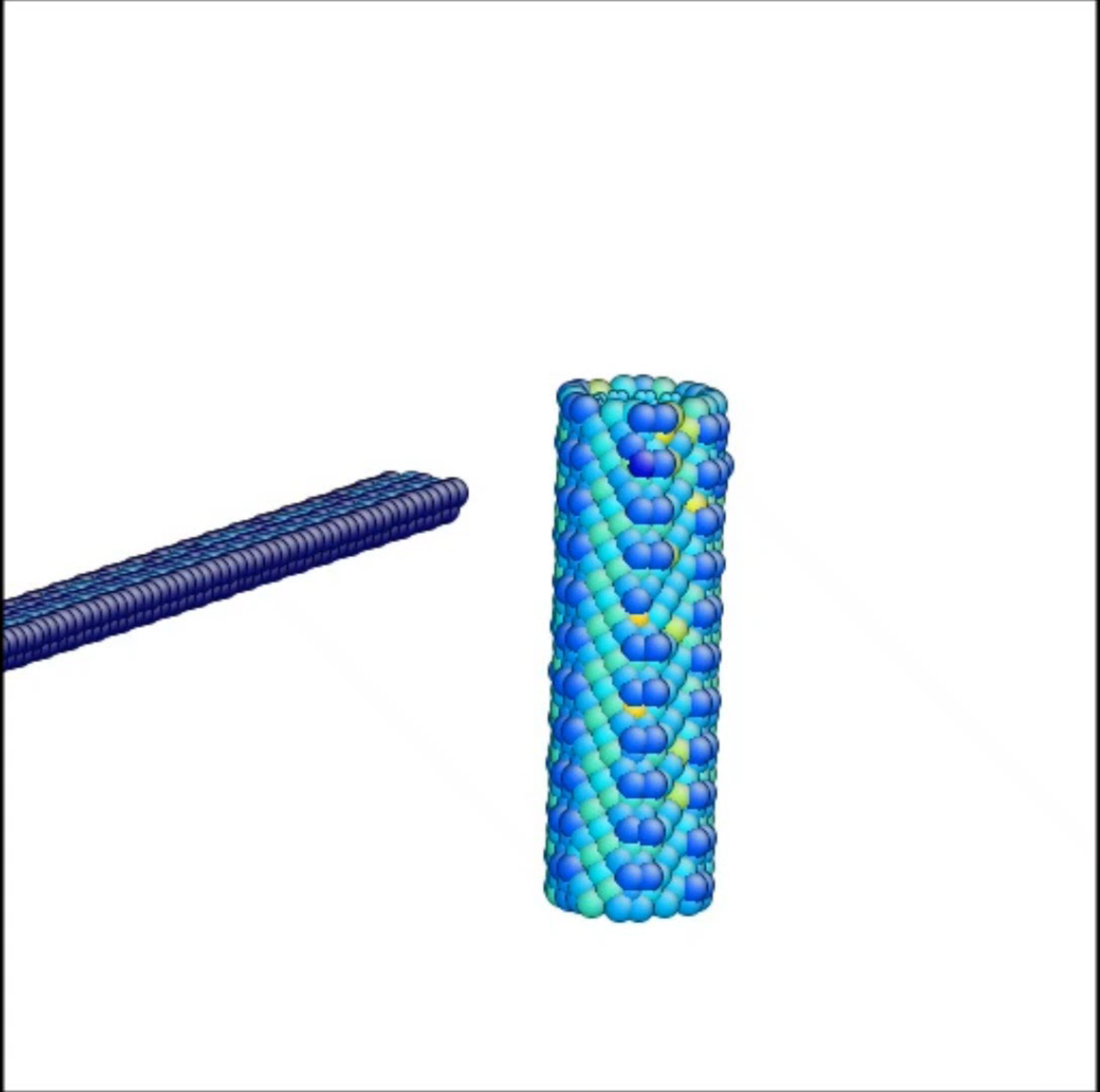



- Unlike 0D obstacle, dislocation **cannot climb over** 1D obstacle even at high temperatures
- 1D/2D obstacles **pin GB differently** from traditional Zener pinning theory

# Enhanced Creep Resistance



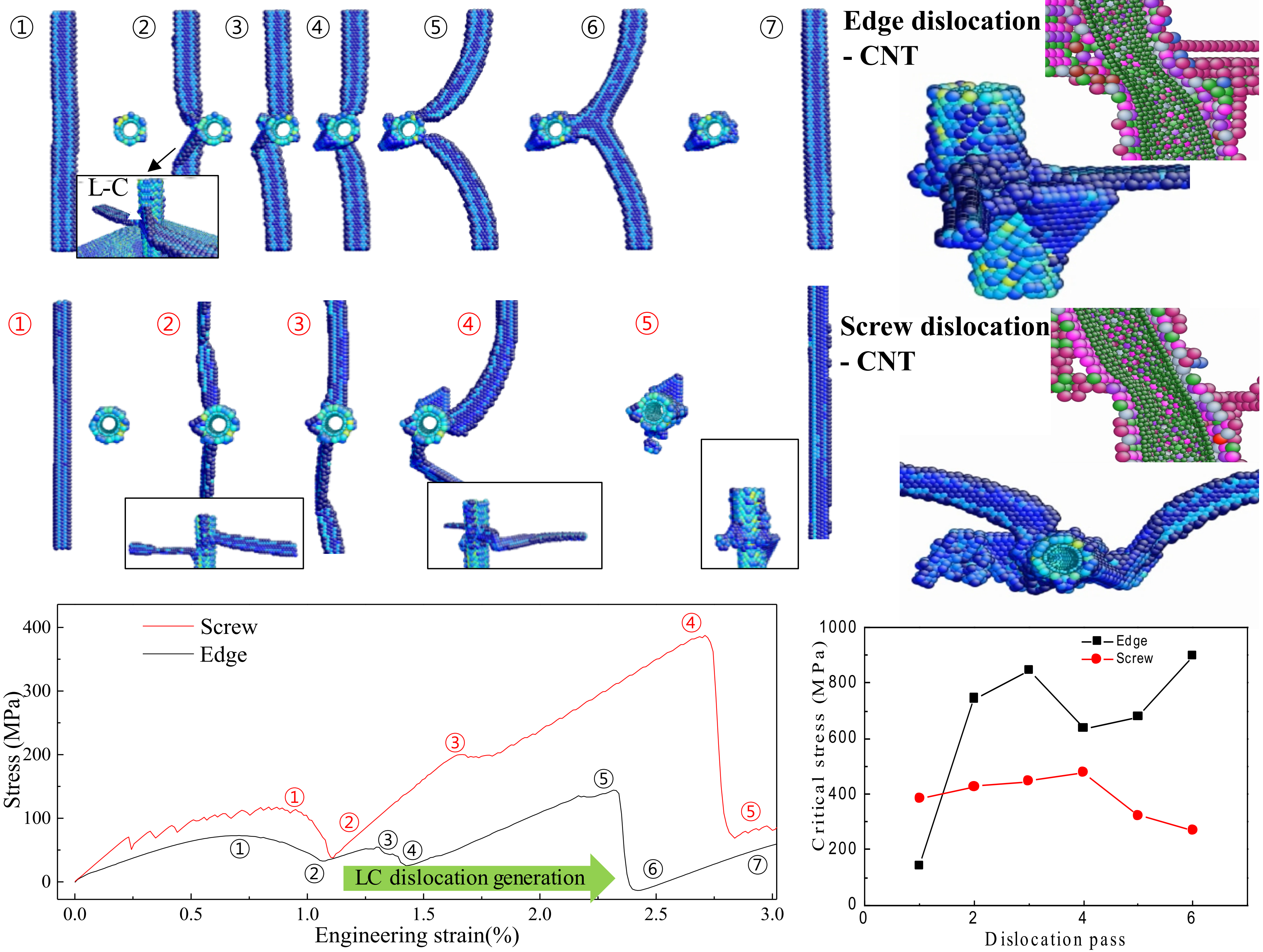

*Extreme Mechanics Letters* **8** (2016) 245



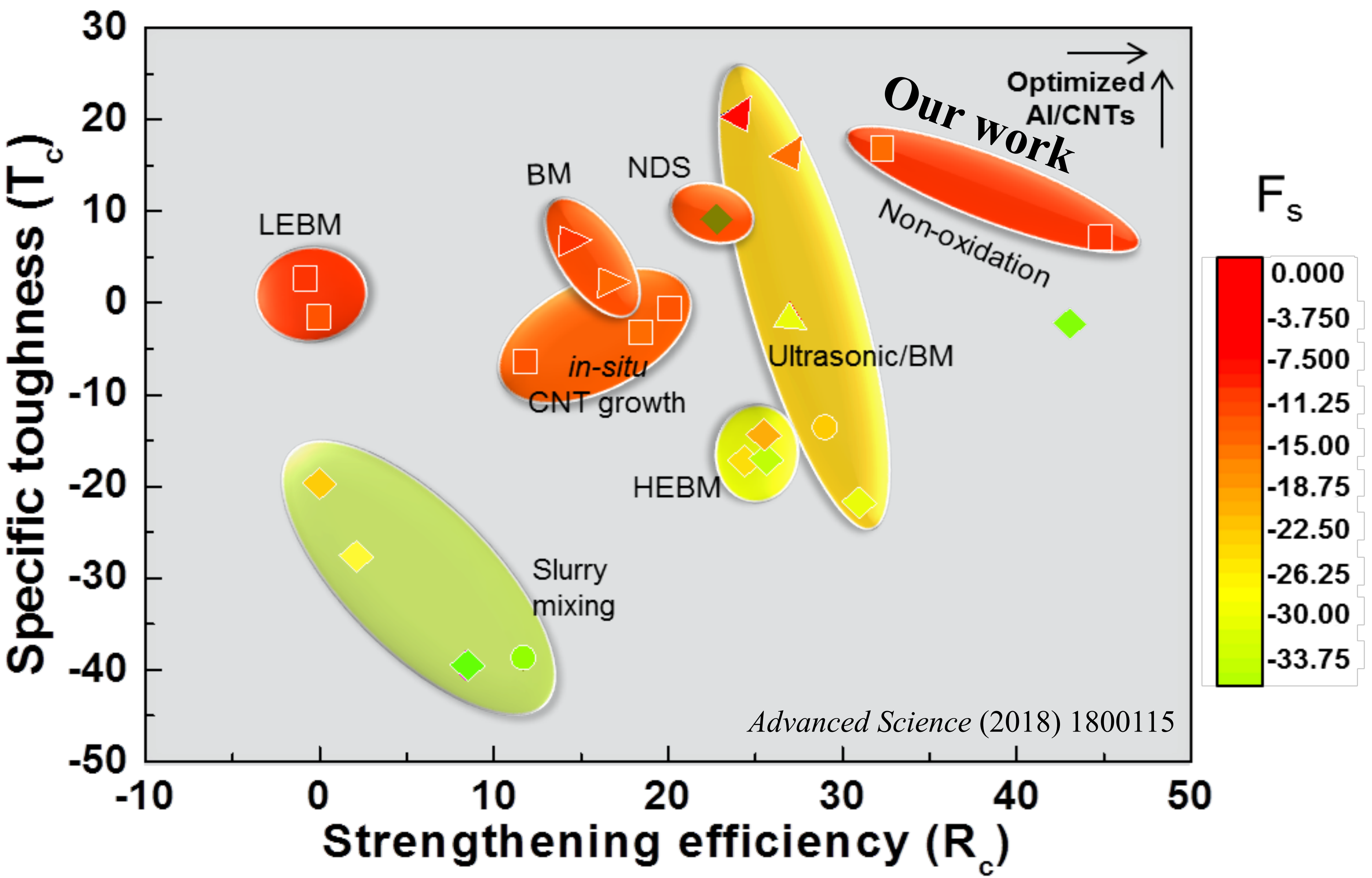
Screw dislocation in Al interacting with CNT

$\rho_{CNT} \sim 10^{14}/m^2$ and act like forest dislocations

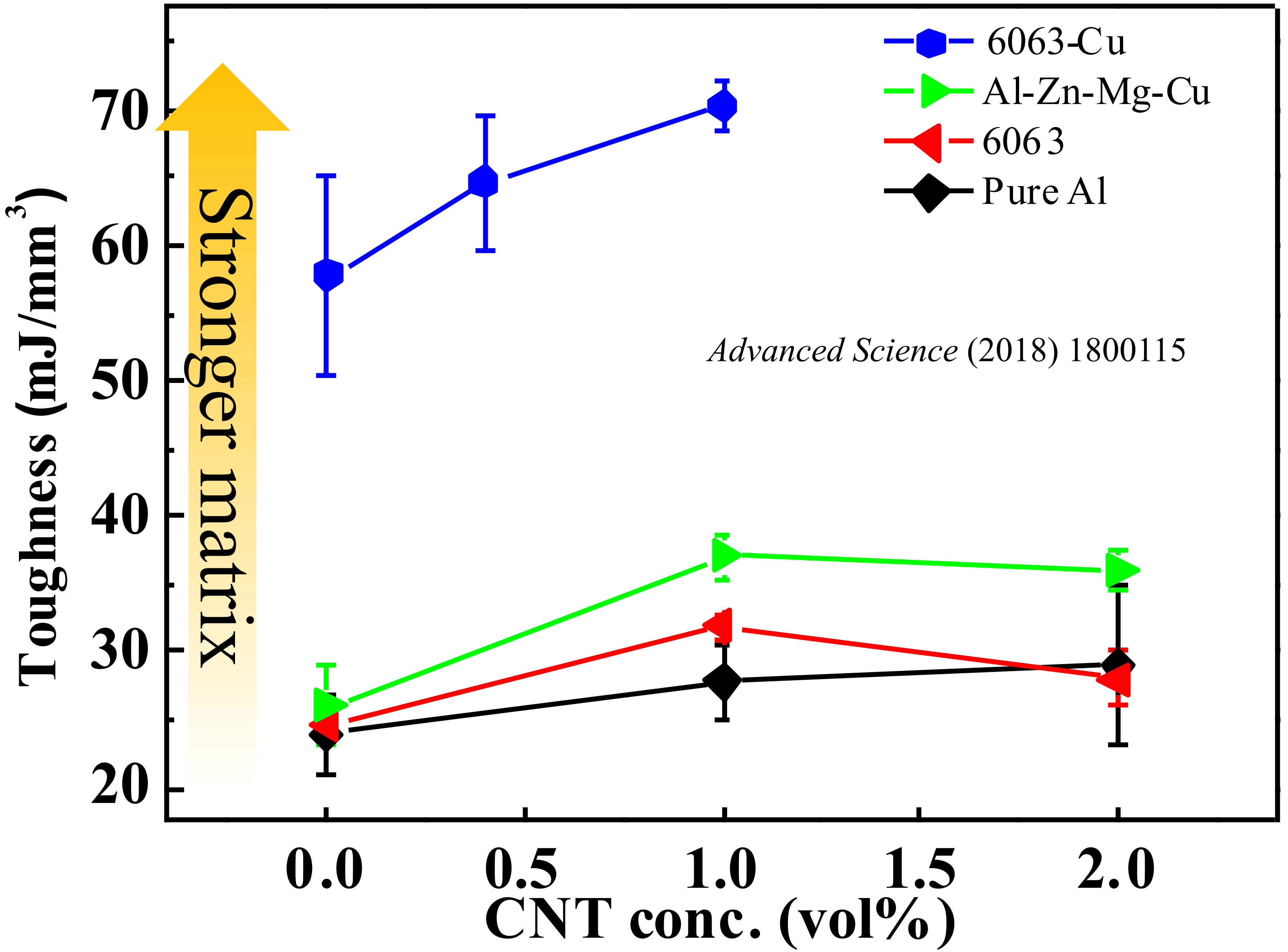
"Taylor-dispersion" hardening



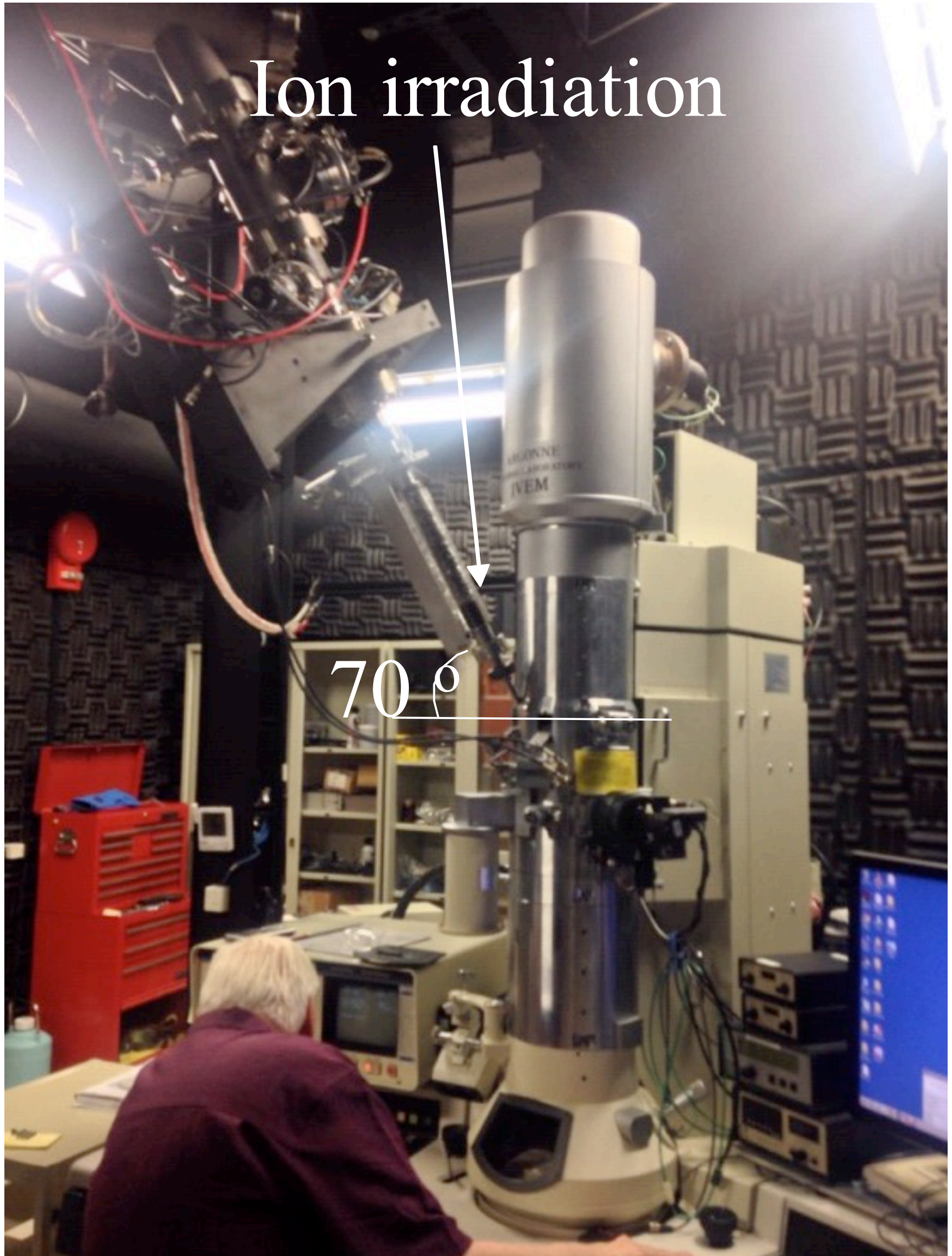
Edge dislocation - CNT
Screw dislocation - CNT
L-C
① ② ③ ④ ⑤ ⑥ ⑦
Screw
Edge
Stress (MPa)
Engineering strain(%)
LC dislocation generation
0 100 200 300 400
0.0 0.5 1.0 1.5 2.0 2.5 3.0
Edge
Screw
Critical stress (MPa)
Dislocation pass
0 200 400 600 800 1000
0 2 4 6



Specific toughness ($T_c$)
Strengthening efficiency ($R_c$)
Optimized Al/CNTs
Our work
Non-oxidation
NDS
BM
LEBM
in-situ CNT growth
Ultrasonic/BM
HEBM
Slurry mixing
$F_s$
0.000
-3.750
-7.500
-11.25
-15.00
-18.75
-22.50
-26.25
-30.00
-33.75
Advanced Science (2018) 1800115



6063-Cu
Al-Zn-Mg-Cu
6063
Pure Al
Stronger matrix
Advanced Science (2018) 1800115
Toughness (mJ/mm$^3$)
70
60
50
40
30
20
0.0
0.5
1.0
1.5
2.0
CNT conc. (vol%)




# Argonne IVEM:

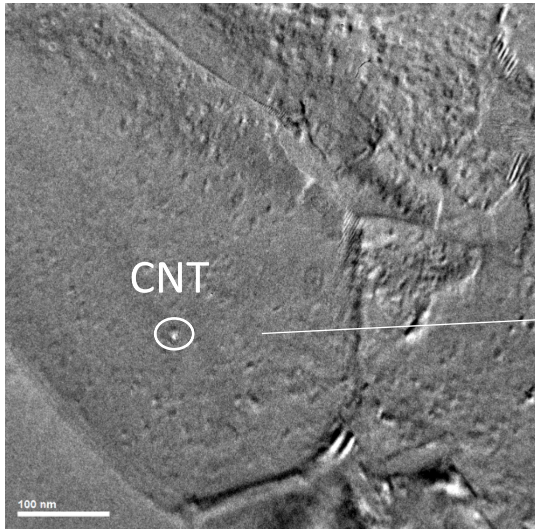
Kangpyo So, Mingda Li, Yang Yang, Meimei Li, June 2016

Hitachi-9000 TEM
150 keV e-beam;
70keV-1MeV Kr ion

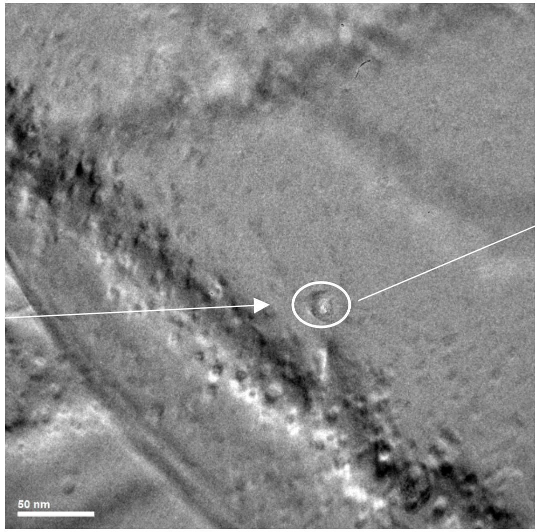
25°C to 400°C

We studied
Al+CNT
Ni based alloy

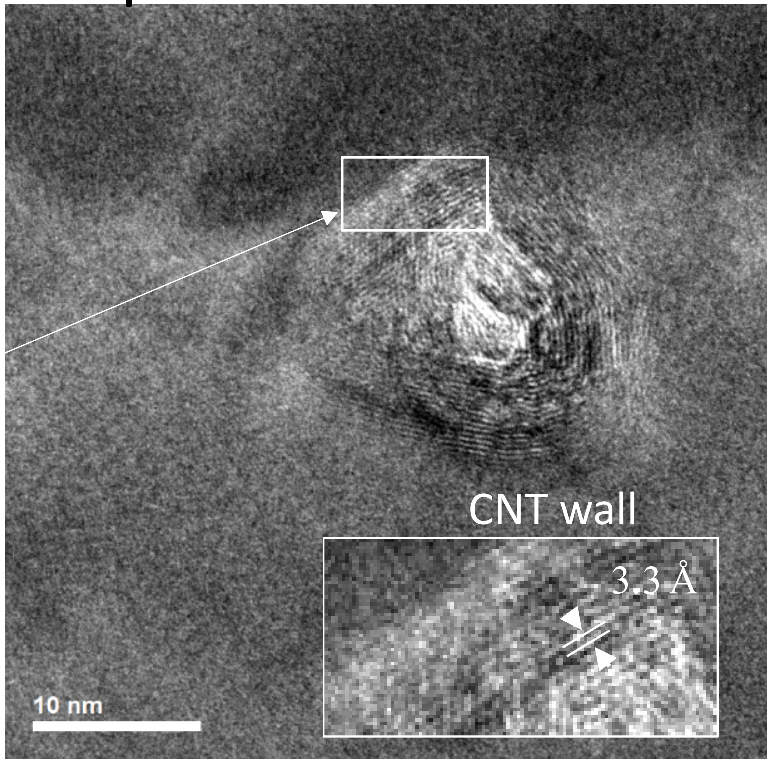
# In-situ irradiation of Al+CNT composite in IVEM

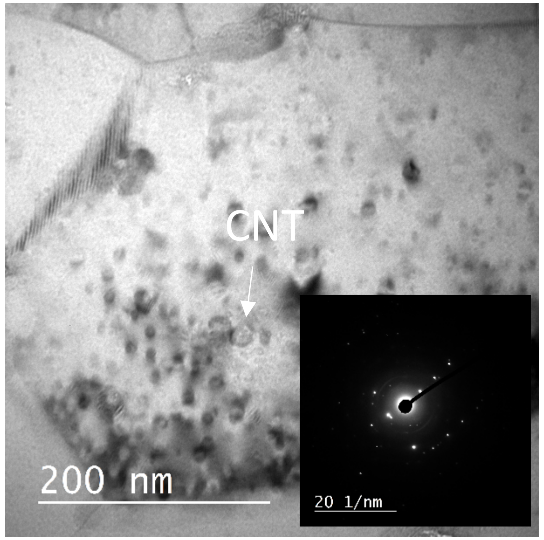



2 vol% CNT in Al




CNT is clearly visible inside Al grain

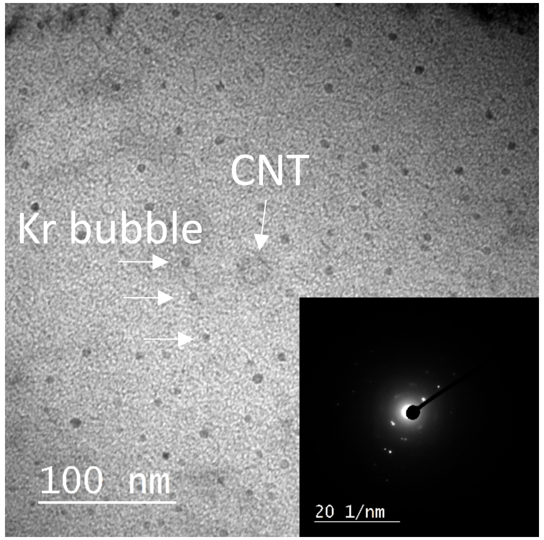



Before irradiation

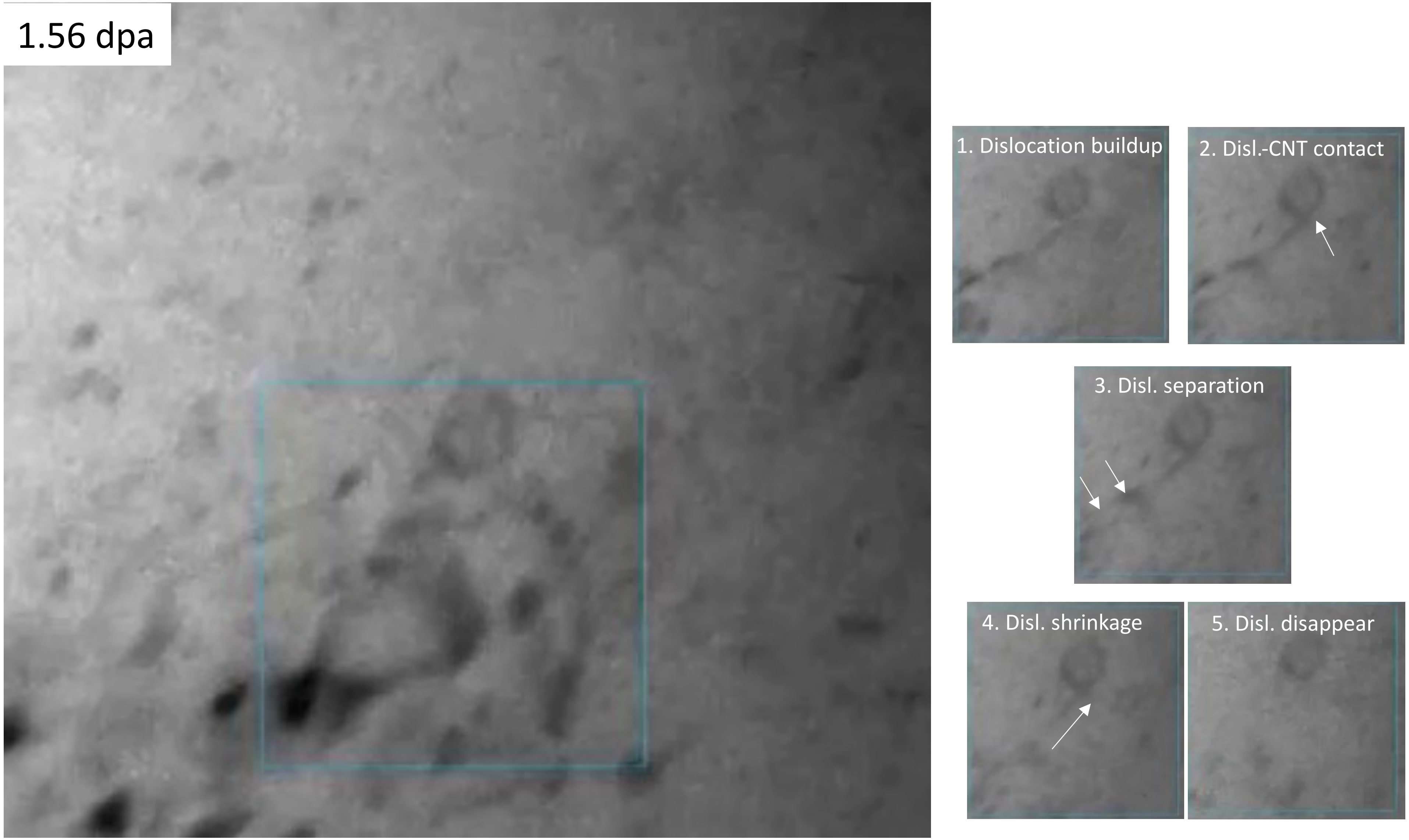
22 dpa

100 KeV
Kr ion irradiation

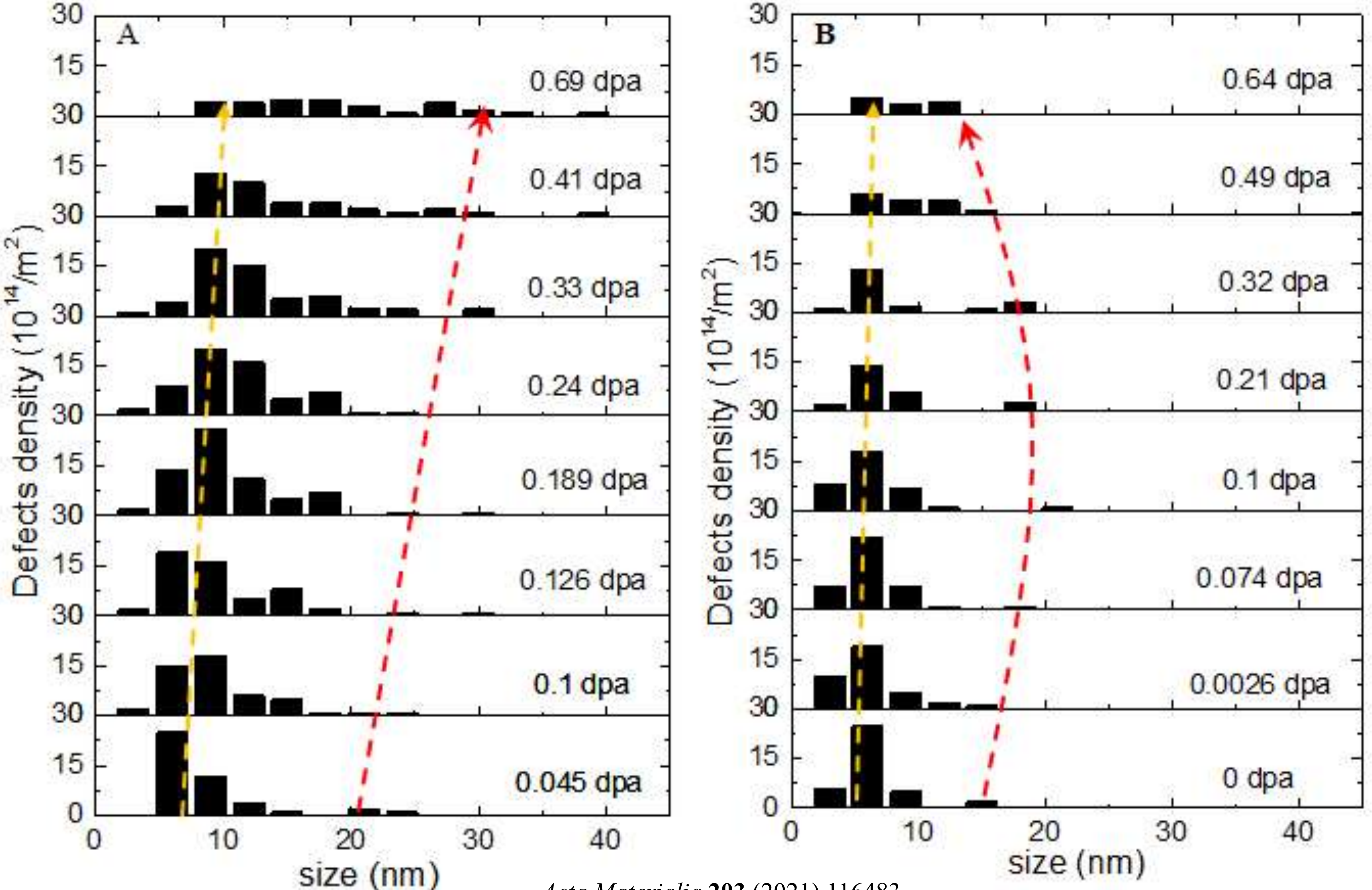



After irradiation

# In-situ observation of CNT-dislocation interaction during irradiation



**Away from CNT** **Near CNT**




*Acta Materialia* **203** (2021) 116483

# Technical Progress/Accomplishments

- Summaries of technical progress/accomplishments for FY-19

  - Setting up the Composites Lab at MIT for sample processing and testing
  - Synthesis of specific samples
  - Establishment of protocols for safety
  - Characterization of the preliminary physical properties for test reactor: Concentration, Thermal conductivity, Specific heat, Melting point, Density, Thermal expansion.

# List of the nanocomposites for neutron irradiation test

| # | Materials | DPA | Temperature (°C) | Number of dog-bone samples | Number of TEM discs |
|---|---|---|---|---|---|
| **1** | Aluminum | 0, 0.7, 1.4, 2.1 | 300 | 16 | 4 |
| **2** | Aluminum + 1D CNT/nanowires | 0, 0.7, 1.4, 2.1 | 300 | 16 | 4 |
| **3** | Zirconium | 0, 0.7, 1.4, 2.1 | 300 | 16 | 4 |
| **4** | Zirconium + 1D CNT/nanowires | 0, 0.7, 1.4, 2.1 | 300 | 16 | 4 |
| **5** | Copper | 0, 0.7, 1.4, 2.1 | 300 | 16 | 4 |
| **6** | Copper +2D Graphene | 0, 0.7, 1.4, 2.1 | 300 | 16 | 4 |
| **7** | Al4SiC4 | 0, 0.7, 1.4, 2.1 | 300 | 16 | 4 |
| **8** | Steel 1 | 0, 0.7, 1.4, 2.1 | 300 | 16 | 4 |
| **9** | Steel 2 | 0, 0.7, 1.4, 2.1 | 300 | 16 | 4 |
| **10** | Steel 1 + oxides/carbides | 0, 0.7, 1.4, 2.1 | 300 | 16 | 4 |
| **11** | Steel 2 + oxides/carbides | 0, 0.7, 1.4, 2.1 | 300 | 16 | 4 |
| **12** | Nickel | 0, 0.7, 1.4, 2.1 | 300 | 16 | 4 |
| **13** | Nickel + 1D CNT/nanowires | 0, 0.7, 1.4, 2.1 | 300 | 16 | 4 |
| **Total number of test samples** | | | | | |

# Physical Properties of materials for irradiation vehicles

| Materials | Concentration | Thermal conductivity (W/m·K) | Specific heat(J/g·K) | Melting point (°C) | Density (g/cm$^3$) | Thermal expansion (um/m·K) |
|---|---|---|---|---|---|---|
| Aluminum | 1 vol% CNT(0.5 wt%) | 193 (LFA) | ~0.9 | 660 | 2.7 | <23.1 |
| Zirconium | ~2vol %( 4.4wt%) CNT | 9.892 (LFA) | ~0.27 | 1855 | 6.5 | <5.7 |
| Copper +2D Graphene | ~1vol% graphene | <401 | ~0.385 | 1084 | 8.96 | <16.5 |
| Al4SiC4 | | 80 | | >2700 | 3.03 | 6.2 |
| Steel 1 + oxides/carbides | ~2wt% $Y_2Ti_2O_7$ | ~24.9 | ~0.46 | 1510 | 7.78 | ~9.9 |
| Steel 2 + oxides/carbides | ~2wt% $Y_2Ti_2O_7$ | ~15 | ~0.5 | 1673 | 7.9 | ~17 |
| Nickel + 1D CNT/nanowires | ~1 vol% CNT | <90.9 | ~0.44 | 1455 | 8.9 | <13.4 |

# Few changes

- No.3 (Zr) and No.4 (Zr/CNT) → No.3 (Fe-16Cr-2Si) and No.4 (Fe-20Cr-2Si)

The oxygen concentration of No.3 (Zr) and No.4 (Zr/CNT) was too high (0.9 at%) which resulted in the brittle phase of Zr matrix. Thus, we replaced Zr and Zr/CNT in the test matrix with Fe-16Cr-2Si and Fe-20Cr-2Si

- No.7 (Al4SIC4) → No.7 (Single Crystal Ni)

The thermal conductivity of the No.7 (Al4SiC4) at high temperature was not fit to the neutron test vehicle. We have changed to Single-crystal Ni.

# Technical Progress/Accomplishments

- List technical progress/accomplishments for FY-20

1. Design and Assembly of Rabbit Capsules for Irradiation of Prototype Metal and Nanocomposite Specimens in the High Flux Isotope Reactor (ORNL)

2. Stability of the dispersoids under irradiation and designing new interface to enhance long term radiation performance

- Investigation of selection criteria of dispersoids: wetting, high temperature stability, reactivity

# 1. Design and Assembly of Rabbit Capsules for Irradiation of Prototype Metal and Nanocomposite Specimens in the High Flux Isotope Reactor (ORNL)

- A HFIR cycle provides between 1.4 - 1.7 dpa per cycle depending on the material and position
- 2 rabbits per dose at 300°C (+/- 20°C)
  - ~.7 dpa - Hydraulic Tube for 13 days (.5 cycle)
  - ~1.4 dpa - Standard position for 1 cycle
  - ~2.1 or 2.8 dpa - either 1 full cycle plus 13 days in Hydraulic Tube position / or 2 full cycle

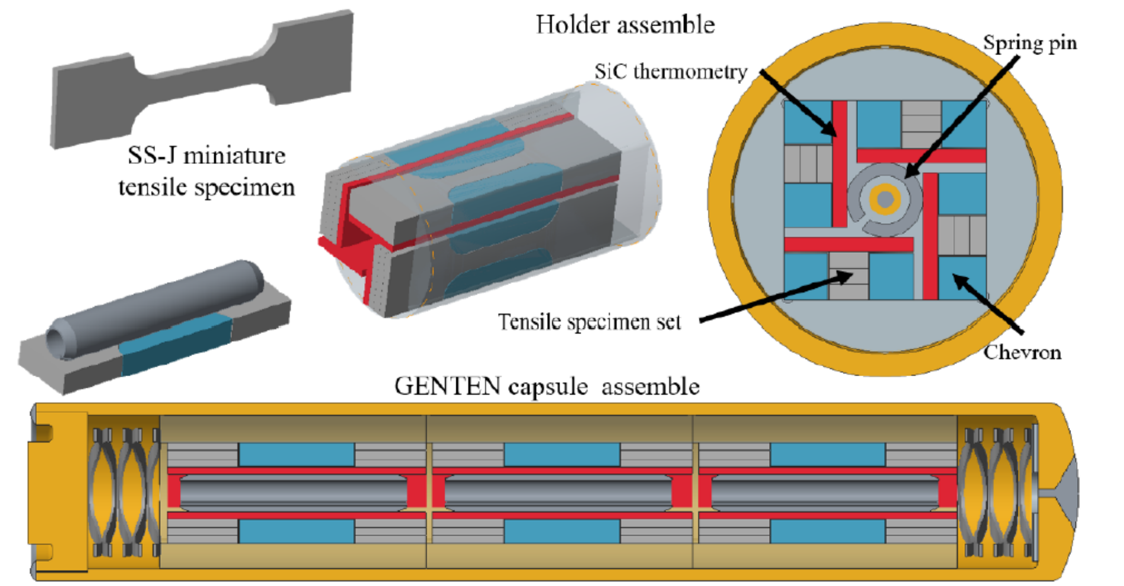


Figure 1. Section view showing irradiation capsule design concept.

Proposed Test Matrix

| DPA | Rabbit ID | Sub Assembly | SS-J2 Tensile 16x4x.5 | MPC1 Coupon (16x4x.25) |
|---|---|---|---|---|
| .7 dpa | JULI01 | Top | 6 | 6 |
| | | Mid | 6 | 6 |
| | | Bottom | 6 | 6 |
| .7 dpa | JULI02 | Top | 6 | 6 |
| | | Mid | 9 | 6 |
| | | Bottom | 6 | 9 |
| Total 0.7 dpa | | | 39 | 39 |
| 1.4 dpa | JULI03 | Top | 6 | 6 |
| | | Mid | 6 | 6 |
| | | Bottom | 6 | 6 |
| 1.4 dpa | JULI04 | Top | 6 | 6 |
| | | Mid | 9 | 6 |
| | | Bottom | 6 | 9 |
| Total 1.4 dpa | | | 39 | 39 |
| 2.1 dpa | JULI05 | Top | 6 | 6 |
| | | Mid | 6 | 6 |
| | | Bottom | 6 | 6 |
| 2.1 dpa | JULI06 | Top | 6 | 6 |
| | | Mid | 9 | 6 |
| | | Bottom | 6 | 9 |
| Total 2.1 dpa | | | 39 | 39 |
| | | | | |
| Total | | | 117 | 117 |

OAK RIDGE National Laboratory

# ANSYS analysis

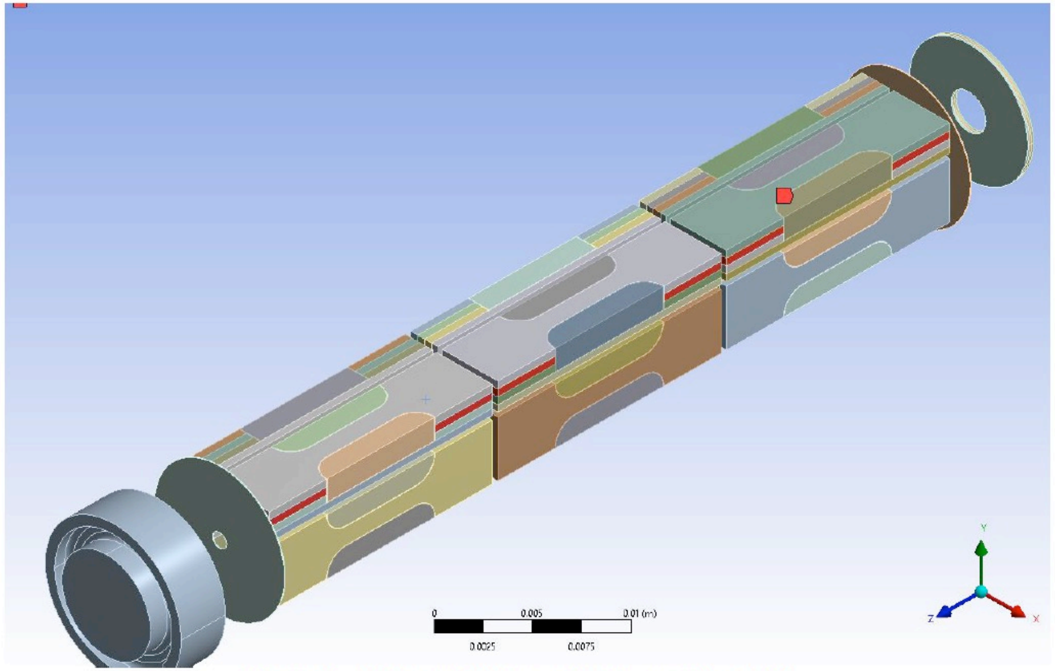


Figure 6. All top middle specimens grouping (T4)

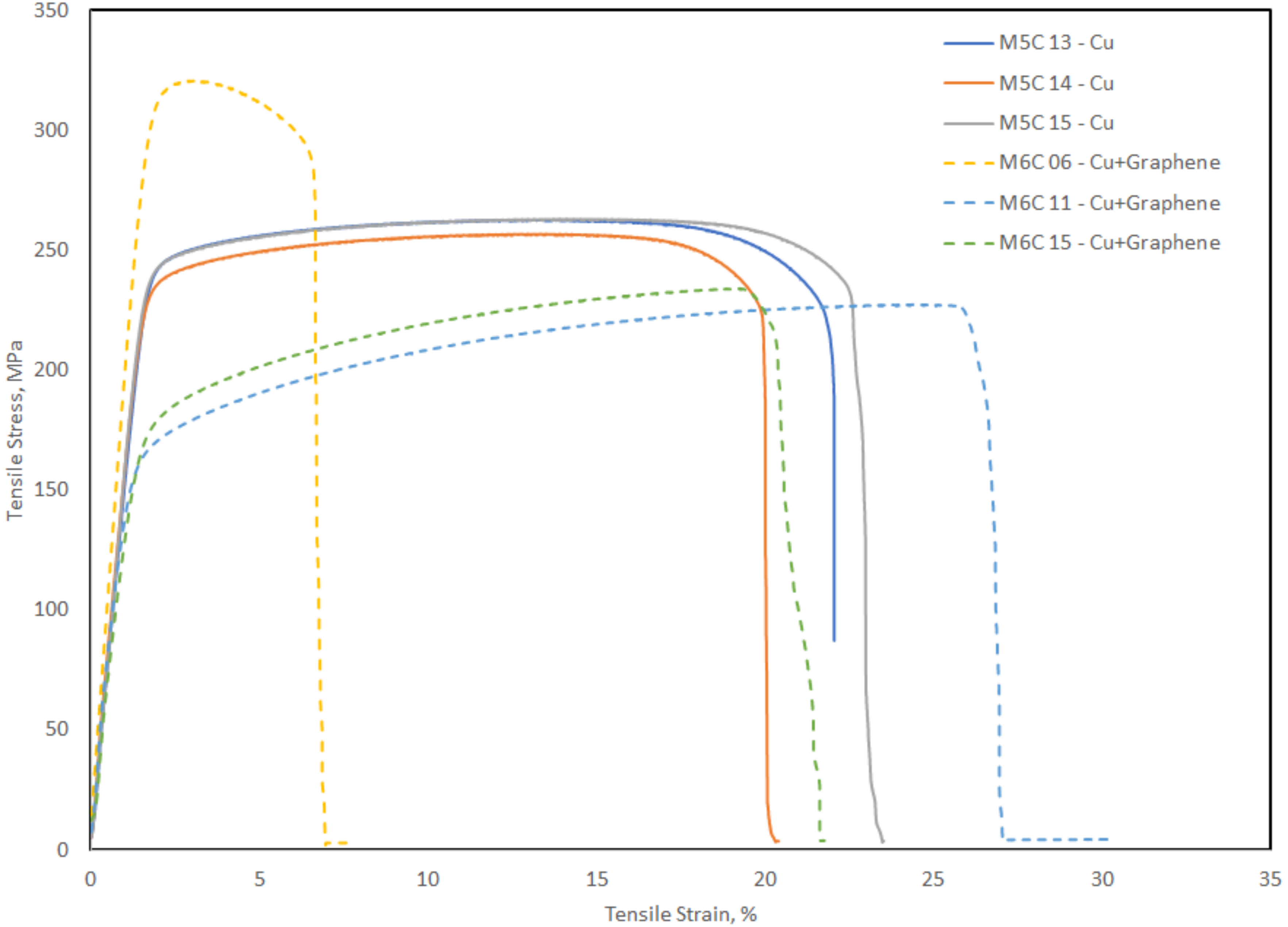
**Table 2-5. ANSYS parametric study results—TRRH position 6**

| Run | Position | OD1 mm | OD2 mm | OD3 mm | T4 °C | T3 °C | T2 °C | T1 °C |
|---|---|---|---|---|---|---|---|---|
| 61 | 6 | 9.30 | 9.30 | 9.30 | 250 | 248 | 258 | 242 |
| 62 | 6 | 9.15 | 9.30 | 9.30 | 276 | 249 | 268 | 306 |
| 63 | 6 | 9.45 | 9.30 | 9.30 | 215 | 246 | 241 | 153 |
| 64 | 6 | 9.30 | 9.15 | 9.30 | 275 | 258 | 311 | 252 |
| 65 | 6 | 9.30 | 9.45 | 9.30 | 210 | 230 | 173 | 223 |
| 66 | 6 | 9.30 | 9.30 | 9.15 | 276 | 313 | 268 | 243 |
| 67 | 6 | 9.30 | 9.30 | 9.45 | 215 | 159 | 242 | 240 |
| 68 | 6 | 9.18 | 9.18 | 9.18 | 320 | 317 | 328 | 310 |
| 69 | 6 | 9.42 | 9.18 | 9.18 | 264 | 313 | 299 | 177 |
| 70 | 6 | 9.18 | 9.42 | 9.18 | 255 | 284 | 201 | 275 |
| 71 | 6 | 9.42 | 9.42 | 9.18 | 214 | 283 | 190 | 165 |
| 72 | 6 | 9.18 | 9.18 | 9.42 | 264 | 184 | 298 | 306 |
| 73 | 6 | 9.42 | 9.18 | 9.42 | 210 | 182 | 269 | 175 |
| 74 | 6 | 9.18 | 9.42 | 9.42 | 214 | 172 | 190 | 274 |
| 75 | 6 | 9.42 | 9.42 | 9.42 | 173 | 171 | 179 | 165 |

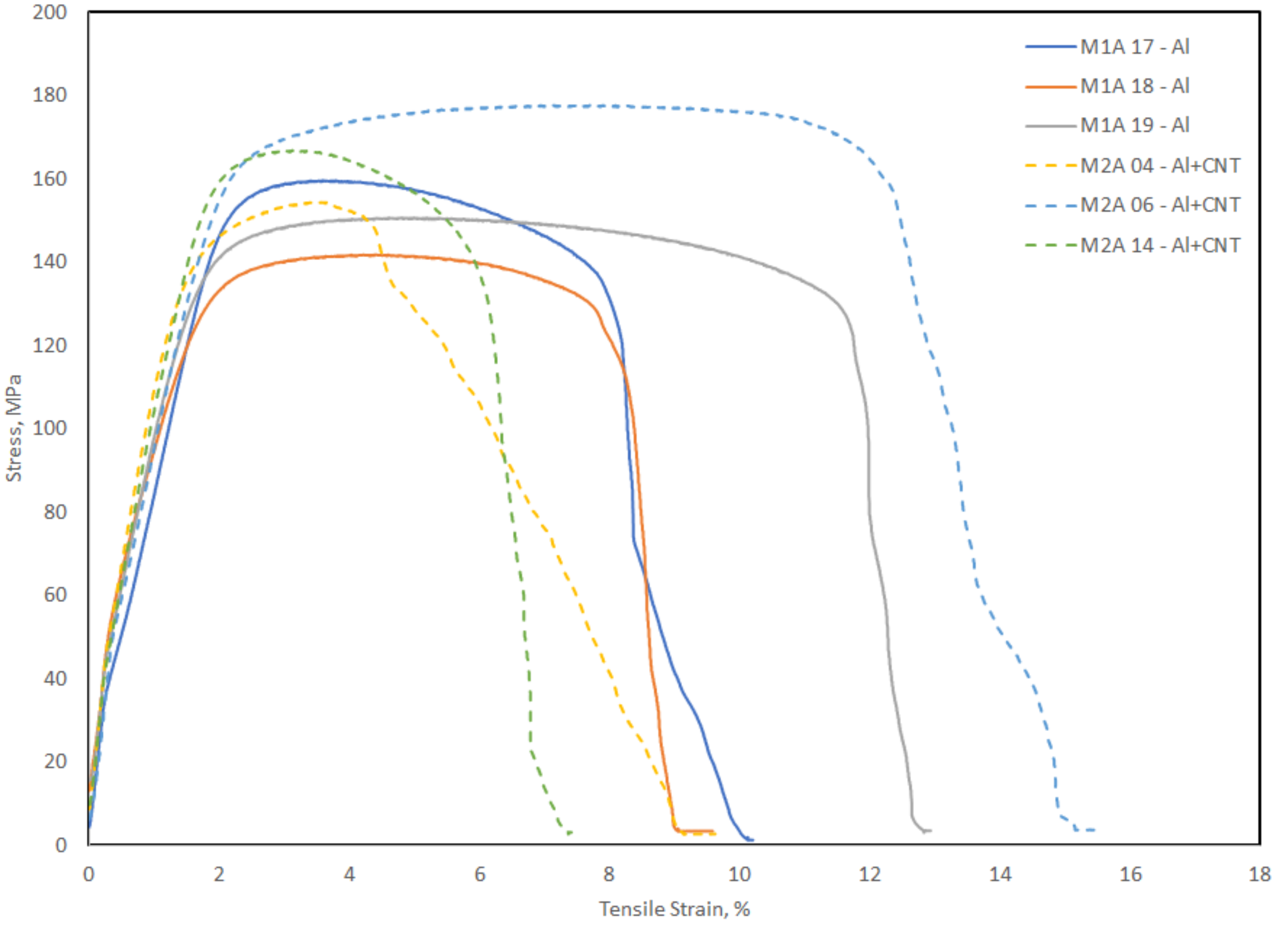
# Part layout for capsules


Neutron irradiation completed!!

# Stress and Stress curve (unirradiated)



# Stress and Stress curve (unirradiated)



Fusion Engineering and Design 157 (2020) 111663

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# Non-contact strain evaluation for miniature tensile specimens of neutron-irradiated F82H by digital image correlation

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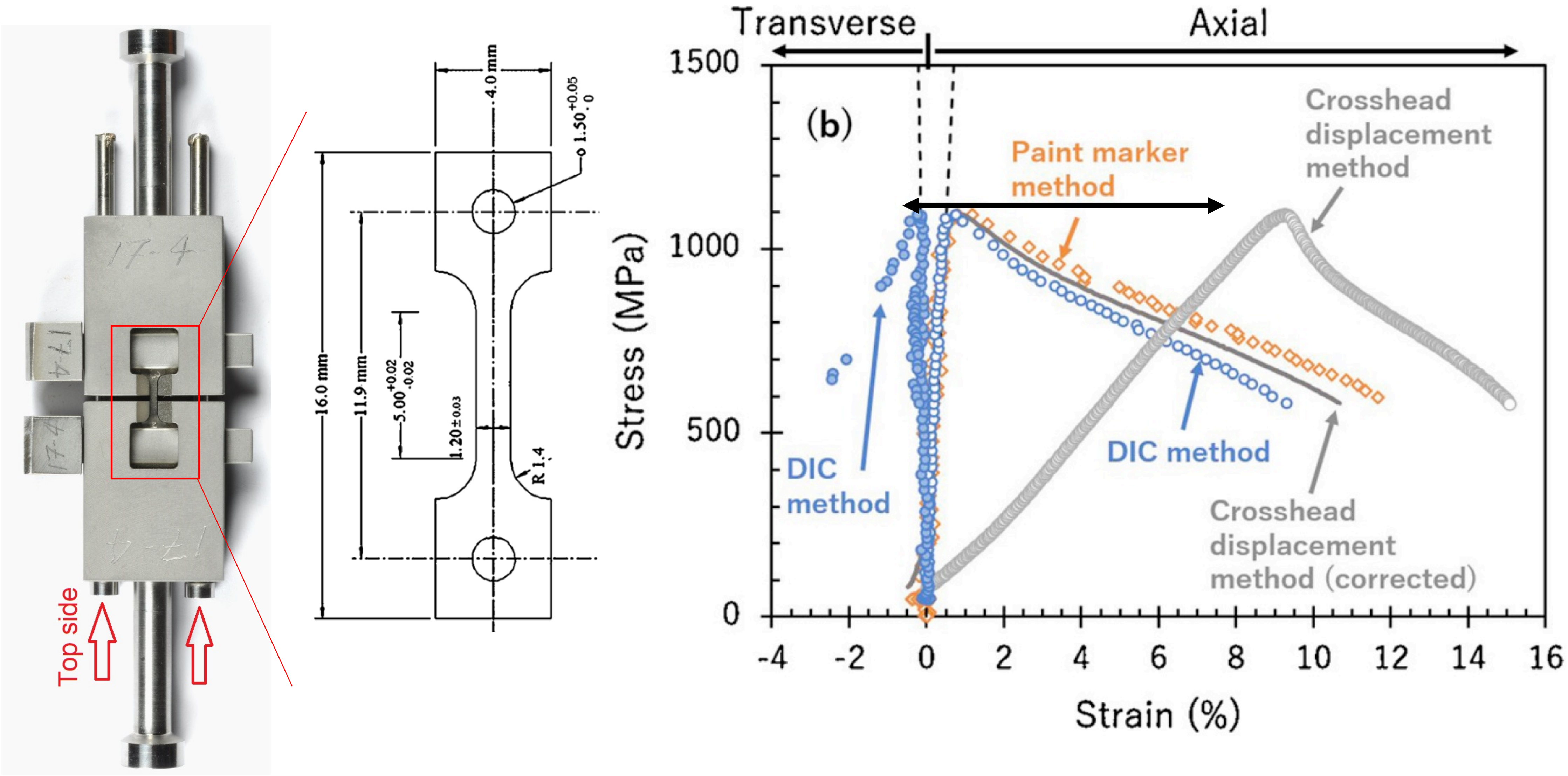
# Load-frame machine compliance need to be corrected



# 2. Selection Criteria of dispersoids

i) high-temperature stability

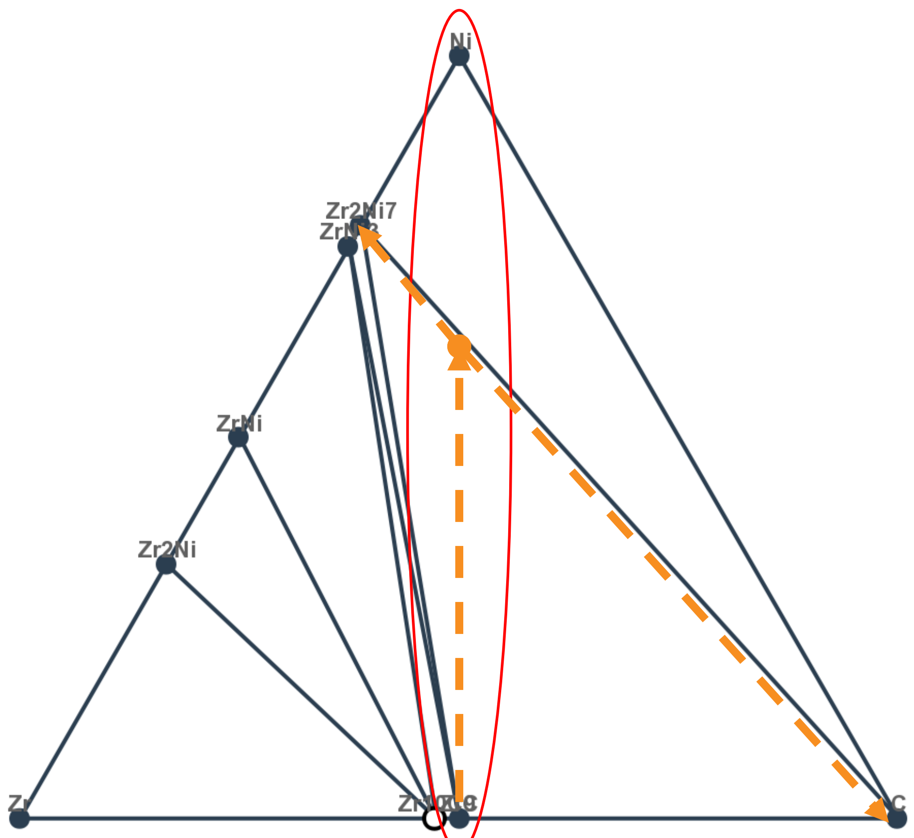
ii) Good wettability

iii) Reactivity

=> investigated different types of oxides, carbides, nitrides

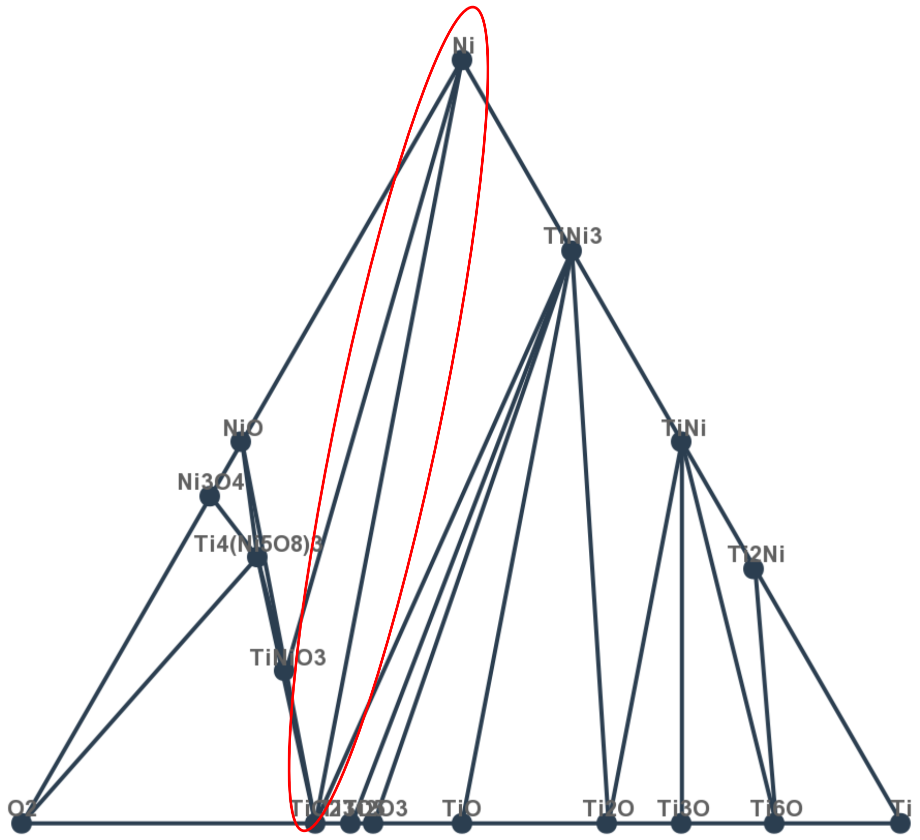
# Reactivity: reaction path

ZrC in Ni




ZiC can create side reaction to $Zr_2Ni_{17}$ + C with Ni

TiO2 in Ni




$TiO_2$ doesn't have a side reaction with Ni

# Formation energy and possible other reaction in Ni.

Data is obtained from the materials projects (https://materialsproject.org/ ).

| NT/NW | Formation/decomposition (eV) | Possible reaction in Ni | Melting temperature($^oC$) |
|---|---|---|---|
| Al2O3 | -3.442 | None | 2,072 |
| BN | -1.472 | None | 2,973 |
| Cr3C2 | -0.11 | None | 1,895 |
| ZrN | -1.87 | None | 2,952 |
| TiO2 | -3.523 | None | 1,843 |
| AlN | -1.595 | None | 2,500 |
| Y2O3 | -3.99 | None | 2,425 |
| SiC | -0.205 | SiNi13, Si12Ni31, SiNi2, SiNi | 2,830 |
| ZrC | -0.808 | Zr2Ni7 | 3,420 |
| TiC | -0.808 | TiNi3 | 3,067 |
| Ti2CN | -1.392 | Complex | |
| WC | -0.122 | Ni4W | 2,870 |
| HfC | -0.943 | HfNi3 | 3,928 |
| Mo2C | -0.103 | Ni3Mo, Ni2Mo | 2,520 |
| V2C | -0.475 | VNi2, VNi3, V6C5 | >2,810 |
| Nb2C | -0.46 | NbNi3 | 3,608 |

# Modeling and Machine Learning prediction

- Preparation of Data: Experimental data from literature (~2300 data points)

| 1 | Me1 | Me2 | | Me3 | | Sub | | | | | | | | | | Comment | Wetting | T | Time | Atmosphere | Pressure | Reaction produ | Comment | Reference |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 522 | Ag | | | | | Ti | 1 | | | | | C | 1.00 | | | | 120 | 1050 | | Vacuum | 1E-3Pa | | | N. Frage, Acta M |
| 523 | Au | | | | | Ti | 1 | | | | | C | 1.00 | | | | 125 | 1150 | | Vacuum | 1E-3Pa | | | N. Frage, Acta M |
| 524 | Sn | | | | | Ti | 1 | | | | | C | 1.00 | | | | 84 | 1150 | | Vacuum | 1E-3Pa | | | N. Frage, Acta M |
| 525 | Au | Cu | 24.3 | | | Ti | 1 | | | | | C | 1.00 | | | | 120 | 1150 | | Vacuum | 1E-3Pa | | | N. Frage, Acta M |
| 526 | Au | Cu | 42.4 | | | Ti | 1 | | | | | C | 1.00 | | | | 119 | 1150 | | Vacuum | 1E-3Pa | | | N. Frage, Acta M |
| 527 | Au | Cu | 61 | | | Ti | 1 | | | | | C | 1.00 | | | | 114 | 1150 | | Vacuum | 1E-3Pa | | | N. Frage, Acta M |
| 528 | Au | Cu | 77.3 | | | Ti | 1 | | | | | C | 1.00 | | | | 104 | 1150 | | Vacuum | 1E-3Pa | | | N. Frage, Acta M |
| 529 | Au | Cu | 92.5 | | | Ti | 1 | | | | | C | 1.00 | | | | 92 | 1150 | | Vacuum | 1E-3Pa | | | N. Frage, Acta M |
| 530 | Cu | | | | | Ti | 1 | | | | | C | 1.00 | | | | 88 | 1150 | | Vacuum | 1E-3Pa | | | N. Frage, Acta M |
| 531 | Au | Ni | 7.5 | | | Ti | 1 | | | | | C | 1.00 | | | | 78 | 1150 | | Vacuum | 1E-3Pa | | | N. Frage, Acta M |
| 532 | Au | Ni | 15.4 | | | Ti | 1 | | | | | C | 1.00 | | | | 68 | 1150 | | Vacuum | 1E-3Pa | | | N. Frage, Acta M |
| 533 | Au | Fe | 4.1 | | | Ti | 1 | | | | | C | 1.00 | | | | 84 | 1150 | | Vacuum | 1E-3Pa | | | N. Frage, Acta M |
| 534 | Au | Fe | 7.5 | | | Ti | 1 | | | | | C | 1.00 | | | | 63 | 1150 | | Vacuum | 1E-3Pa | | | N. Frage, Acta M |
| 535 | Ag | Ti | 0.98 | | | Ti | 1 | | | | | C | 1.00 | | | | 84 | 1050 | | Vacuum | 1E-3Pa | | | N. Frage, Acta M |
| 536 | Ag | Ti | 1.27 | | | Ti | 1 | | | | | C | 1.00 | | | | 71 | 1050 | | Vacuum | 1E-3Pa | | | N. Frage, Acta M |
| 537 | Ag | Ti | 2.69 | | | Ti | 1 | | | | | C | 1.00 | | | | 54 | 1050 | | Vacuum | 1E-3Pa | | | N. Frage, Acta M |

# Featurization

- Generate features using Matminer

matminer

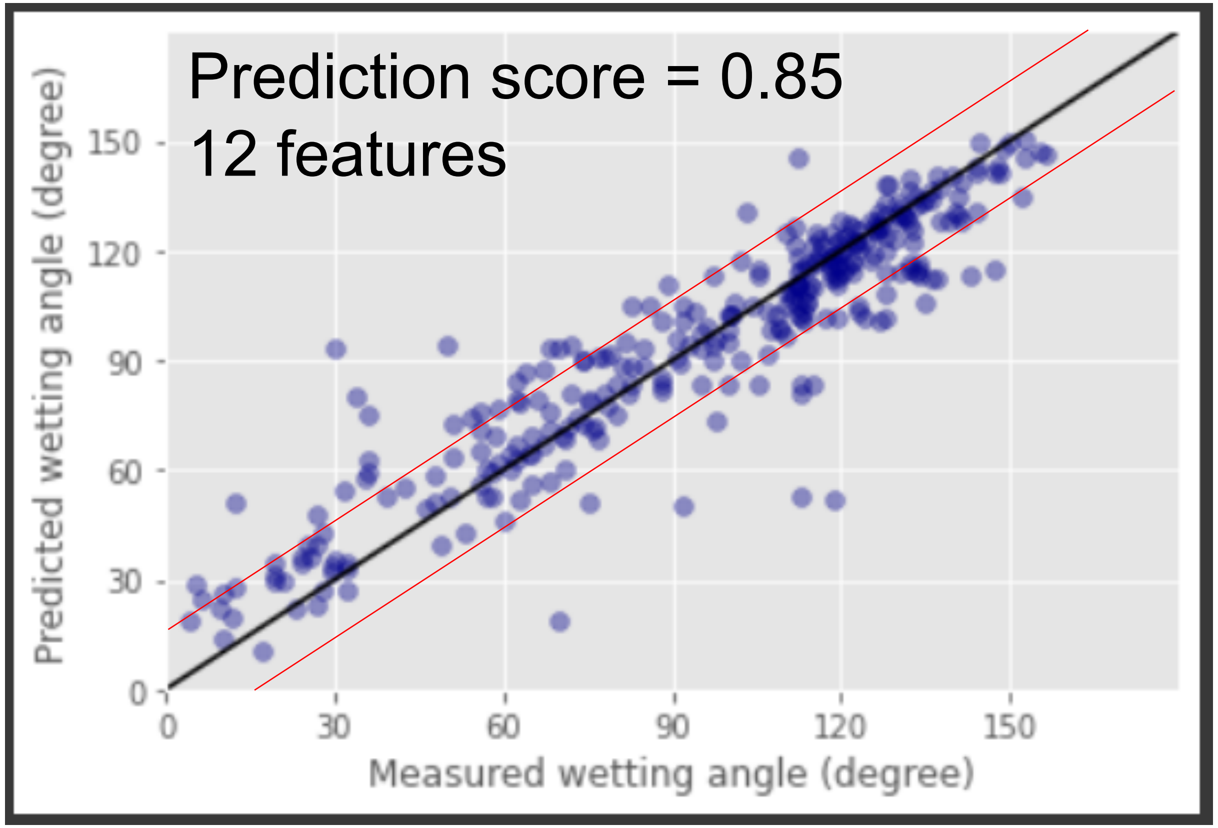
$Y_{0.9}Ba_{0.1}Cu_1O_4$ → Elemental properties → Compound properties (average, range, std, max, min)

| | | | | |
|---|---|---|---|---|
| Atomic Number | Mendeleev Number[9] | Atomic Weight | Melting Temperature | Column |
| Row | Covalent Radius | Electronegativity* | # s Valence Electrons | # p Valence Electrons |
| # d Valence Electrons | # f Valence Electrons | Total # Valance Electrons | # Unfilled s States† | # Unfilled p States† |
| # Unfilled d States† | # Unfilled f States† | Total # Unfilled States† | Specific Volume of 0 K Ground State‡ | Band Gap Energy of 0 K Ground State‡ |
| Magnetic Moment (per atom) of 0 K ground state‡ | Space Group Number of 0 K Ground State‡ | | | |

*L. Ward et al., npj Computational Materials 2 (2016) 16028.*

# Training and Testing of the Wetting Angle Model

- Divided the collected experimental data into subsets
- Took a subset with ~1000 data points of metal-oxide/fluoride wettability
- Used 70% of this subset for the training of the model



# Search for Candidate Materials

| | Metal | Substrate |
|---|---|---|
| 0 | Al | Ac2O3 |
| 1 | Al | Ag1Al1F3 |
| 2 | Al | Ag1Al1F3 |
| 3 | Al | Ag4Al4O8 |
| 4 | Al | Ag1Al1O3 |
| ... | ... | ... |
| 28699 | Al | O8Zr4 |
| 28700 | Al | O4Zr2 |
| 28701 | Al | O4Zr2 |
| 28702 | Al | O8Zr4 |
| 28703 | Al | O8Zr4 |

28704 rows × 2 columns

List of pairs

| | Metal | Substrate | T |
|---|---|---|---|
| 0 | Al | Ac2O3 | 93 |
| 1 | Al | Ag1Al1F3 | 93 |
| 2 | Al | Ag1Al1F3 | 93 |
| 3 | Al | Ag4Al4O8 | 93 |
| 4 | Al | Ag1Al1O3 | 93 |
| ... | ... | ... | |
| 28699 | Al | O8Zr4 | 193 |
| 28700 | Al | O4Zr2 | 193 |
| 28701 | Al | O4Zr2 | 193 |
| 28702 | Al | O8Zr4 | 193 |
| 28703 | Al | O8Zr4 | 193 |

631488 rows × 49 columns

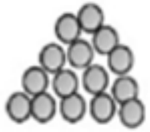
Featurization

| | Metal | Substrate | Temp | theta_pred |
|---|---|---|---|---|
| 0 | Al | Ac2O3 | 933.47 | 128.954733 |
| 1 | Al | Ag1Al1F3 | 933.47 | 133.420695 |
| 2 | Al | Ag1Al1F3 | 933.47 | 128.954733 |
| 3 | Al | Ag4Al4O8 | 933.47 | 128.954733 |
| 4 | Al | Ag1Al1O3 | 933.47 | 133.420695 |
| ... | ... | ... | ... | ... |
| 28699 | Al | O8Zr4 | 1933.47 | 135.089938 |
| 28700 | Al | O4Zr2 | 1933.47 | 135.089938 |
| 28701 | Al | O4Zr2 | 1933.47 | 117.249954 |
| 28702 | Al | O8Zr4 | 1933.47 | 131.061663 |
| 28703 | Al | O8Zr4 | 1933.47 | 131.061663 |

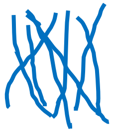
631488 rows × 6 columns

# Experimental validation

## Fabrication of Ni-Titanium oxide nanowire composite

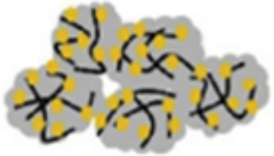


Raw pure Ni powders (50 μm)



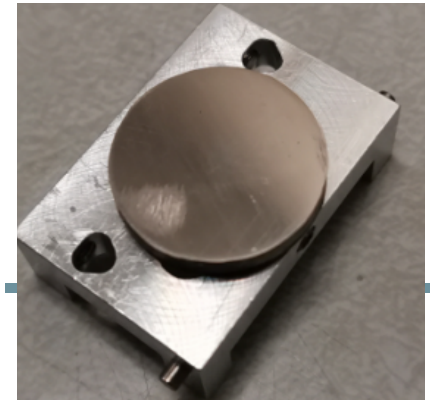
$TiO_2$ nanowires (industrial grade, Φ < 10 nm, length ~ 100 nm)

High energy ball milling (600 rpm, 30 mins in total)



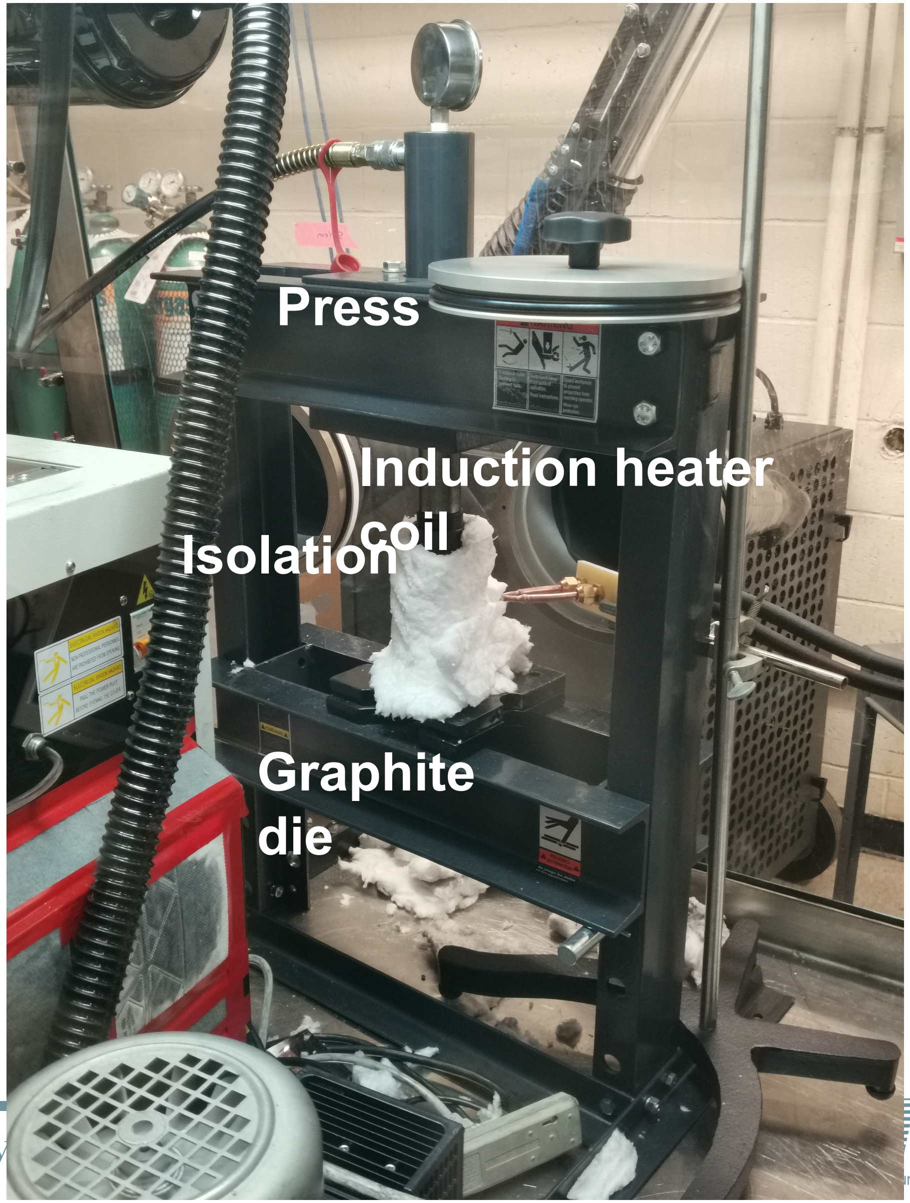
Powder mixtures

Sintering (Hot press: 85 MPa 45 mins, 1100 C)

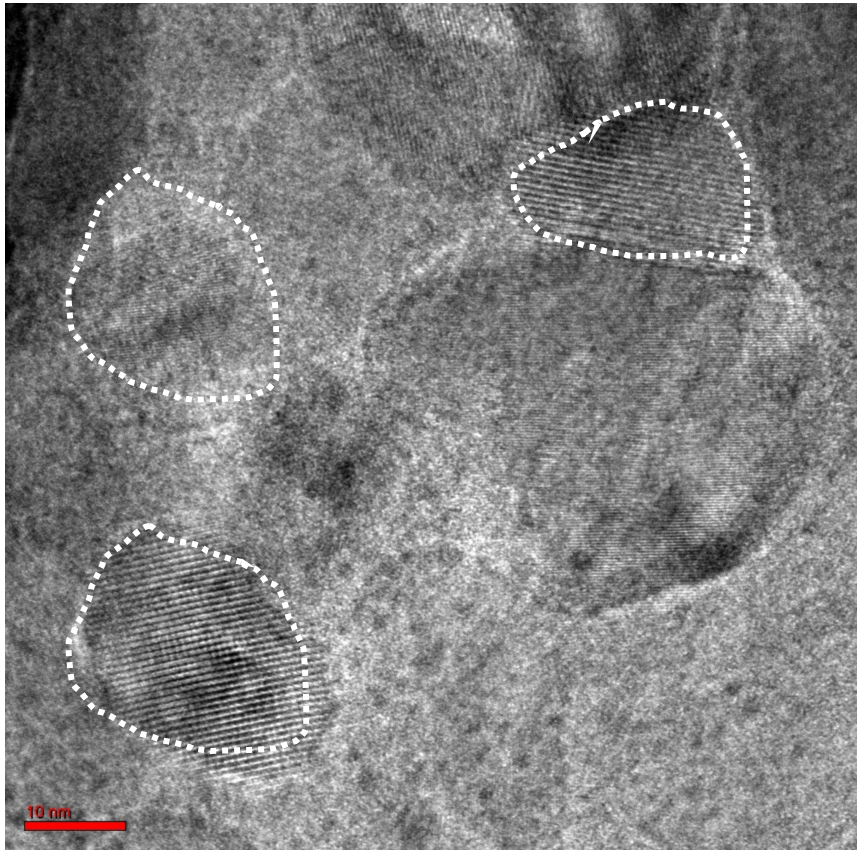


Φ 20 mm, ~10 gram

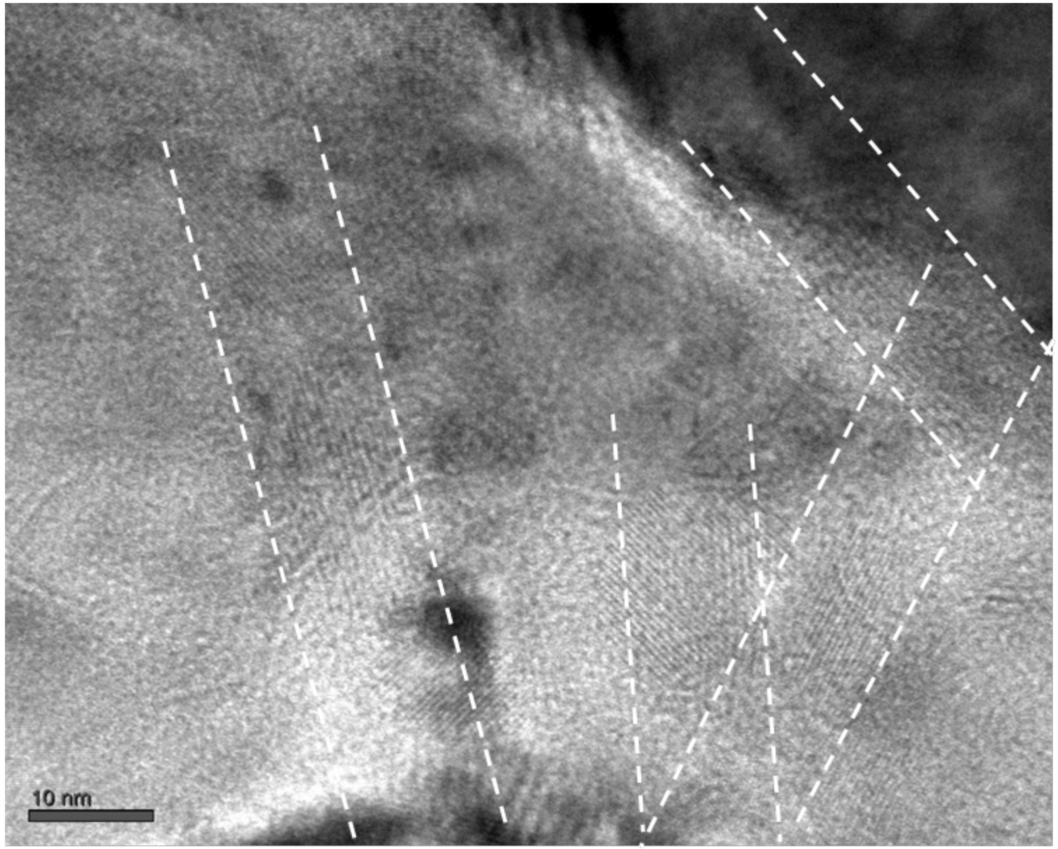
## Sintering facility



# $TiO_2$ nanowires dispersed in Ni matrix




Cross-sectional view of
TiO2 nanowire




longitudinal sectional view of
TiO2 nanowire

# Project Impacts

- 7 Journal Publications to date

[1] Nanotube/nanowire as effective defect sinks in metals: atomistic simulations and in situ ion radiation transmission electron microscopy, *Acta Materialia* **203** (2021) 116483
[2] Additive manufacturing for energy: A review, *Applied Energy* **282** (2021) 116041
[3] Superconducting Cu/Nb nanolaminate by coded accumulative roll bonding and its helium damage characteristics, *Acta Materialia* **197** (2020) 212
[4] Radiation-resistant metal-organic framework enables efficient separation of krypton fission gas from spent nuclear fuel, *Nature Communications* **11** (2020) 3103

- Conference Papers: nothing to report

- Conference Presentations:

Kang Pyo So, P. Cao, Y. Yang, J. G. Park, M. Li, Y. Long, J. Hu, M. Kirk, M. Li, E. M. Bringa, Y. H. Lee, M. P. Short, J. Li "Nanotube/nanowire as effective defect sinks in metals: atomistic simulations and in situ ion radiation transmission electron microscopy," TMS (2019), MiNES (2019), MITAB (2020) -> invited in journal *Applied Energy*

- Patents: nothing to report

- Involvement: nothing to report

# Milestones

## Schedule:

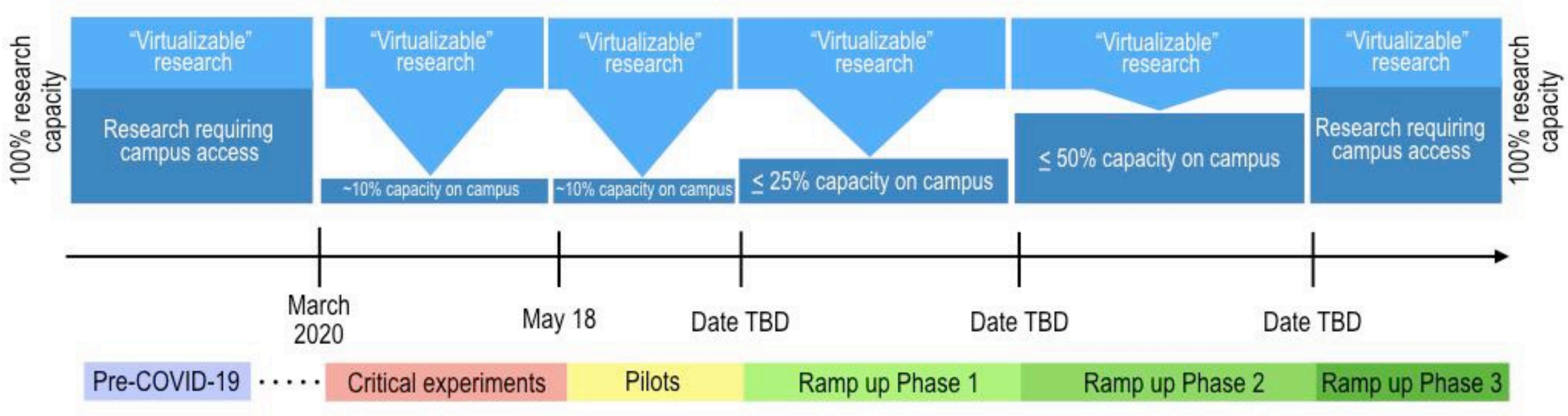
| | Yr1 – first half | Yr1 – second half | Yr2 – first half | Yr2 - second half | Yr3 – first half | Yr3- second half |
|---|---|---|---|---|---|---|
| Sample preparation and neutron radiation planning | ■ | ■ | | | | |
| Neutron radiation in ATR | | | ■ | ■ | | |
| TEM in IMCL | | | | | ■ | ■ |
| APT in CAES | | | | | ■ | ■ |
| Mechanical testing in IMCL | | | | | ■ | ■ |

# Milestones and Deliverables for FY-20

| Deliverables | Status | Start Date | Finish Date | Revised Finish Date | Actual Finish Date |
|---|---|---|---|---|---|
| Synthesis and preliminary characterization of 13 types of samples | Completed Late | 10/1/2018 | 9/30/2019 | 12/30/2019 | 3/31/2020 |
| Provided Four quarterly reports | Completed | 01/01/2020 | 10/30/2020 | | |
| Published two papers | Completed | 01/01/2020 | 11/27/2020 | | |

# Issues and Concerns

- MIT create a protocol for rapid response to the COVID-19 crisis. The MIT administration established policies to significantly scale back research activities on campus.




Response to this project: we have been continuing our research activities through remote meetings, literature surveys, data analysis, modeling, theoretical approach, and limited lab activities during COVID periods.

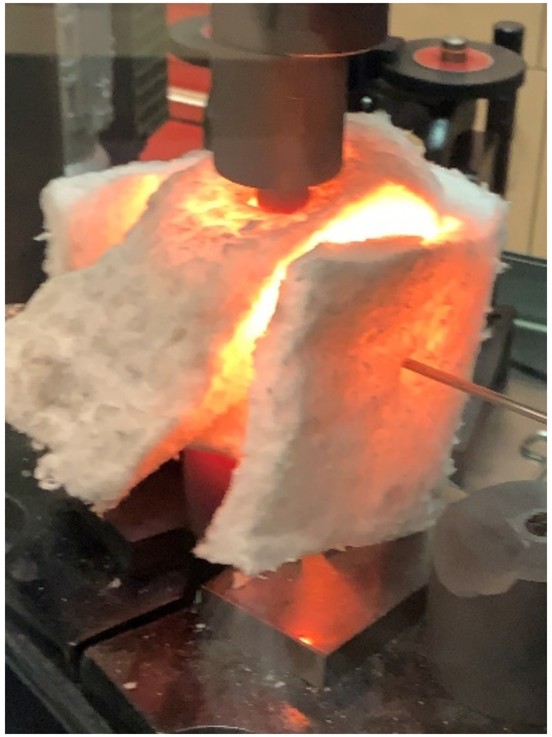
# Milestones and Deliverables for FY-21

- Deliverables:

| Technical reports | Date |
|---|---|
| Final Technical Report on Nanodispersion Strengthened Metallic Composites with Enhanced Neutron Irradiation Tolerance | 12/29/2021 |
| Revealing of the mechanical and thermal properties | 9/30/2021 |
| Radiation properties and mechanism characterization | 9/30/2021 |
| Characterization of the microstructure: dispersion of 1D and 2D reinforcement | 9/30/2021 |
| Optimization of the composite structure: grain and interface | 9/30/2021 |

# Possible Areas/Industries/Programs (and Readiness) for Adoption

- Include estimated Technology Readiness Level (TRL)



We have been working on the market discovery and commercialization for this technology (TRL 4 to 5) to transfer to the industry. The 3D printing market could be the lowest hanging fruit and most impactful industrial areas.

# Special thanks to our Oak Ridge National Lab collaborators

Kory Linton

Annabelle Le Coq

Xiang Chen

Ben Garrison

# Idaho National Lab collaborators

Cheng Sun

Mitch K. Meyer

# Contact Information and Questions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