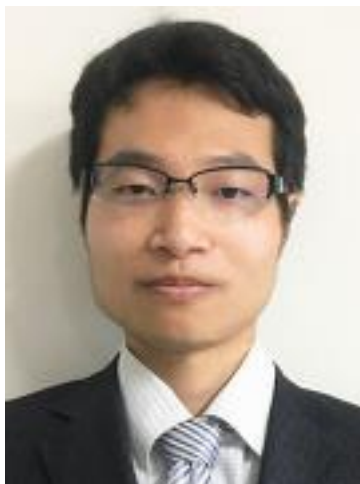


Development of High Entropy Alloys for Fusion Reactor

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April 12th, 2024



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Materials Science and Engineering
University of Tennessee, Knoxville

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Education:

- PhD, Energy Science, Kyoto University, Japan
- MS, Materials Science and Engineering, USTB, China
- BS, Materials Science and Engineering, USTB, China

Research interest:

- Nuclear materials
- Irradiation damage
- Advanced microscopy
- Physical metallurgy

Technology:

- TEM
- EBSD
- Nano-indentation
-

Extended interest:

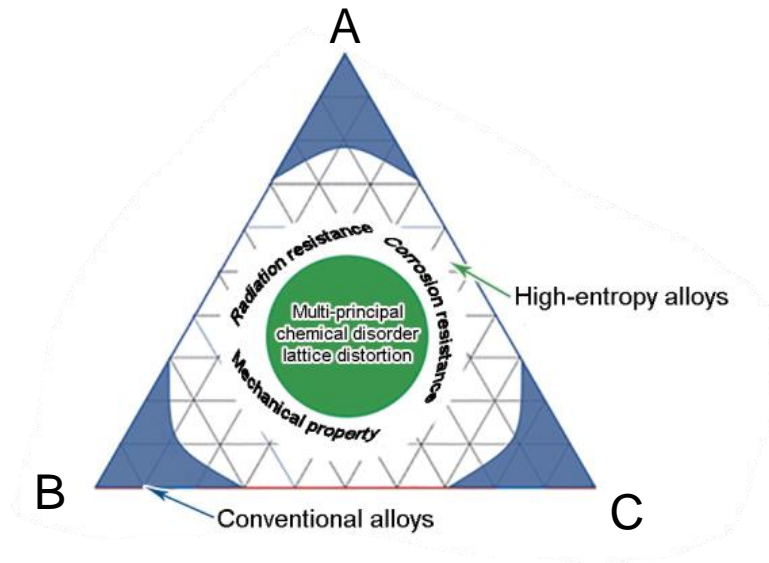
- computational material science
- machine learning

Researched topics:

- Ion-irradiation effects in pure tungsten
- Helium effects in FeCrAl ODS steels
- Ion and neutron irradiation in multiple steels
- Aging embrittlement in FeCrAl ODS steels
- Advanced TEM techniques

What is HEA

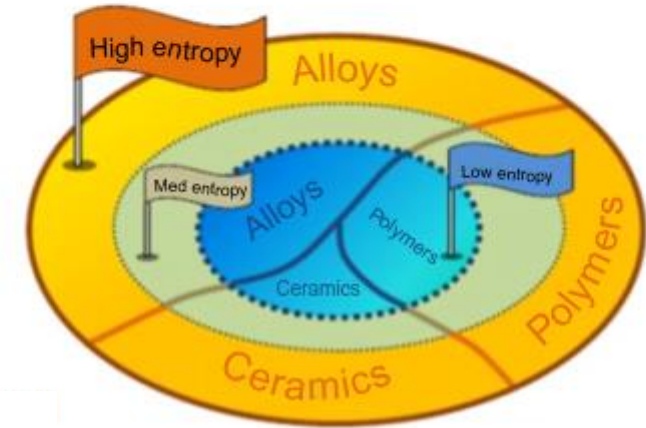
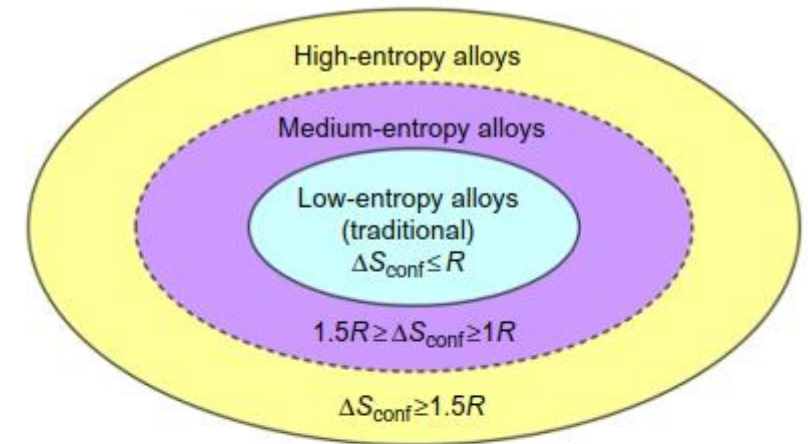
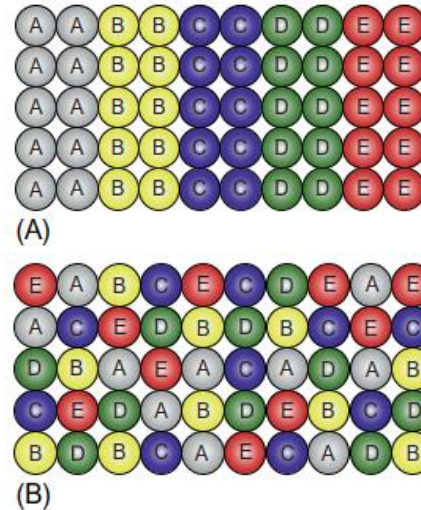
~ It's more like an idea rather than specific alloys



$$\Delta S_{\text{conf}} = -R \sum_{i=1}^n X_i \ln X_i$$

Fraction of each elements

Murty, et al, 2019



Entropy max when equimolar:

$$\Delta S_{\text{conf}} = -k \ln w = -R \left(\frac{1}{n} \ln \frac{1}{n} + \frac{1}{n} \ln \frac{1}{n} + \dots + \frac{1}{n} \ln \frac{1}{n} \right) = -R \ln \frac{1}{n} = R \ln n$$

Table 2.1 Configurational entropies of equiatomic alloys with constituent elements up to 13

N	1	2	3	4	5	6	7	8	9	10	11	12	13
ΔS_{conf}	0	0.69R	1.1R	1.39R	1.61R	1.79R	1.95R	2.08R	2.2R	2.3R	2.4R	2.49R	2.57R

Definition is Not Absolute!

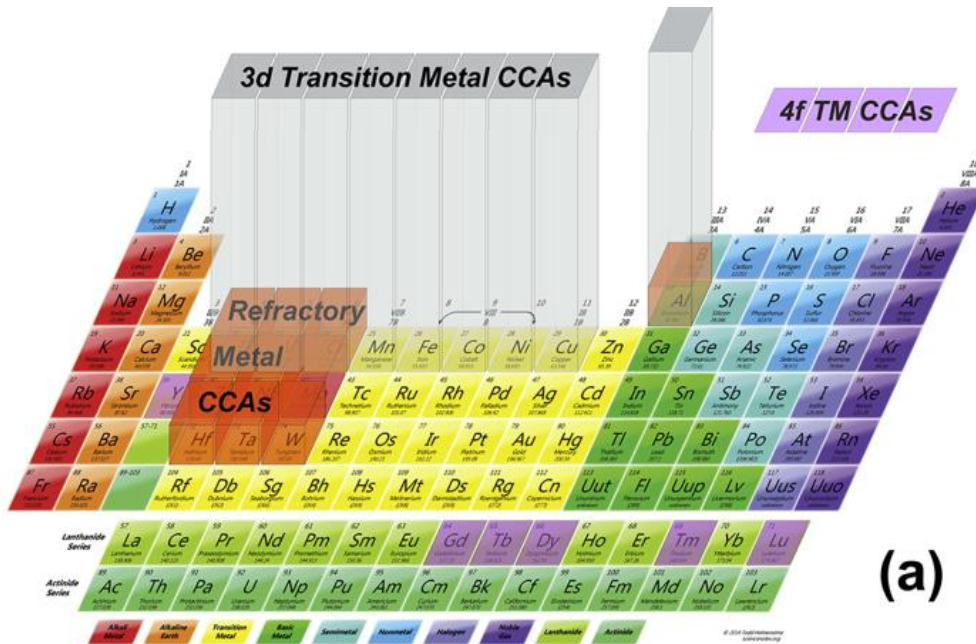
The element selection in alloy designs

Hume-Rothery Rules:

1. Solvent and solute atoms have similar atomic size.
2. Solvent and solute atoms have similar valence and electronegativity, or electron/atom ratio.

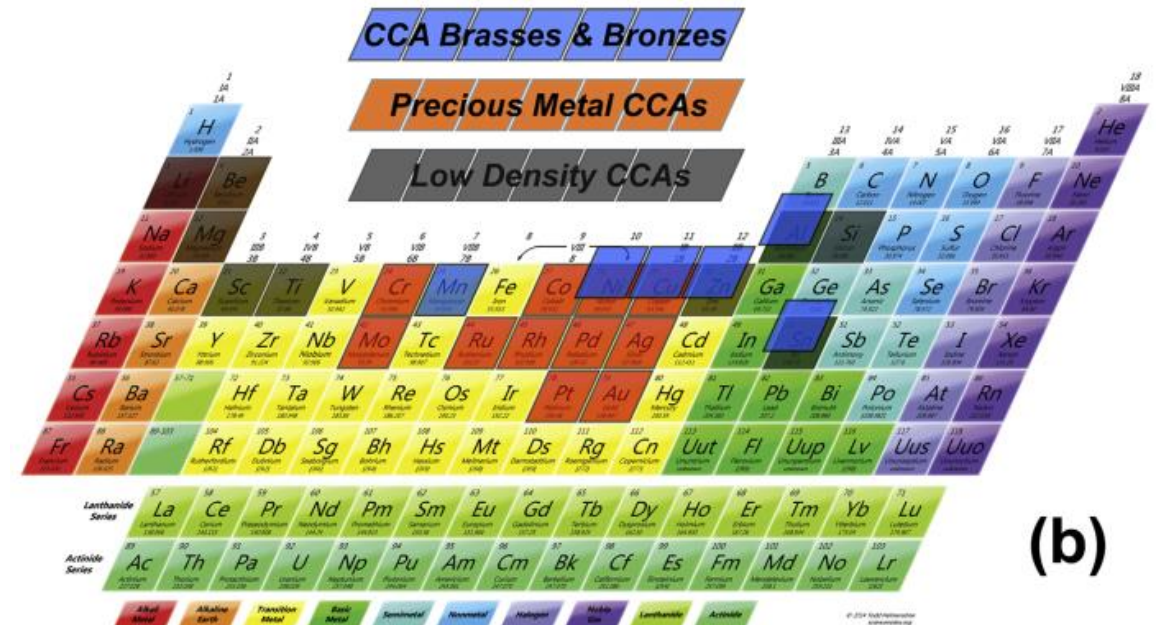
- ✓ Cr Mn Fe Co Ni (Cantor Alloy) c/o Al
- ✓ Ti V Cr – Y Zr Nb – Hf Ta W (RHEA)

- Al – Mn Ni Cu Zn- Sn
- Cr Co Ni Cu – Mo Ru Rh Pd Ag – Pt Au
- Li Be – Mg Al Si – Sc Ti Zn - Sn



(a)

Miracle, et al, 2017



(b)

Four core effects of HEA

1. *High-entropy effect:* ☁ ~ not universal!

Assure simple formation of solid solutions
instead of compounds or segregated phases.

$$\downarrow \Delta G^{\text{mix}} = \Delta H^{\text{mix}} - T \Delta S^{\text{mix}} \uparrow$$

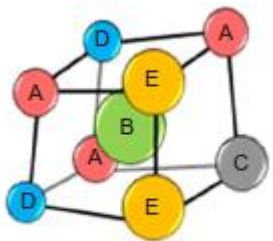
Especially at high temperature

~Key effects in irradiation resistance?

2. *Severe lattice distortion effect:*

☁ ~ not significant!

5-components alloy



BCC
Severe lattice
distortion

- Increased hardness and strength due to solution hardening
- Low electrical conductivity
- Reduced thermal effects on properties, in sensitive to temperature

3. *Sluggish diffusion effect:* ☁ ~ not proved yet!

Slower diffusion and higher activation energy

interstitials are difficult to migrate, so more I-V recombination will occur.

4. *Cocktail effects:*

☁ ~ not unique!




Mixture of atoms

↓
Mixture of properties

Diffusion coefficient




~ Critical in defect evolution



Contents lists available at ScienceDirect

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journal homepage: www.elsevier.com/locate/actamat



By invitation only: overview article

A critical review of high entropy alloys and related concepts

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^b UES, Inc., 4401 Dayton-Xenia Road, Beavercreek, OH, USA

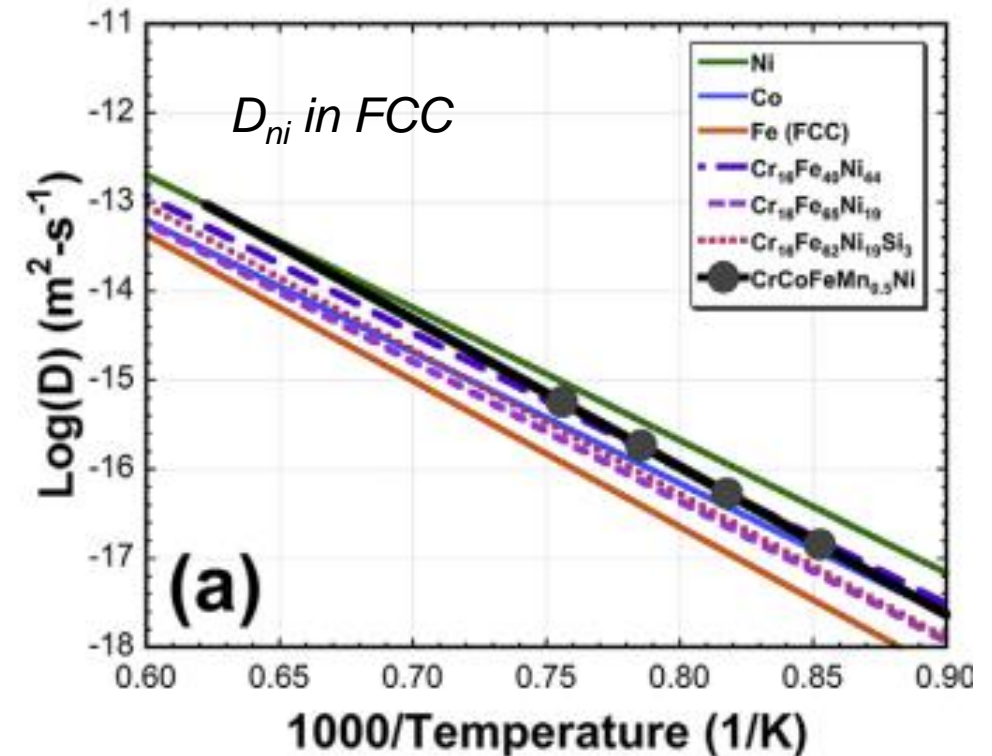
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Multicomponent

ABSTRACT

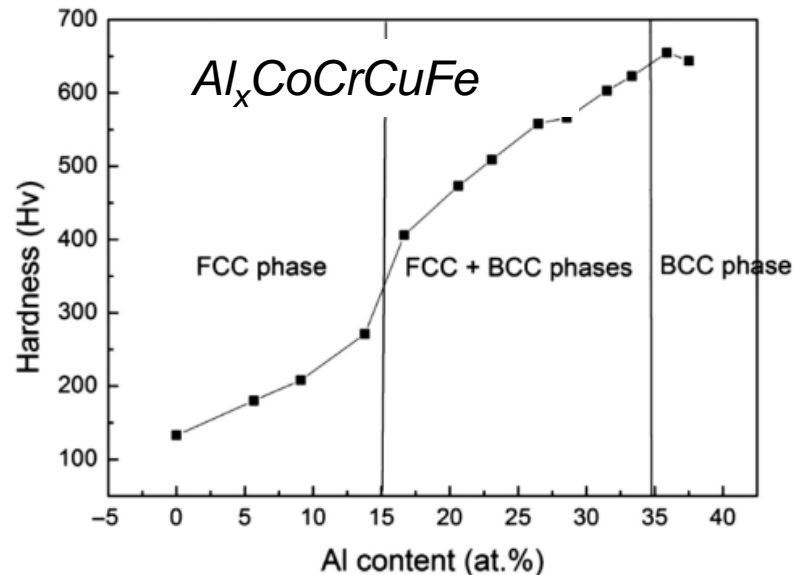
High entropy alloys (HEAs) are barely 12 years old. The field has stimulated new ideas and has inspired the exploration of the vast composition space offered by multi-principal element alloys (MPEAs). Here we present a critical review of this field, with the intent of summarizing key findings, uncovering major trends and providing guidance for future efforts. Major themes in this assessment include definition of terms; thermodynamic analysis of complex, concentrated alloys (CCAs); taxonomy of current alloy families; microstructures; mechanical properties; potential applications; and future efforts. Based on detailed analyses, the following major results emerge. Although classical thermodynamic concepts are unchanged, trends in MPEAs can be different than in simpler alloys. Common thermodynamic perceptions can be misleading and new trends are described. From a strong focus on 3d transition metal alloys, there are now seven distinct CCA families. A new theme of designing alloy families by selecting elements to achieve a specific, intended purpose is starting to emerge. A comprehensive microstructural assessment is performed using three datasets: experimental data drawn from 408 different alloys and two computational datasets generated using the CALculated PHase Diagram (CALPHAD) method. Each dataset emphasizes different elements and shows different microstructural trends. Trends in these three datasets are all predicted by a 'structure in – structure out' (SISO) analysis developed here that uses the weighted fractions of the constituent element crystal structures in each dataset. A total of 13 distinct multi-principal element single-phase fields are found in this microstructural assessment. Relationships between composition, microstructure and properties are established for 3d transition metal MPEAs, including the roles of Al, Cr and Cu. Critical evaluation shows that commercial austenitic stainless steels and nickel alloys with 3 or more principal elements are MPEAs, as well as some established functional materials. Mechanical properties of 3d transition metal CCAs are equivalent to commercial austenitic stainless steels and nickel alloys, while some refractory metal CCAs show potential to extend the service strength and/or temperature of nickel superalloys. Detailed analyses of microstructures and properties allow two major HEA hypotheses to be resolved. Although the 'entropy effect' is not supported by the present data, it has



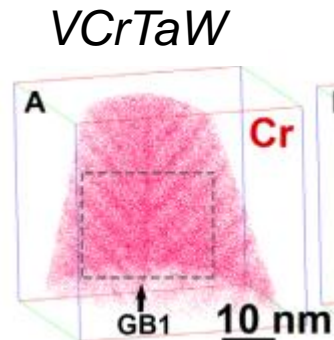
Plus, measured data from Tsai, et al, AM, 2013

Phases and Structure

1. Single FCC
 - a. Ordered FCC ~ e.g. $L1_2$
 - b. Disordered FCC
2. Single BCC
 - a. Ordered BCC ~ e.g. B_2 and DO_3
 - b. Disordered BCC ~ e.g. A_2
3. Multiphase
 - a. Dual-phase ~ e.g. FCC+B2
 - b. Others
4. Metastable
 - a. ~ e.g. FCC \rightarrow HCP
5. Others

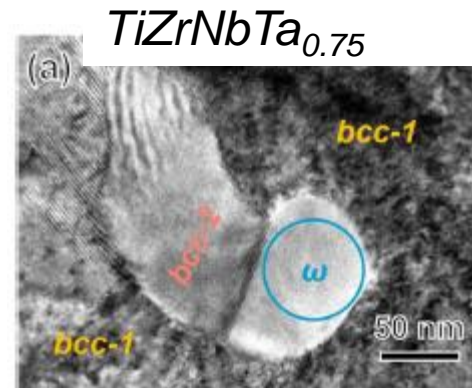


~ could be much more complicated

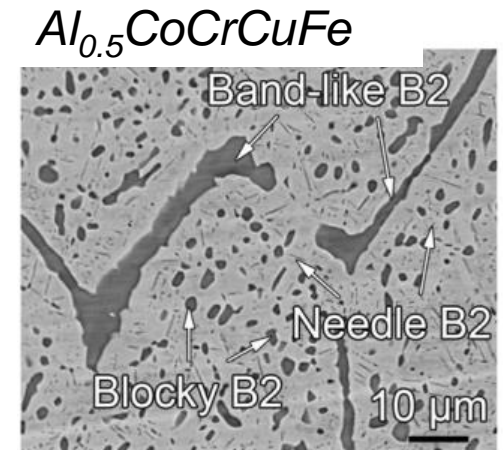


El-Atwani, et al, 2019

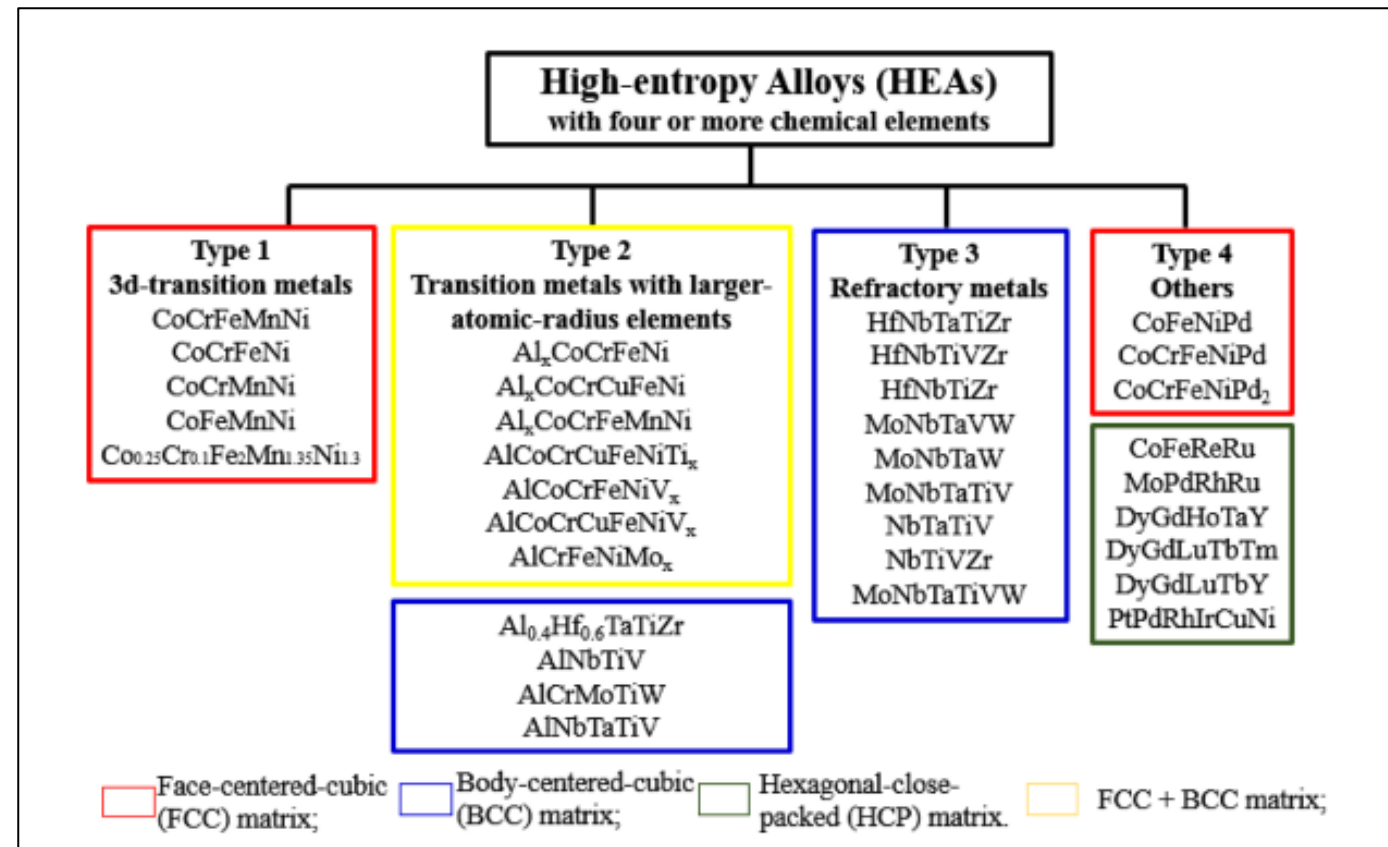
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Zhao, et al, 2022



Diao, et al, 2017



HEA pros and cons

➤ In academy:

Mechanically oriented HEAs

Category

- Nanoprecipitates HEAs
- TWIP HEAs
- TRIP HEAs
- Eutectic HEAs
- Dual-phase HEAs
- Ultrafine grained HEAs
- Lightweight HEAs
- Metallic-glass HEAs
- ...

Mechanism

- TWIP/ TRIP
- Multi-component precipitation
- Heterostructure
- Ordered interstitial complexes
- Dislocation shear mode

High-entropy materials

Advantage

- Overcoming the trade-off of strength-toughness
- Low temperature ductility
- Thermal stability
- Corrosion resistance
- Irradiation resistance
- Self-sharpening

Mechanism analysis

Functionally oriented HEAs

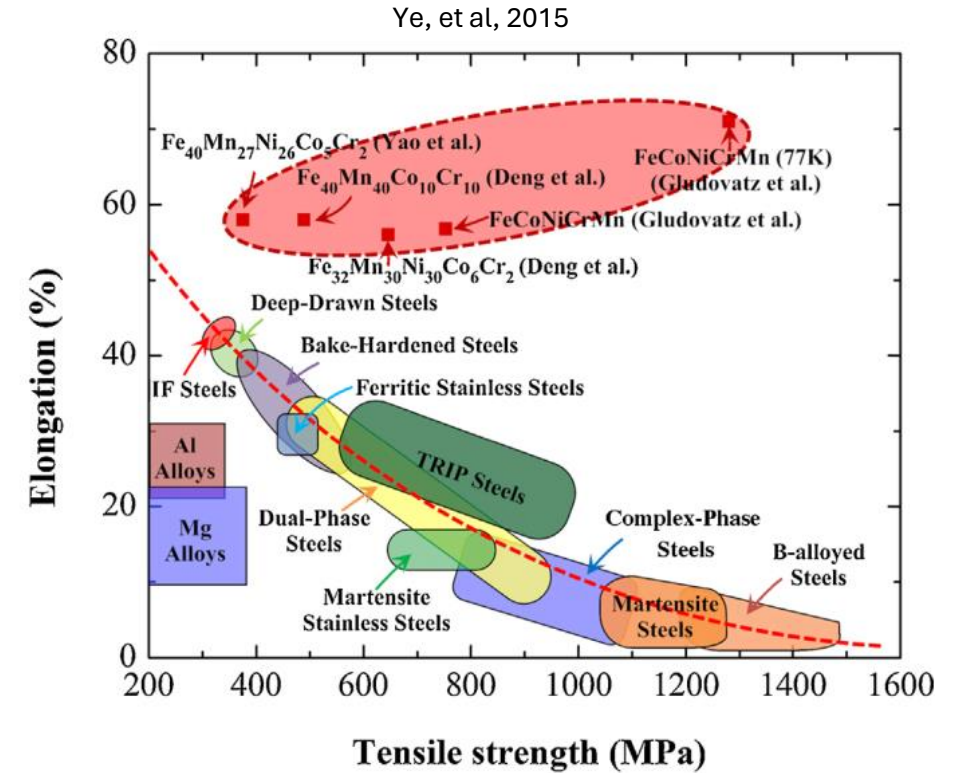
Category

- Anti-irradiation HEAs
- Soft-magnetic HEAs
- Corrosion-resistance HEAs
- Thermoelectric HEAs
- Superconducting HEAs
- High-entropy ceramics
- ...

Mechanism

- Anti-irradiation
- Self-healing,
- Short electronic free path
- ...

Abbreviations: Twinning-induced plasticity (TWIP), Transformation-induced plasticity (TRIP).



➤ In industry: None !

- High cost *Difficult in recycling?*
- Manufacturing methods
- Thermal stability
- Data not sufficient
- ...

For nuclear applications



~ Generally, at **low** technique readiness level (**TRL**)

➤ Low cross section HEA~ *fission reactors*

- ~ *TiVZrNb (BCC)*

Co-free HEA • ~ *CrMnFeNi (FCC)*

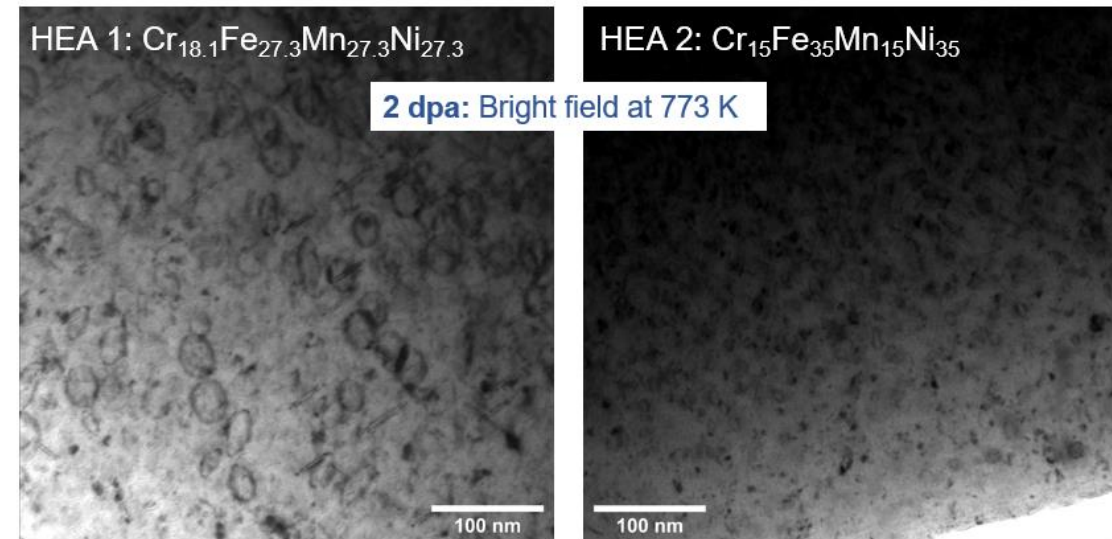
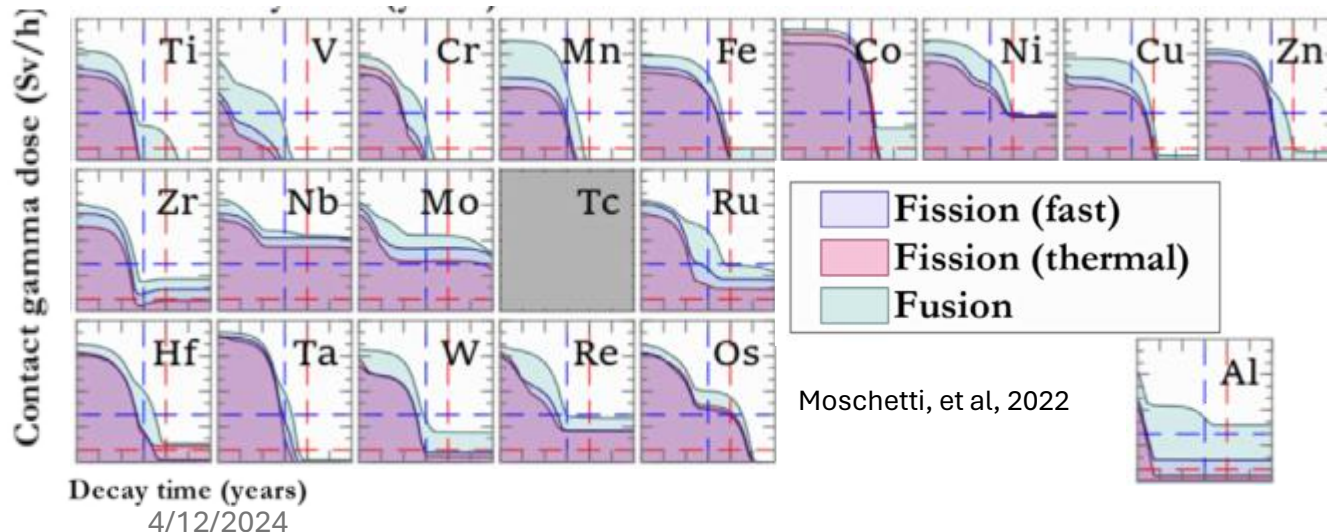
➤ Reduced activation HEA~ *fusion reactors*

- ~ *TiVZrTa (BCC)*
- ~ *VCrTaW (BCC)*

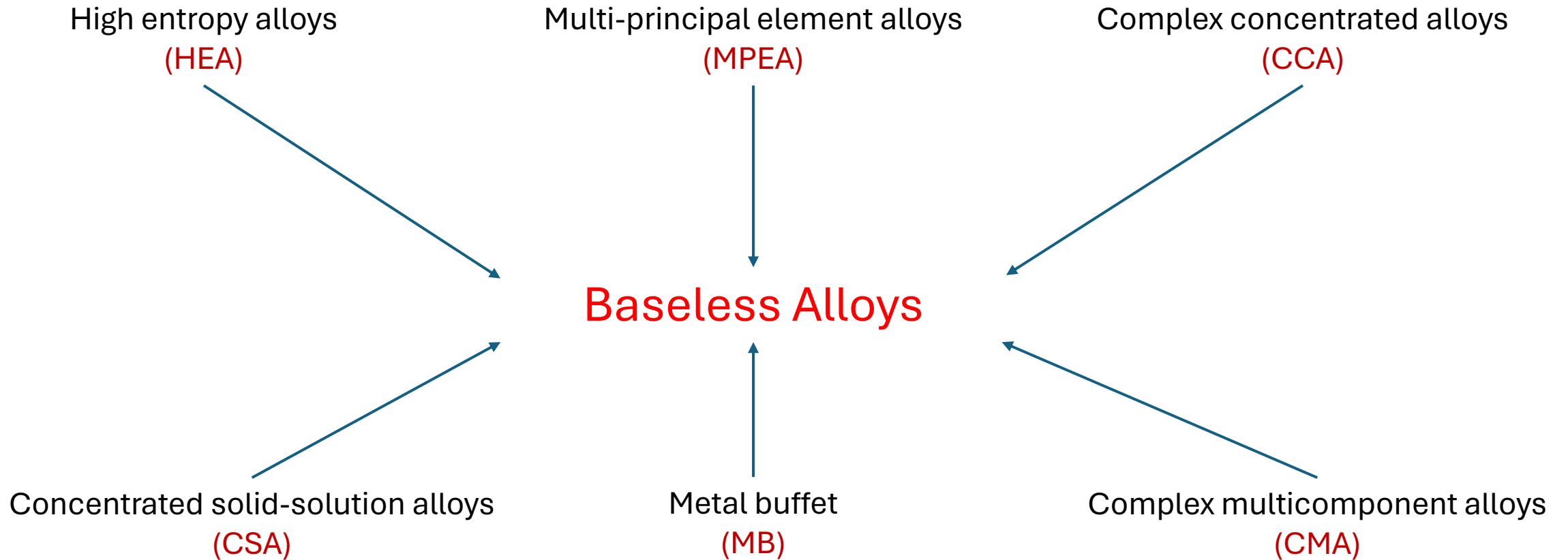
Challenges and Future works:

1. Element designs ~ *machine learning assistance*
2. Fabrication/manufacturing ~ *additive manufacturing*
3. Database ~ *corrosion and irradiation*
4. Mechanism ~ *microstructure analysis*

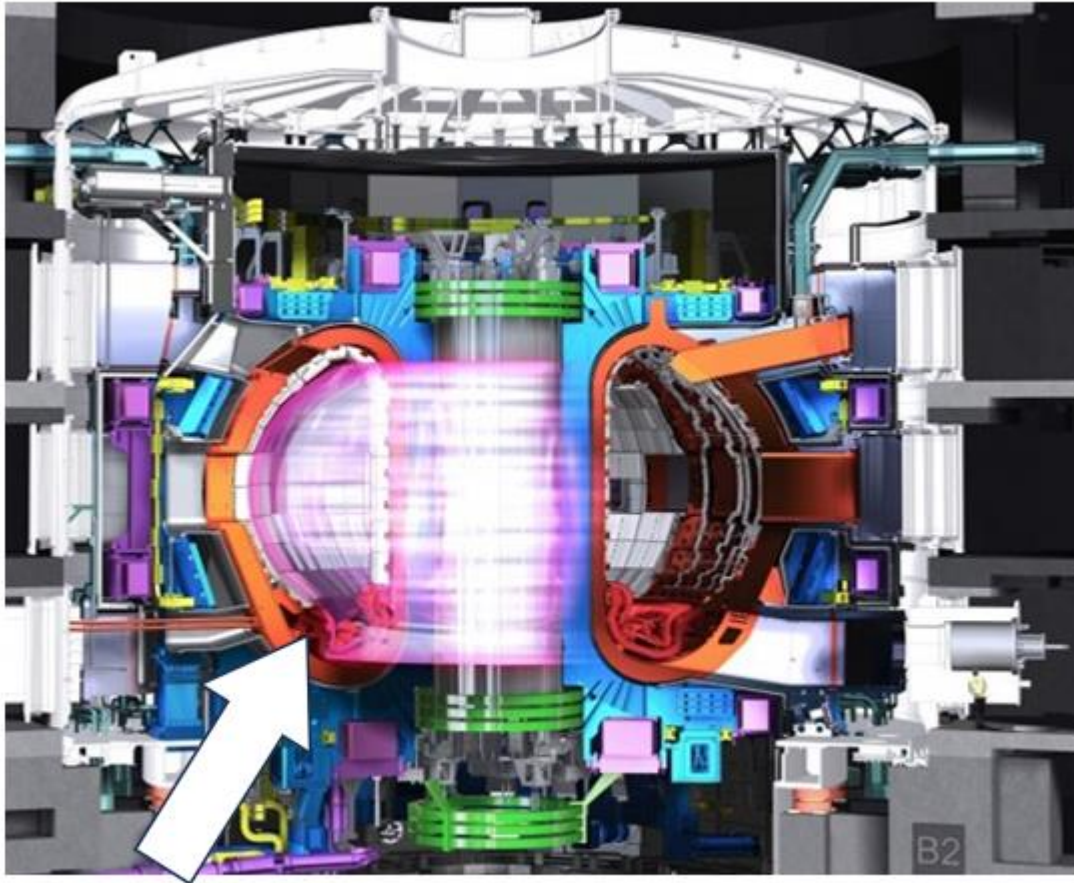
~e.g. Effect on **defect mobility** is **not conclusive**



Additional:



International thermonuclear experimental reactor (ITER)



divertor

<https://www.iter.org/proj/inafewlines>

Prototype panel for First Wall

Be
CuCrZr
ss316L



<https://fusionforenergy.europa.eu/news/european-production-of-iter-first-wall-panels-starts/>

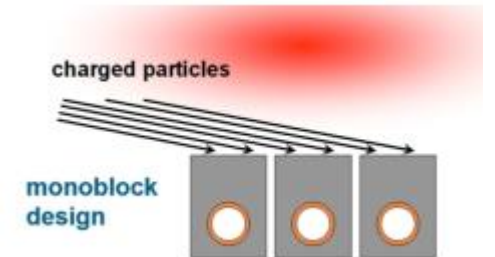
Divertor

Linke, Matter Radiate. Extrem.
2019

W
CuCrZr

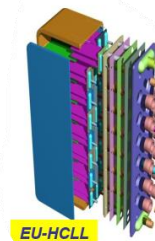


Mono-block

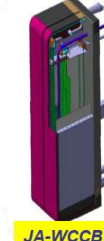


Tritium breeder module

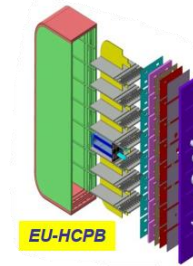
RAFM



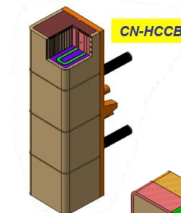
EU-HCLL



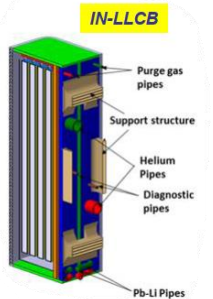
JA-WCCB



EU-HCPB



KO-HCCR




IN-LLCB

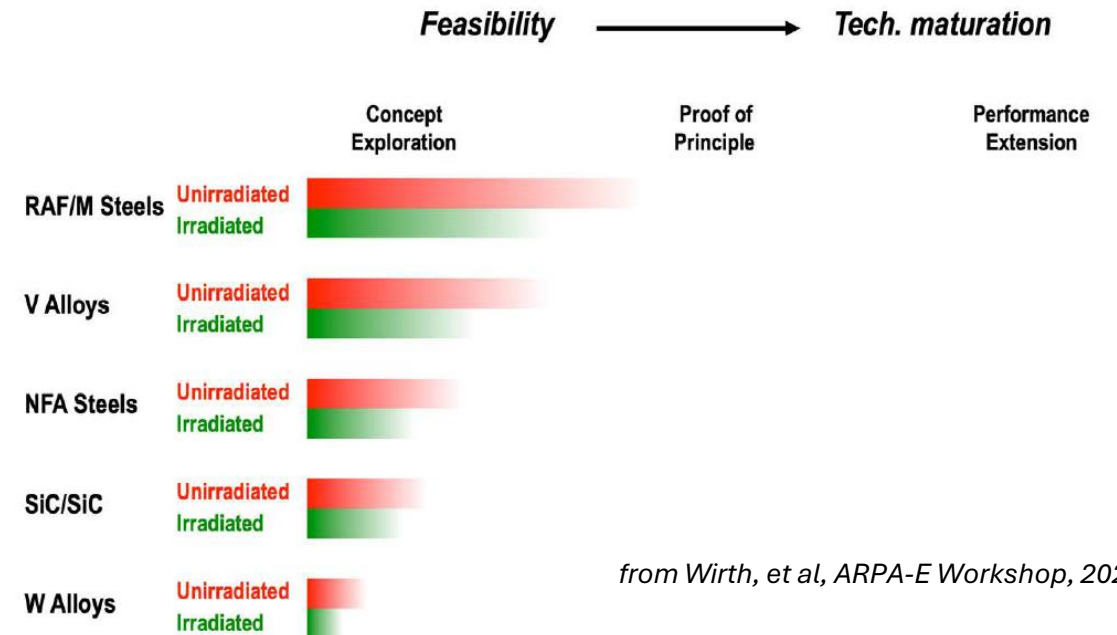
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The demonstration power plant (DEMO)

DEMO and Power Reactor beyond ITER in neutron damage - but heat flux issues are comparable.

	ITER	DEMO	Reactor
Fusion Power	0.5 GW	2.5 – 5 GW	2.5 - 5 GW
Heat flux (first wall) (divertor)	0.1-0.3 MW/m² ~ 10 MW/m²	0.5 MW/m² ~15-20 MW/m²	0.5 MW/m² ~20 MW/m²
Neutron Load (FirstWall)	0.78 MW/m²	< 2 MW/m²	~ 2 MW/m²
Integrated Neutron Load (First Wall)	0.07MW.year/m² (3 years operation)	5 - 8 MW.year/m²	10 - 15 MW.year/m²
Displacement per atom (dpa)	< 3 dpa	50 - 80 dpa	100 - 150 dpa
	<div>Increasing Materials challenge</div> 		
Transmutation product rates at first wall	~10 appm Helium / dpa ~45 appm H / dpa		

Fusion Materials Current Readiness



from Wirth, et al, ARPA-E Workshop, 2023

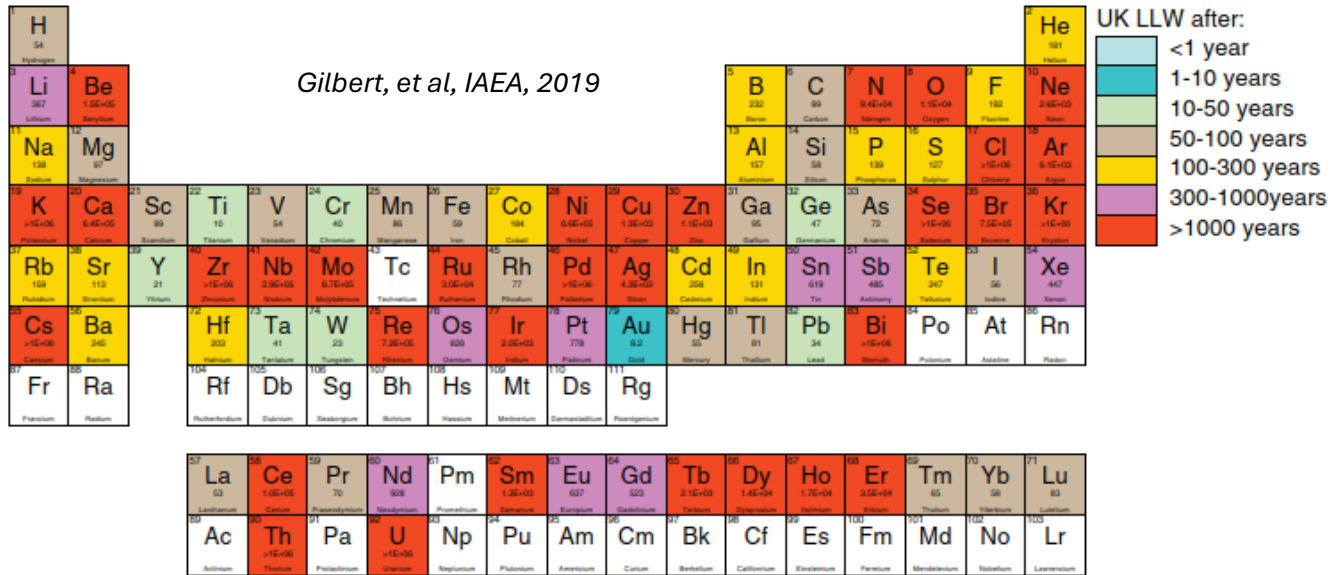
The low activation



~ not consistent in different evaluations

Time to LLW
after DEMO divertor body exposure
(phase 2c ≈ 5 years pulsed operation)

Gilbert, et al, IAEA, 2019



W-HEA → W (smart) alloy

V-HEA → V-4Cr-4Ti

Moschetti, et al, JNM, 2022

Long active:

- Al, Ni, Mo, Nb

High and long-lived gamma dose rate:

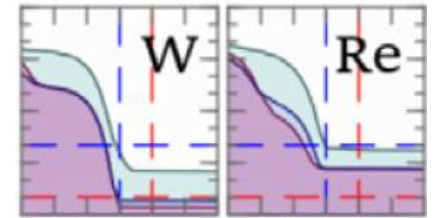
- Co, Ag, Sn, Sb

Large neutron cross section:

- Hf

Low activation:

- Ti, Cr, V



High initial gamma dose with fast cooling rate

- (Hf), Ta, W

High activation but acceptable:

- Zr, Fe

Application dependent (may not low activation):

- Mn, Y, Rh

forbidden elements

Requirements

- Room temperature ductility after 50 dpa of irradiation damage and helium generation;
- Sufficiently high thermal conductivity to remove up to 10 MW/m² of heat;
- Activation below 10,000 Sieverts per hour (Sv/hr) to enable remote handling;
- Swelling below 1% to maintain dimensional stability; and
- Tritium retention and plasma erosion lower than current state-of-the-art (SoA) materials.

Table 2. Unirradiated material property constraints for plasma-facing component materials in Category A.

ID	Metric Name	Acceptable Baseline Value	Category A Target Value (Plasma-Facing Component)
A1	Thermal conductivity	>170 W/m-K @ 20°C (Pure tungsten)	340 W/m-K @ 20°C (2x improvement)
A2	Plasma erosion	<3.4 μm/hr @ 650°C and 10e22 ion/m ² -s of plasma ion flux (Erosion of 30 mm thickness in 1 year)	85 nm/hr @ 650°C and 10e22 ion/m ² -s of plasma ion flux (40x improvement)
A3	Tritium solubility	<5e20 tritium/vPa-m ³ @ 650°C (Pure tungsten)	2e20 tritium/vPa-m ³ @ 650°C (2x improvement)
A4	Tritium diffusivity	>1.6e-8 m ² /s @ 650°C (Pure tungsten)	3.2e-8 m ² /s @ 650°C (2x improvement)

Table 3. Unirradiated material property constraints for structural materials in Category B.

ID	Metric Name	Acceptable Baseline Value	Category B Target Value (Structural)
B1	Thermal conductivity	>33 W/m-K @ 20°C (RAFM steel)	66 W/m-K @ 20°C (2x improvement)
B2	High temperature yield strength	>280 MPa @ 650°C (RAFM steel)	560 MPa @ 650°C (2x improvement)
B3	Tritium solubility	<4e21 tritium/vPa-m ³ @ 650°C (RAFM steel)	2e21 tritium/vPa-m ³ @ 650°C (2x improvement)
B4	Tritium diffusivity	>8.2e-9 m ² /s@ 650°C (RAFM steel)	1.6e-8 m ² /s@ 650°C (2x improvement)

Table 4. Material product scalability requirements.

ID	Metric Name	Value	Description and Rationale
M1	Total product amount	250 mm x 250 mm	Create enough material product to cover a surface area of at least 250 mm x 250 mm.
M2	Minimum unit size	50 mm x 50 mm x 5 mm	Show parts can be made in tile sizes to build a first-wall component.
M3	Manufacturing tolerance	<0.25 mm	Show parts can be made with commercial machining precision.
M4	Manufacturing reliability	Rejection rate <10%	Show parts can be consistently manufactured without excessive waste.
M5	Average raw material unit cost	<\$1000/kg	Ensure the new material is not composed of excessively rare minerals or elements.

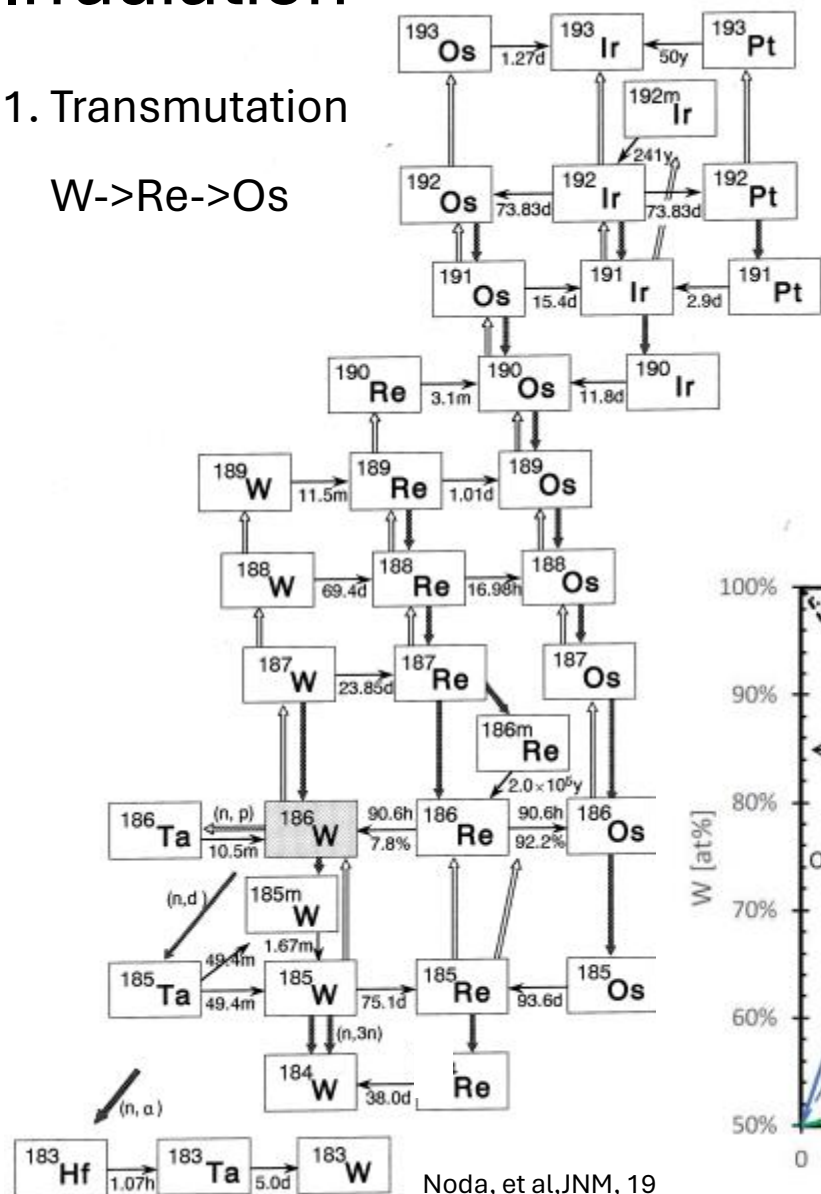


~ Database: experiments needed

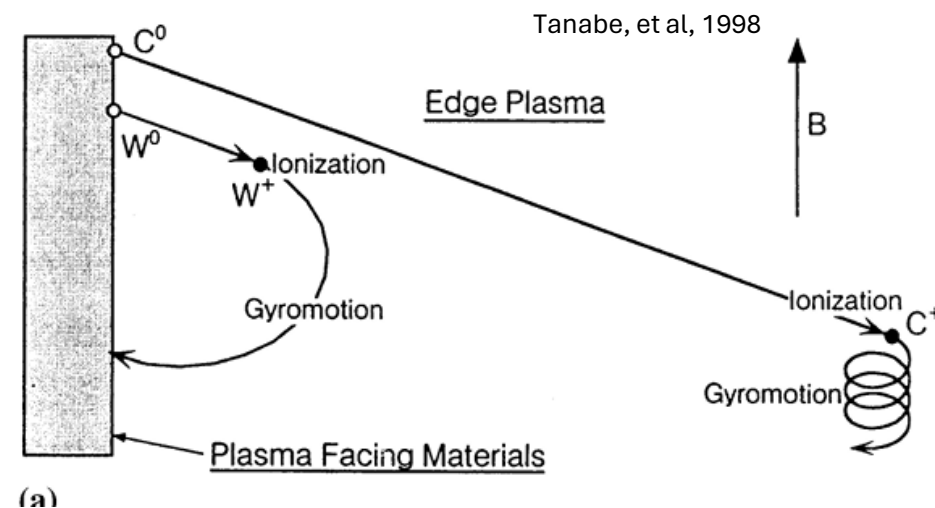
Irradiation

1. Transmutation

W→Re→Os



2. Sputtering

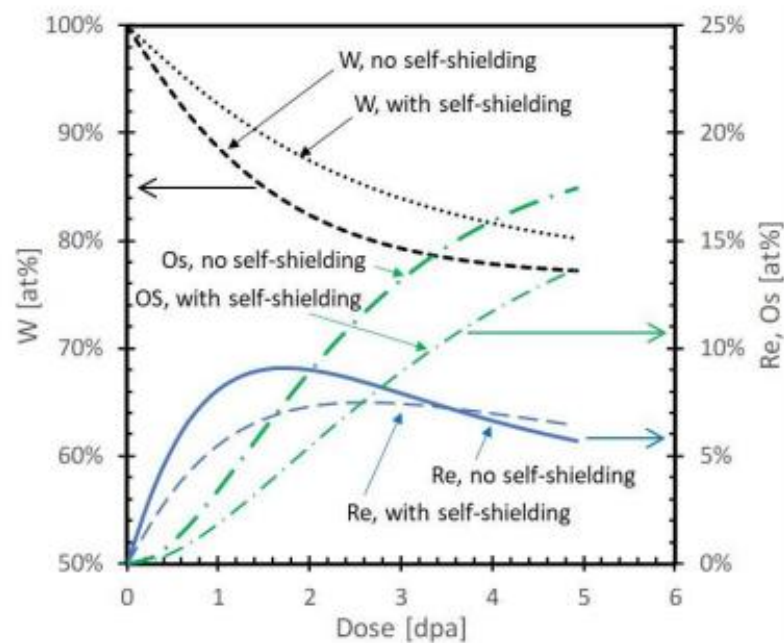


Plasma contamination

➤ High Z vs low Z

Sputtering resistance

➤ SiC design abandoned



Katoh, et al, 2019

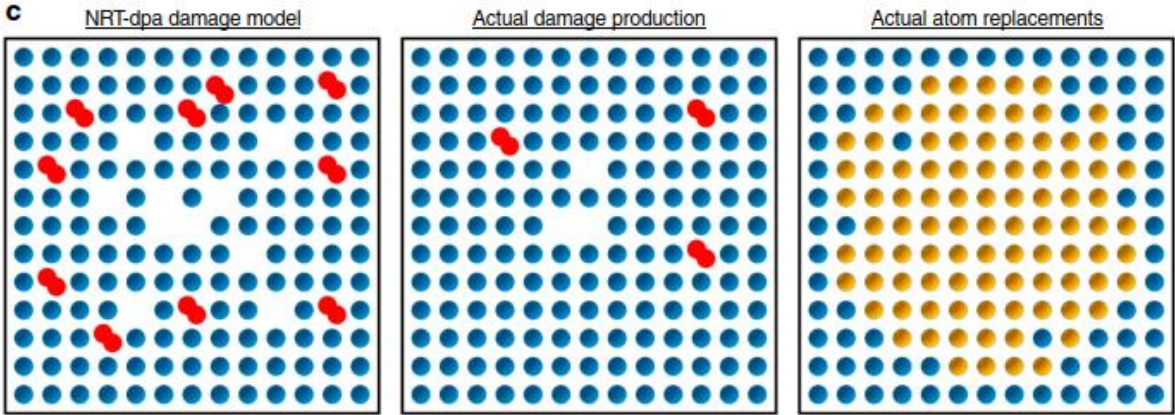
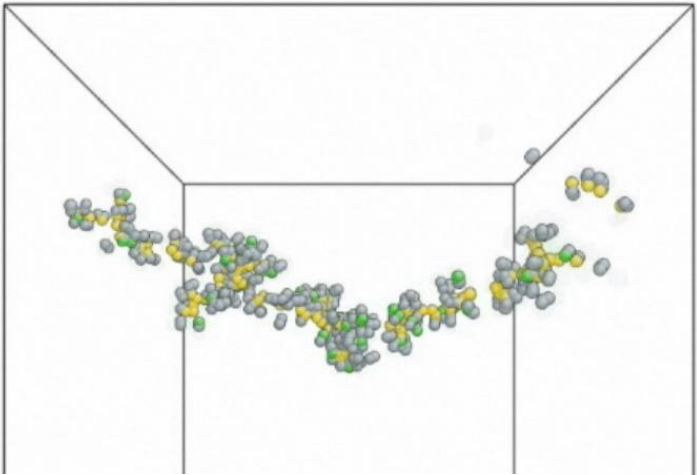
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Irradiation

3. Short-term damage

- Replacement
- I-V pairs

3.3 Cascade formation and cooling in an Fe-10%Cr alloy, In this MD simulation, the yellow spheres are vacancies, the grey are iron interstitials and the green are chromium interstitials. Chromium is modeled as the larger solute, and after cooling, the remaining interstitial population is predominantly iron atoms as their distortion of the lattice is less than that from the oversized chromium atoms. (courtesy, B. Wirth, University of California, Berkeley)



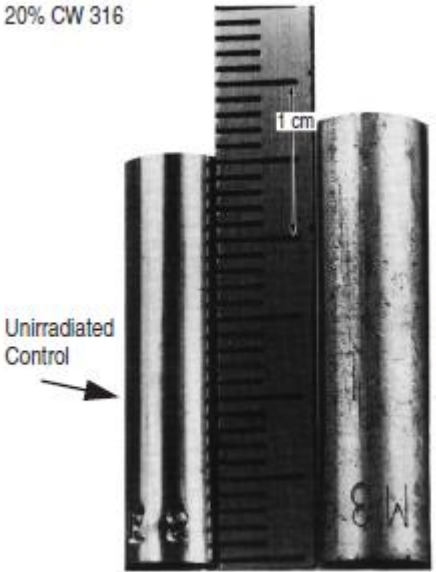
Nordlund, et al, 2018

- amorphous
- disordering

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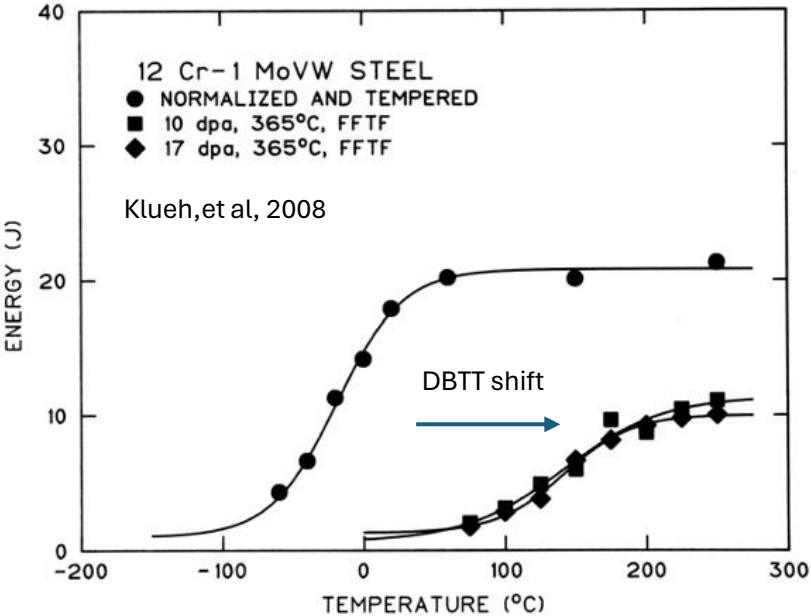
4. Long-term damage

20% CW 316



Dislocation loop
Cavity

Allen, JOM, 2008



Chemical depletion and precipitate

5. Irradiation Induced Precipitate

The coupling between the point defect fluxes produced under irradiation and the solute fluxes will generate the precipitation/segregation of solute at point defects sinks. This mechanism can occur in both **under-saturated and over-saturated systems**.



~ Irradiation modified precipitates

~ High Entropy -> phase stability

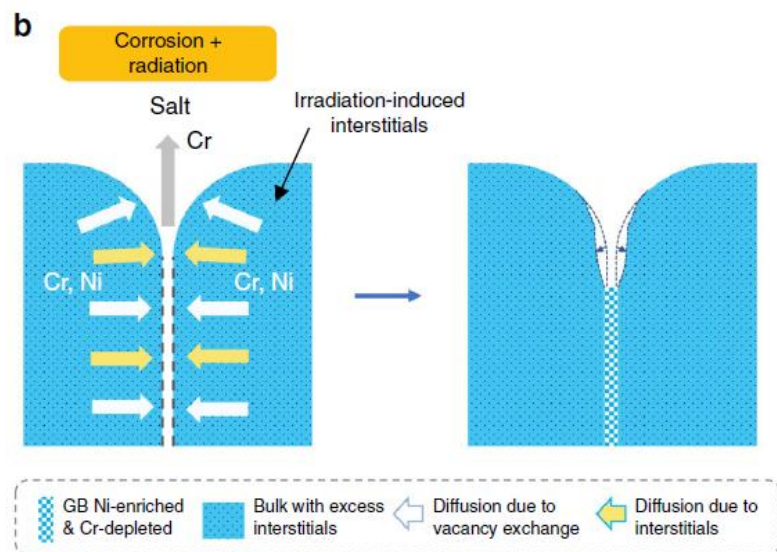
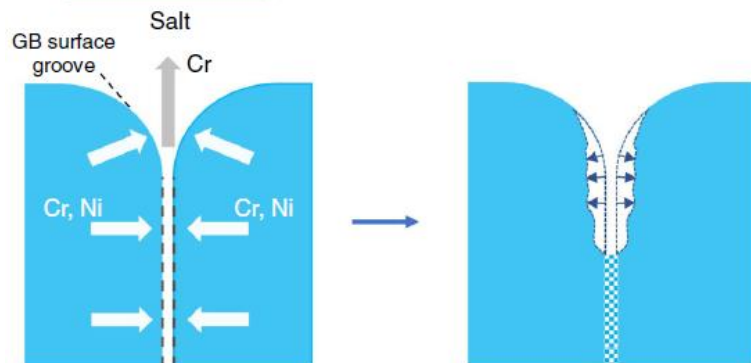
6. Irradiation Enhanced Precipitate

The irradiation is to accelerate the evolution of the system towards its equilibrium state. This mechanism can only appear in **over-saturated alloys**.

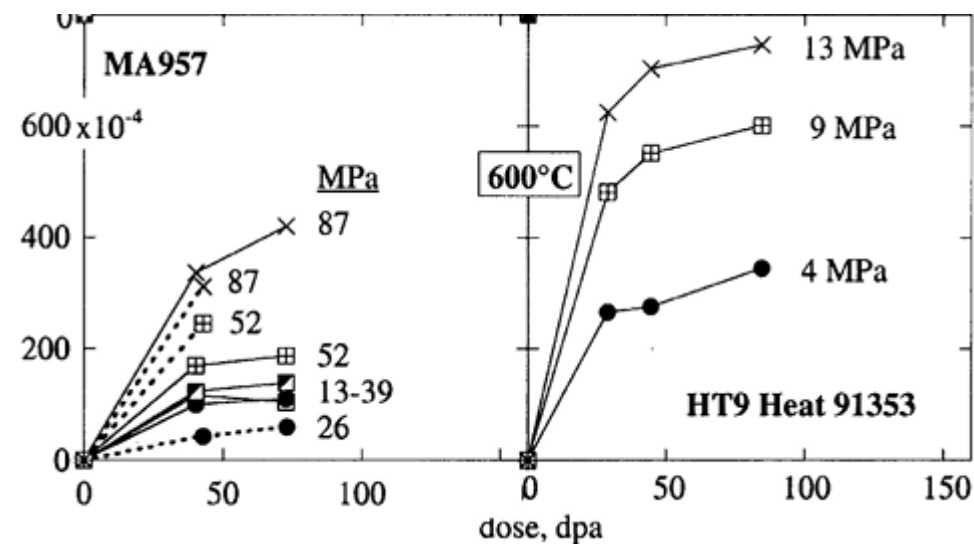
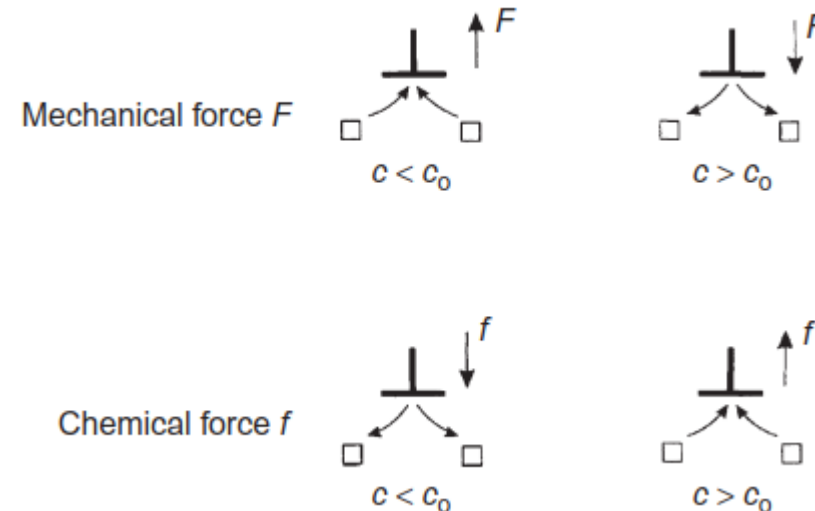
Synergetic effects

7. Irradiation + Corrosion

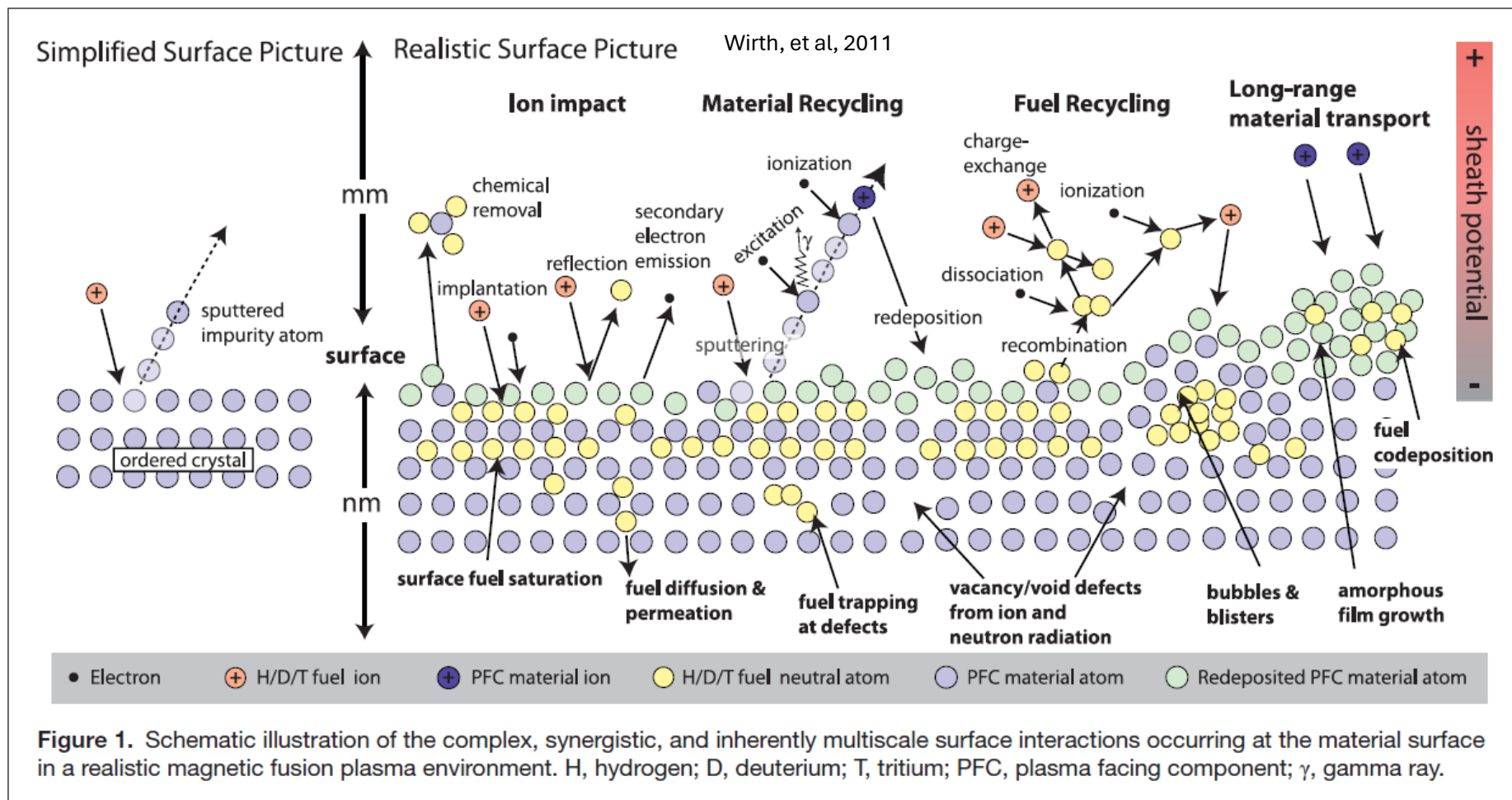
a Corrosion only Acceleration vs Deceleration



8. Irradiation + Creep



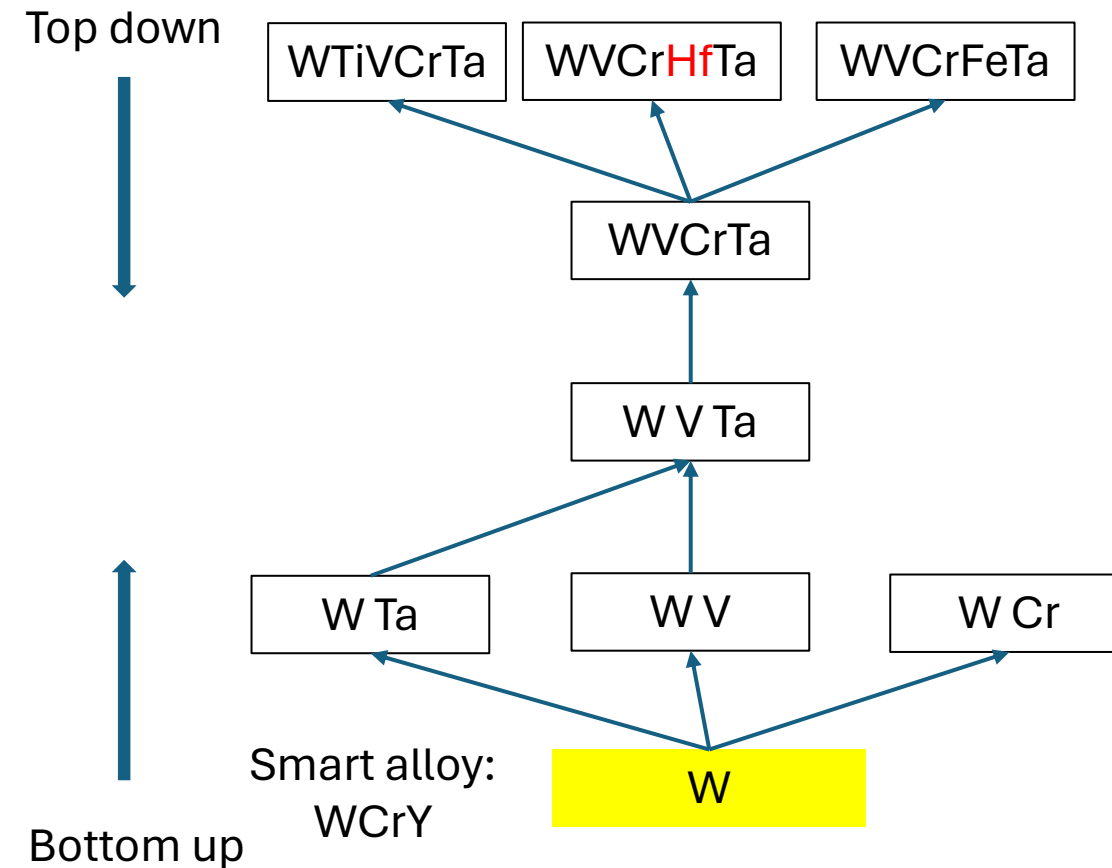
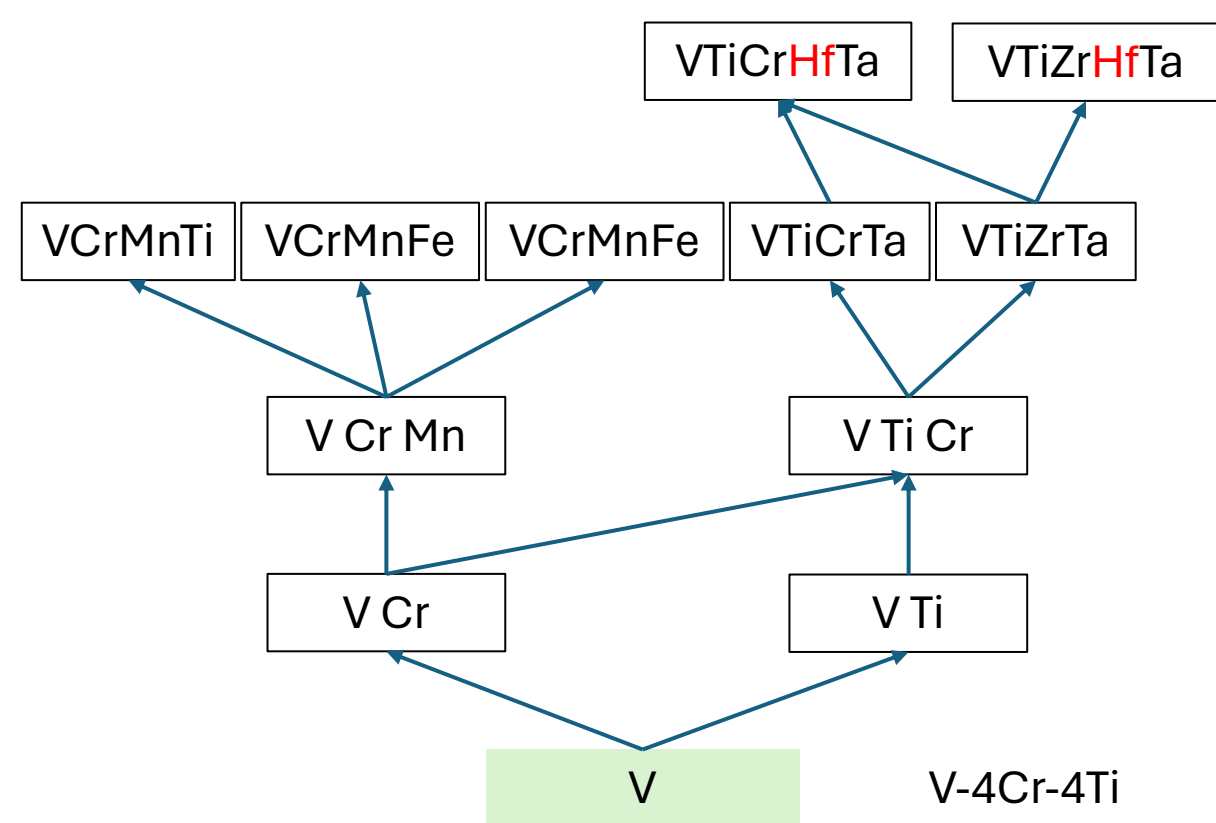
Irradiation: 9. Fuel Retention



Low activation HEA

Wirth, et al, 2011

Ti, V, Cr, Mn, Fe, Y, Zr, (Hf), Ta, W, Si



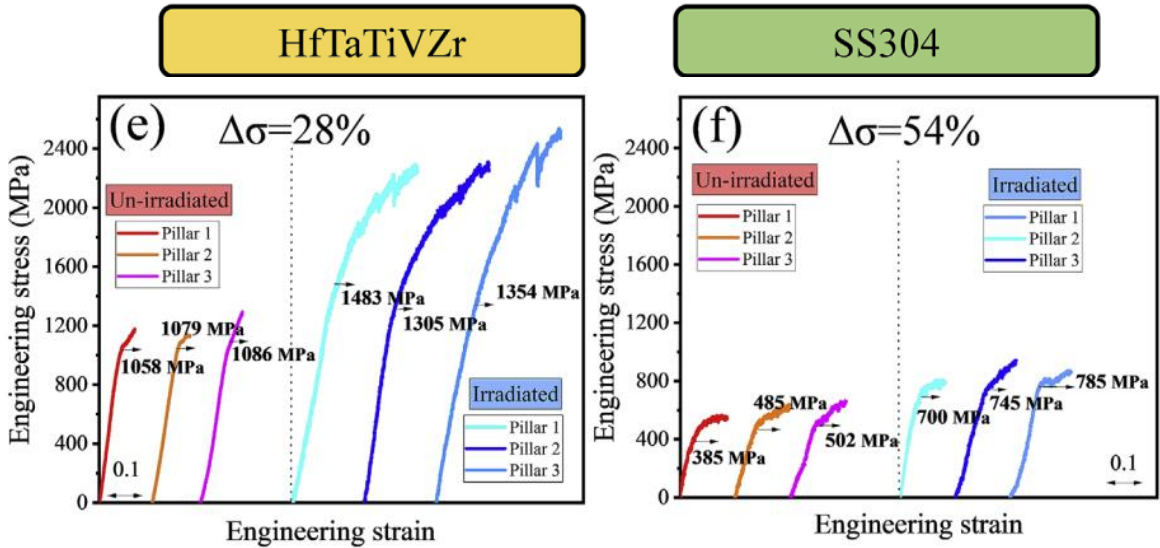
Irradiation effects

~ BCC vs FCC

~ temperature ~ grain size

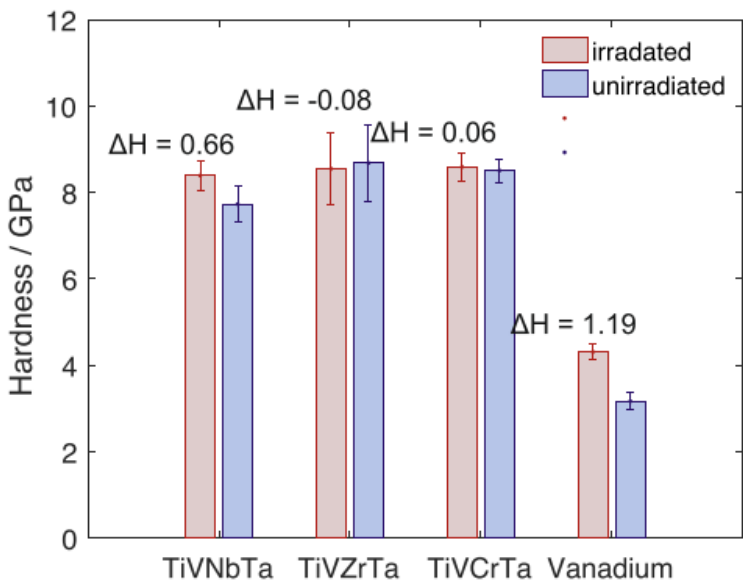
Sadeghilaridjani, et al, 2020

Ni²⁺ irradiation, peak 40dpa, RT



Kareer, et al, 2019

V⁺ irradiation,
peak 3.6dpa,
500°C



Zhang

Alloys		Temperature (K)	Fluence (ions/cm ²)	Peak He Concentration (at.%)	Average Size (nm)
HEAs	TiVTa [57]	973	1 × 10 ¹⁷	5.0	13.4
	TiVNbTa [57]	973	1 × 10 ¹⁷	5.0	8.1
	TiZrNbV [46]	1023	6 × 10 ¹⁶	3.9	12.5
	TiZrNbVMo [46]	1023	6 × 10 ¹⁶	3.9	10.4
	NiCo [58]	973	6.4 × 10 ¹⁶	3.6	25.1
	NiCoCr [58]	973	6.4 × 10 ¹⁶	3.6	34.1
	NiCoFeCrMn [58]	973	6.4 × 10 ¹⁶	3.6	85.6
CMs	V-4Cr-4Ti [59]	573	5 × 10 ¹⁶	4.0	2.7
	ODS [60]	723	1 × 10 ¹⁷	5.6	3.9
	RAFM [63]	773	3 × 10 ¹⁶	5.7	5.1
	GH3535 [62]	923	1 × 10 ¹⁷	5.0	2.3
	Ni-SiC [64]	923	6 × 10 ¹⁶	3.5	8.1

Zhang, et al, 2023

Alloys		Temperature (K)	Fluence (×10 ¹⁶ ions/cm ²)	Hardening Fraction (%)
HEAs	TiZrNb [46]	1023	6	17.3
	TiZrNbV [46]	1023	6	41.3
	TiZrNbVMo [46]	1023	6	23.6
CMs	ODS [60]	723	5	48.1
	V-Cr-Ti [59]	573	5	52.0
	RAFM [65]	773	3	85.9
	CLAM [19]	773	3	61.1

Issues

fuel trap:

D in W-HEA shows 5 times more than in W

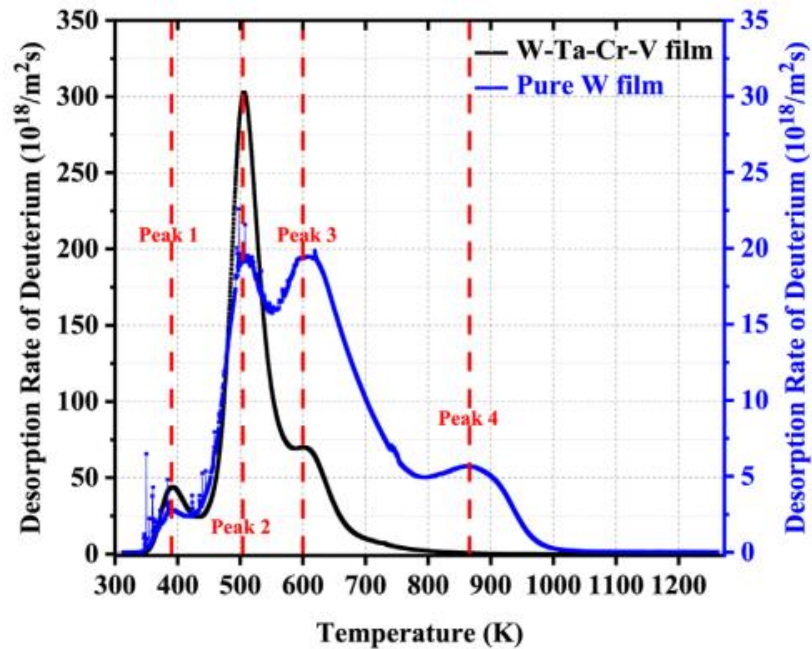


Fig. 8. TDS spectra of W film and W-Ta-Cr-V film irradiated by 110 eV deuterium plasma to a fluence of 2.63×10^{24} D/m².

Shi, et al, JNM, 2022

Grain boundaries?

aging, phase issues:

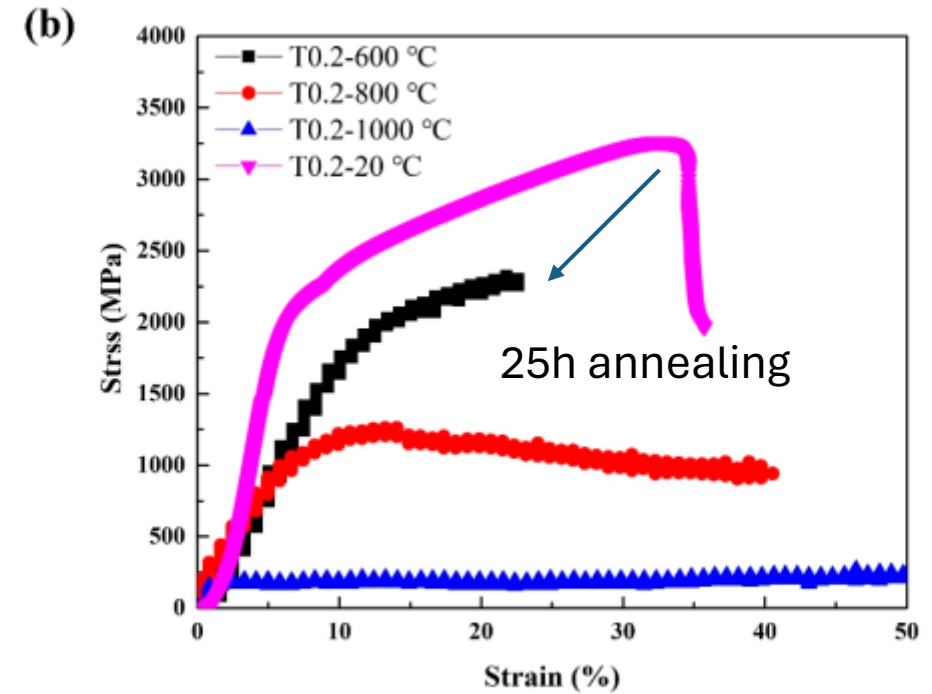


Figure 6. The compressive stress-strain curves of (a) VCrFeTa_{0.1}W_{0.1} and (b) VCrFeTa_{0.2}W_{0.2} alloys at different temperatures with a diameter of 3 mm.

Zhang, et al, entropy, 2018

Study examples

nature communications

Article

<https://doi.org/10.1038/s41467-023-38000-0>

A quinary WTaCrVHf nanocrystalline refractory high-entropy alloy withholding extreme irradiation environments

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Check for updates

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In the quest of new materials that can withstand severe irradiation and mechanical extremes for advanced applications (e.g. fission & fusion reactors space applications, etc.), design, prediction and control of advanced material beyond current material designs become paramount. Here, through a combined experimental and simulation methodology, we design a nanocrystalline refractory high entropy alloy (RHEA) system. Compositions assessed under extreme environments and in situ electron-microscopy reveal both high thermal stability and radiation resistance. We observe grain refinement under heavy ion irradiation and resistance to dual-beam irradiation and helium implantation in the form of low defect generation and evolution, as well as no detectable grain growth. The experimental and modeling results—showing a good agreement—can be applied to design and rapidly assess other alloys subjected to extreme environmental conditions.



~ Ultrafine grains

~ Coating



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Helium implantation damage resistance in nanocrystalline W-Ta-V-Cr high entropy alloys

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ABSTRACT

Nanocrystalline W-Ta-Cr-V high entropy alloys have shown promising properties as nuclear fusion materials with enhanced radiation resistance to heavy ion irradiation and negligible radiation hardening. In this work, we investigate the performance of the alloy under low energy helium (He) implantation up to a fluence of $1.25 \times 10^{17} \text{ cm}^{-2}$ at 1223 K. We observe a uniform high density of very small ($\sim 2\text{--}3 \text{ nm}$) bubbles grown at a slow rate along with enhanced He bubble damage resistance, further marked by no preferential bubble formation on the grain boundaries, even at much higher fluences compared to previously implanted tungsten grades. First principle calculations of He formation and migration en-

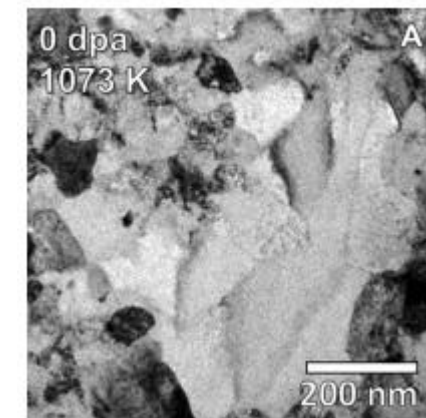
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MATERIALS SCIENCE

Outstanding radiation resistance of tungsten-based high-entropy alloys

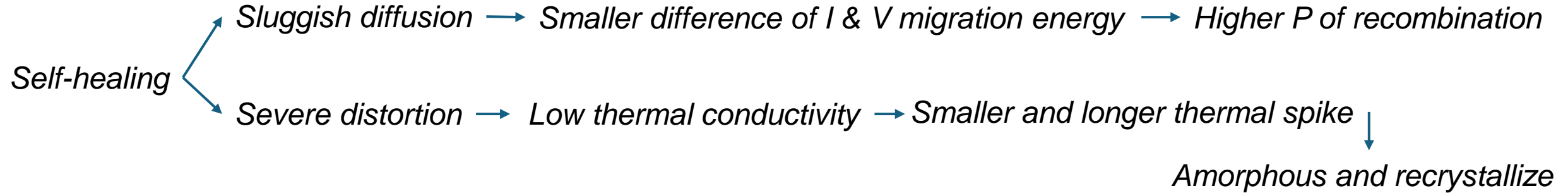
O. El-Atwani^{1,*}, N. Li², M. Li³, A. Devaraj⁴, J. K. S. Baldwin², M. M. Schneider¹, D. Sobieraj⁵, J. S. Wróbel⁵, D. Nguyen-Manh⁶, S. A. Maloy¹, E. Martinez^{7,*}

A body-centered cubic W-based refractory high entropy alloy with outstanding radiation resistance has been developed. The alloy was grown as thin films showing a bimodal grain size distribution in the nanocrystalline and ultrafine regimes and a unique 4-nm lamella-like structure revealed by atom probe tomography (APT). Transmission electron microscopy (TEM) and x-ray diffraction show certain black spots appearing after thermal annealing at elevated temperatures. TEM and APT analysis correlated the black spots with second-phase particles rich in Cr and V. No sign of irradiation-created dislocation loops, even after 8 dpa, was observed. Furthermore, nanomechanical testing shows a large hardness of 14 GPa in the as-deposited samples, with near negligible irradiation hardening. Theoretical modeling combining ab initio and Monte Carlo techniques predicts the formation of Cr- and V-rich second-phase particles and points at equal mobilities of point defects as the origin of the exceptional radiation tolerance.

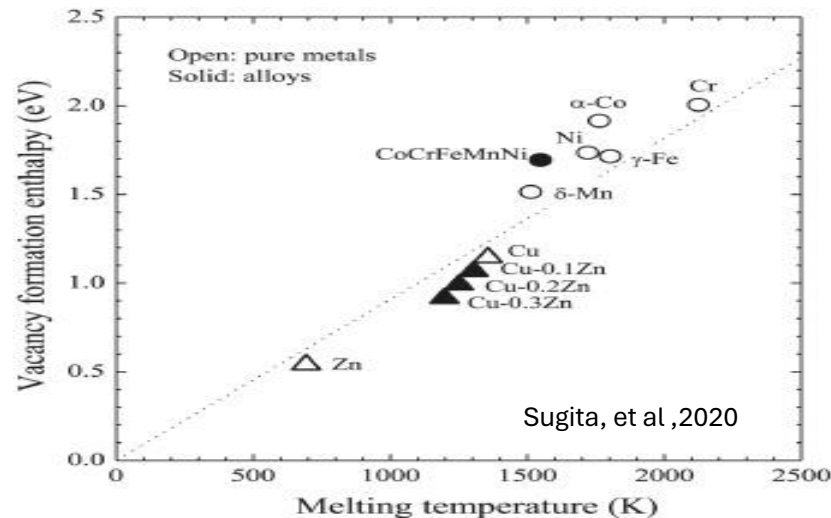


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Mechanism studies

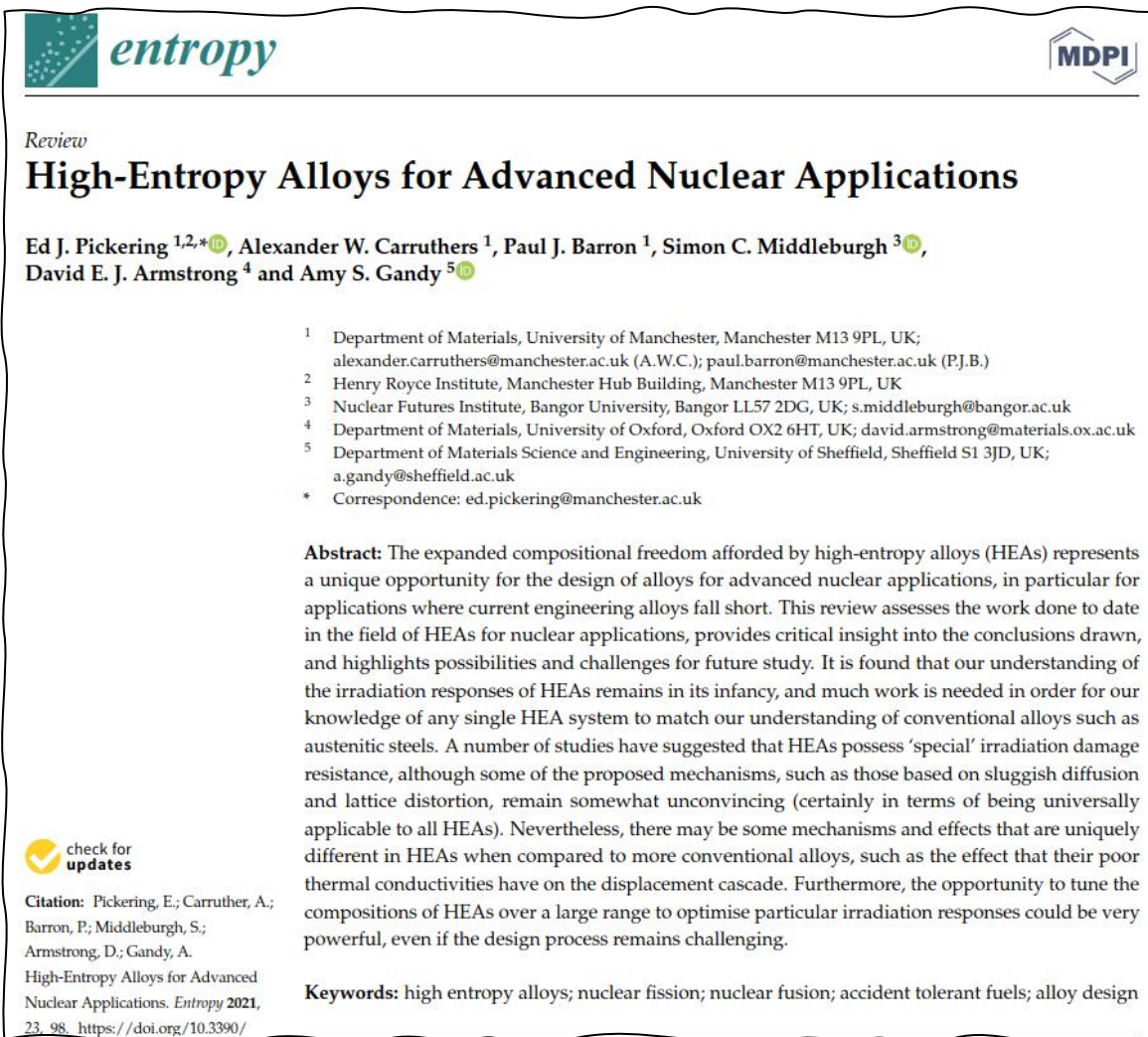


1. No conclusive experimental evidence that diffusion is sluggish in HEA
2. Measurements of lattice distortion of single-phase HEA have not found abnormally distorted lattice
3. A recent study should little difference in vacancy formation energy of CrFeCoNiMn
4. Some trends, like precipitate and depletion, and grain refinement, no different in concentrated alloys



- ~ Composition dependence
- ~ Temperature dependence
- ~ Flux & Beam type dependence

Future challenge



1. Element selection
2. Impurity problem
3. Modeling Challenge

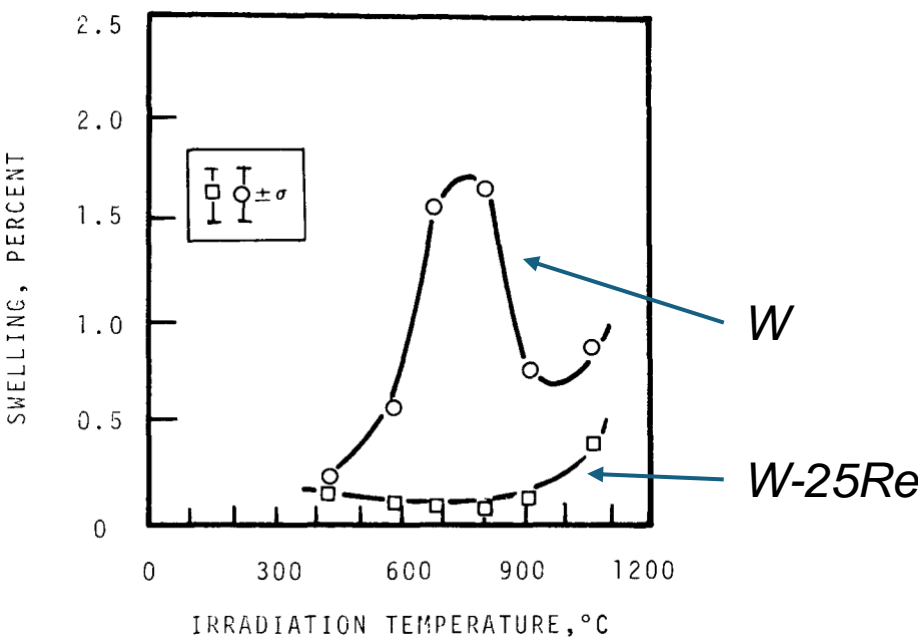
7. Remaining Questions

Some key remaining questions, which researchers may wish to direct their attention to, include:

- **Do HEAs possess enhanced intrinsic damage resistance versus conventional alloys?** There is little indication that sluggish diffusion is likely to be significant in general, but are there changes in cascade dynamics and defect energetics brought about by HEA compositional complexities that are beneficial? For instance, it has been suggested that the reduced thermal conductivities generally possessed by HEAs may prolong the thermal spike and change the size and shape of the cascade, leading to more opportunity for defect recombination. Is this generally true for all HEAs? This deserves more attention.
- **Do refractory BCC HEAs possess special irradiation damage resistance, versus less-concentrated BCC alloys and/or FCC HEAs?** If so, what is the mechanism for this? There has been some suggestion that the defect energetics in some BCC refractory HEAs may be conducive to superior irradiation damage resistance, but further confirmation of whether this true more generally is needed.
- **How can we best design HEAs for nuclear applications?** Designing HEAs for enhanced irradiation tolerance is still in its infancy, and there's not yet been a study that's sought to tune, for example, the defect formation energies, or sink strengths or biases, through the optimisation of HEA compositions. Design efforts will almost certainly require the use of advanced atomic-scale models to predict irradiation response, and there remain challenges associated with their construction.

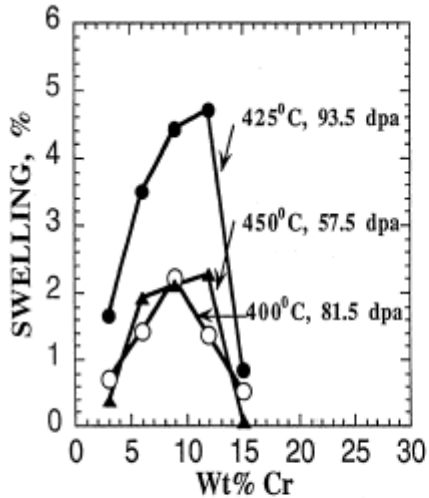
Example

Fast neutron irradiation



Matolich,1974

Fast neutron irradiation



- Originally assigned dpa levels are too large.

T, °C	dpa
400	81.5 → 35.1
425	93.5 → 48.3
450	57.5 → 34.8

Fig. 13. Dependence of swelling in Fe-Cr alloys on chromium level, temperature, and dpa in EBR-II for Fe-Cr alloys, showing correction of dpa assignments.

Garner,2000

Comparison

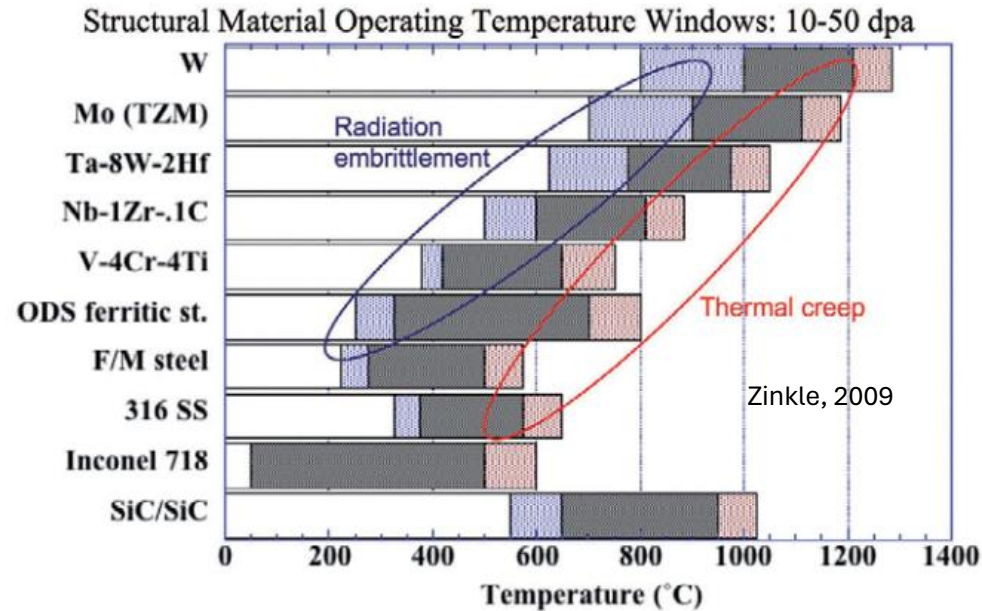


Fig. 5 Estimated operating temperature windows (dark shaded region)^{30,79} for structural materials in nuclear energy systems for damage levels of 10 to 50 dpa. The light blue and red regions represent lower and upper temperature uncertainty bands.

~ HEA?

- Bulk material fabrication
- Joining and welding
- Operation temperature window
- Long term mechanical property
- Fuel retention
- Irradiation resistance
- Thermal conductivity

Summary

- 1. HEA, special mechanical property, a huge number of selections, four core effects*
- 2. Fusion reactor materials, extreme environment, low activation requirements, conventional alloy issues*
- 3. Low activation HEA, V-HEA, W-HEA, development in infancy, debates and challenges*