Direct view of self healing in creep alloys

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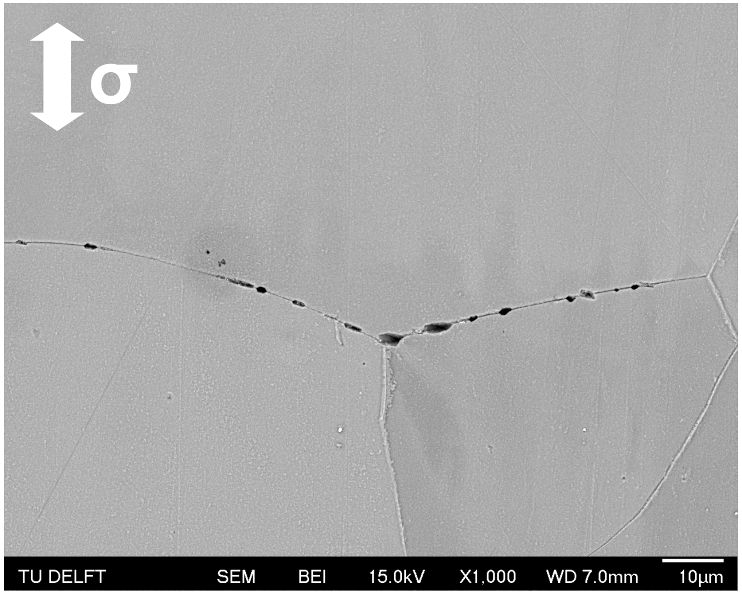
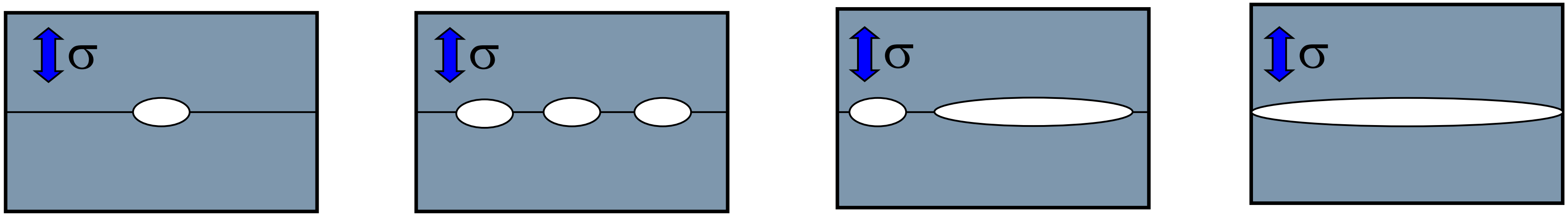
Dutch Materials 2018, Utrecht, The Netherlands

Creep damage in metals:



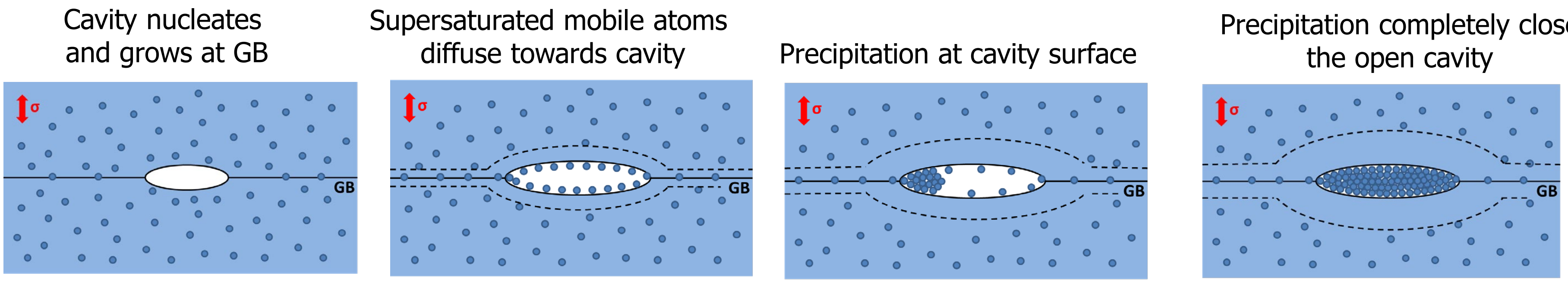
- time-dependent deformation
- occurs for $T > 0.4\ T_{melting}$ (in K)
- under constant load
- Damage preferentially nucleate and grow at grain boundary (GB)



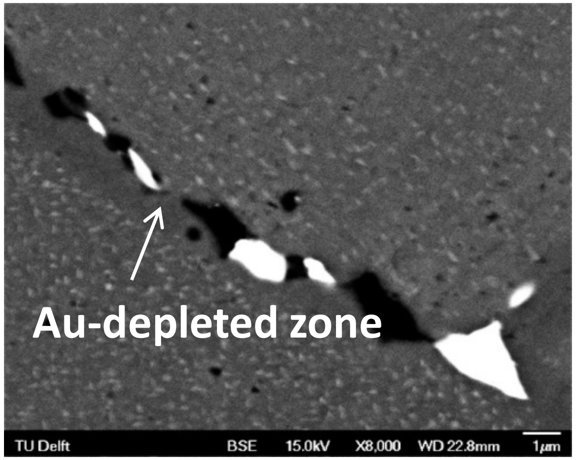
Isolated | Oriented | Micro crack | Macro crack

Self healing approach: manage the damage

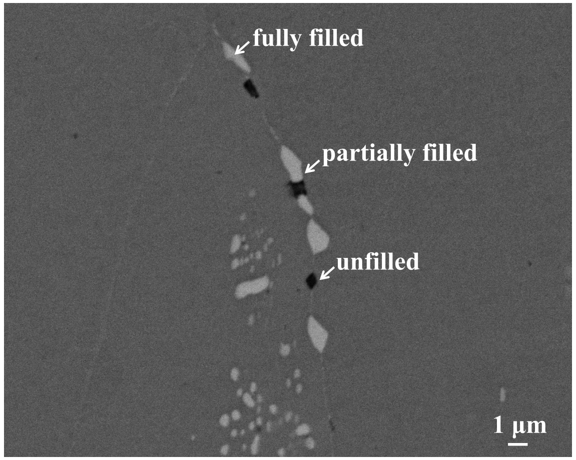
mobile solute atoms → precipitation at free crack surface



Self healing model alloys



Fe-Au



Fe-Mo

S. Zhang, Adv. Eng. Mater., 2015.
S. Zhang, Metall. Mater. Trans. A, 2016.

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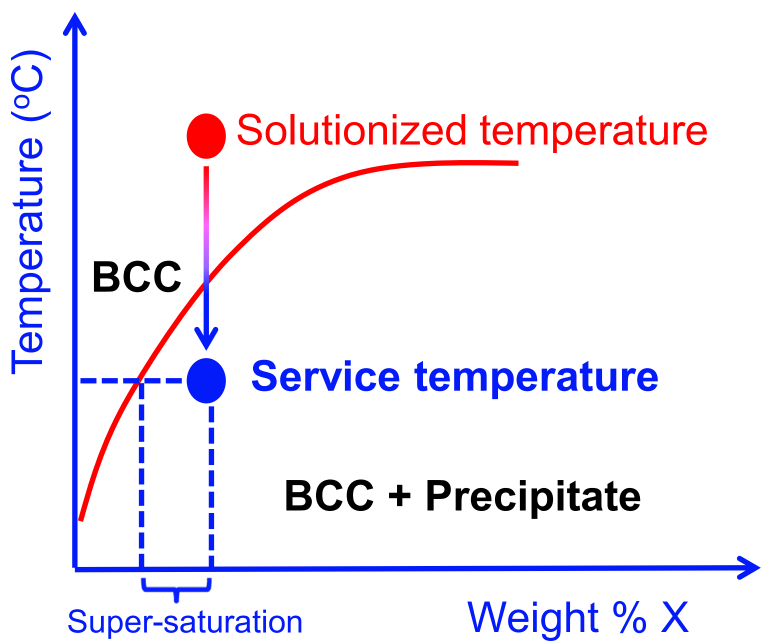
Requirements of element X to be healing agent

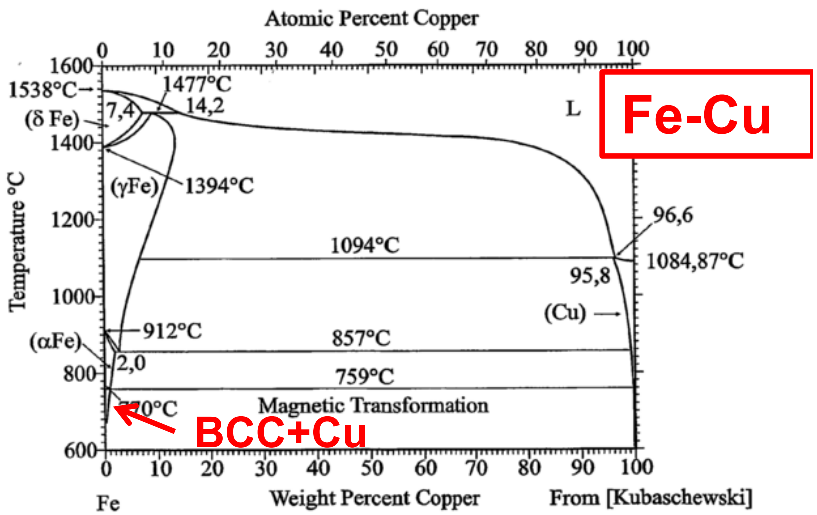
Schematic phase diagram of potential self healing Fe-X alloy

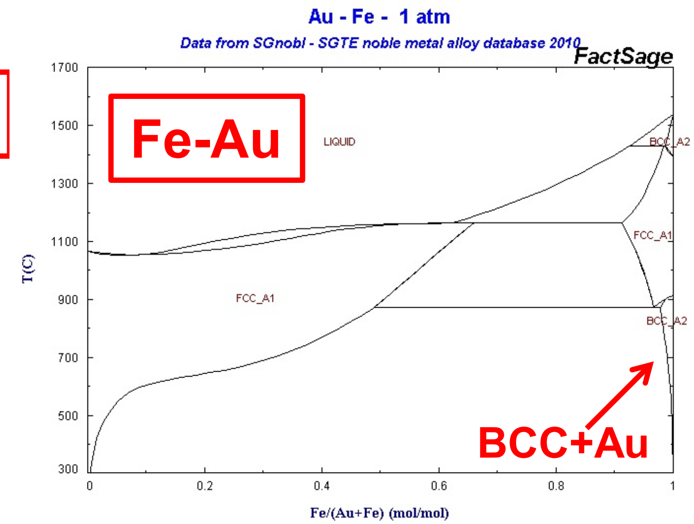


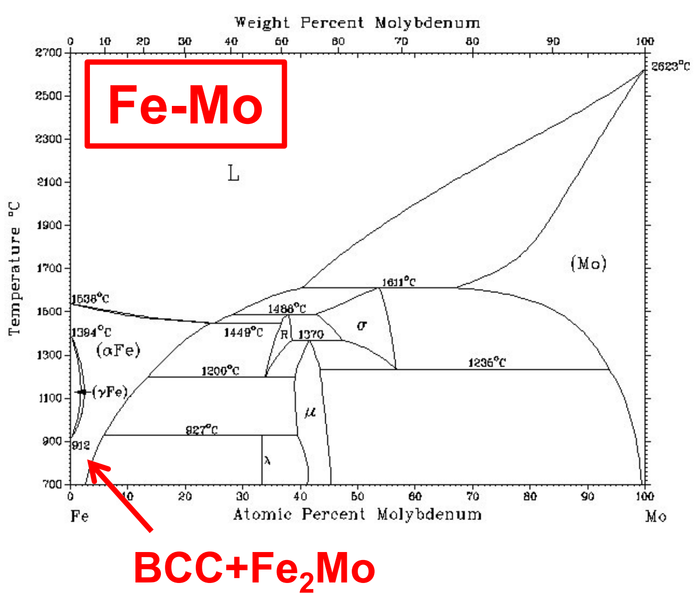
- Could be solutionized at high *T*
- Supersaturation: $w_X > w_X$ (sol.)
- Diffusivity: D(X) > D(Fe)
- A tendency to precipitate at free surface

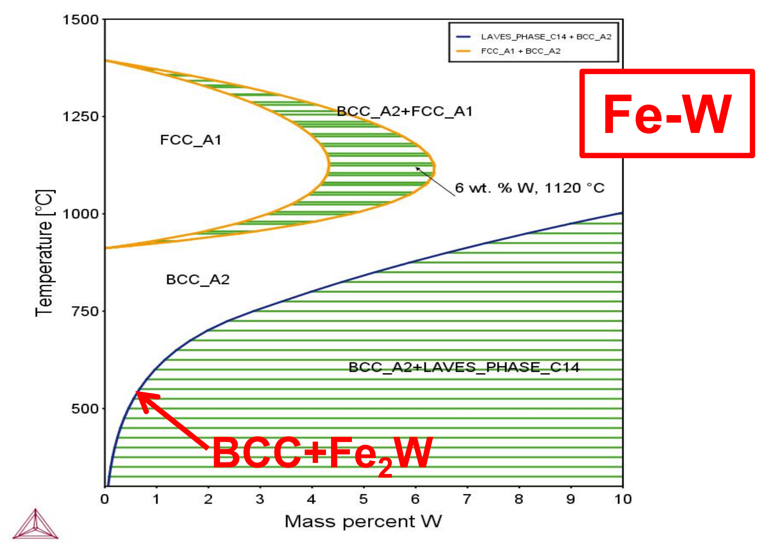
- Atomic radius: R(X) > R(Fe)
- Atomic volume: V(X of prec.) > V(Fe)

Preferred X elements for self healing Fe-X alloys

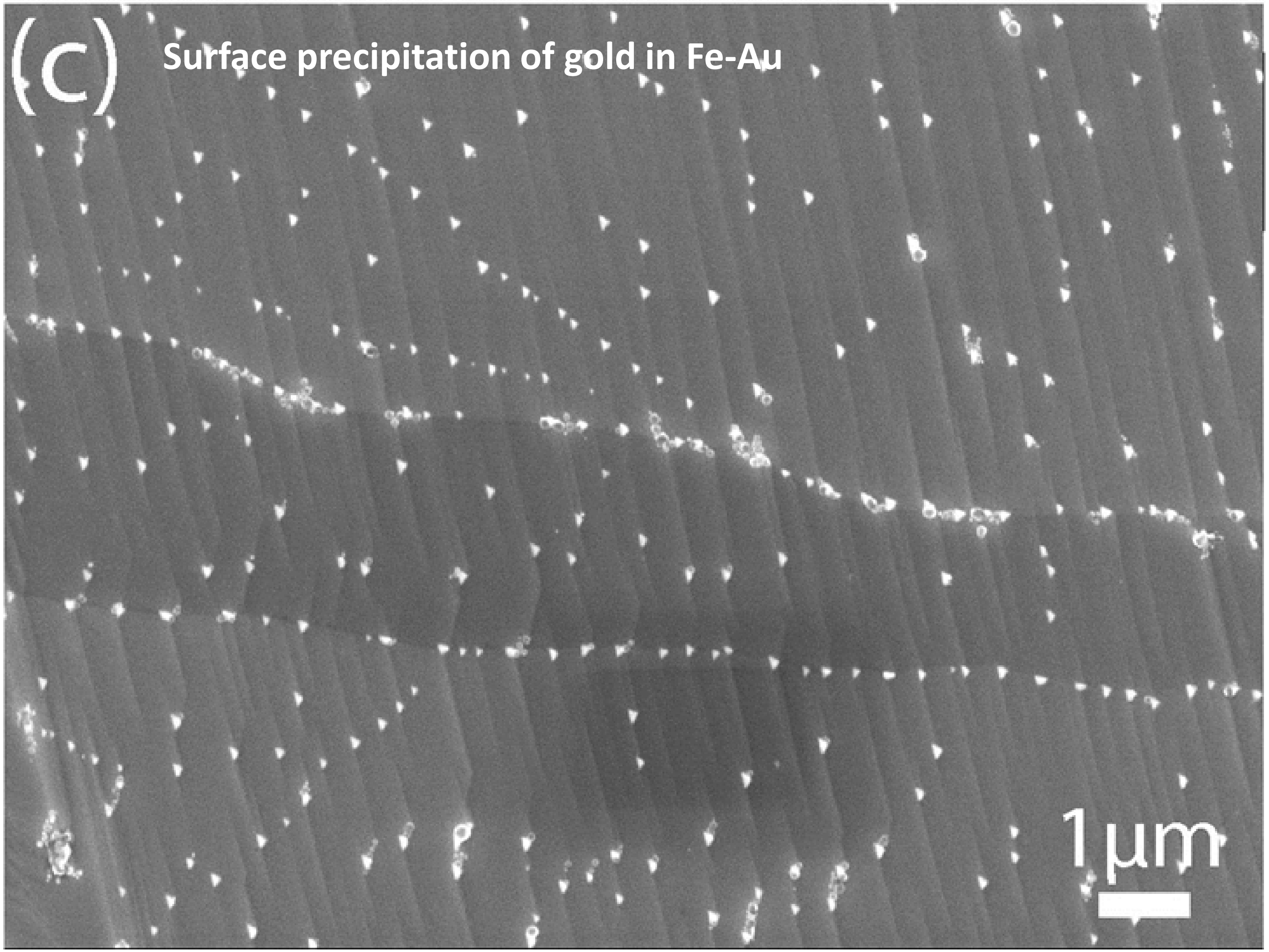








Nucleation at free surface than in the matrix is easier for larger atoms, e.g. T = 550 °C, Fe-(sat.+1) at.% X alloys

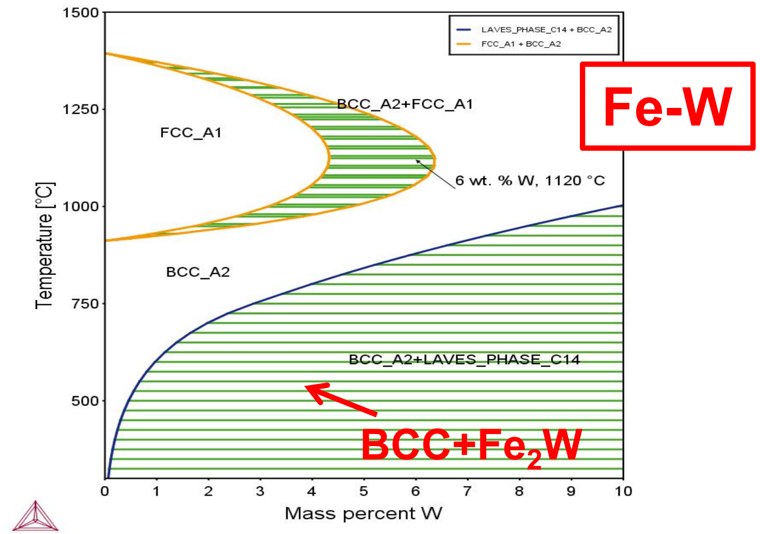


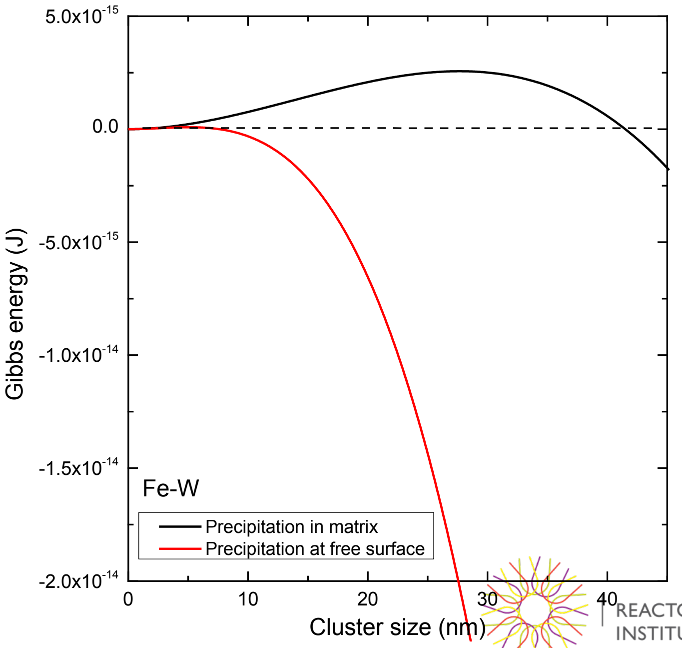
W.W. Sun et al, Metall. Mater. Trans. A, 2017.

Advantages of W as healing agents in steels

- Be able to be tuned into supersaturation state
- $D_W > D_{Fe}$ (diffusivity)*
- $R_W \approx 1.10\ R_{Fe}$, $R_{Fe2W} \approx 1.06\ R_{Fe}$ (atomic radius)

- **Less expansive than Au**
- **Lower neutron activation than Mo**
- **Widely used in most advanced creep-resistant steels as solid solution strengthening solute**





*C.D. Versteylen et al, Phys. Rev. B, 2017

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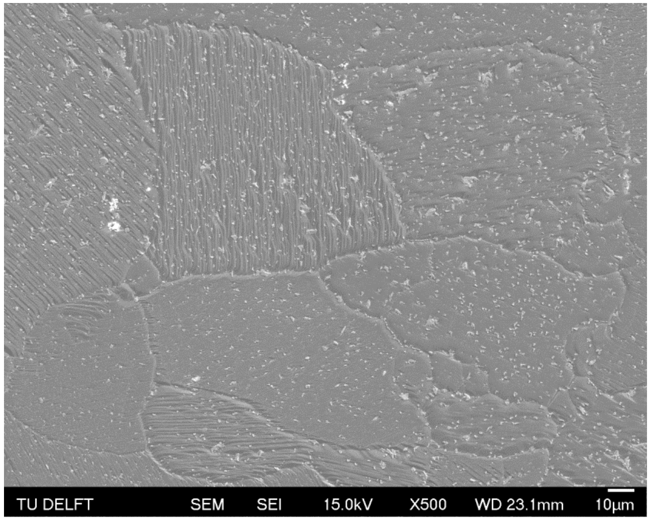
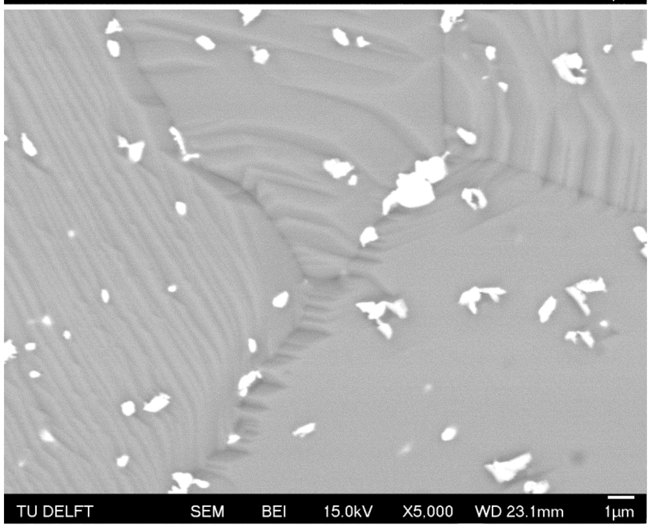
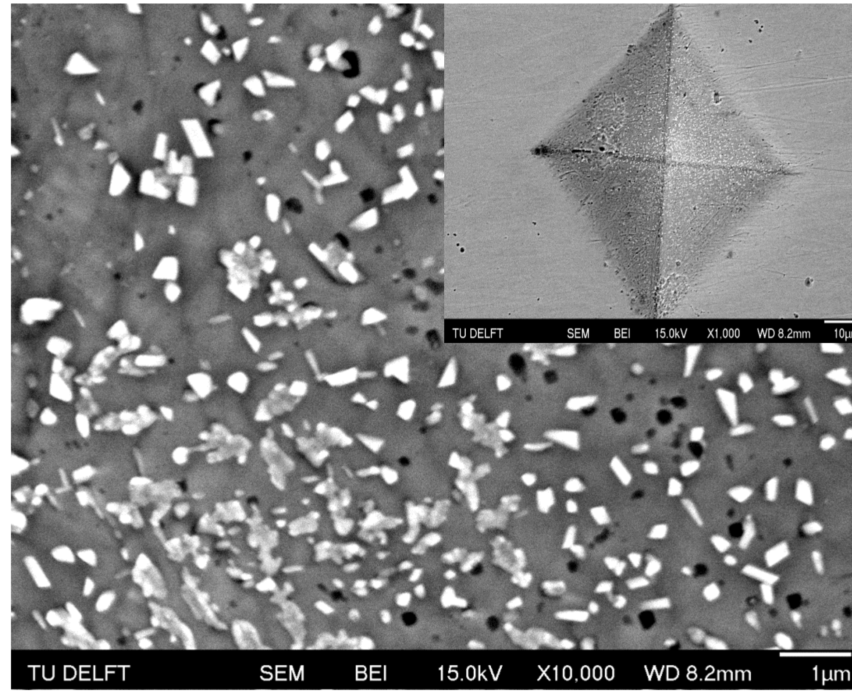
Precipitation of Laves Fe_2W at external surface in Fe-W alloys





Surface precipitation without damage
(ageing at 700 ^{o}C for 30 min)



Surface precipitation at surface indentation damage
(ageing at 600 ^{o}C for 140 h)

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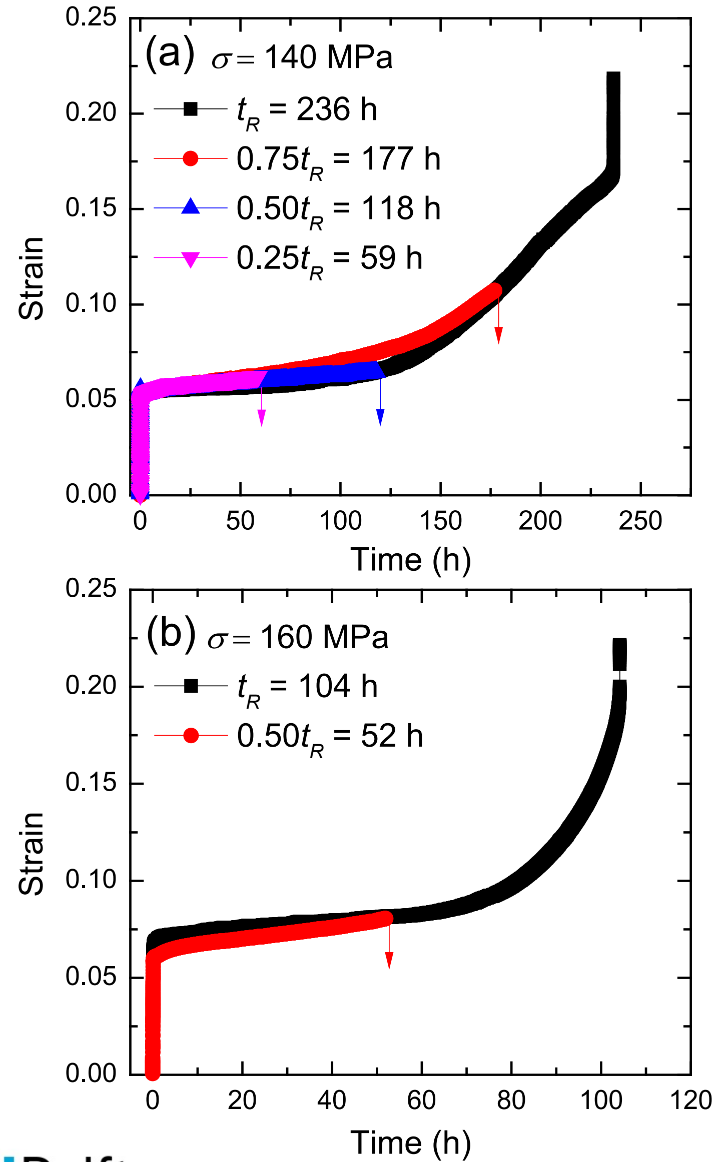
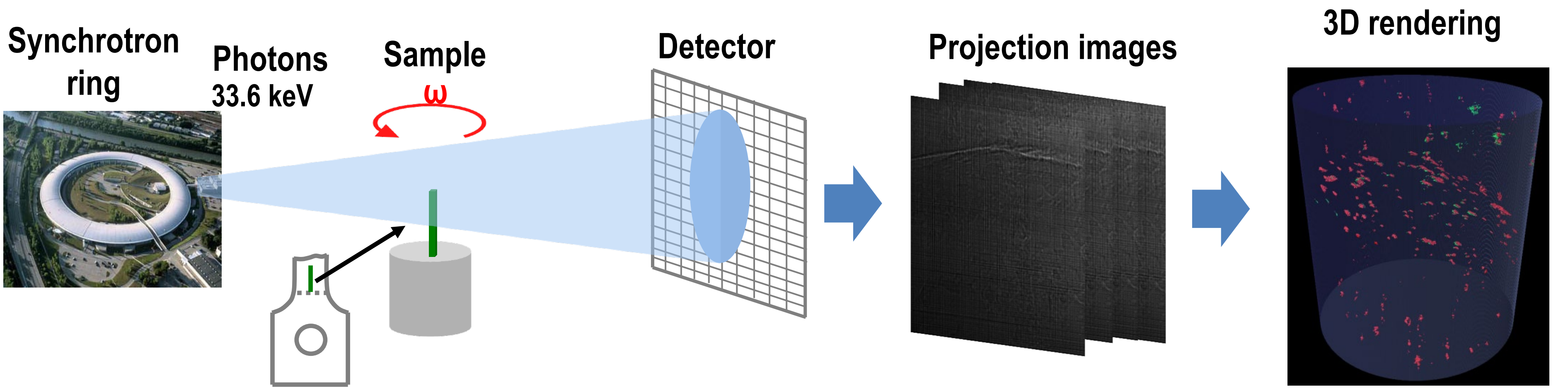
Creep tests of Fe-3.8 wt. % W



Samples for synchrotron X-ray nano-tomography

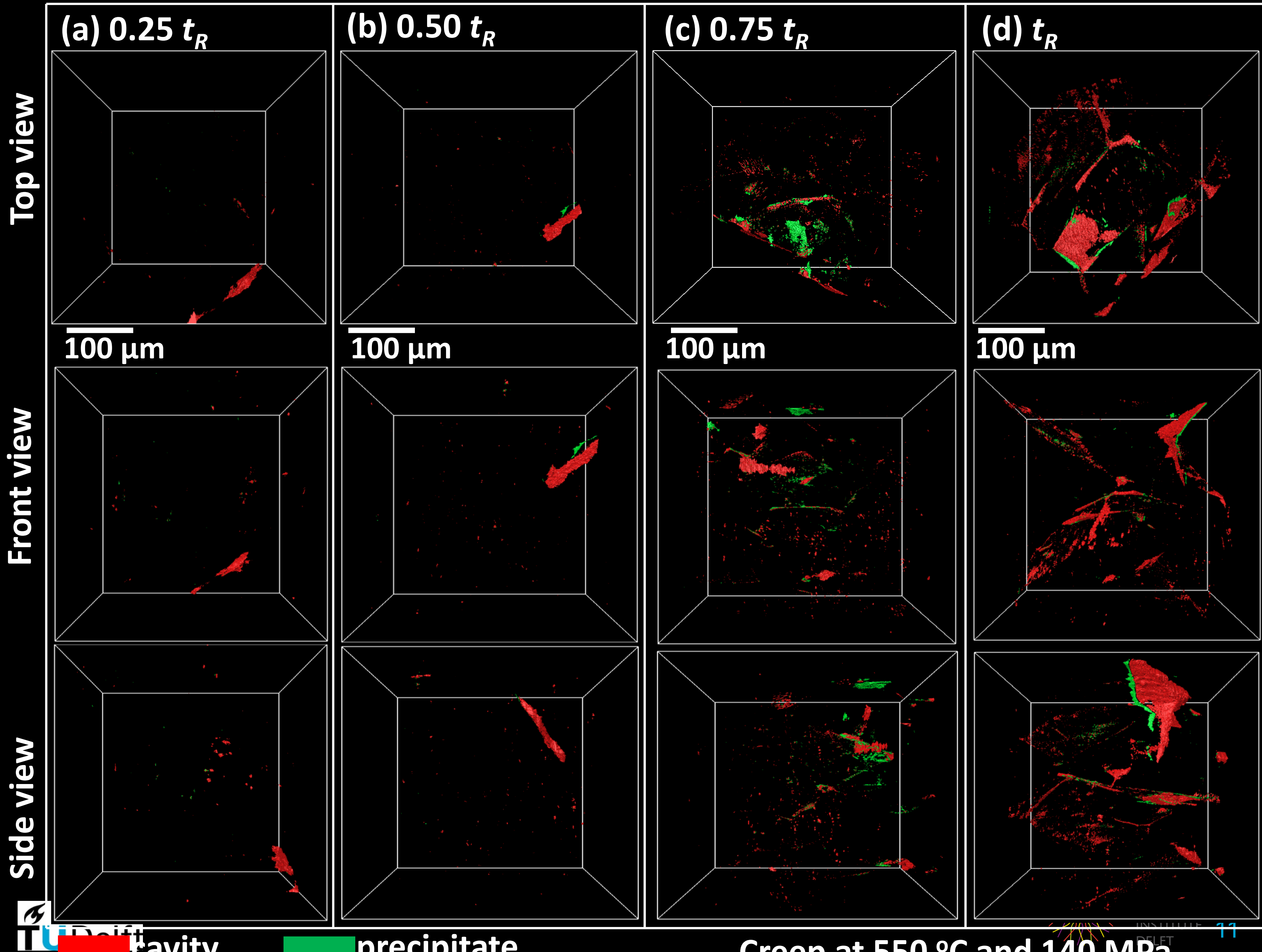
Sample	*T* (°C)	*σ* (MPa)	t_{creep} (h)	Scanning resolutions
S1	550	140	t_R = 236 h	100 nm & 30 nm
S2	550	140	$0.75t_R$ = 177 h	100 nm & 30 nm
S3	550	140	$0.5t_R$ = 118 h	100 nm & 30 nm
S4	550	140	$0.25t_R$ = 59 h	100 nm & 30 nm
S5	550	160	t_R = 104 h	100 nm & 30 nm
S6	550	160	$0.5t_R$ = 52 h	100 nm & 30 nm

Synchrotron X-Ray Tomography on Fe-4W alloy after creep tests



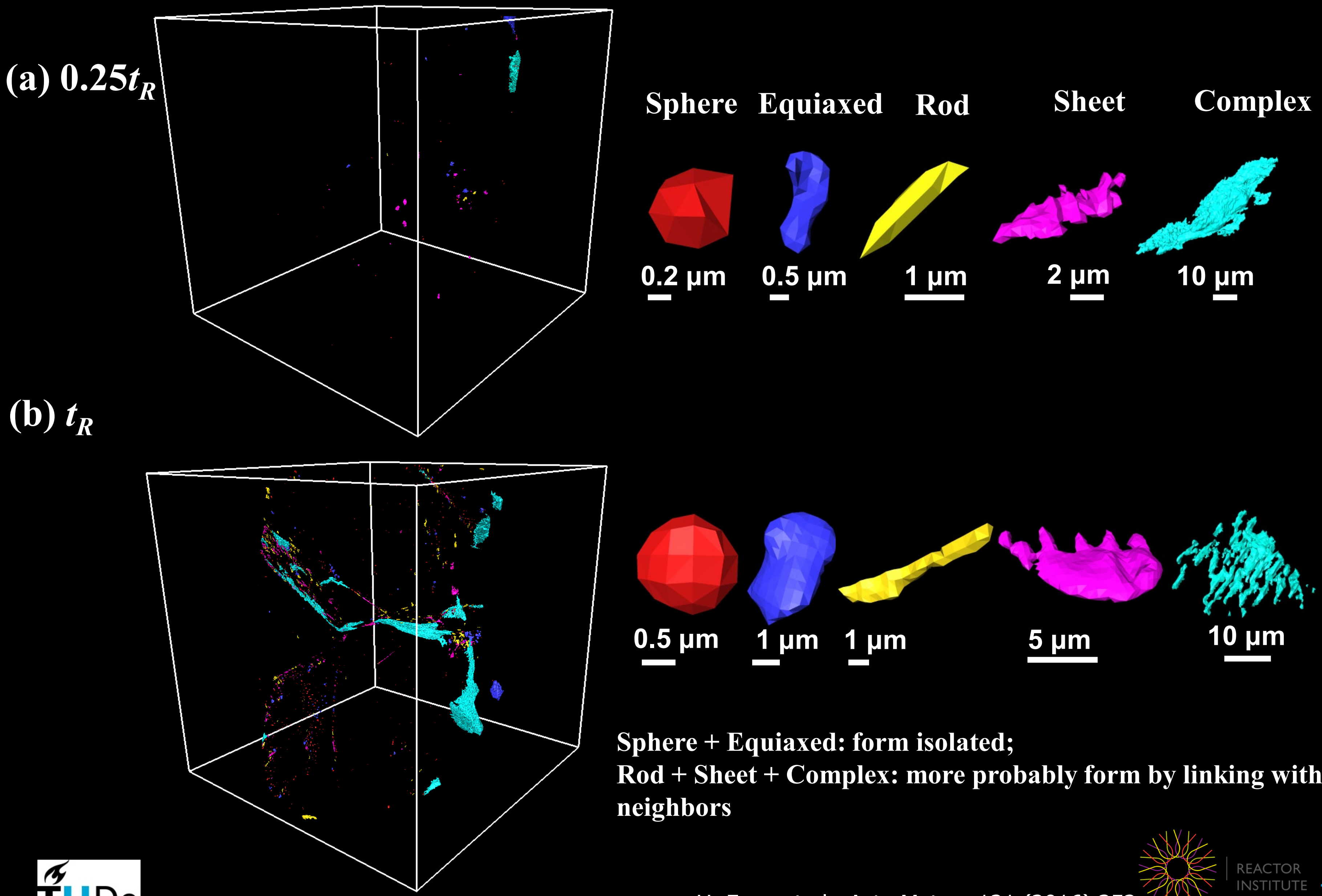
3D image with a nanometer (30 and 100 nm) resolution

- ESRF ID16A-NI nano-imaging beamline
- Exposure time: 1 s
- 1800 projections with 3216 × 3216 pixels
- One measurement renders 3216 slices with 3216 × 3216 pixels
- 3D rendering and visualization by FEI Avizo 8.1
- Voxel size: 100 nm and 30 nm



Creep at 550 °C and 140 MPa

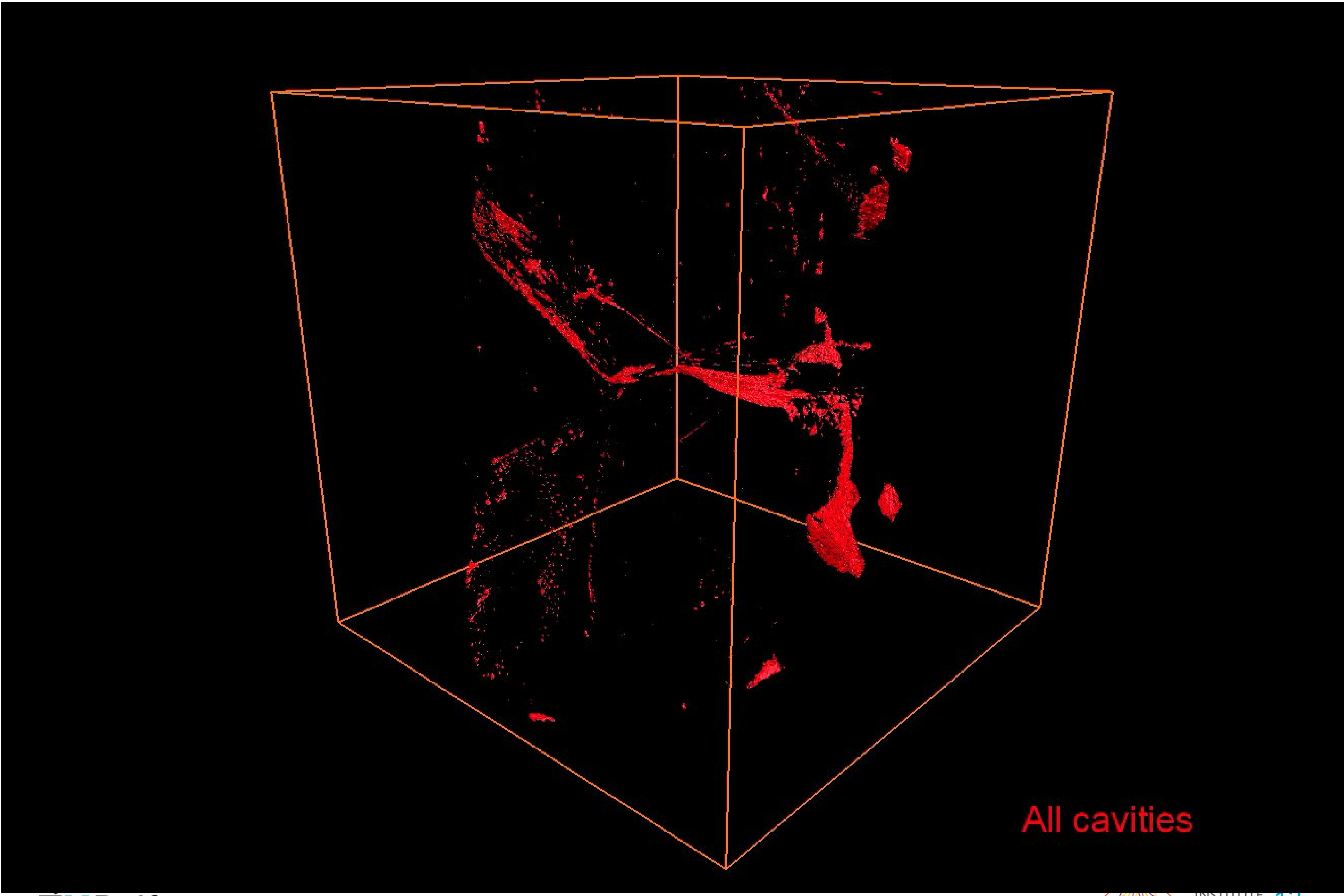
Identification of cavities in different shapes using complexity (Ω_3), elongation (E) and flatness (F)



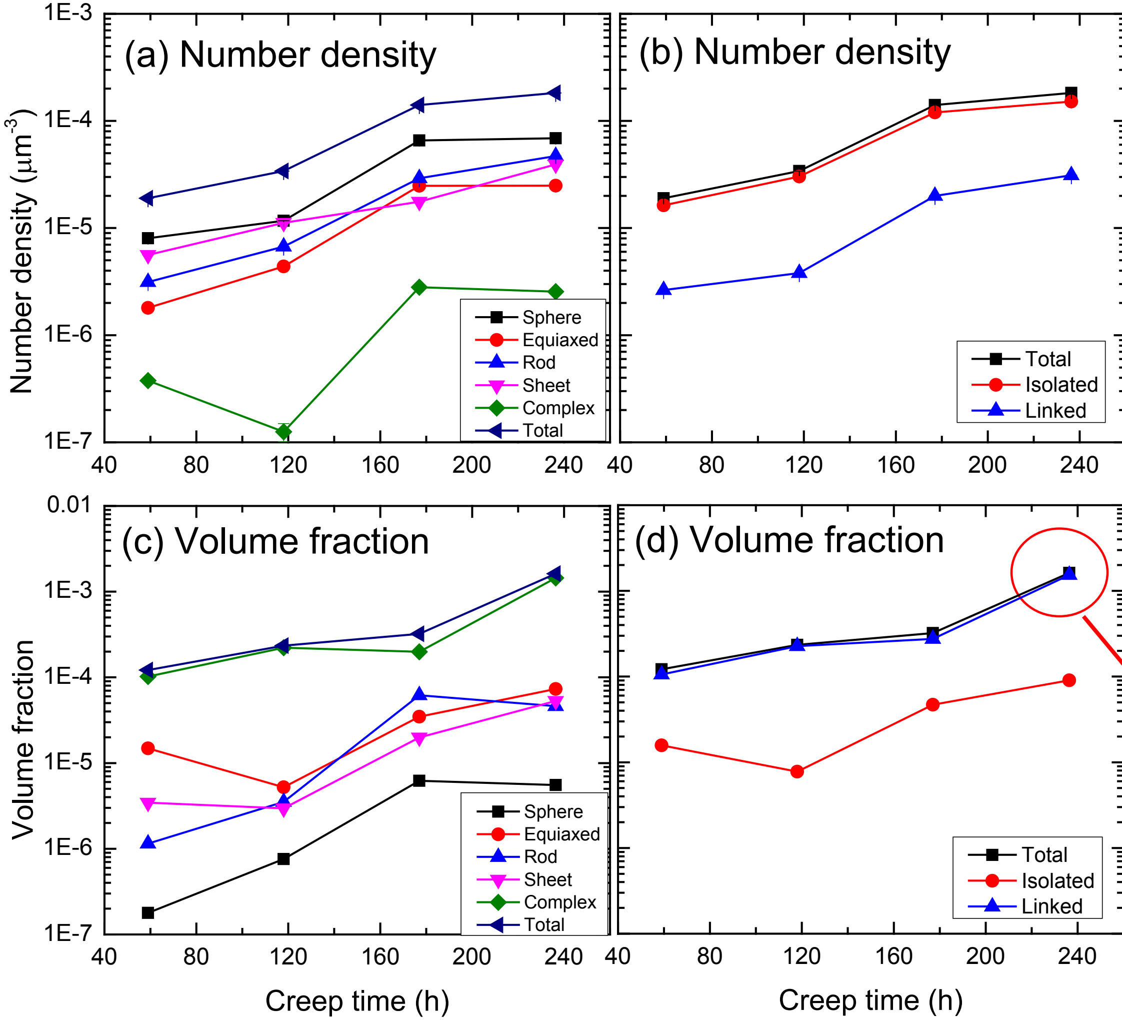
Sphere + Equiaxed: form isolated;
Rod + Sheet + Complex: more probably form by linking with neighbors

H. Fang et al., Acta Mater. 121 (2016) 352.

Movie showing shape classification of cavities



By shape classification and comparing cavity size to cavity spacing, we can identify isolated and linked cavities.



Linked cavities fulfill two criteria:

1) Shape is rod, sheet or complex

2) Major axes of cavity > λ_{cavity}

High number density of non-linked cavities

Low volume fraction of non-linked cavities

Coalescence of cavity increases by one magnitude at failure

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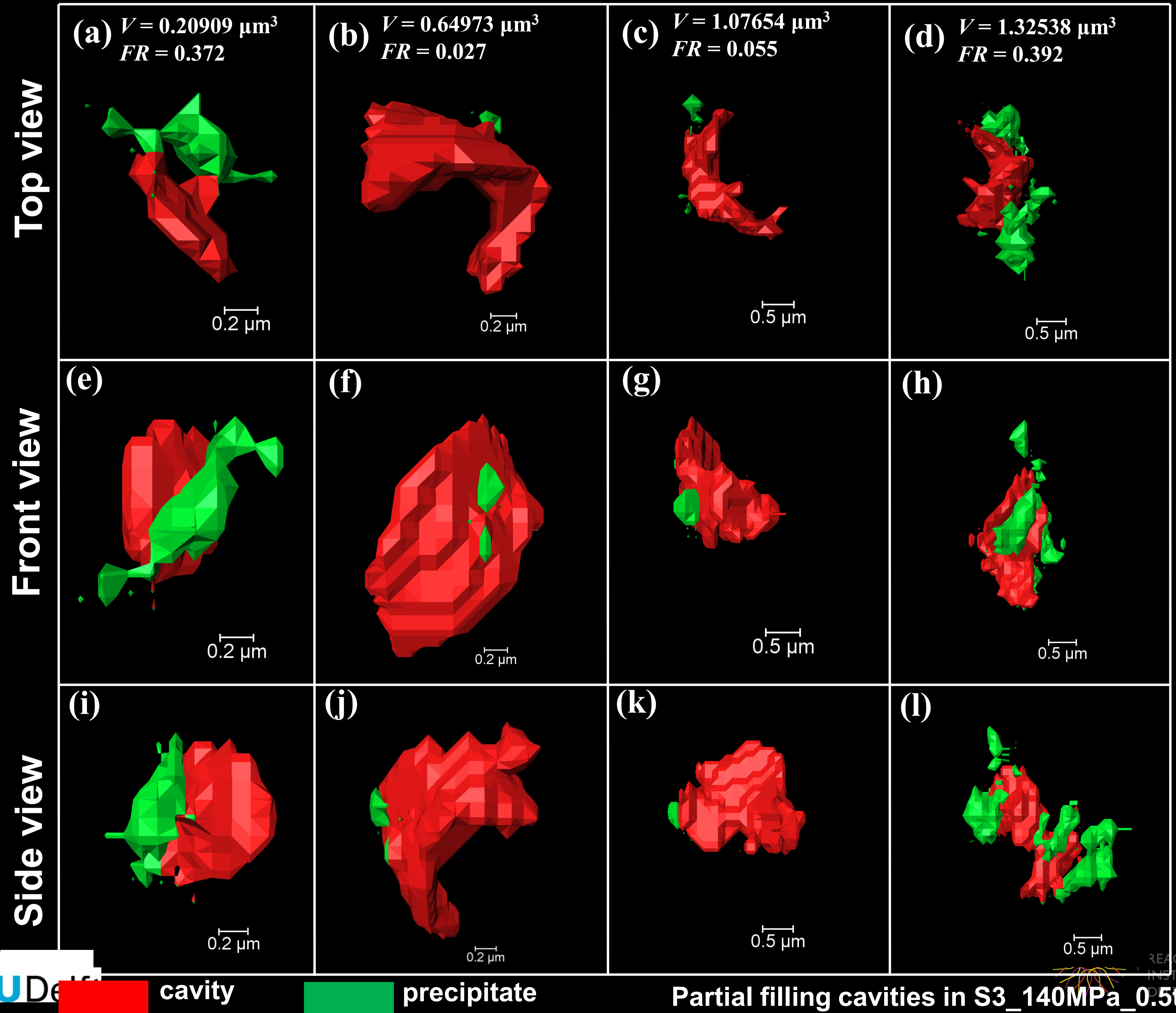
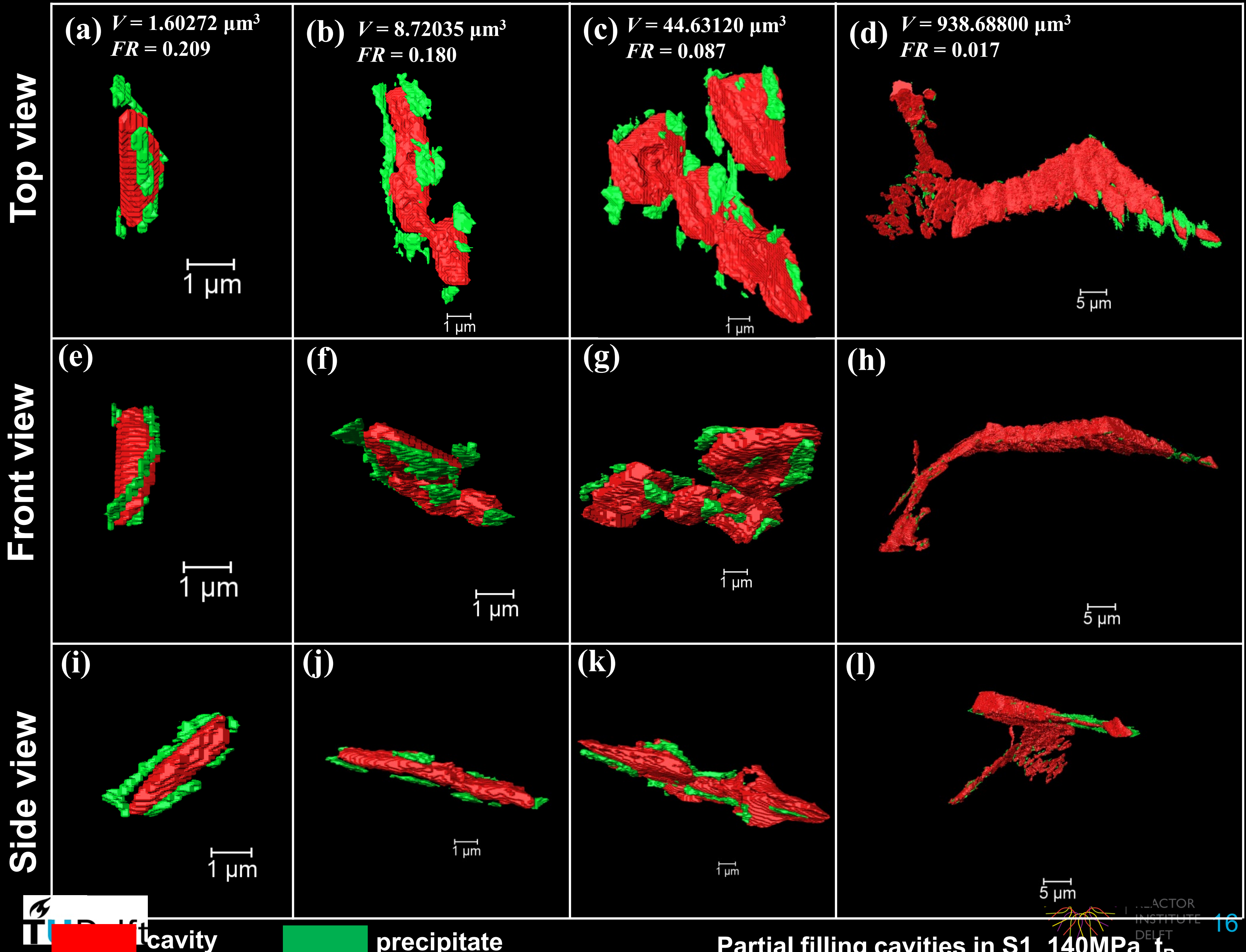
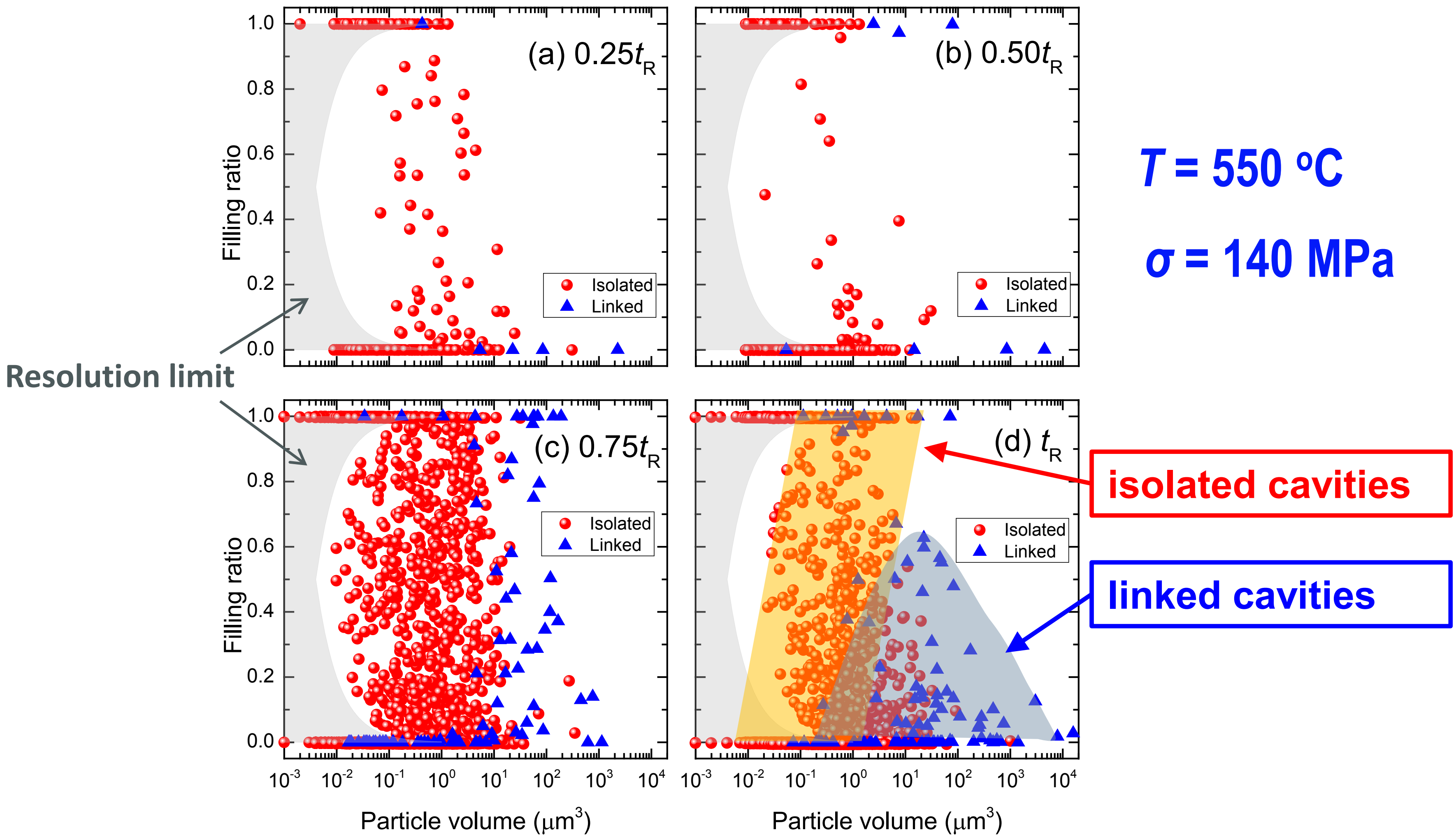


Partial filling cavities in S3_140MPa_0.5t_R



Partial filling cavities in S1_140MPa_t_R

Filling ratio as a function of particle (= cavity+precipitate) volume



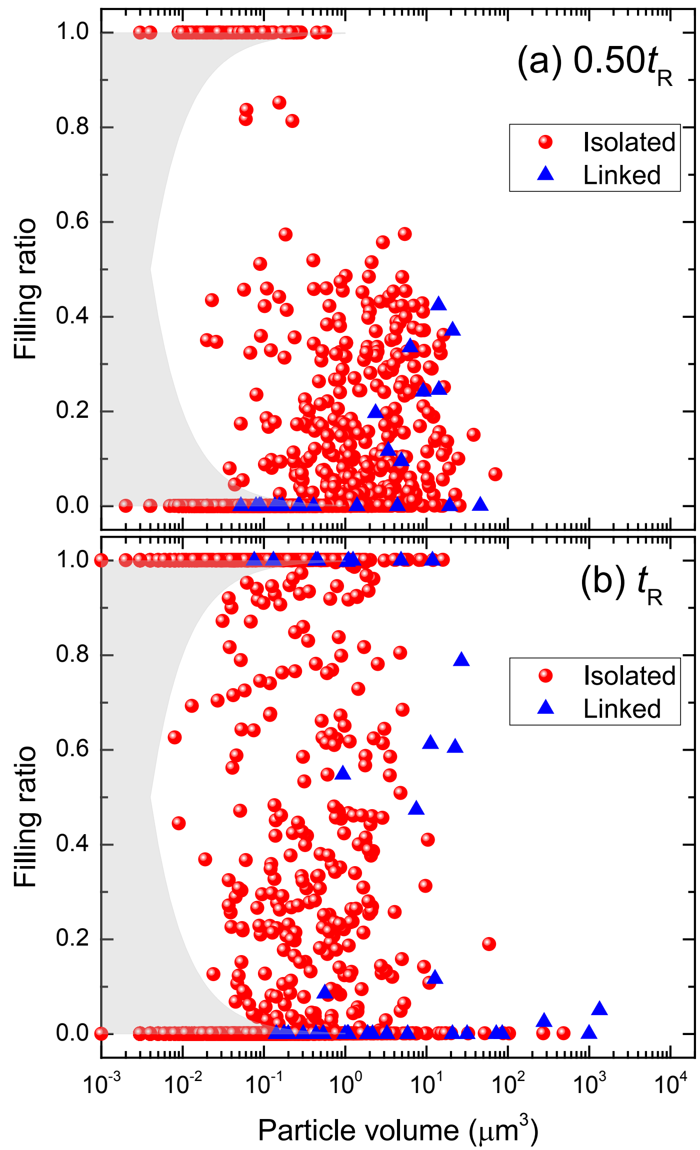
***T* = 550 °C**

***σ* = 140 MPa**

Yellow region: filling ratio continuously increases until fully filled.

Blue region: filling ratio increases to some extent and then drops down.

Filling ratio as a function of particle (= cavity+precipitate) volume



T = 550 °C

σ = 160 MPa

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Conclusions

- Creep cavities can be filled autonomously by precipitation of supersaturated solute in *bcc* iron matrix
- Self healing is most efficient in Fe-Au, but Fe-W provides a promising (and technologically more relevant) alternative for self healing alloys
- The filling behavior for isolated and linked cavities is distincly different
- X-ray nanotomography provides a unique insight in the damage formation and healing operation in these alloys

Aknowledgements

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