Enhancing irradiation tolerance of steels via nanostructuring by innovative manufacturing techniques

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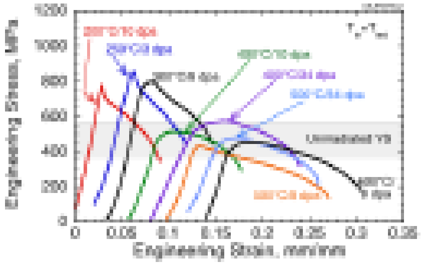
Outline

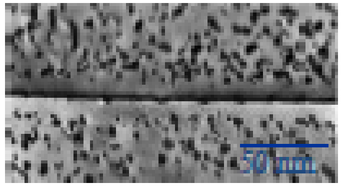
- **Background**
- **Manufacturing, microstructure and mechanical properties**
- **Ion irradiation effects**
- **Neutron irradiation effects**

Radiation Damages in Structural Materials and Fuel Cladding

- **Radiation hardening and embrittlement (<0.4 T_M, >0.1 dpa)**

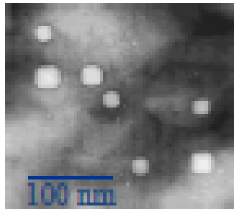


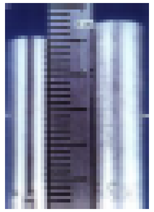
- **Phase instabilities from radiation-induced precipitation (0.3-0.6 T_M, >10 dpa)**



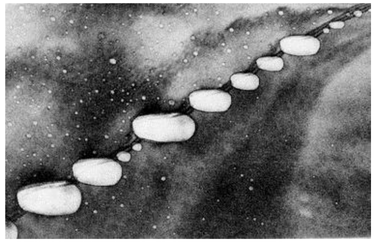
- **Irradiation creep (<0.45 T_M, >10 dpa)**

- **Volumetric swelling from void formation (0.3-0.6 T_M, >10 dpa)**





- **High temperature He embrittlement (>0.5 T_M, >10 dpa)**



Zinkle SJ, Was GS, Acta Mater 61 (2013) 735

- Life extension of current reactors and development of advanced reactors require improved irradiation tolerance
- How to improve irradiation tolerance: 1) engineer microstructure of existing materials or 2) develop new materials

Nanostructuring to improve mechanical strength

Strengthening mechanisms:

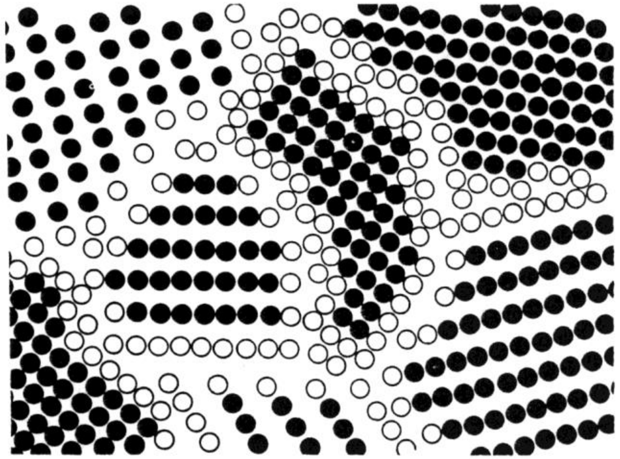
A. Work hardening: dislocation-dislocation interaction

B. Solid solution strengthening: solute-dislocation interaction

C. Particle strengthening: dislocation-particle interaction

Including precipitate strengthening and dispersion strengthening

D. **Grain boundary strengthening**: dislocation-grain boundary interaction



Hall-Petch relationship:

$\sigma_y = \sigma_0 + k_y \cdot d^{-1/2}$

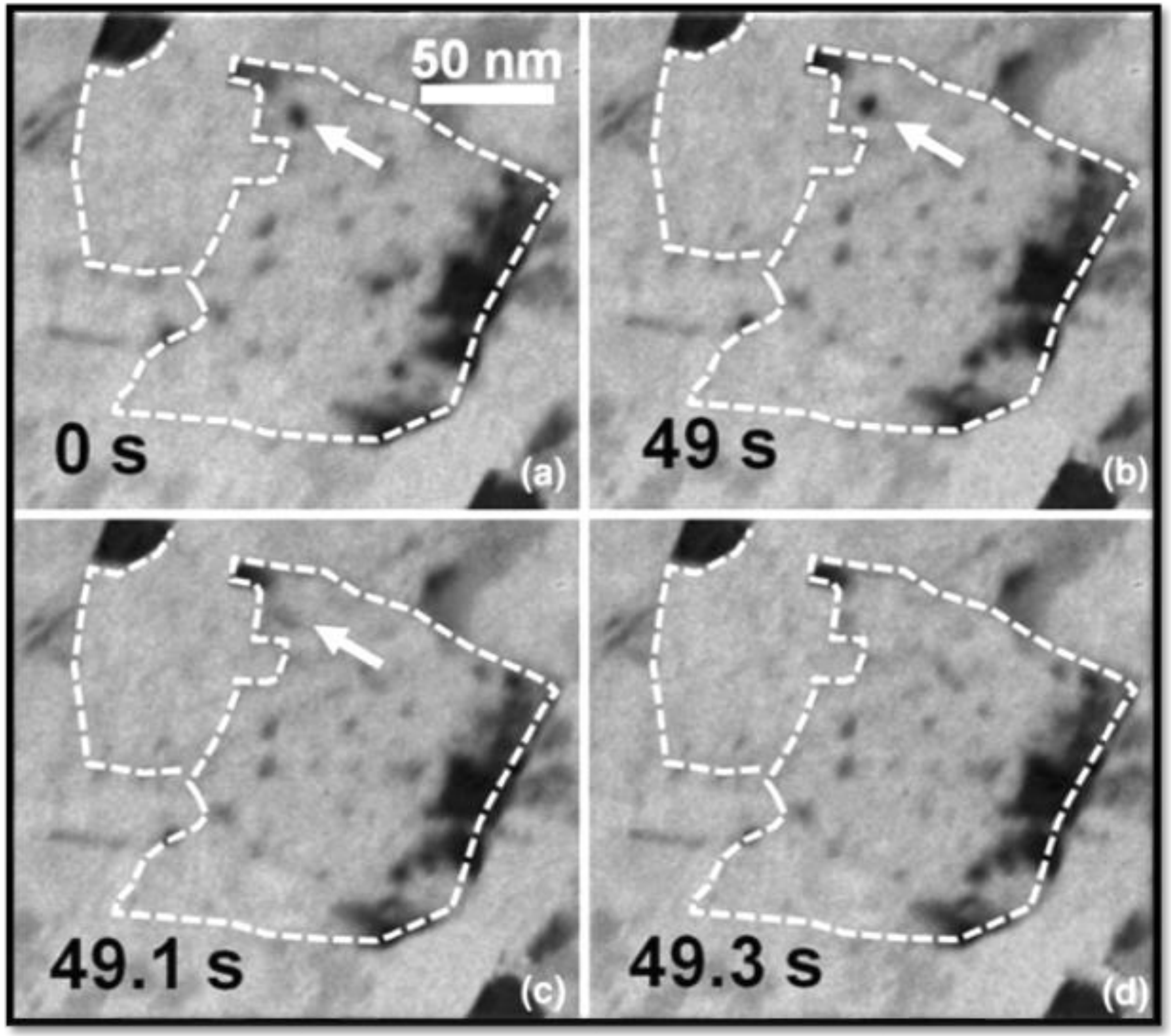
σ_0, k_y : material constants

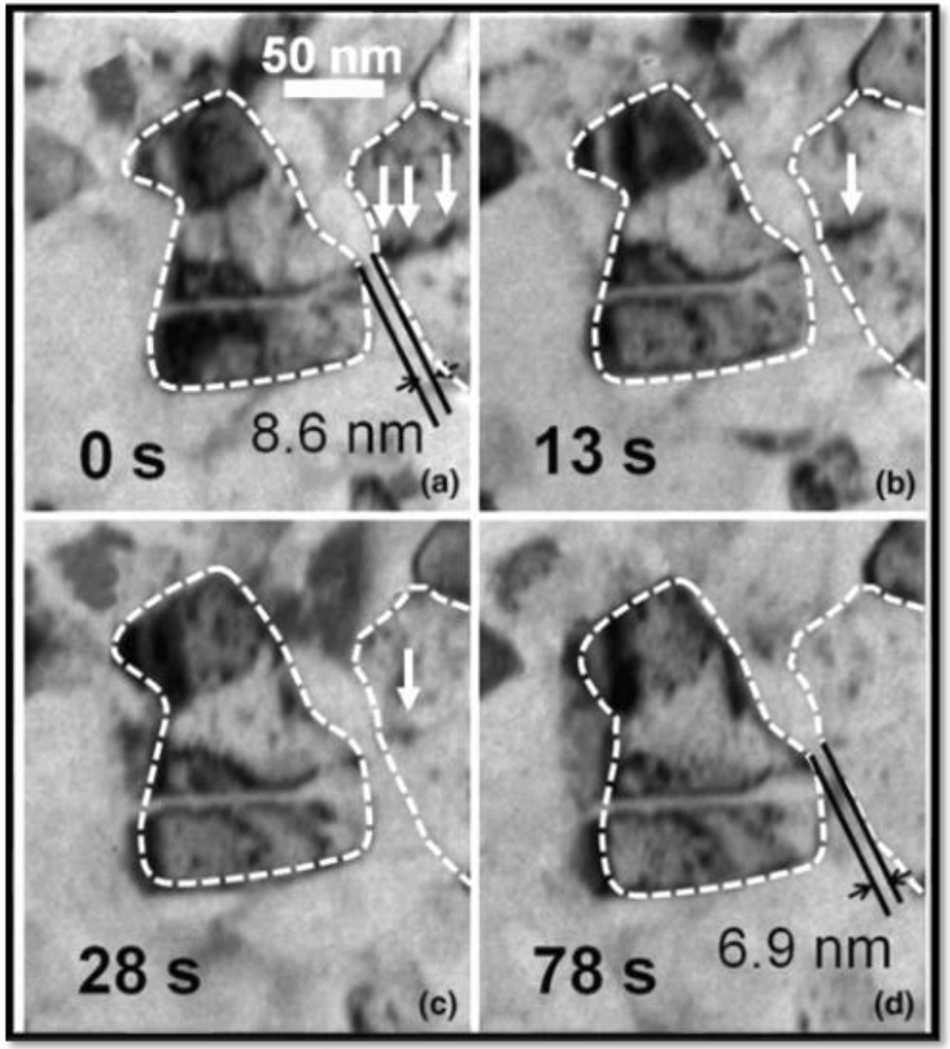
Nanocrystalline material: single or multiple-phase polycrystals with structural features (typically grains) smaller than 100 nm

- D=5 nm, fraction of GBs=50%
- D=100 nm~1 μm, **ultrafine grained materials**; D=1~10μm, fine grained materials; D>10 μm, coarse grained conventional materials

H. Gleiter, in Proceedings of the second Risϕ International Symposium on Metallurgy and Materials Science,1981, Denmark: Risϕ National Laboratory, Roskilde

GBs as Sinks for Irradiation Defects



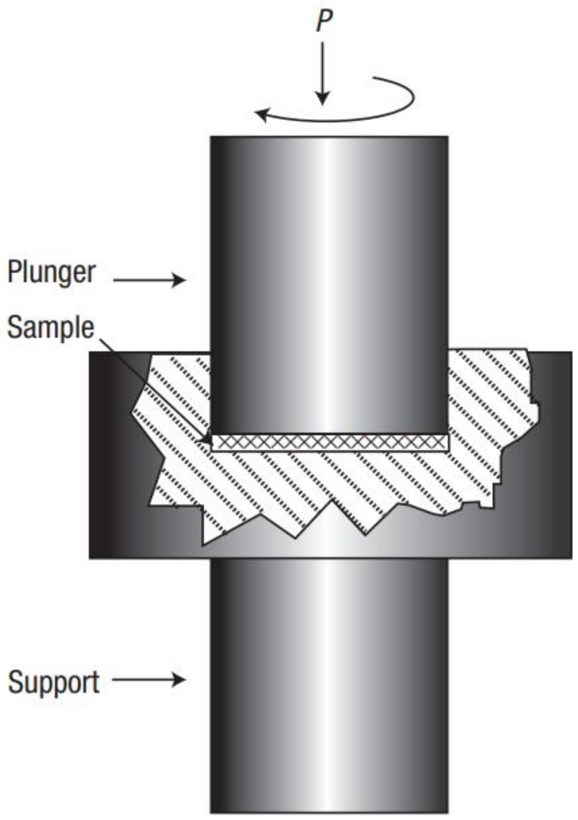


- In-situ TEM imaging during ion irradiation of NC Ni films
- Grain boundaries as sinks for irradiation-induced dislocation loops and segments
- Anticipated enhanced irradiation tolerance in NC or UFG materials

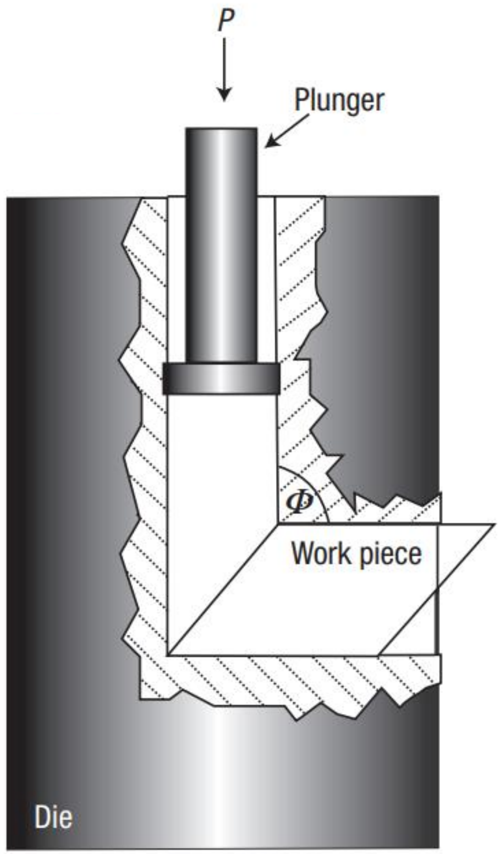
Sun C, et al., Metall Mater Trans A 44 (2013) 1966

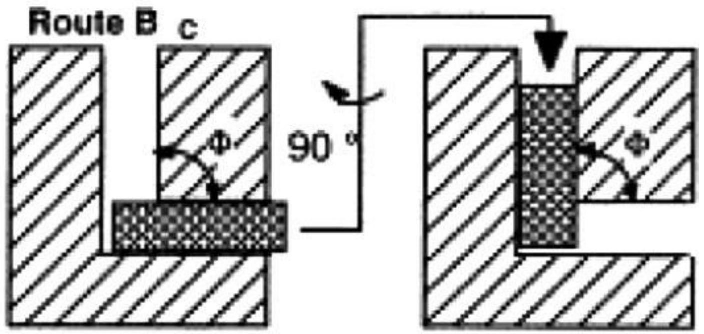
Nanostructuring through severe plastic deformation

High pressure torsion (HPT)

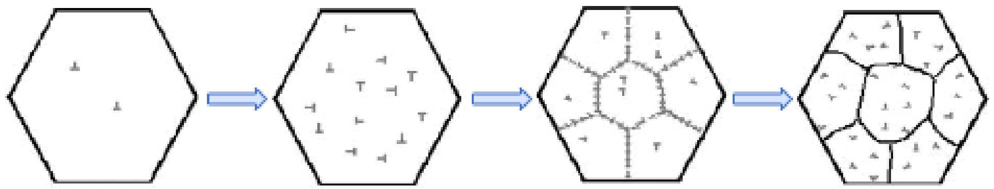


Equal Channel Angular Pressing (ECAP)





Grain refinement process



-Valiev, Ruslan. "Nanostructuring of metals by severe plastic deformation for advanced properties." *Nature materials* 3, no. 8 (2004): 511-516.

Advanced manufacturing using severe plastic deformation

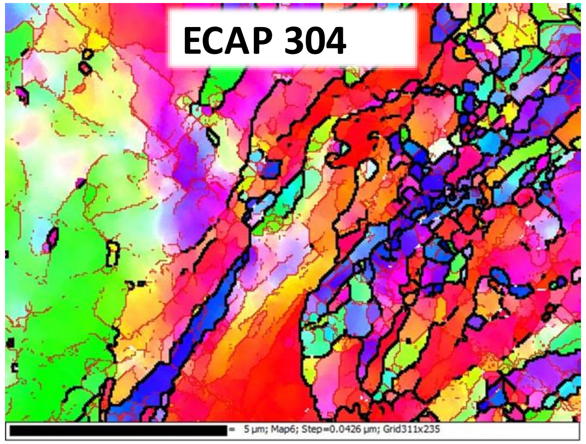
Element	SS304	SS316	G91	Kanthal-D
Fe	Balance	Balance	Balance	Balance
Cr	17.22	16.18	8.38	20.57
Ni	9.56	12.24	0.17	0.26
C	0.03	0.02	0.11	0.026
Mo	0.12	2.47	0.9	-
Al	-	-	-	4.79
V	0.04	0.04	0.2	0.03
Ti	0.26	0.32	-	0.02
Cu	0.16	0.23	0.17	0.02
Si	0.24	0.37	0.46	0.24
W	0.04	0.04	-	-
P	0.03	0.03	0.01	-
Mn	-	-	0.43	0.18
Nb	-	-	0.06	-

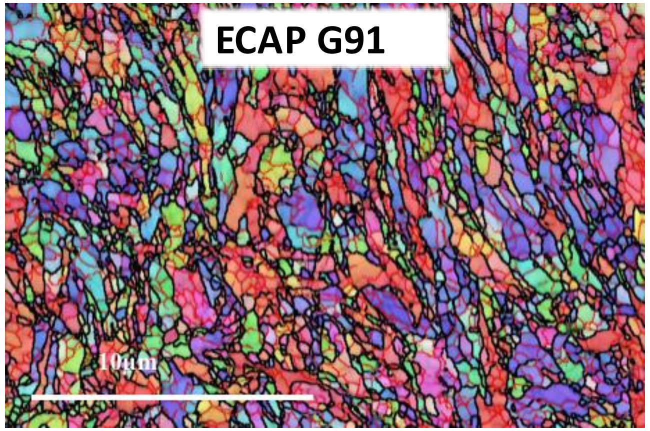
Material	Technique	Temp (°C)	# of passes/turns
SS 304	HPT	300	10
SS 316	HPT	300	10
Grade 91	HPT	300	10
Kanthal D	HPT	300	10
SS 304	ECAP	450	6
SS 316	ECAP	380	6
Grade 91	ECAP	300	6
Kanthal D	ECAP	520	6

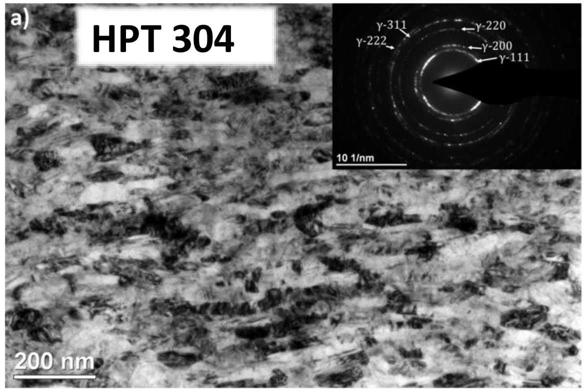
- HPT was performed at 300 ^{o}C for all the alloys.
- ECAP manufacturing temperature is material specific to prevent cracking.
- Deformation at elevated temperatures suppressed deformation induced martensitic transformation: 304 and 316 fully austenitic after SPD

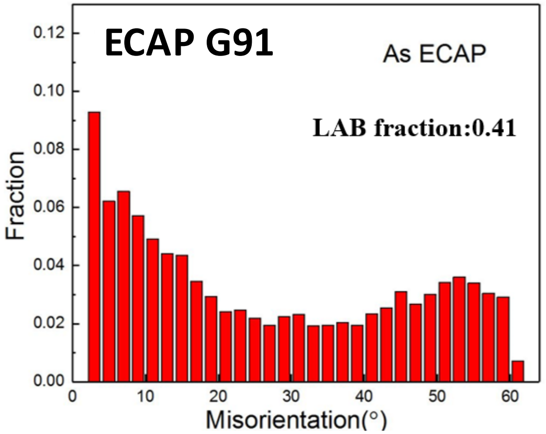
Hoffman AK, Wen HM, et al., *Materials Letters* 243 (2019) 116–119.
Arivu M, Wen HM, et al., *Materials Letters* 253 (2019) 78–81.
Duan JQ, Wen HM, et al., *Materialia* 6 (2019) 100349.

Microstructures obtained by SPD

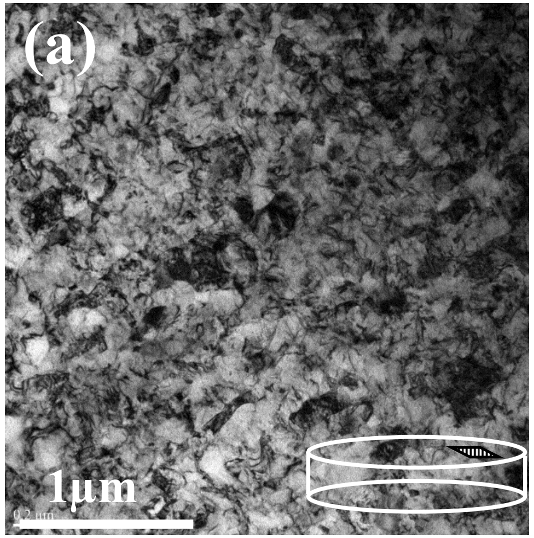


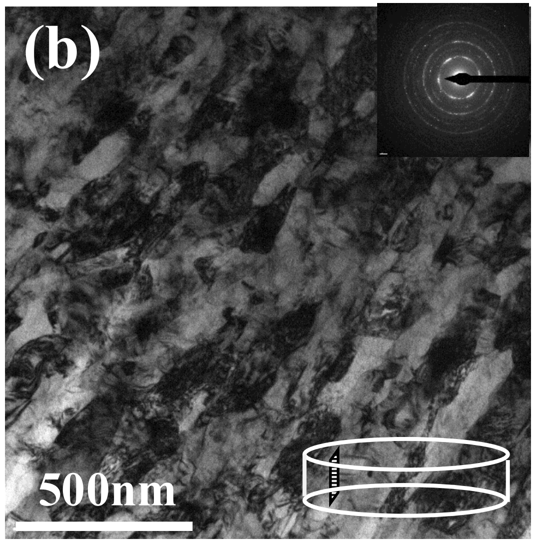


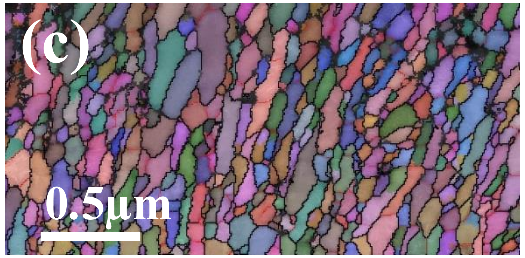


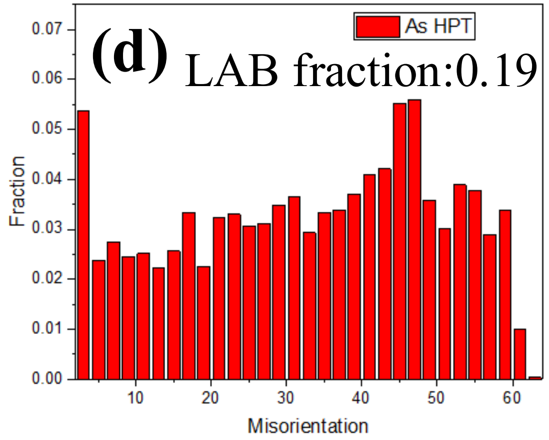


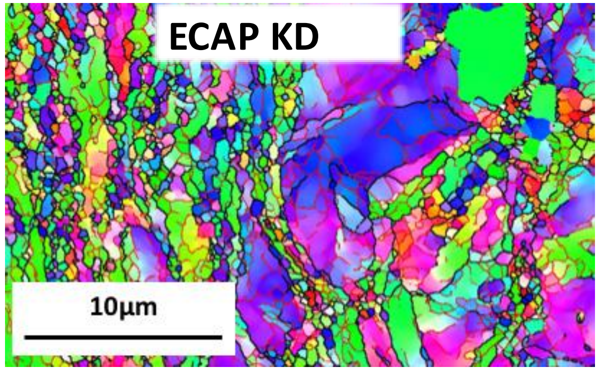
HPT G91

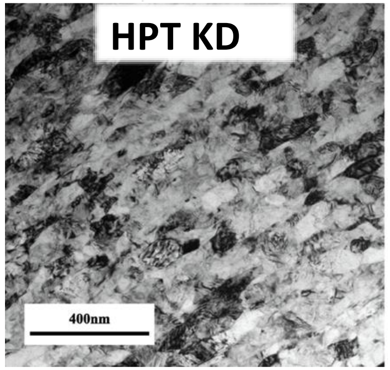








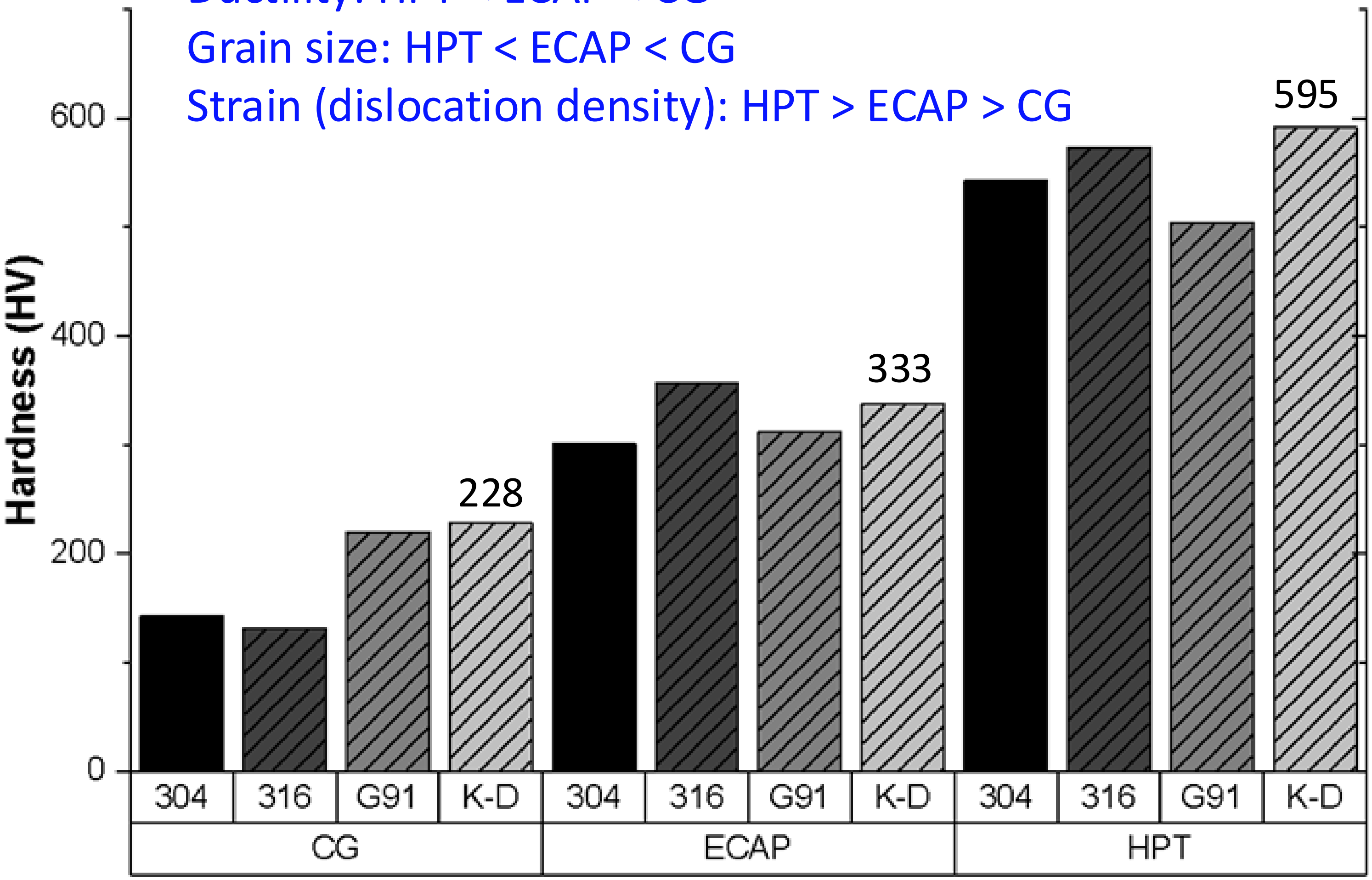


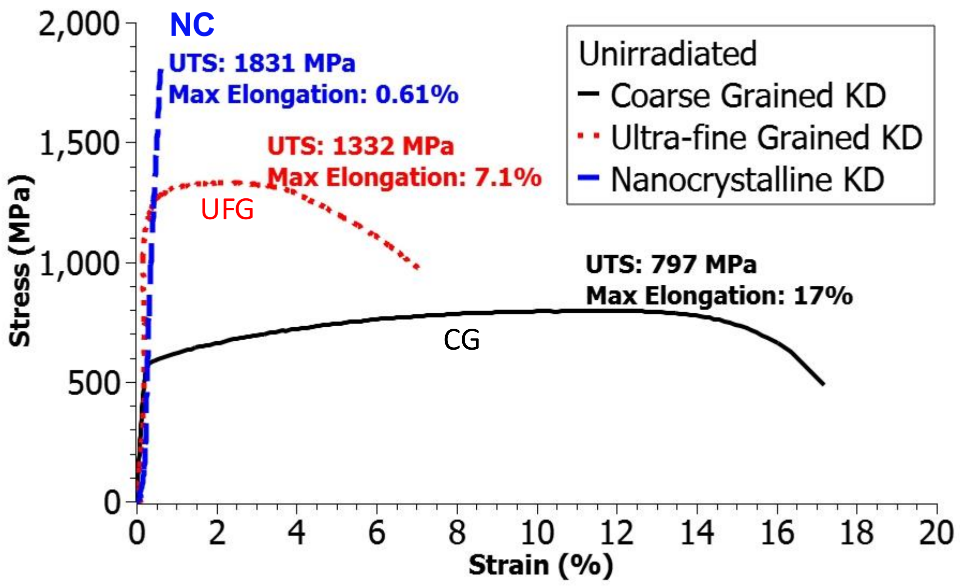


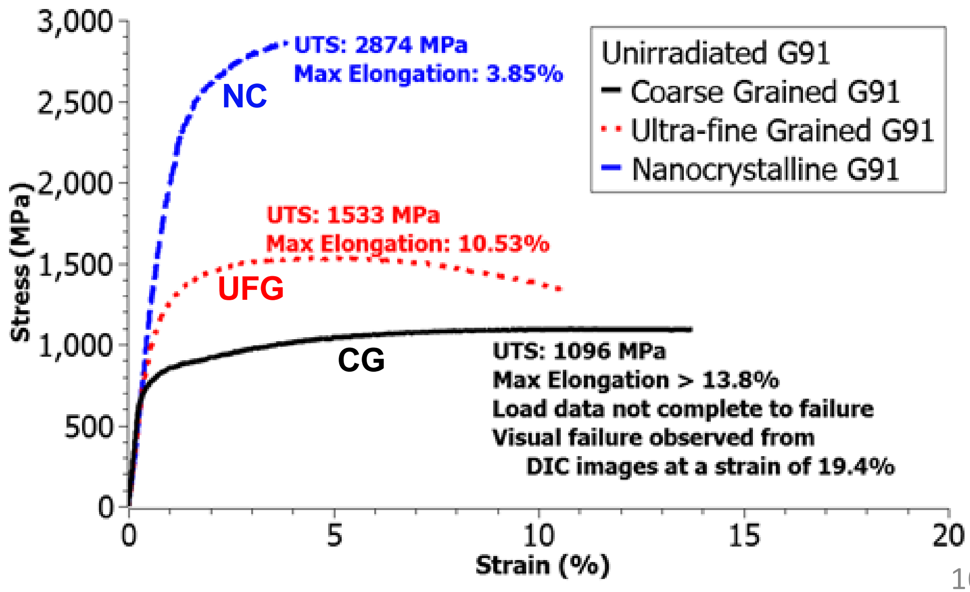
- HPT: ~100 nm average grain size, homogeneous microstructure, low LAGB fraction
- ECAP: ~400 nm average grain size, inhomogeneous microstructure, significant LAGB fraction.
- HPT + ECAP: high dislocation density

Mechanical Properties of Nanostructured Steels

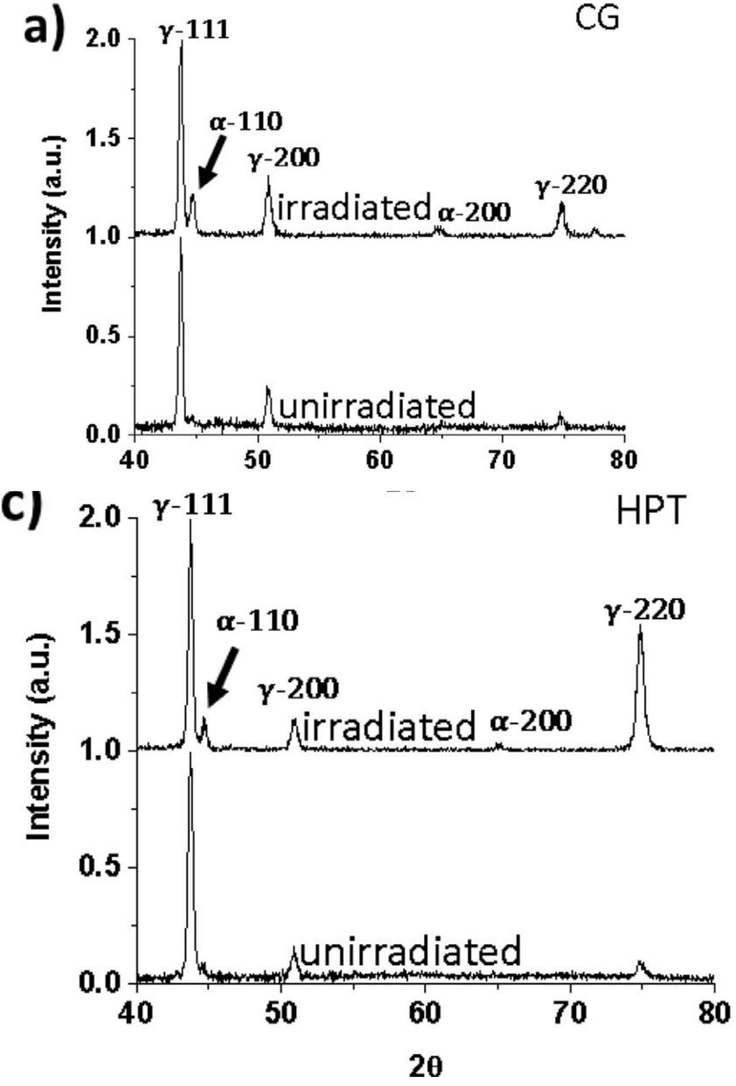
Hardness/strength: HPT > ECAP > CG
Ductility: HPT < ECAP < CG
Grain size: HPT < ECAP < CG
Strain (dislocation density): HPT > ECAP > CG

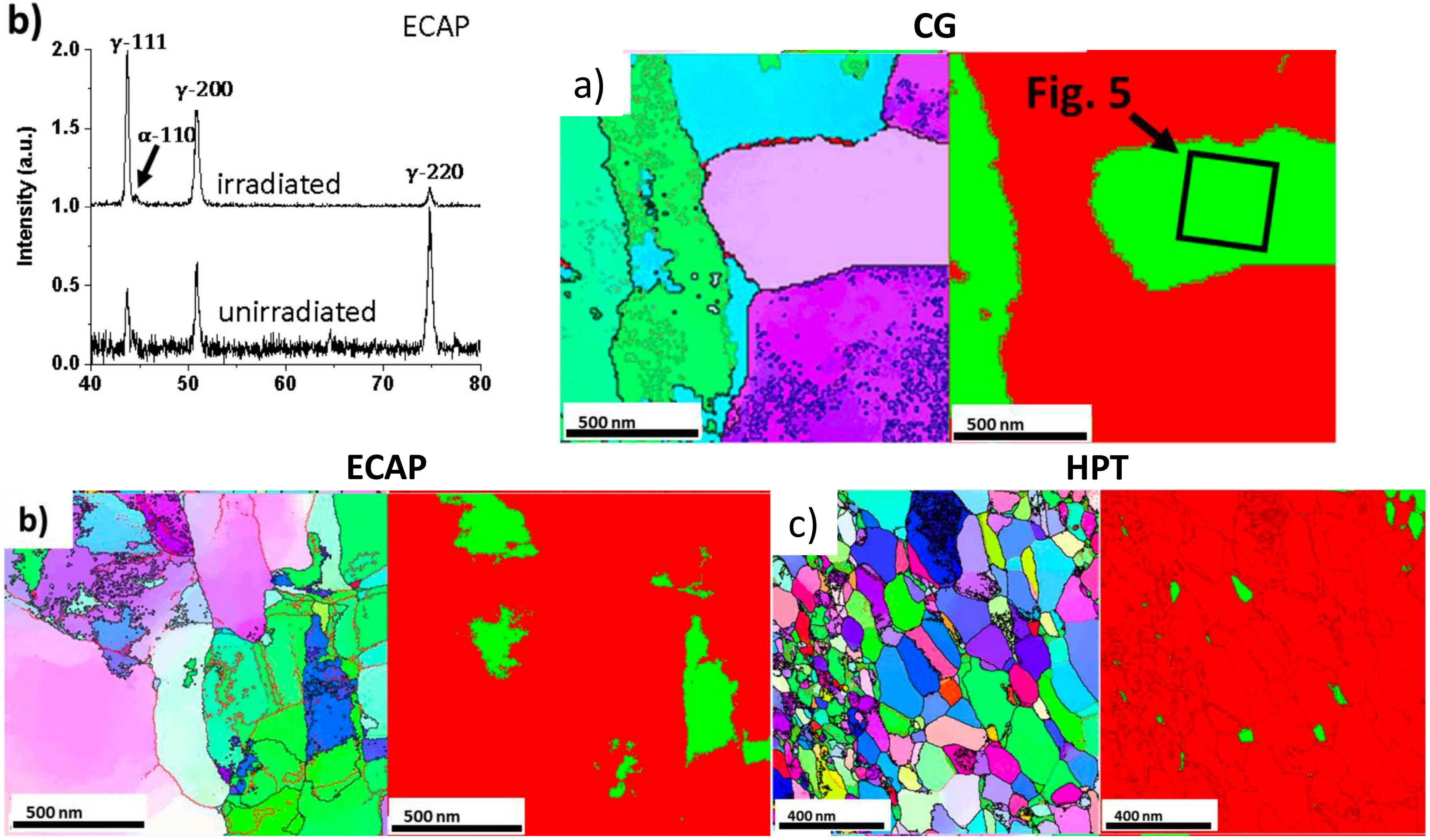






Enhanced Austenite Stability of Nanostructured 304

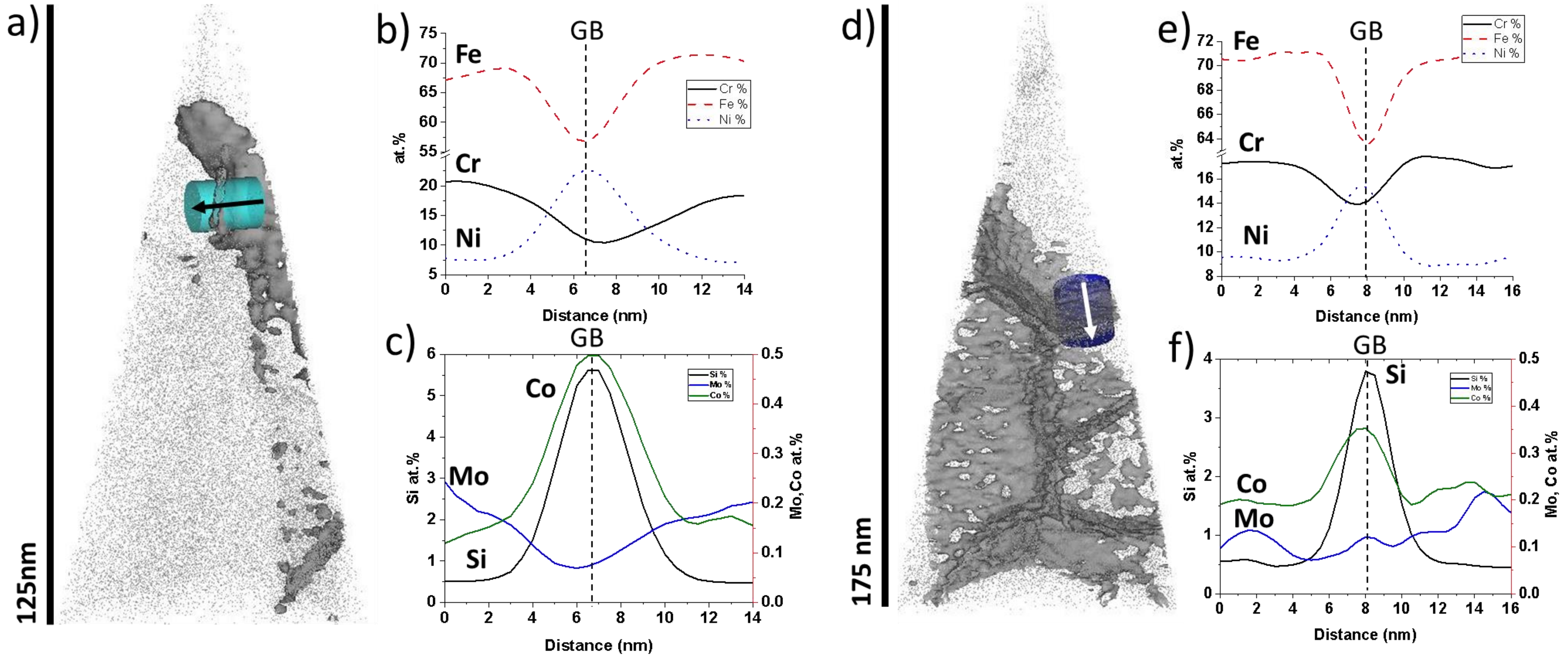




- **Nanostructured steels resistant to radiation induced austenite to ferrite transformation**

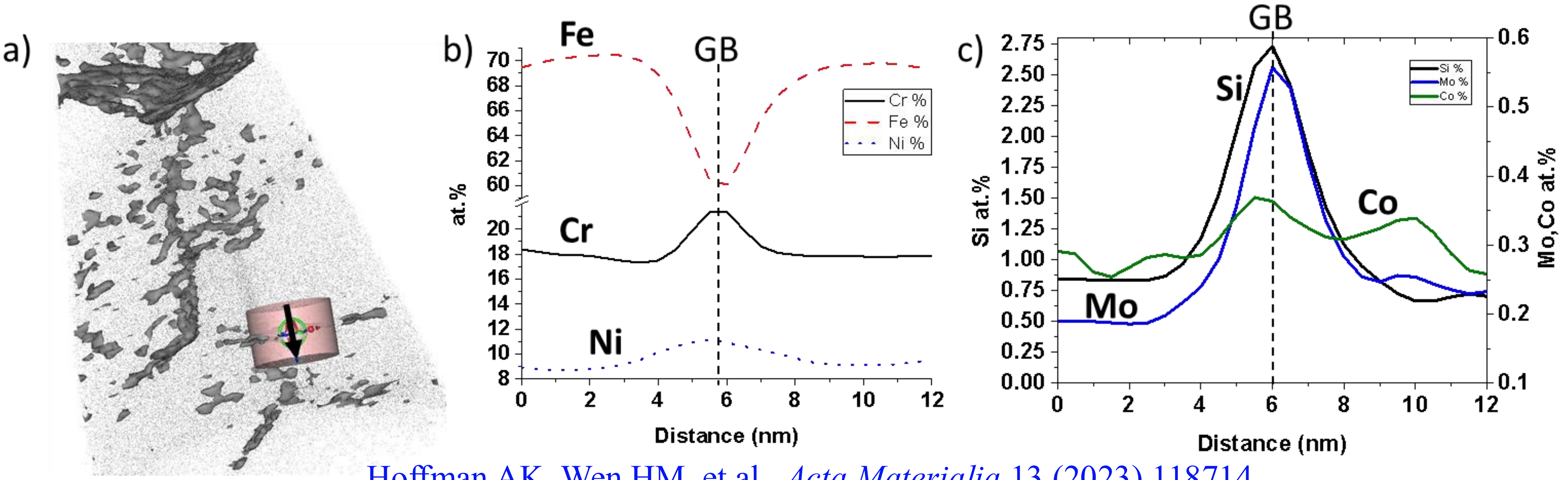
Hoffman AK, Wen HM, et al., *Materialia* 13 (2020) 100806.

RIS comparison between UFG and NC 304



- RIS observed with Cr and Fe depletion and Ni enrichment at GBs
- UFG region (left) showed more severe RIS than NC region (right)
- Limited Mo depletion seen in UFG region but no depletion of Mo observed in NC region

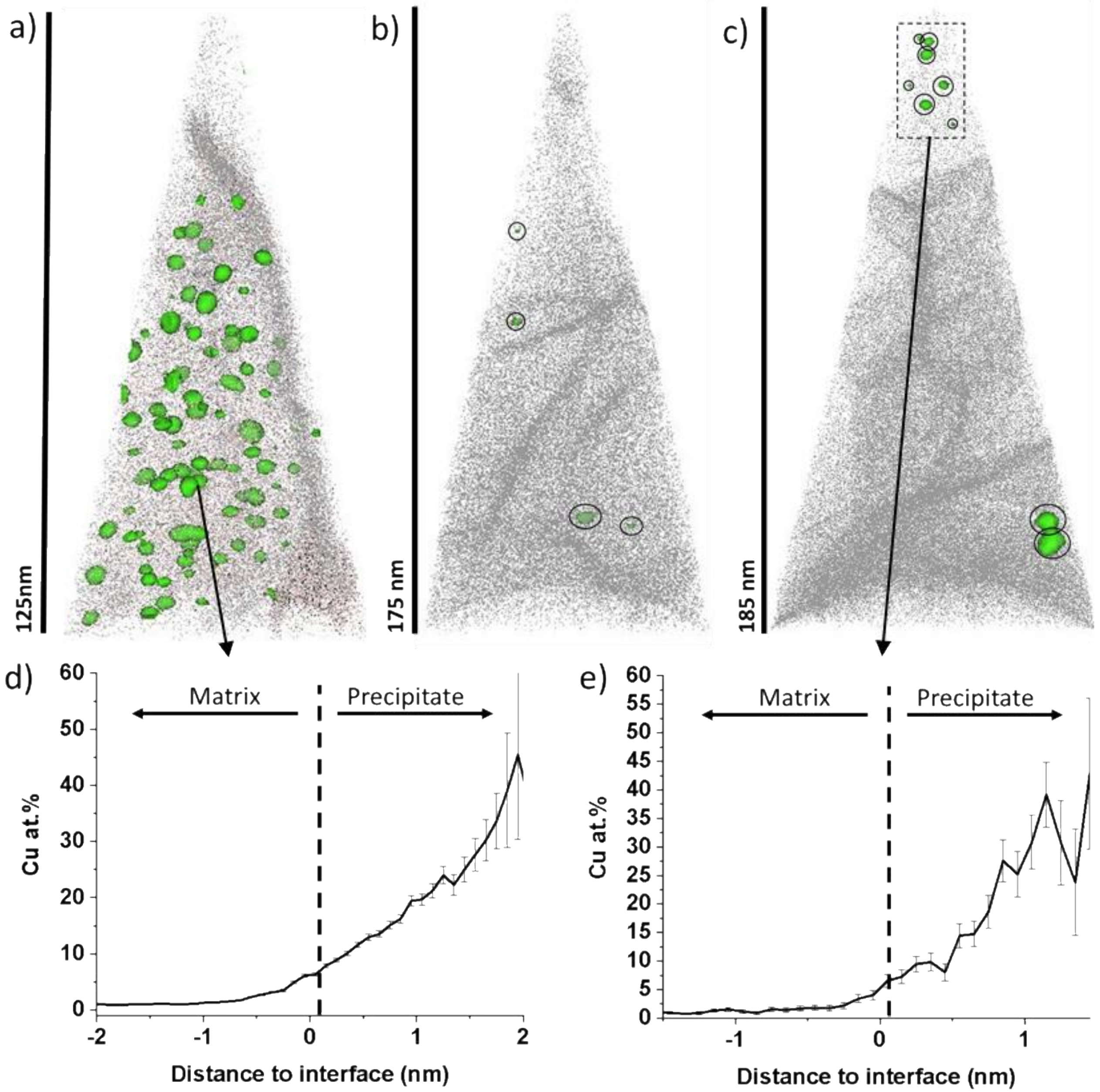
Enhanced resistance to segregation in NC 304



Hoffman AK, Wen HM, et al., *Acta Materialia* 13 (2023) 118714.

GB Description	# of GBs	Δat.% Fe	Δat.% Cr	Δat.% Ni	Δat.% Si	Δat.% Mo	Δat.% Co
Annealed	6	-3.5±2.4	+1.5±1.0	0	+0.6±0.4	1.2±0.7	0
Irradiated UFG	3	-11.6±6.0	-7.3±1.6	+13.8±3.0	+3.7±1.8	0	+0.2±0.1
Irradiated NC Grains	18	-4.3±1.4	-3.9±1.4	+5.3±1.8	+2.2±0.6	0	+0.1±0.04
Irradiated NC Cr enriched	4	-6.6±2.6	1.5±0.9	+2.0±1.0	+2.0±0.3	+0.4±0.1	0

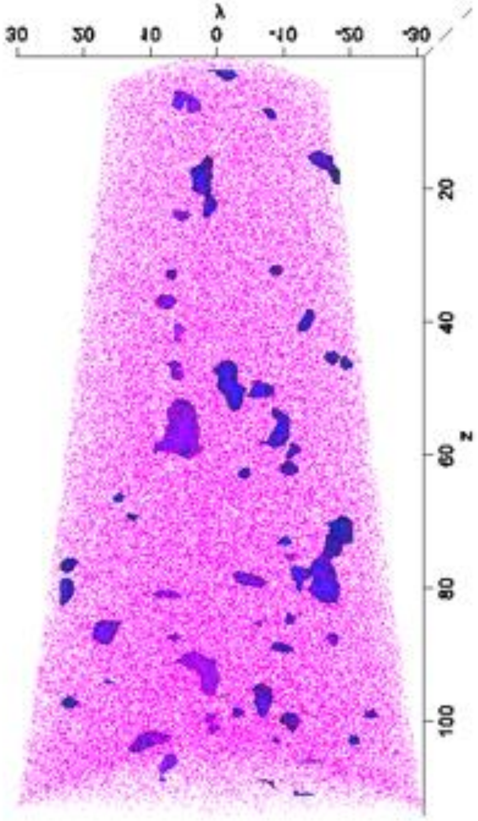
Precipitation comparison between UFG and NC 304



- UFG contains many Cu precipitates which are assumed to nucleate on small dislocations or loops
- NC regions have significantly fewer to no Cu precipitates
- Reduced irradiation-induced segregation/precipitation by reducing grain size

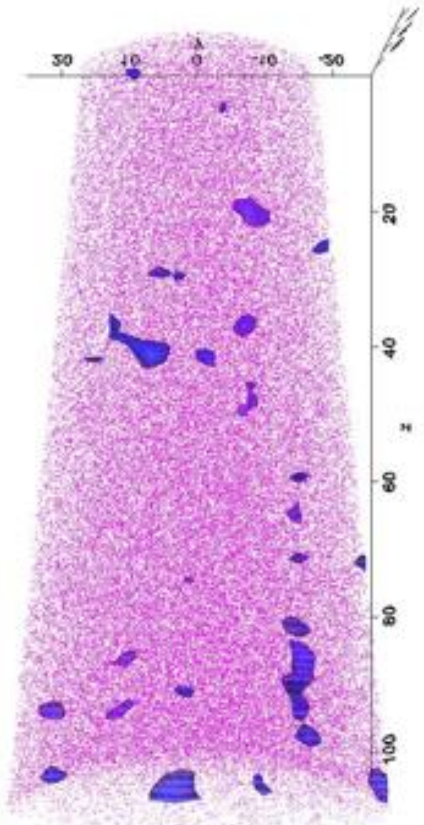
Reduced irradiation-induced α’ Cr precipitation in nanostructured Kanthal-D steel

CG KD 5DPA 500C



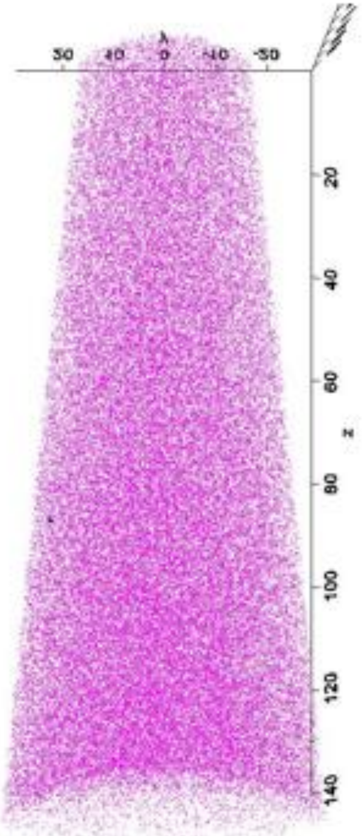
Fe atoms with 30 at.% Cr iso-surface

ECAP KD 5DPA 500C



Fe atoms with 30 at.% Cr iso-surface

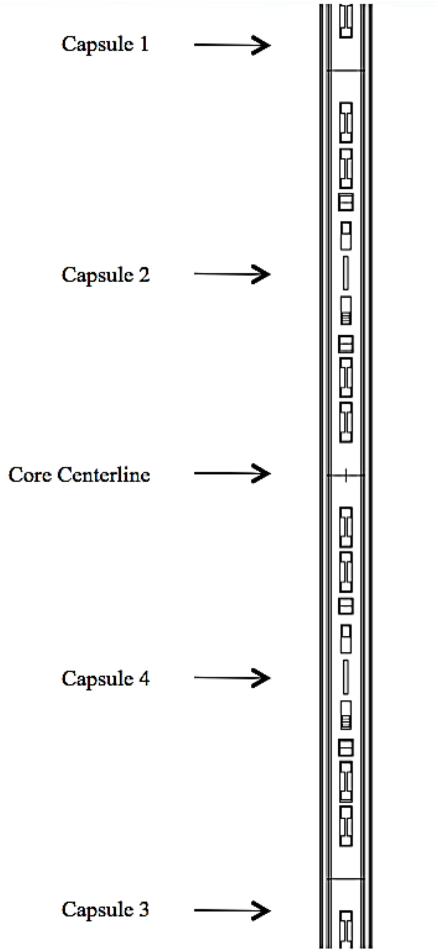
HPT KD 5DPA 500C

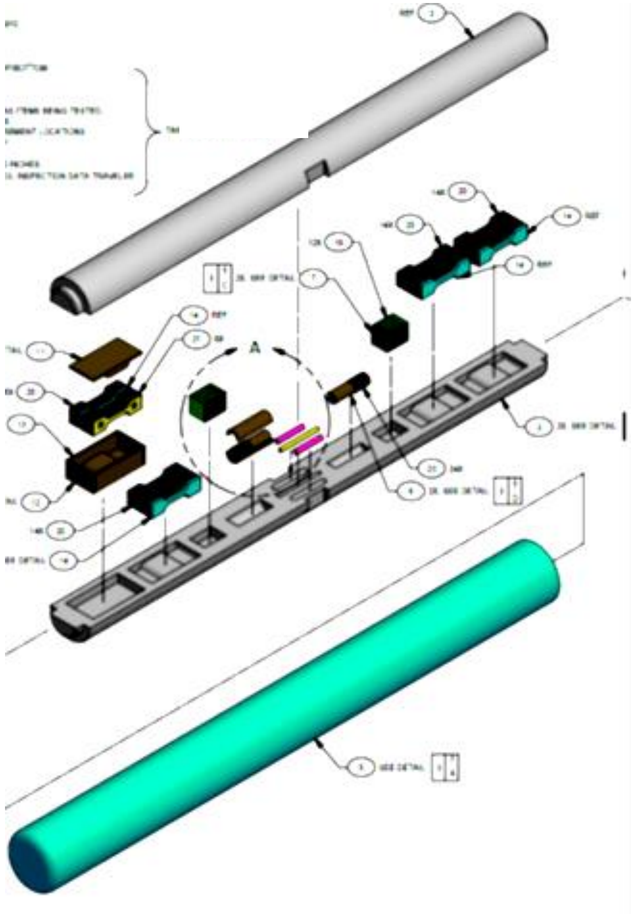


Fe atoms with 30 at.% Cr iso-surface

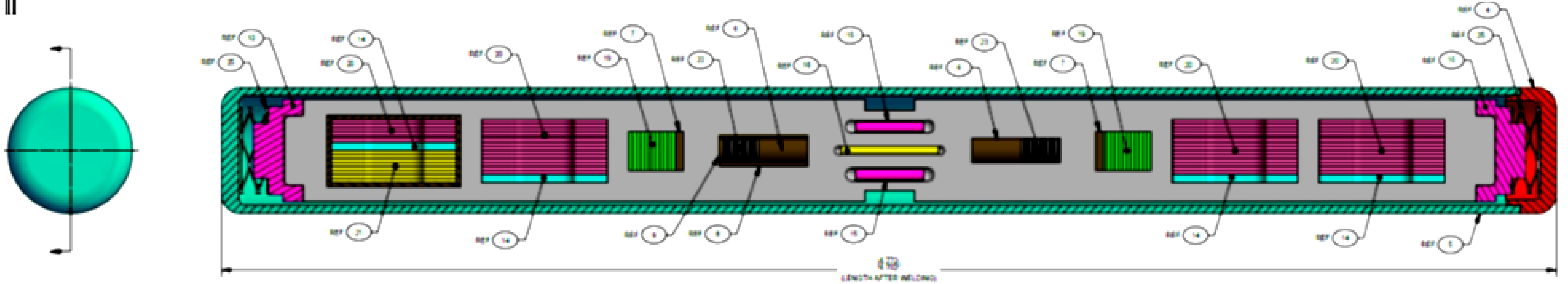
- Samples ion irradiated
- α’ Cr precipitation can be observed in CG and ECAP KD
- HPT KD showed no precipitation

Neutron irradiation

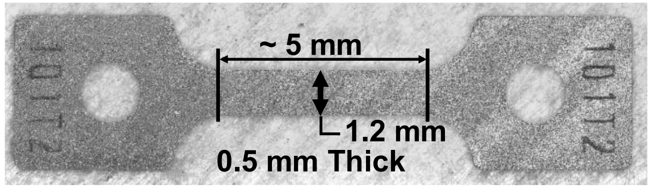


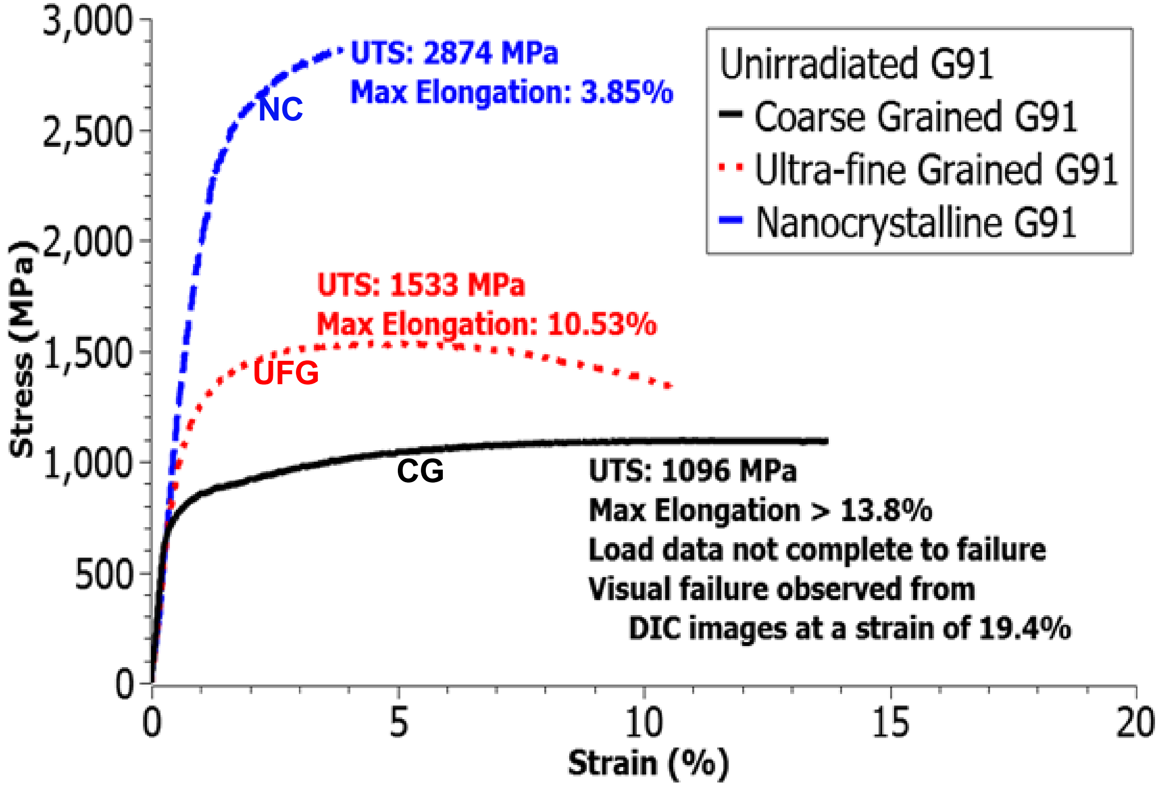


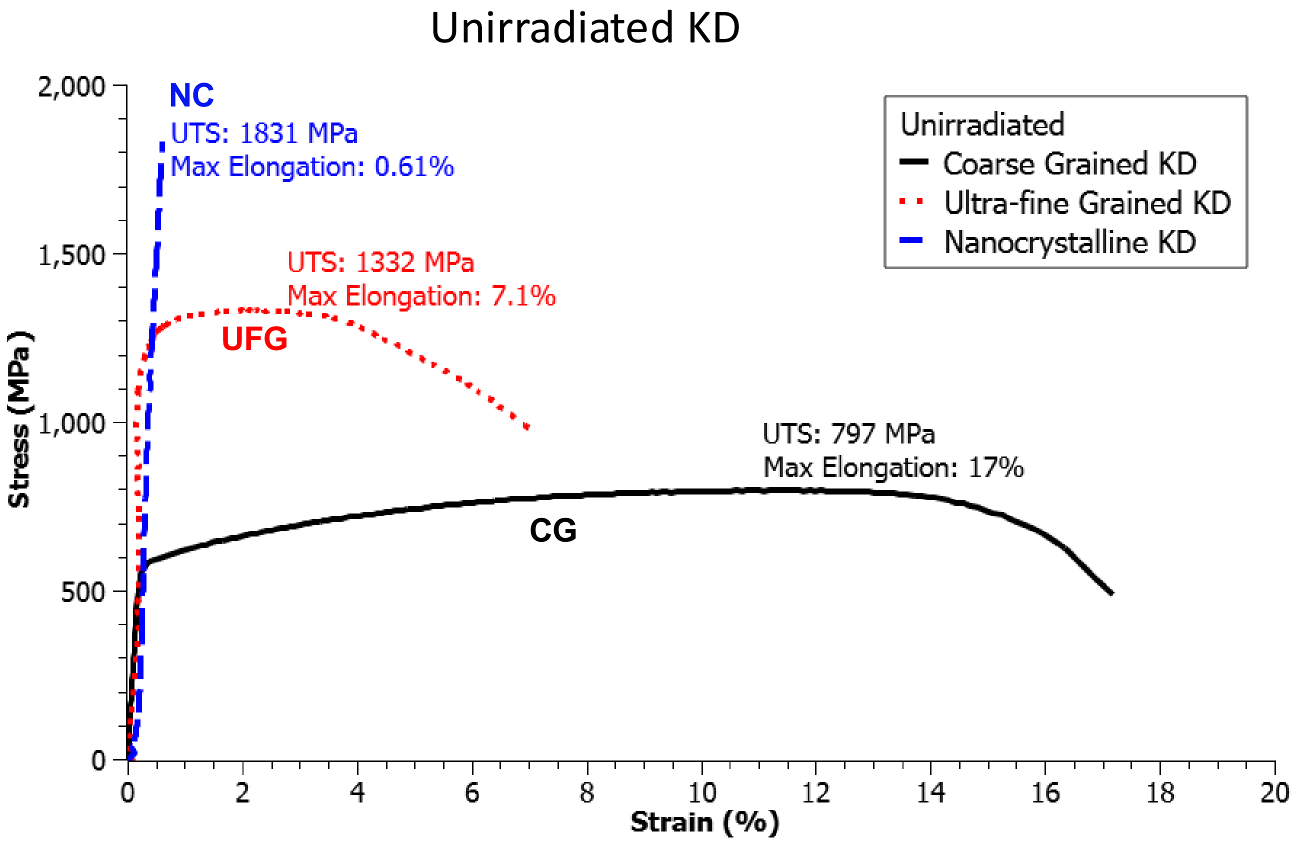
- Four irradiation conditions and capsules
  - Capsule 1 at 300 ^{o}C to 2 DPA
  - Capsule 2 at 300 ^{o}C to 6 DPA
  - Capsule 3 at 500 ^{o}C to 2 DPA
  - Capsule 4 at 500 ^{o}C to 6 DPA
- Tensile, hardness and TEM specimens
- ~500 specimens in total
- Post-irradiation examination completed for KD and Grade 91



Tensile behavior of unirradiated nanostructured KD and G91 steels

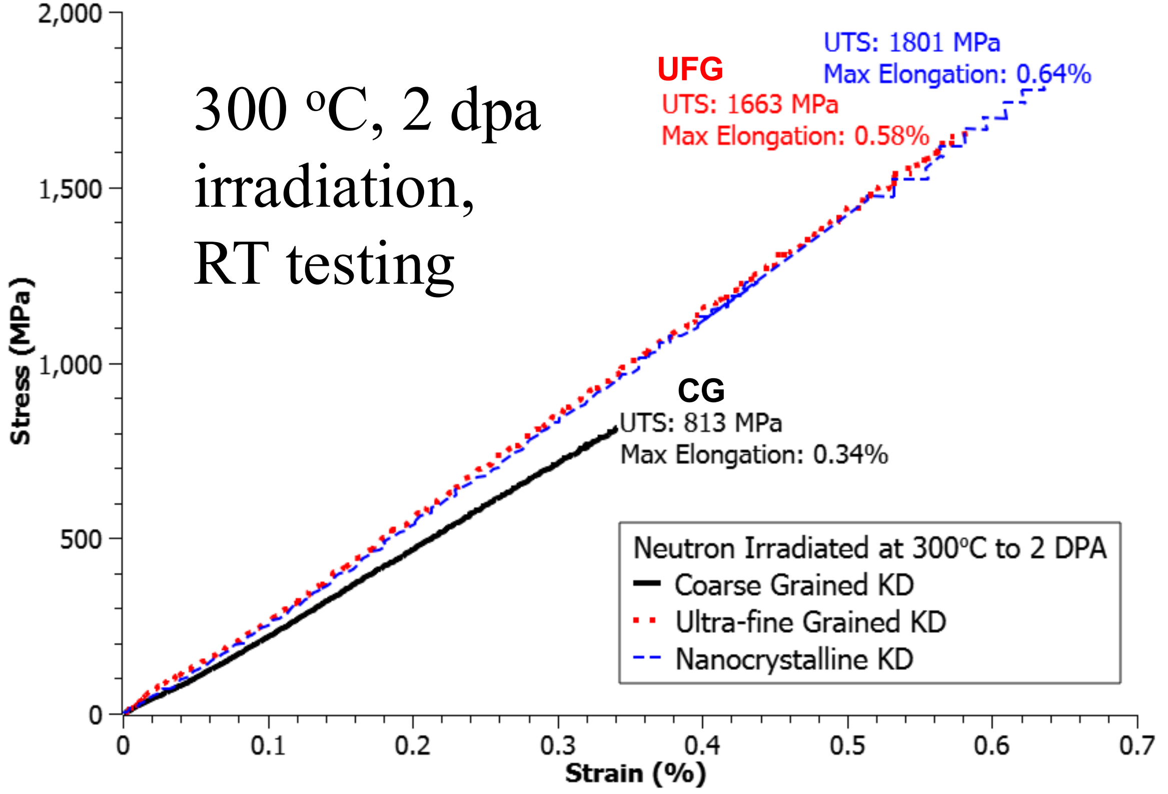


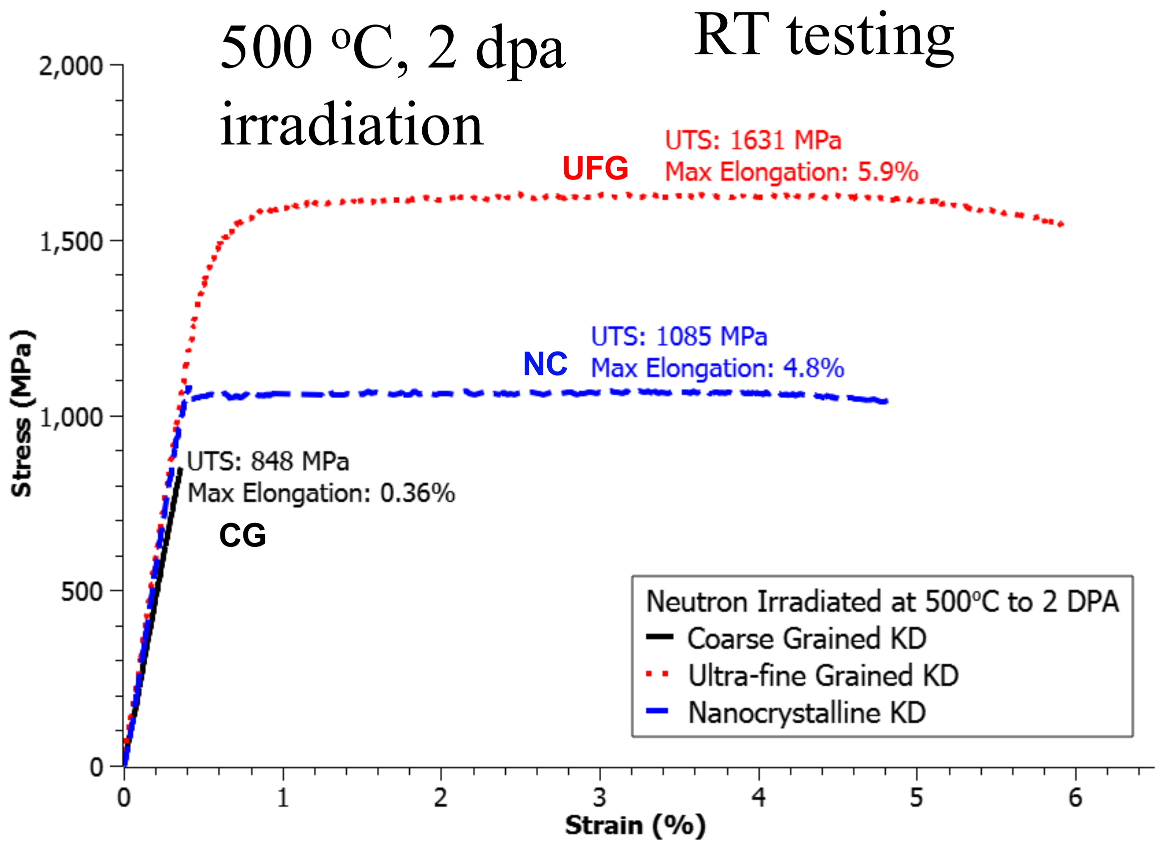




- Nanostructured steels are stronger and less ductile than CG steels.
- CG KD and Grade 91 steels have good ductility.

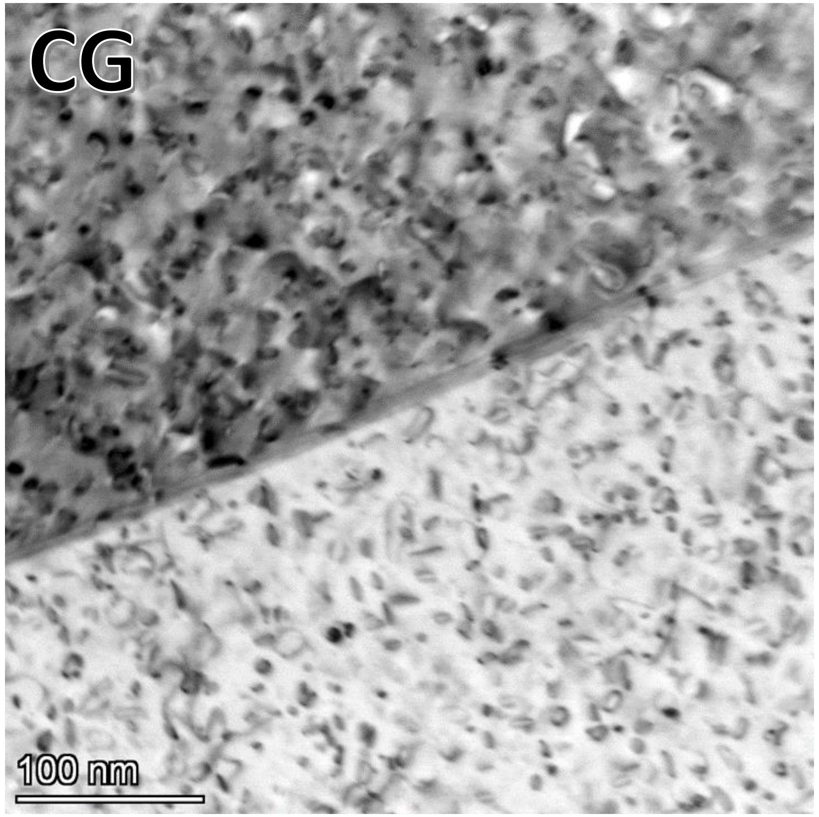
Tensile behavior of neutron irradiated nanostructured KD steel

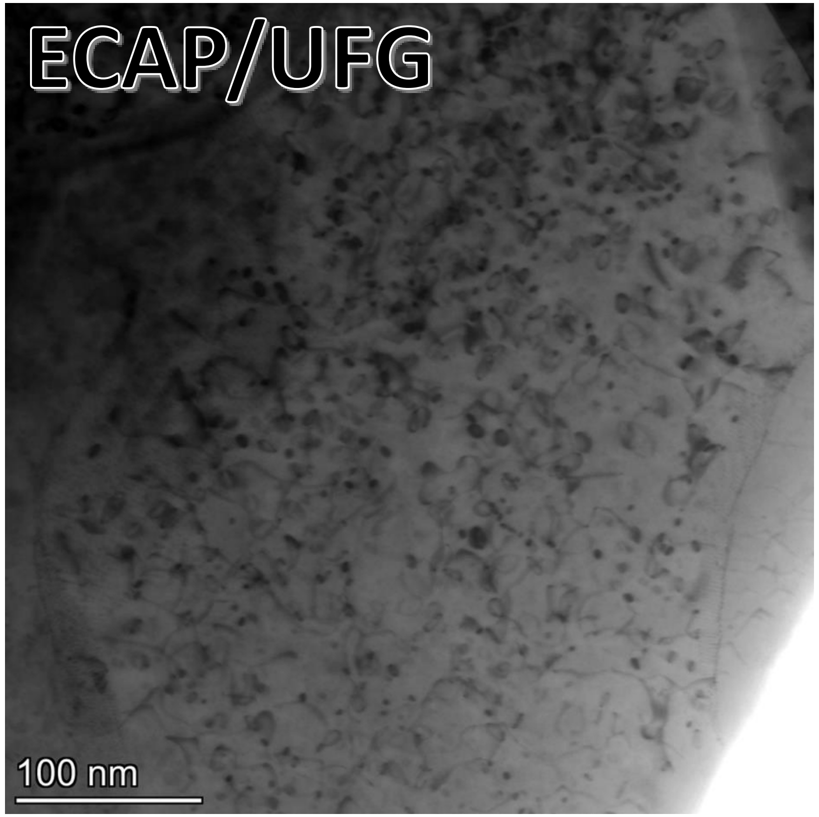


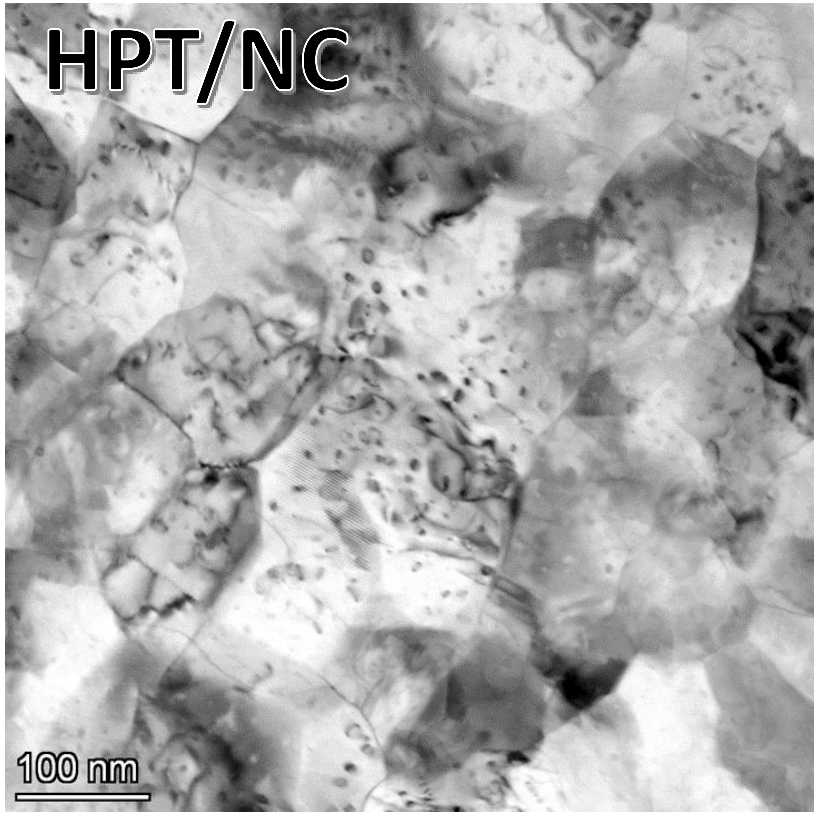


- CG KD becomes very brittle after irradiation at 300 and 500 °C.
- Ductility of NC KD significantly improved after irradiation
- UFG and NC KD retained ductility much better than CG KD
- UFG and NC KD performed better than CG KD

TEM of neutron irradiated nanostructured KD steel – 300 ^{o}C, 2 dpa



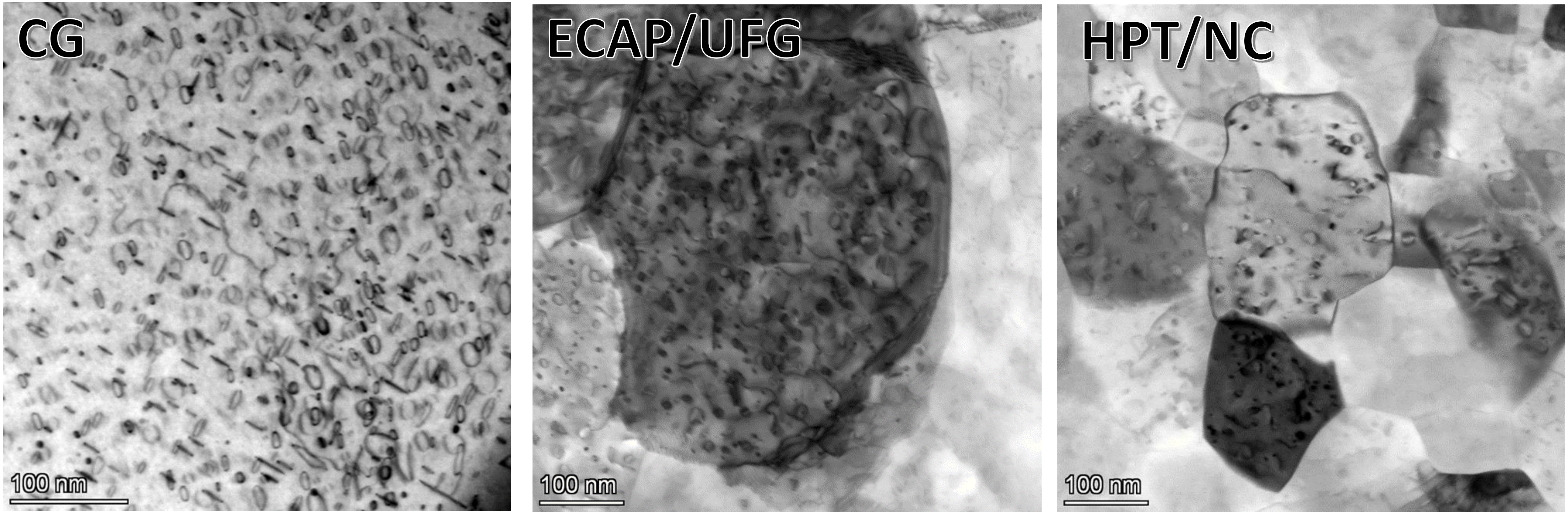




KD neutron irradiated to 2 DPA at 300 °C

- Less dislocation loops in HPT
- Nanocrystalline grain size maintained after irradiation
  - Some grain growth in NC with average grain size of ~123 nm likely due to added effects of irradiation

TEM of neutron irradiated nanostructured KD steel – 500 °C, 2 dpa

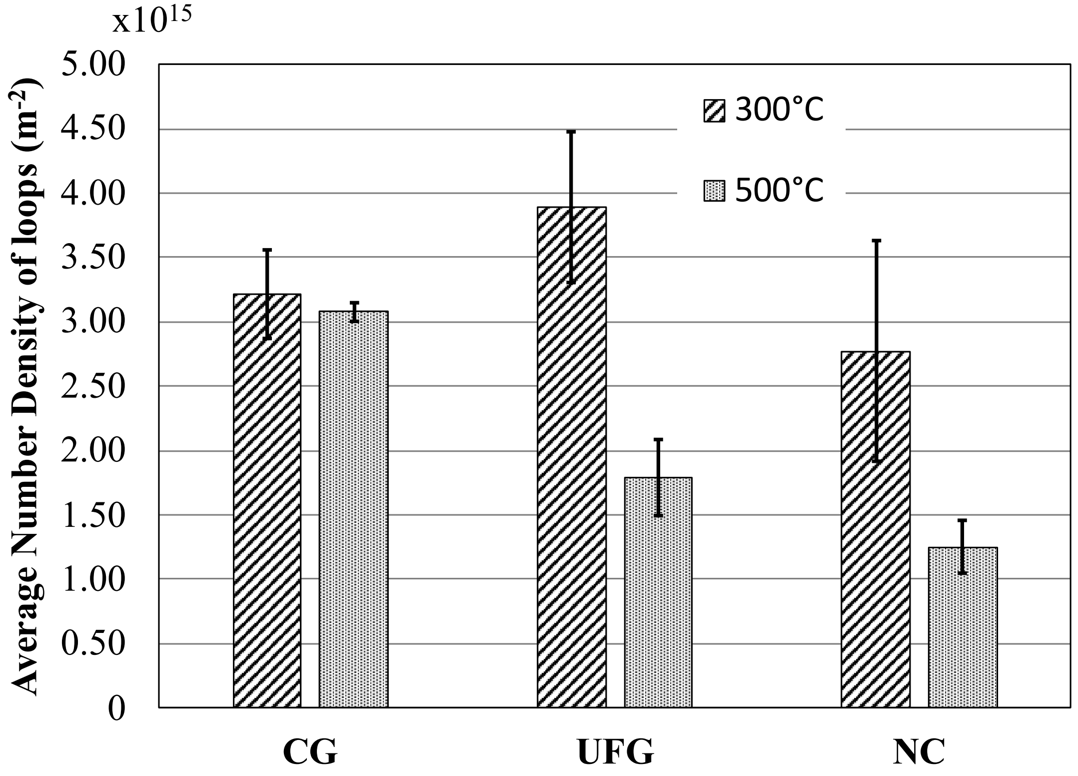


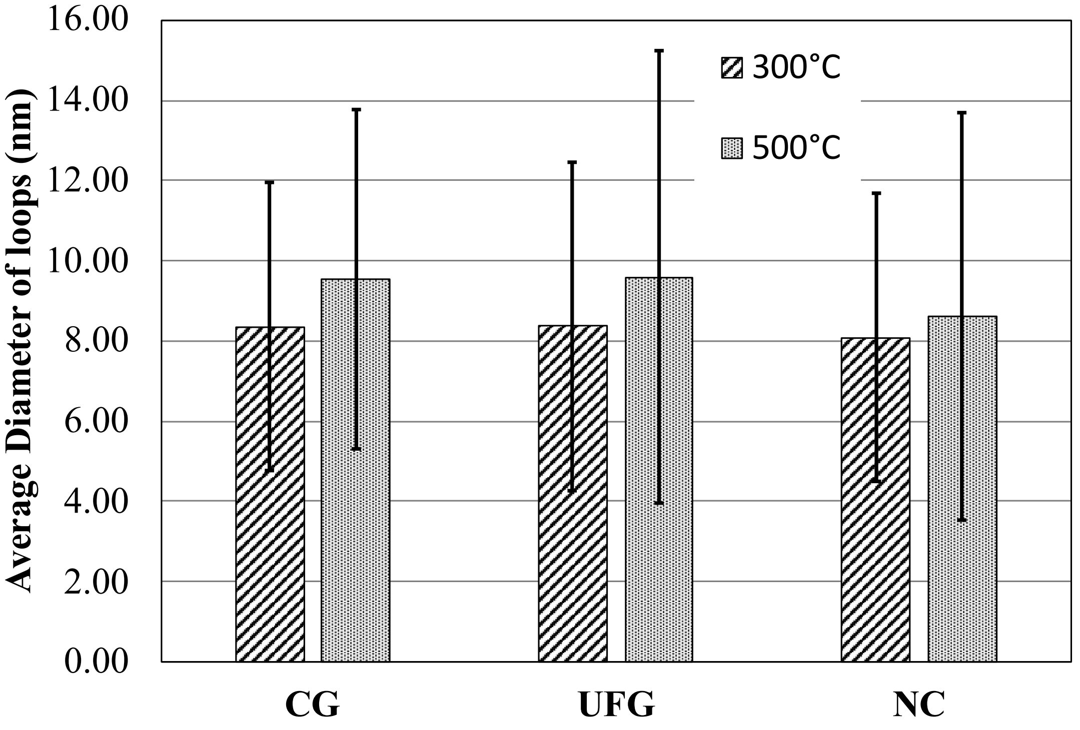
KD neutron irradiated to 2 DPA at 500 °C

- Less dislocation loops in HPT
- Grain growth in HPT (average grain size of ~143 nm) and reduced dislocation density
  - Suspected reasons for increased ductility
  - Better microstructural stability compared to that found in thermal aging studies

Dislocation Loops in Neutron Irradiated KD –2 dpa

- Quantification of dislocation loops
  - No distinguishable trend in density after irradiation at 300 °C
  - Reduced density of loops with decreasing grain size in the 500 °C condition
  - Primary contributor to the embrittlement of the material

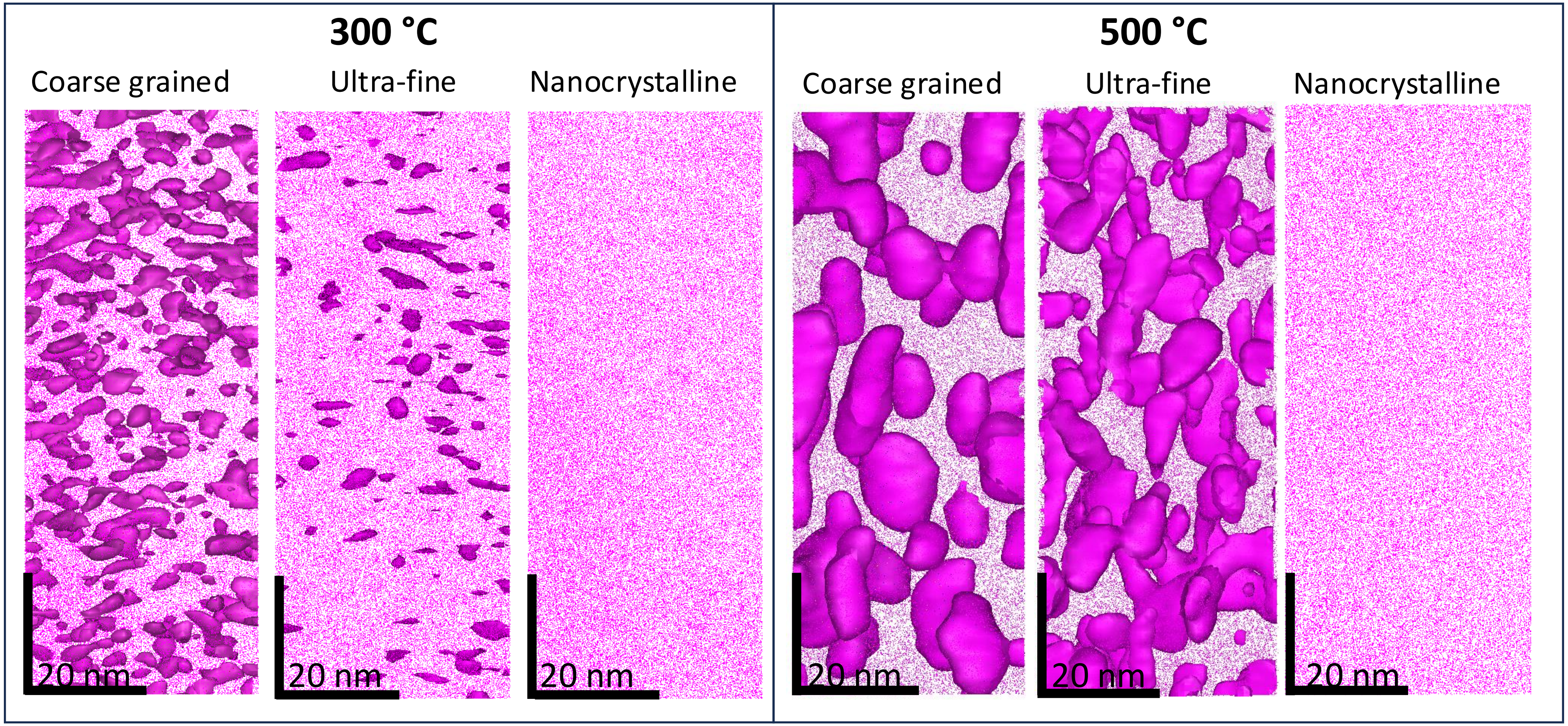




APT of Neutron Irradiated CG, UFG, and NC KD –2 dpa

30 at.% Cr Isosurfaces from KD samples irradiated to 2 DPA

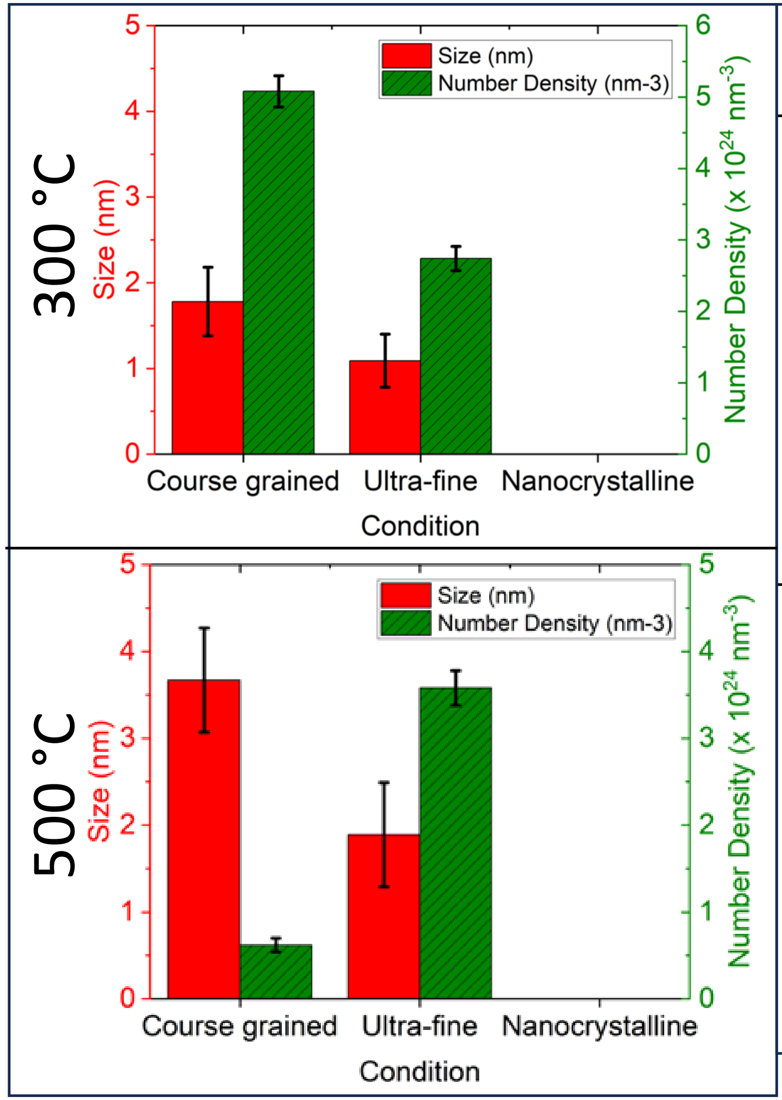
- α’-Cr precipitates are common in FeCrAl alloys
- Decreased grain sizes reduce irradiation assisted precipitation

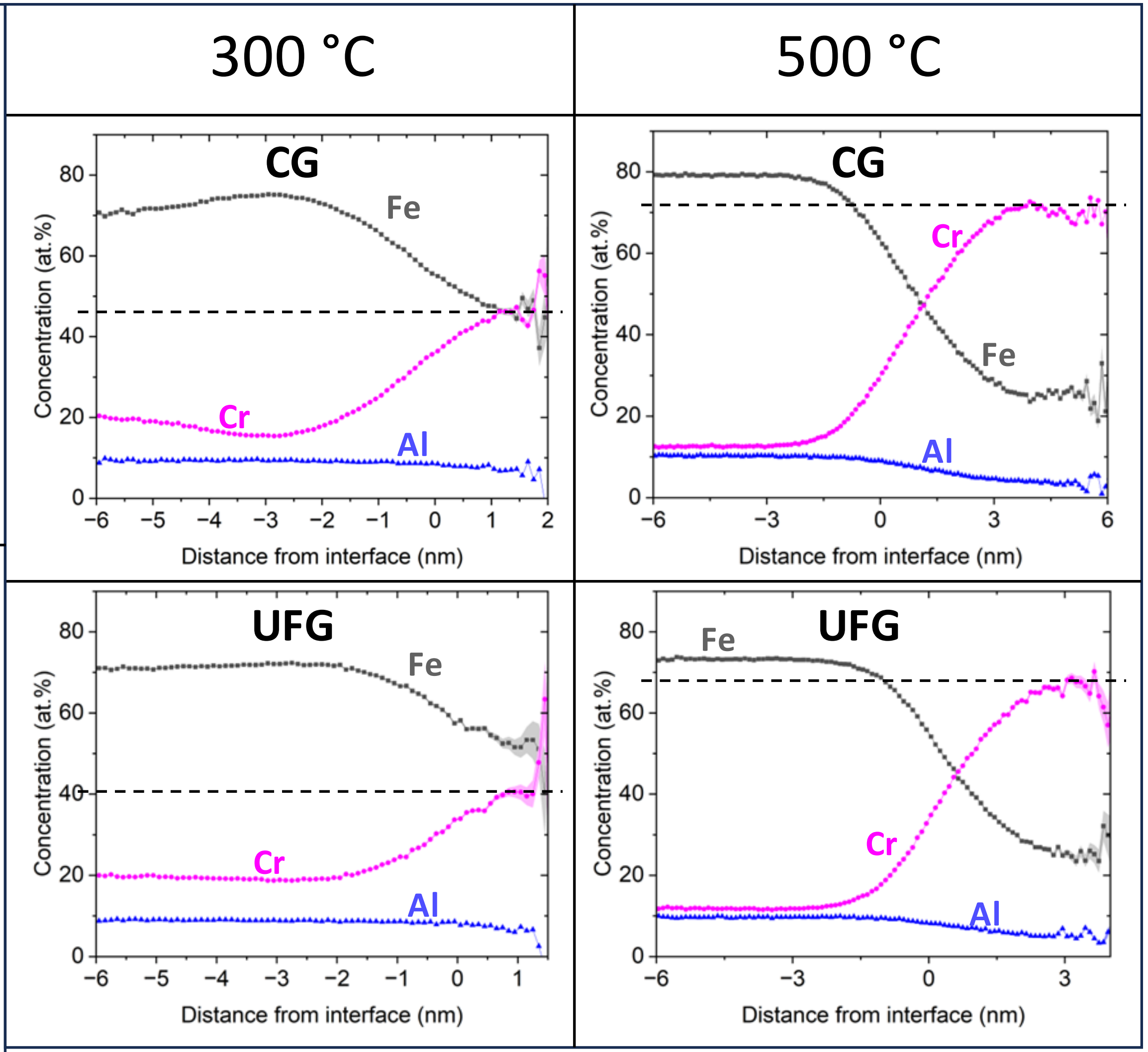


- No α’ precipitation observed in NC KD after irradiation
- Cr-rich clusters after irradiation at 300 °C are not believed to be α’ precipitates

APT of Neutron Irradiated CG, UFG, and NC KD –2 dpa

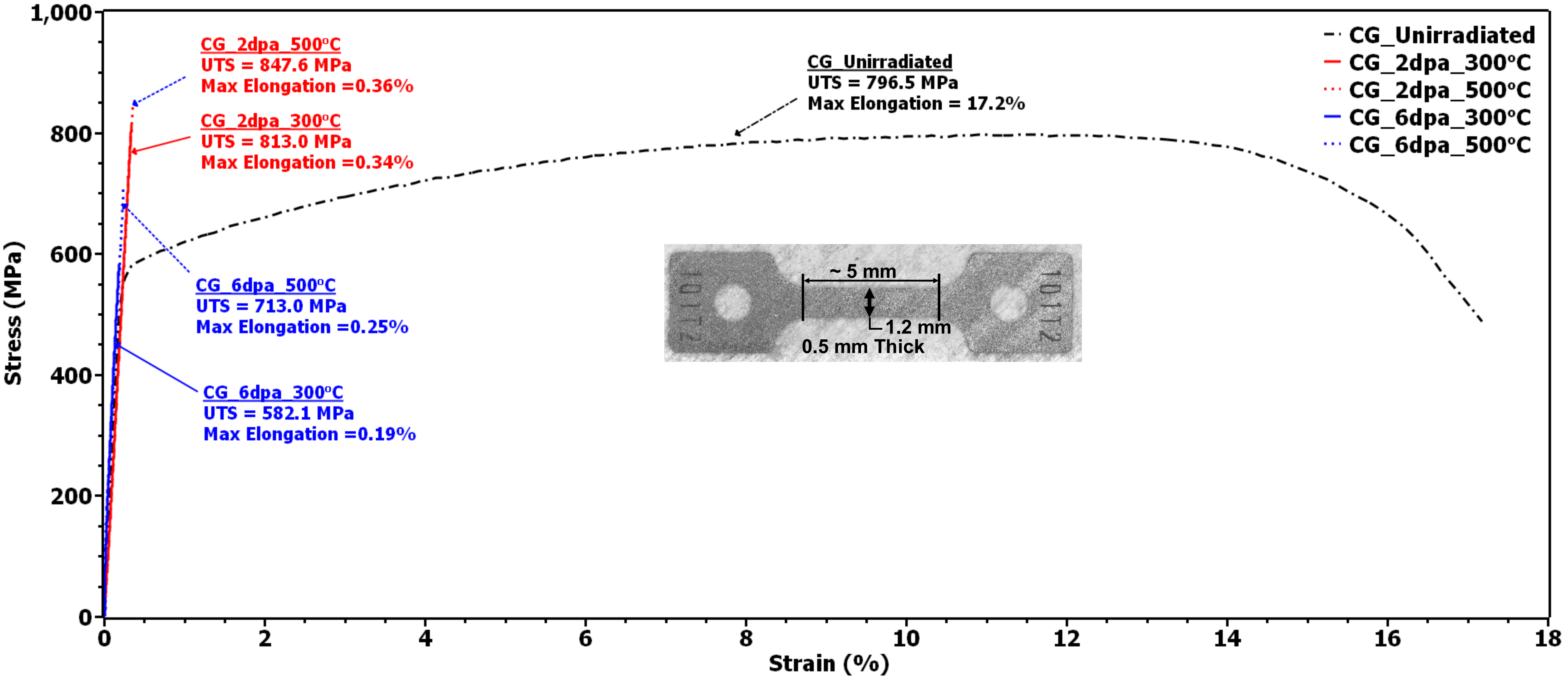
- Generally, as grain size decreases, size and number density of precipitates / clusters decrease
- Al partitions away from α’ which is seen in 500 °C condition





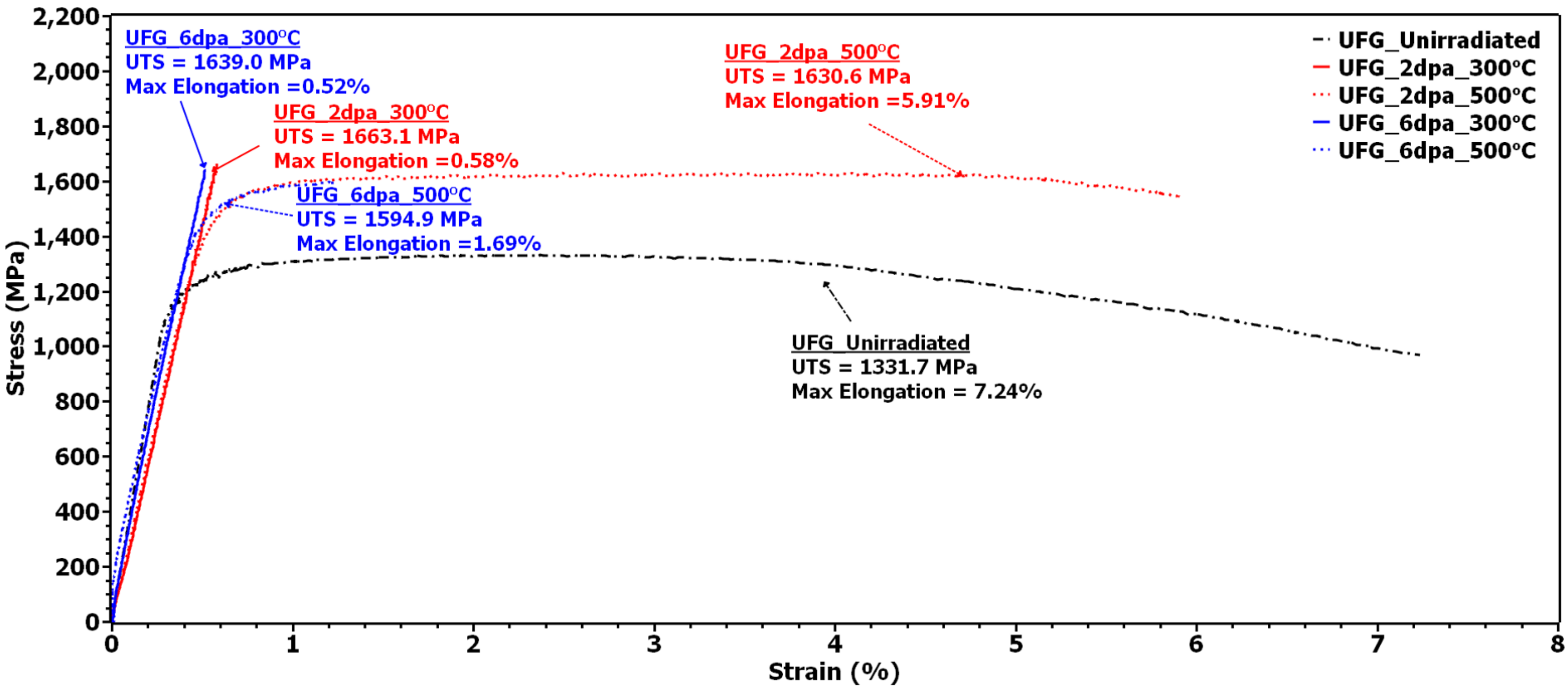
- Higher Cr concentration in precipitates after irradiation at 500 °C
- Slow kinetics limit precipitation at 300 °C → only clusters of Cr atoms

Tensile Behavior of Unirradiated and Irradiated CG KD (2 dpa and 6 dpa)



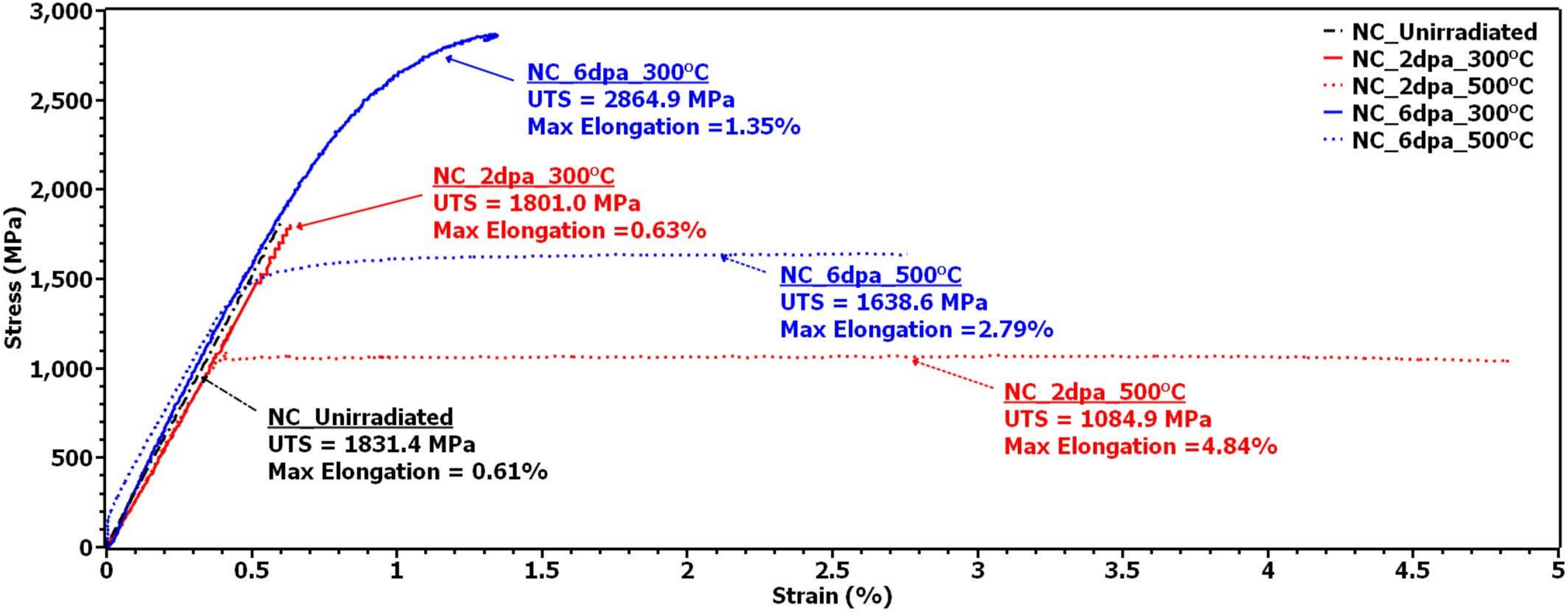
- CG KD exhibits irradiation embrittlement, regardless the irradiation temperature and dose.
- The strength and strain to failure are notably lower after irradiation to 6 dpa compared to 2 dpa.

Tensile Behavior of Unirradiated and Irradiated UFG KD (2 dpa and 6 dpa)



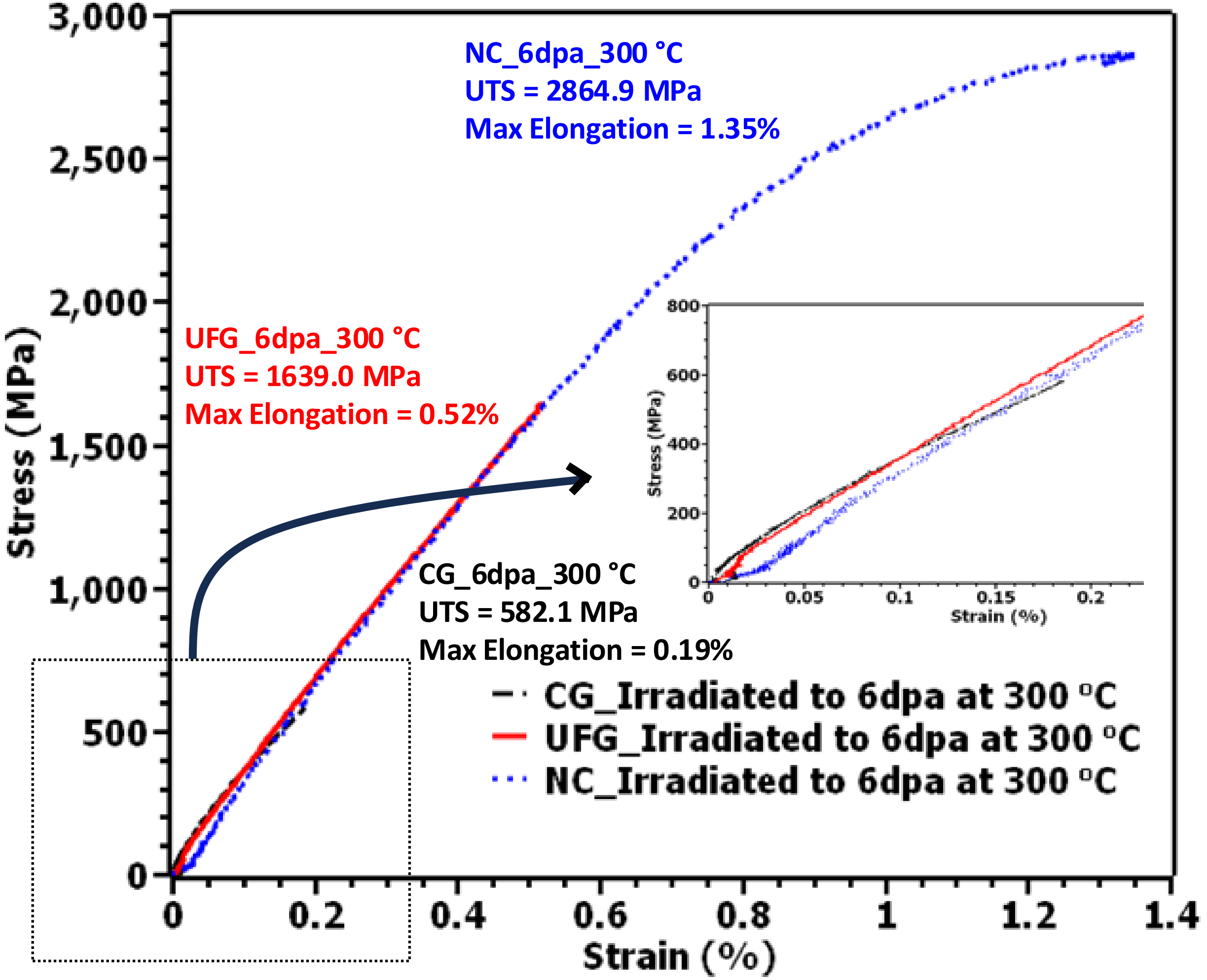
- UFG KD shows irradiation embrittlement at 300 °C but retains ductility at 500 °C.
- Increase in irradiation dose results in significant reduction in ductility at 500 °C.

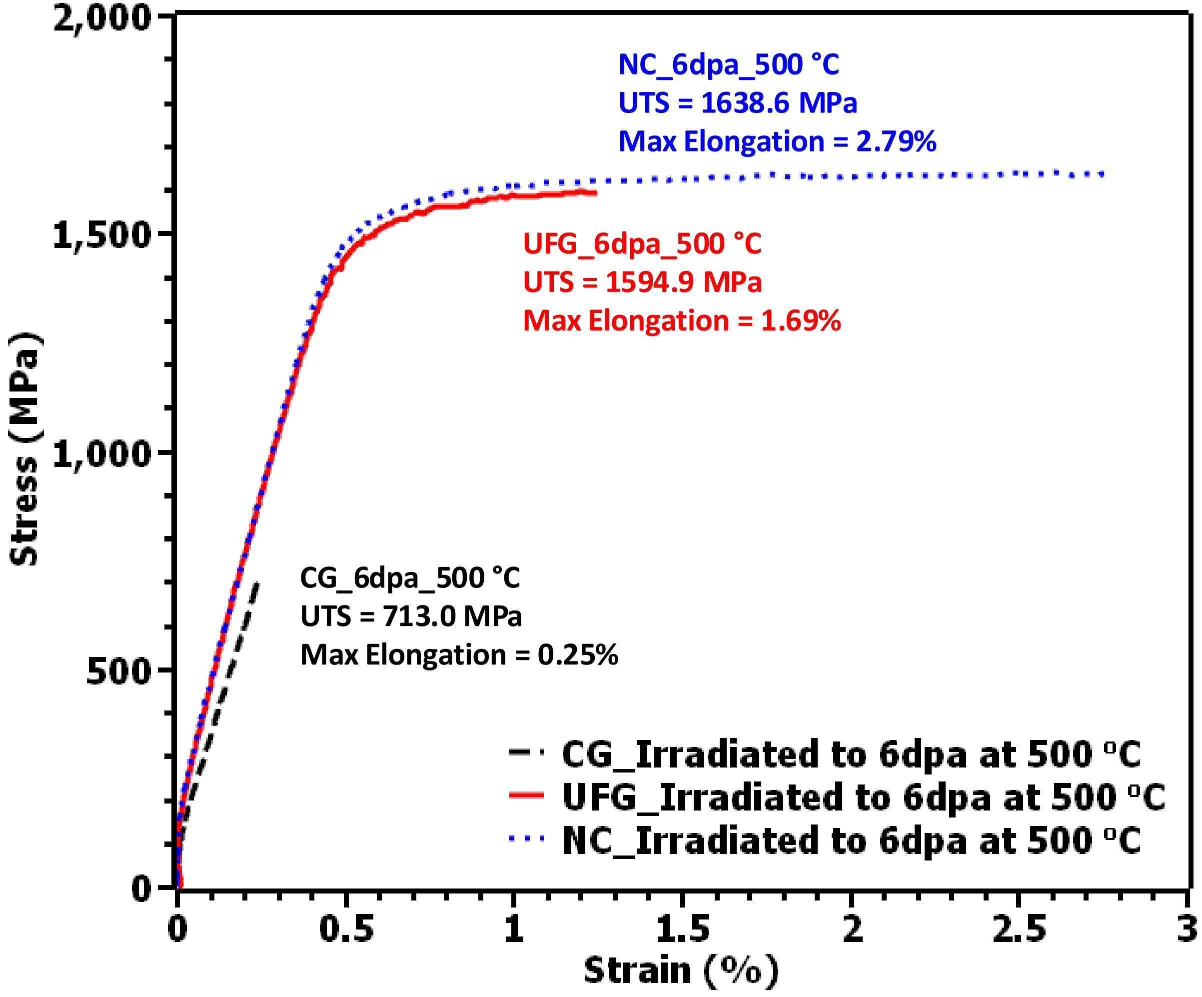
Tensile Behavior of Unirradiated and Irradiated NC KD (2 dpa and 6 dpa)



- NC KD exhibits enhanced ductility after all irradiation conditions.
- NC KD shows increased strength and ductility after 6 dpa at 300 °C.
- At 500 °C, irradiation to 2 dpa reduces strength but significantly enhances ductility; after 6 dpa, ductility decreases while strength increases by ~50%.

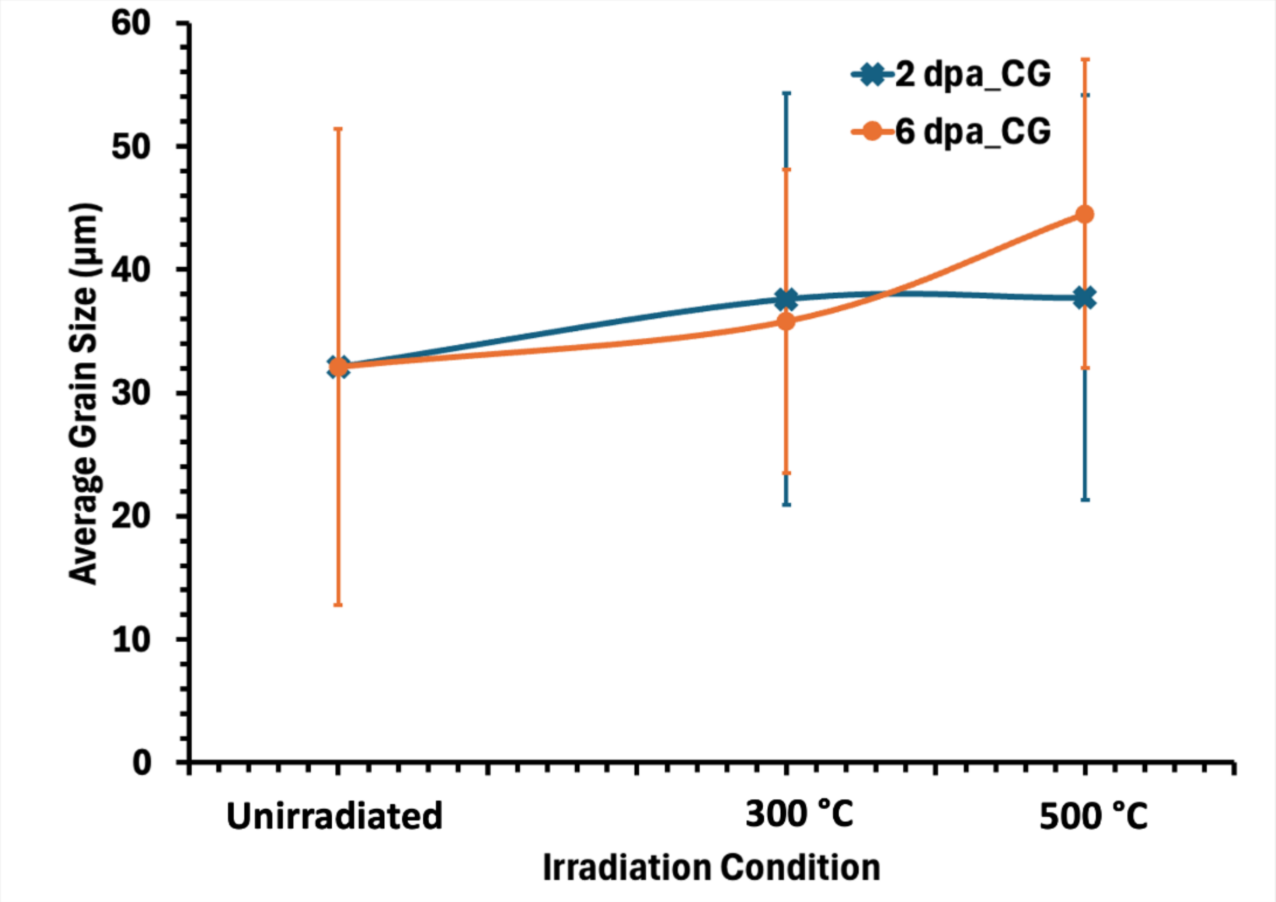
Tensile Behavior of 6 dpa Irradiated KD with Different Grain Sizes

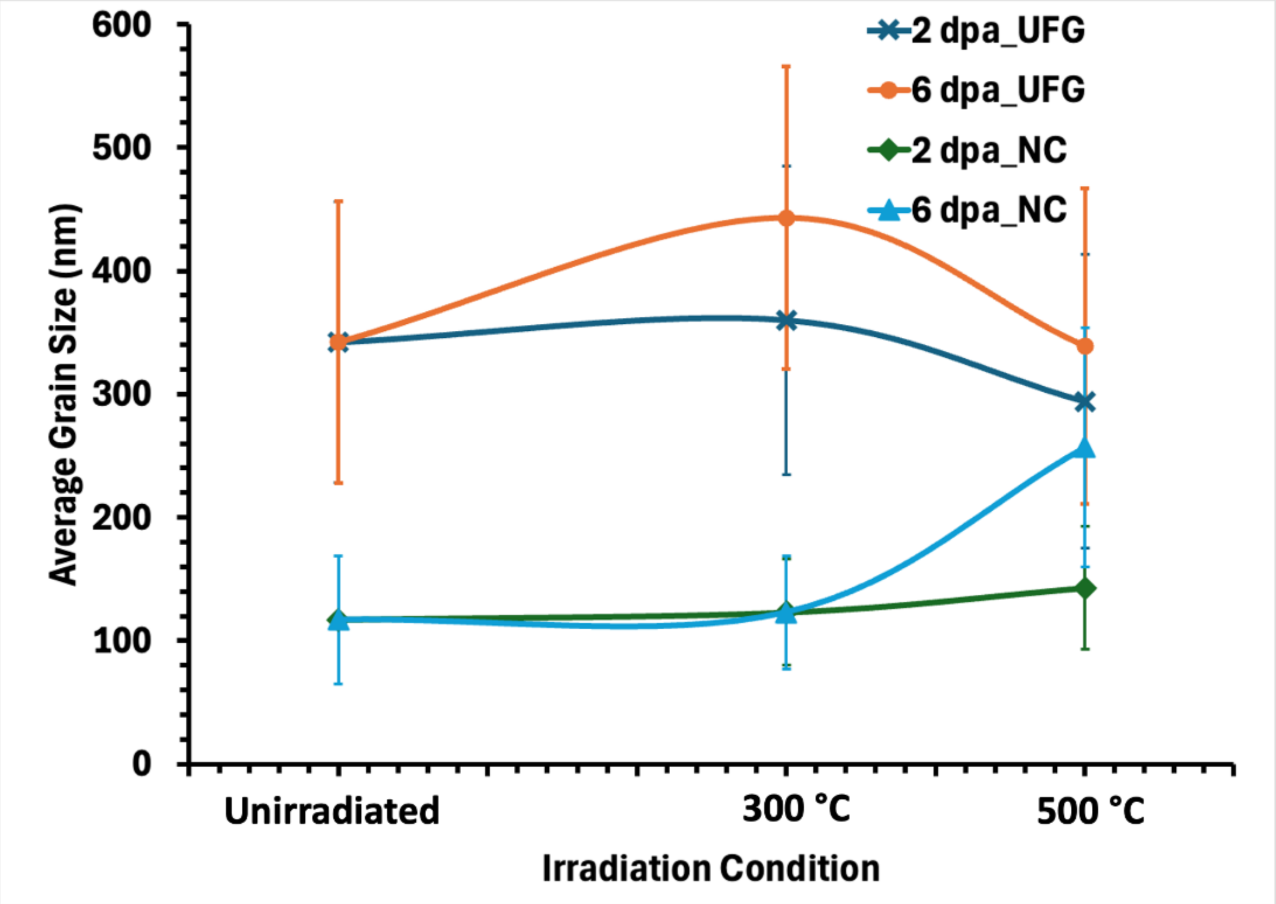




- After irradiation to 6dpa, NC KD is strongest and most ductile, followed by UFG KD; CG KD worst
- NC KD and UFG KD much stronger and more ductile than CG KD, although they are less ductile before irradiation.
- Reduction in grain size improves resistance to irradiation embrittlement.

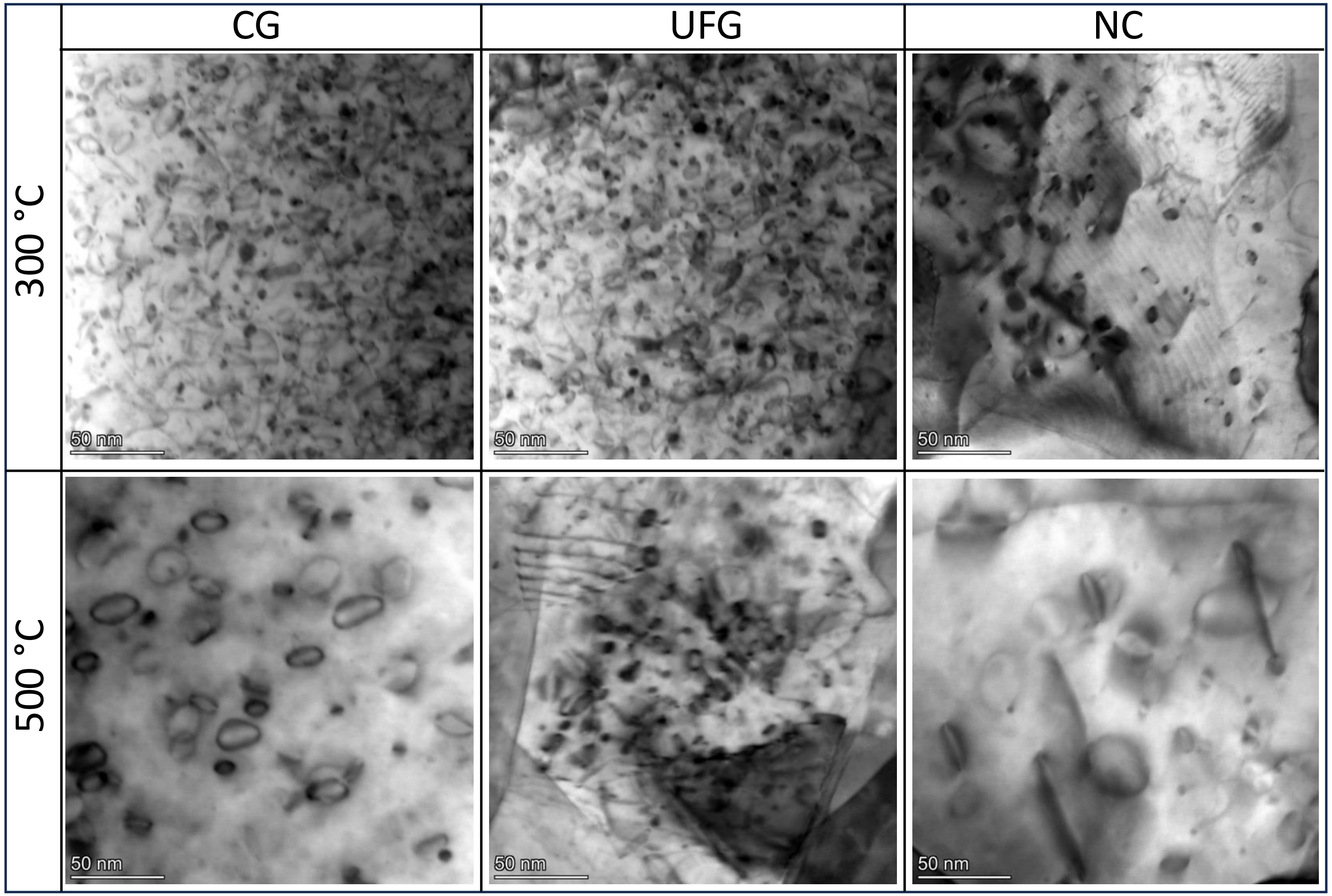
Quantification of Grain Size after Neutron Irradiation of KD to 2 and 6 dpa

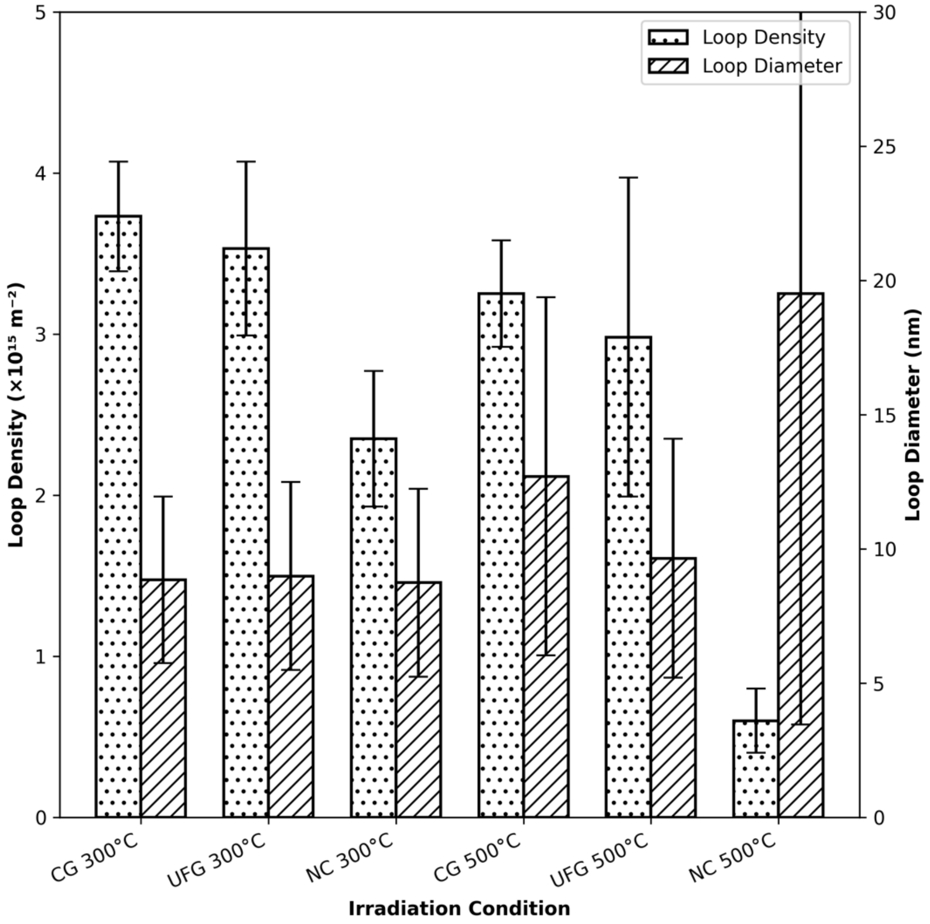




- The UFG KD exhibits a slight increase in grain size after irradiation to 6 dpa compared with 2 dpa.
- The grain size of the NC KD increases by ~80% after irradiation to 6 dpa at 500 °C compared with 2 dpa.
- Overall, there is limited grain growth in UFG and NC KD after elevated temperature irradiation.

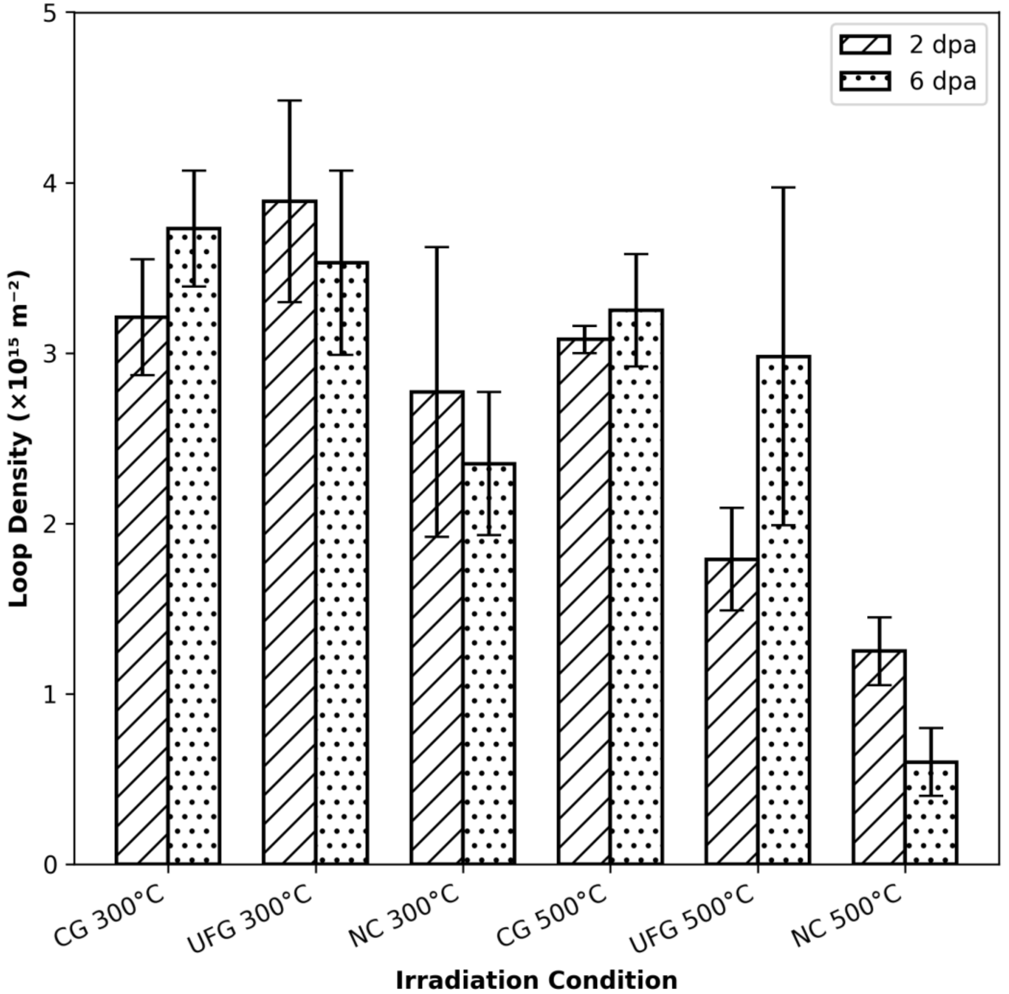
TEM of KD Neutron Irradiated to 6 dpa

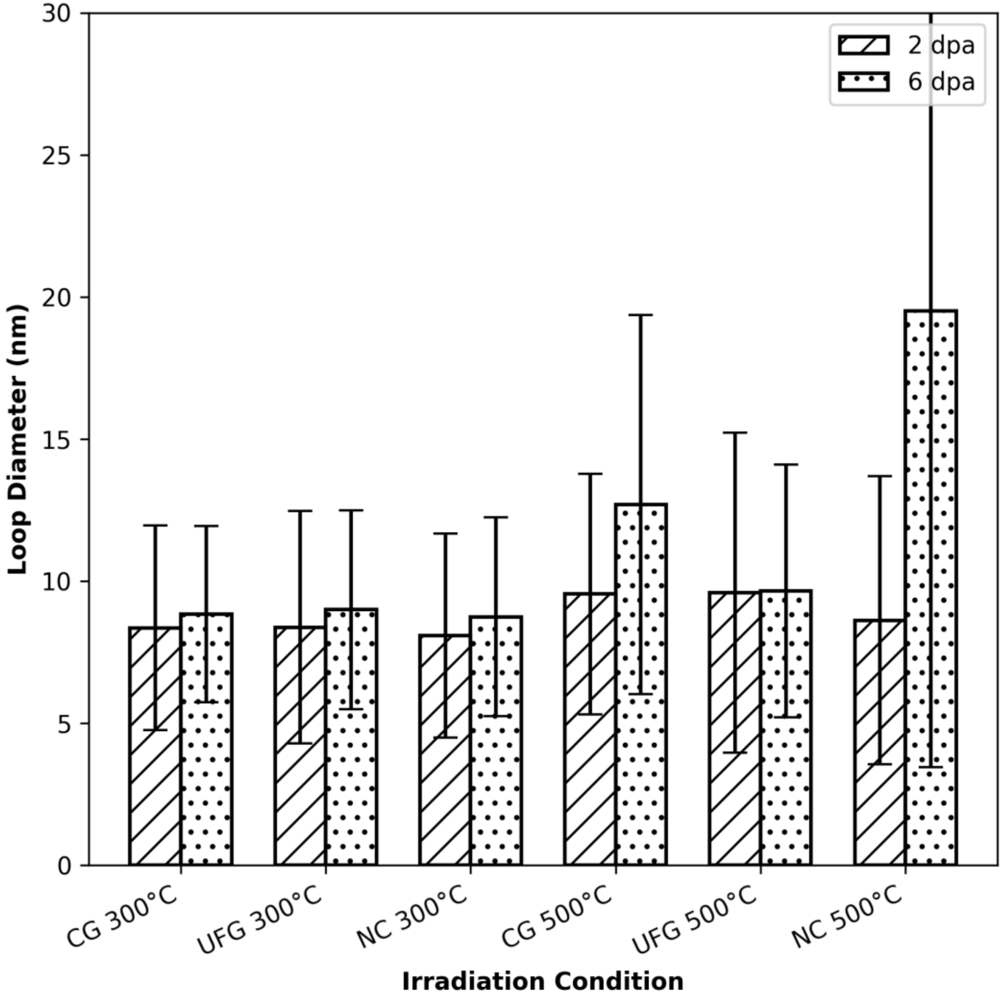




- As grain size decreases, the dislocation loop density is reduced.
- As irradiation temperature increases, loops are larger with lower number density.
- The loop diameter shows significant variation for samples irradiated at 500 °C.

Comparison of Irradiation-induced Dislocation Loops after 2 and 6 dpa

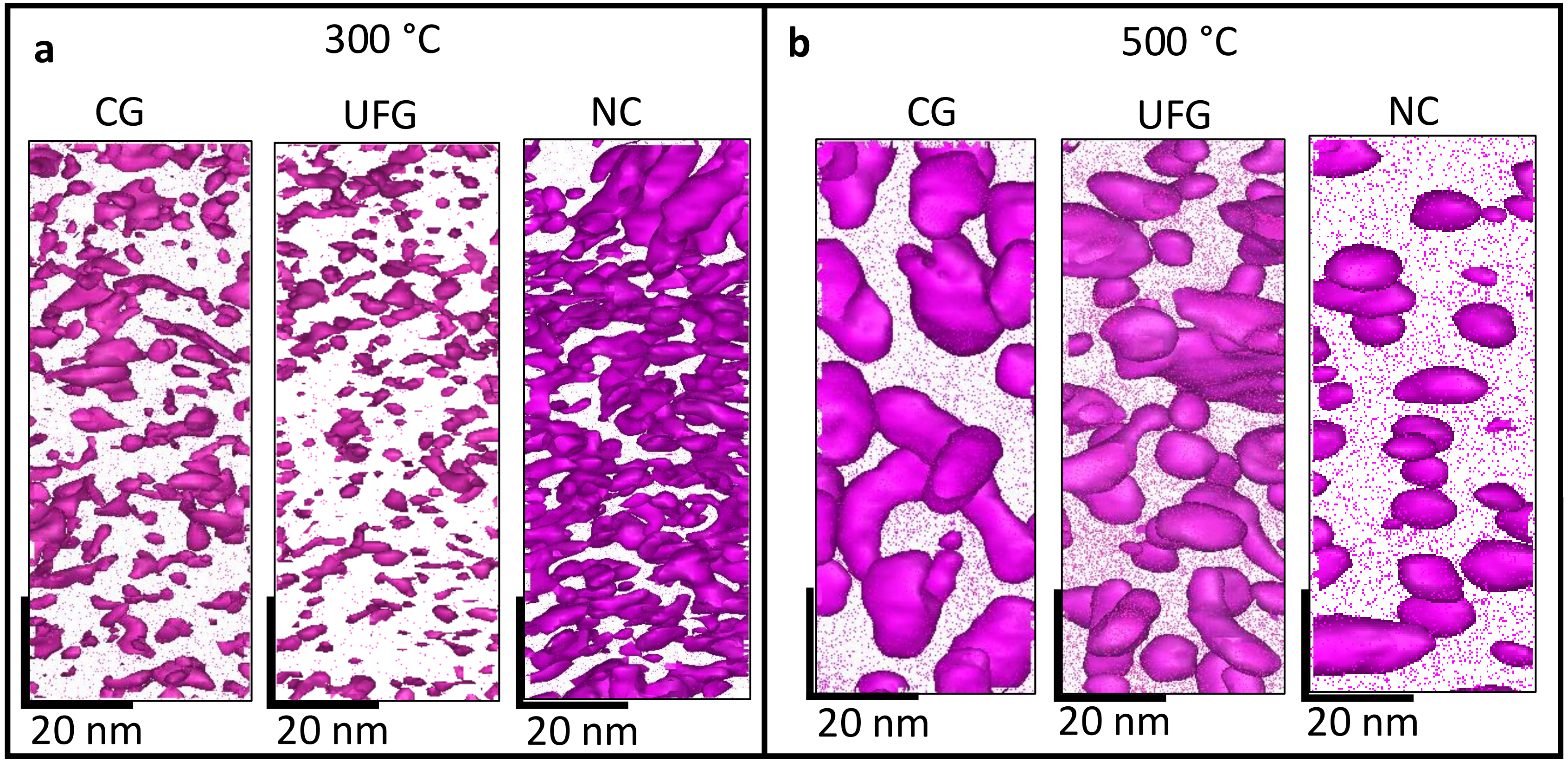


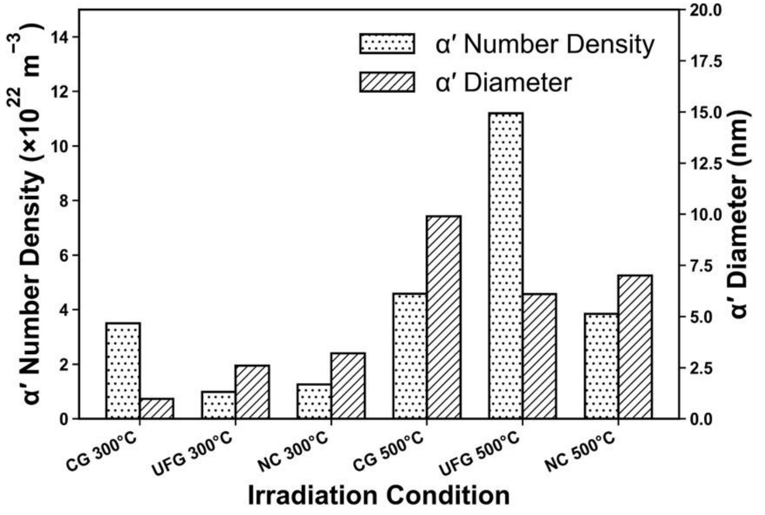


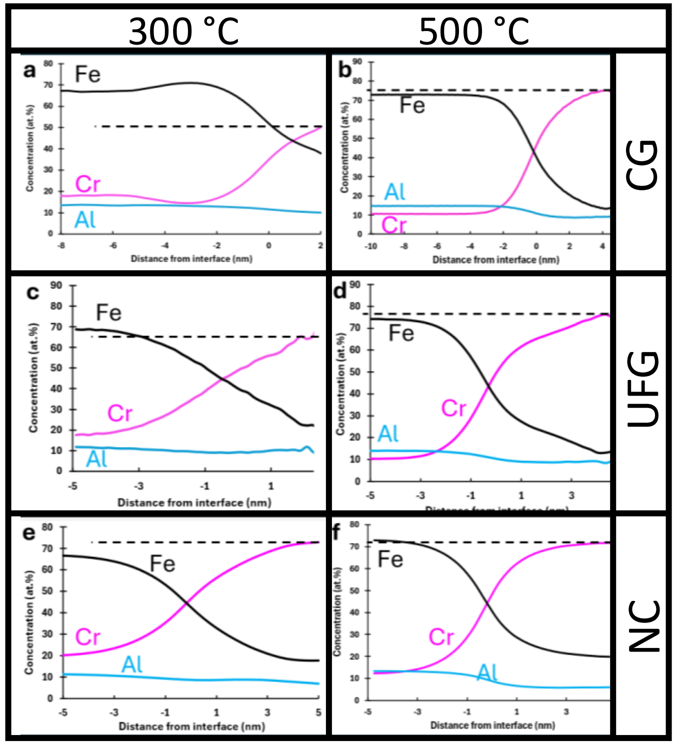
- For the NC KD, loop density reduces after 6 dpa irradiation at both 300 and 500 °C.
- For CG KD, loop density increases at 6 dpa but for UFG KD it depends on the temperature.
- The loop diameter increases with an increase in the irradiation dose from 2 dpa to 6 dpa.

APT of Neutron Irradiated KD after 6 dpa

30% Cr isoconcentration surface







- More α′ precipitation at 6 dpa than at 2 dpa.
- α′ precipitation even in NC KD at 6 dpa.
- More α′ precipitation at 500 °C than at 300 °C.
- The NC KD at 6 dpa still shows lower α′ density than CG and UFG.

Summary and Conclusions

- **Microstructure and mechanical property of UFG/NC steels**
  - **Grain size: ECAP samples ~400 nm (UFG); HPT sample ~100 nm (NC)**
  - **Strength: NC>UFG>CG; Ductility: NC<UFG<CG.**
- **Thermal stability of UFG/NC steels**
  - **Thermally stable up to 600 °C (HPT) or 650 °C (ECAP) for austenitic steels, 500 °C (HPT) or 550 °C (ECAP) for ferritic steels**
- **Thermal aging of FeCrAl steel**
  - **Reduction in grain size reduces α' Cr precipitation and decreases aging embrittlement**
- **Irradiation behavior of UFG/NC steels**
  - **Reduced irradiation-induced hardening in UFG steels compared to CG ones**
  - **Nanostructured austenitic steels more resistant to irradiation-induced FCC-BCC transition**
  - **Small grain sizes reduce irradiation-induced segregation and precipitation**
  - **Very limited irradiation induced grain growth in NC steels**
  - **Nanostructured steels more resistant to irradiation embrittlement**
  - **Nanostructuring increases both strength and ductility of irradiated steels**

Enhancing irradiation tolerance of steels via nanostructuring by innovative manufacturing techniques

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Questions?