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Novel Co 20 Cr 15 Fe 26 Mn 17 Ni 22 ultra-fine grained high-entropy alloy

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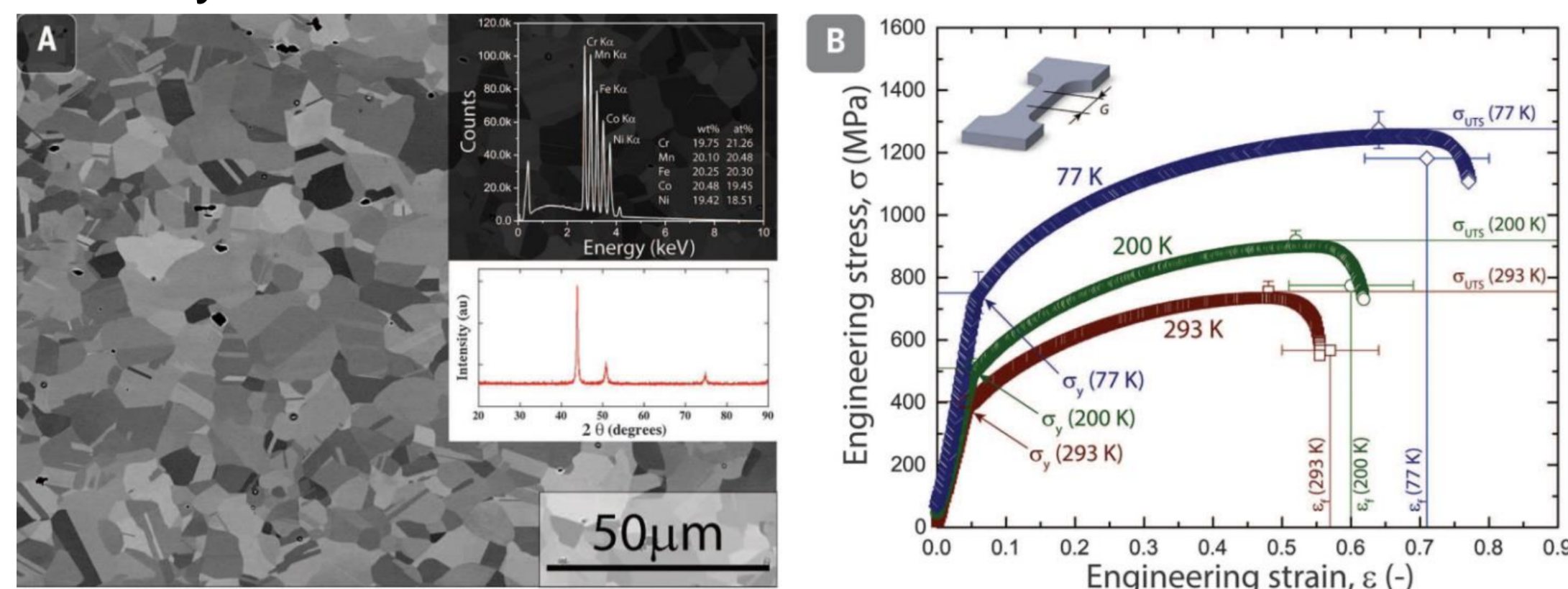
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Origins of the alloy

NOVEL COMPOSITION: based on equiatomic CoCrFeMnNi

- Reference alloy (Yeh, Cantor, 2004)
- Face-Centered Cubic structure (lattice parameter $a = 3.6\text{\AA}$)
- Yield strength of $\sim 240\text{MPa}$ (recrystallized) and $\sim 760\text{MPa}$ (cold-rolled)
- High ductility: $>60\%$

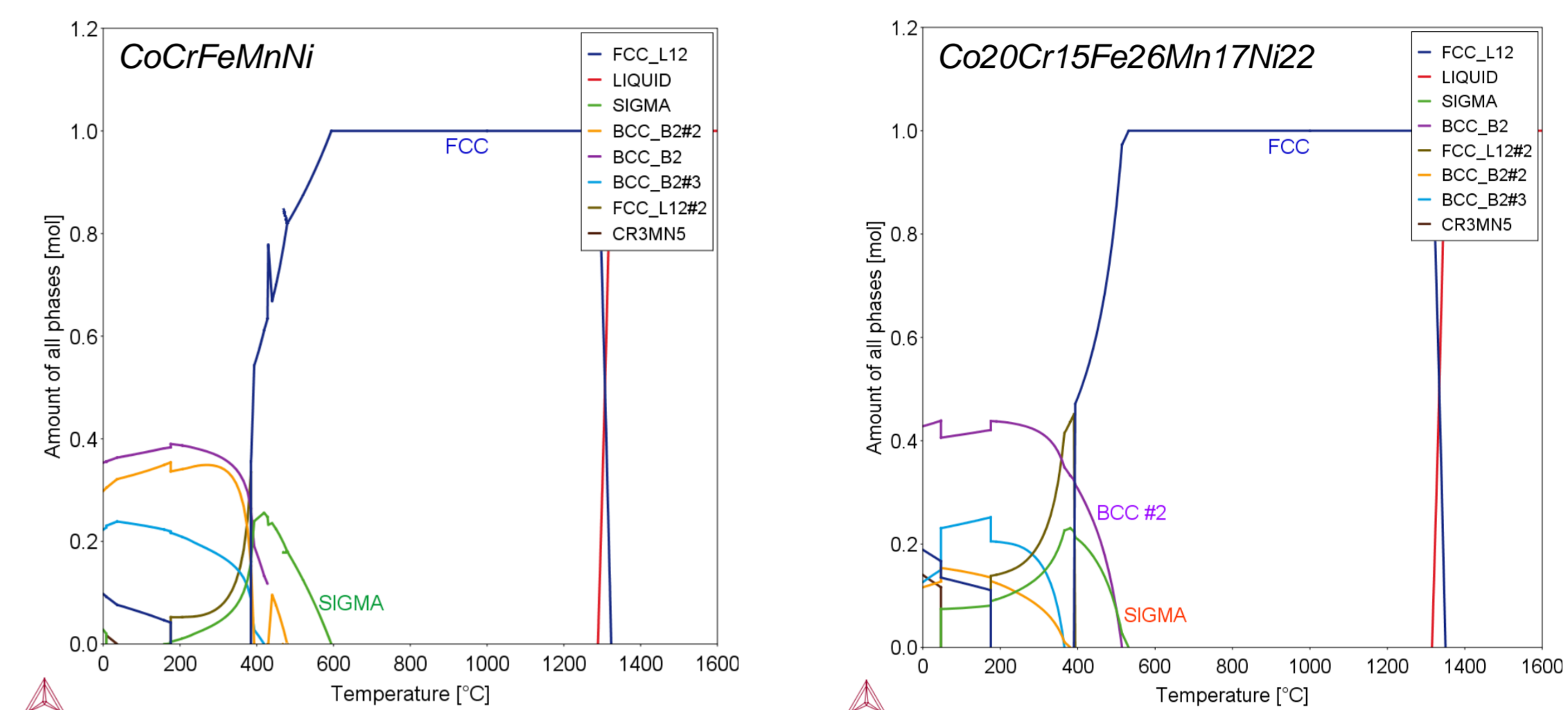


Recrystallized structure and stress-strain curves of CoCrFeMnNi, Gludovatz et al., 2014

- This composition was taken as a reference and optimized with Thermo-Calc software

THERMO-CALC : optimization of 'reference' equiatomic alloy

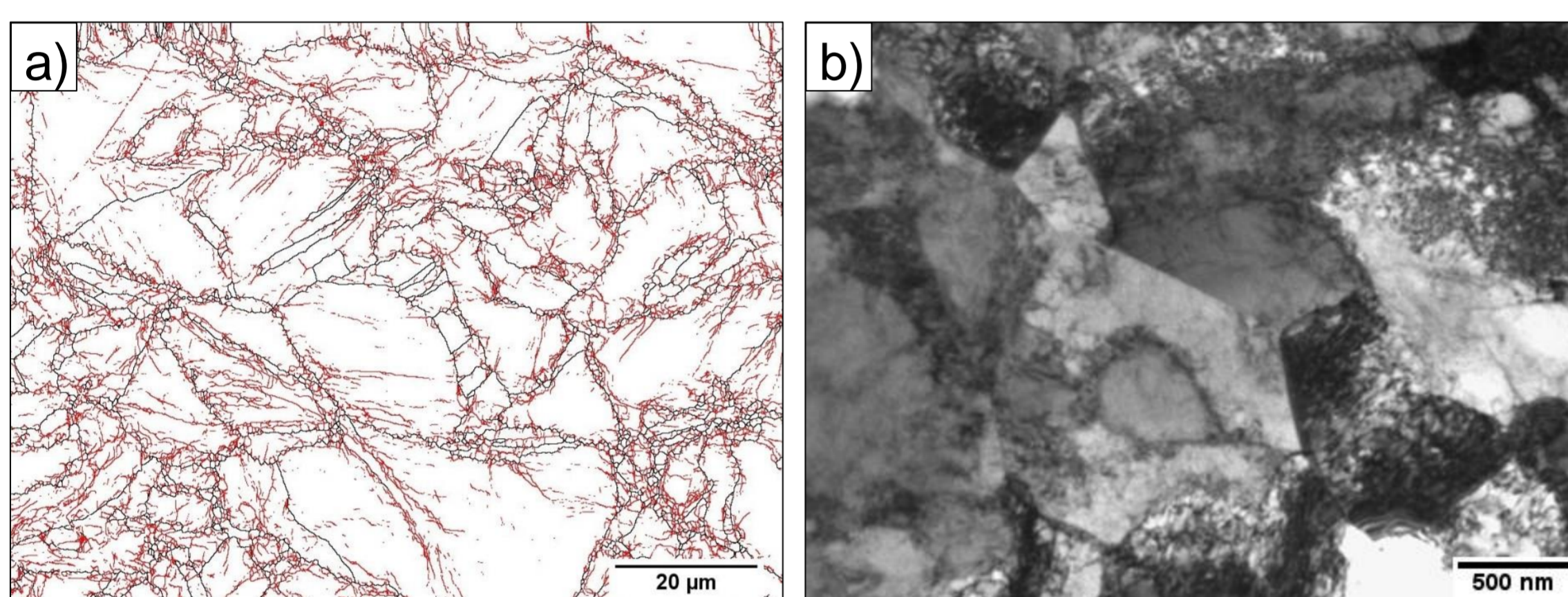
- FCC area increased to assure phase stability
- Intermetallics (especially σ phase) were avoided
- Amount of Cr decreased to lower temperature of phase transformations
- Amount of Ni increased (FCC stabilizing element)
- **Novel alloy called A3S® : austenitic super stainless steel**



Amount of phase vs temperature (HEA1 database)

Facility of nanostructuration

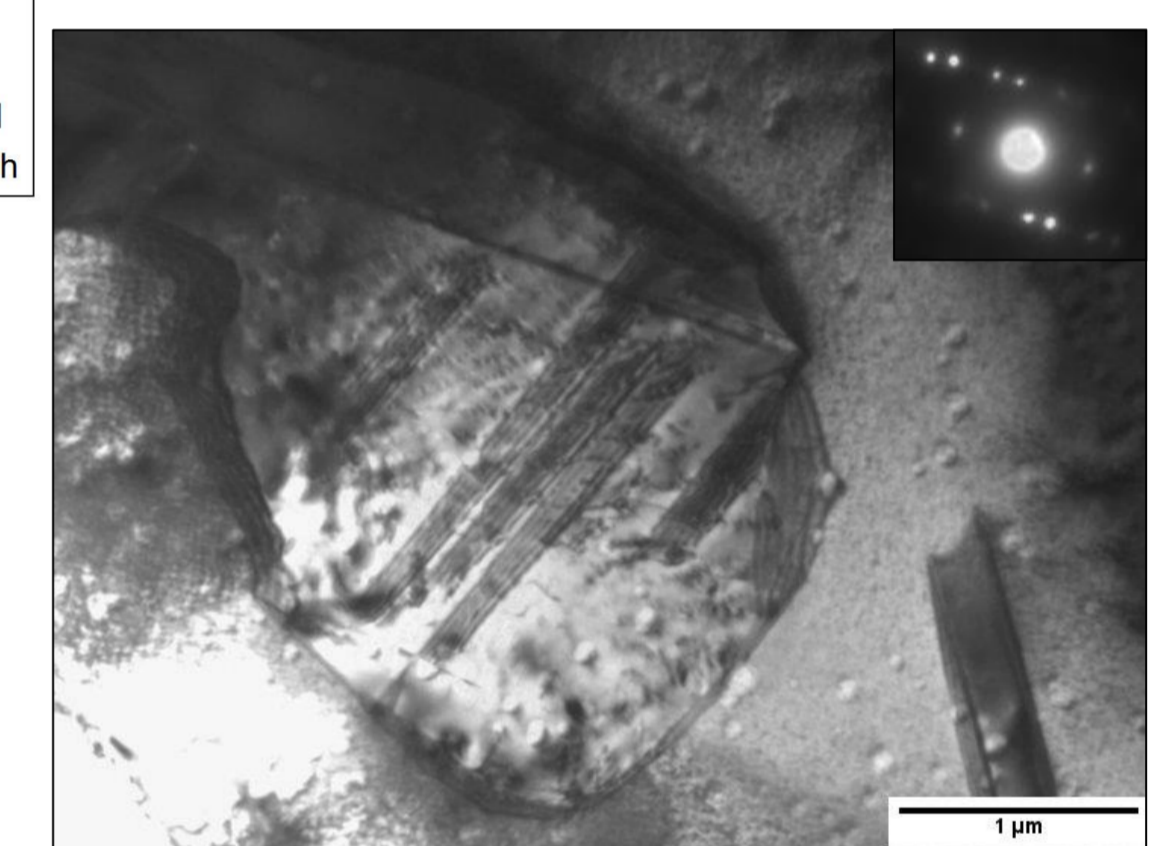
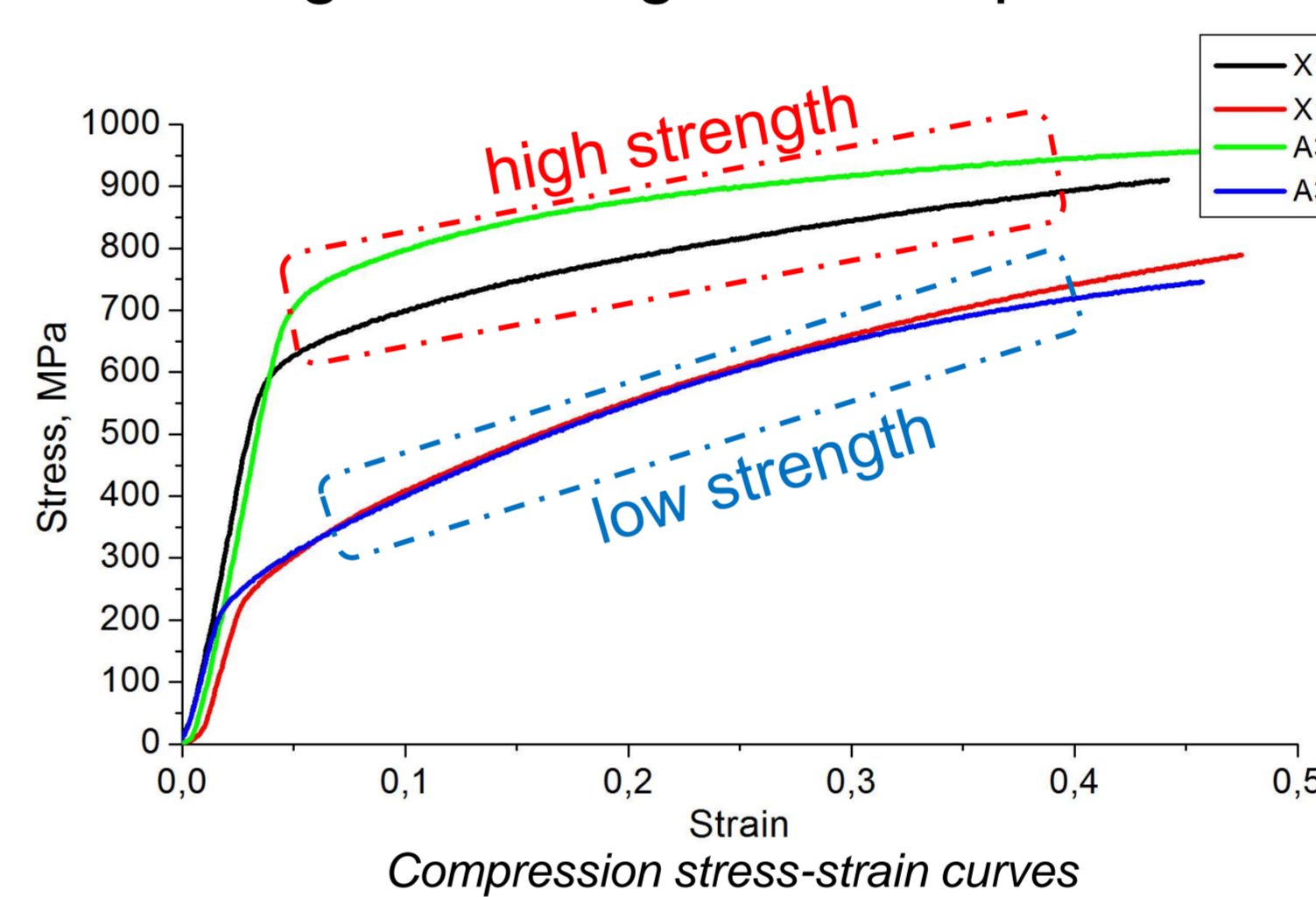
- After **hot forging**, nanostructure/UFG structure is formed in both alloys
- Many low-angle grain boundaries in grains, nanometric size of cells
- High density of dislocations



a) EBSD and b) TEM imaged of as-forged A3S

- After annealing ($1000^\circ\text{C}/2\text{h}$) : only high-angle grain boundaries, high density of twins

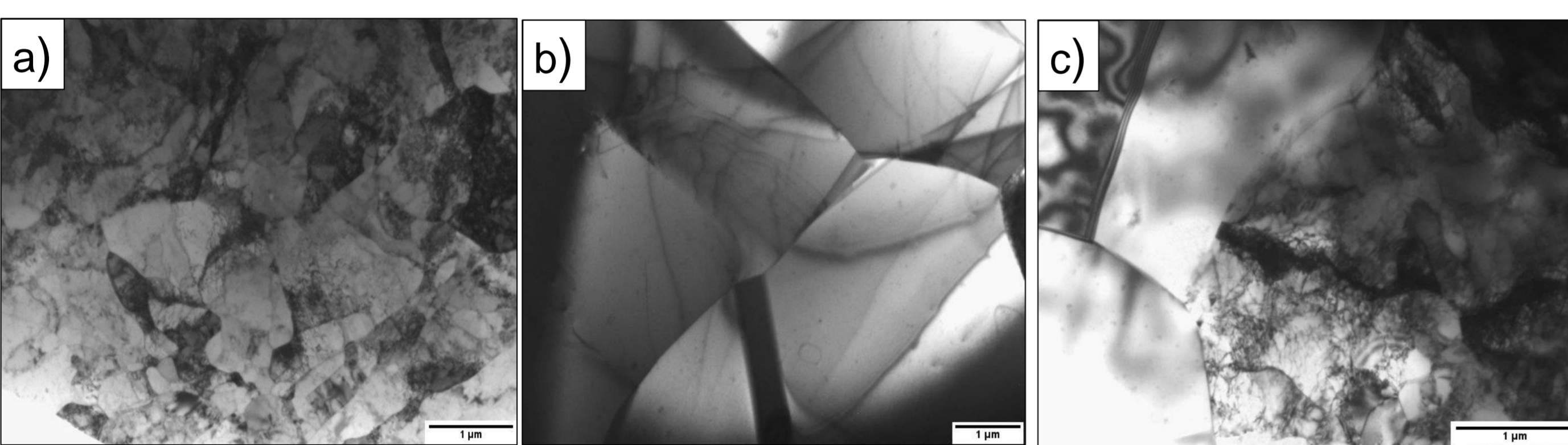
- Due to two different types of microstructure, two modes of deformation can be distinguished:
 - **high strength**: high ($\sim 750\text{MPa}$ for A3S and $\sim 600\text{MPa}$ for X1) yield strength, low work hardening coefficient
 - **low strength**: low ($\sim 250\text{MPa}$) yield strength, high work hardening coefficient
- A3S has higher strength than equiatomic alloy; in X1 - nanotwins



TEM bright field images of as-forged X1

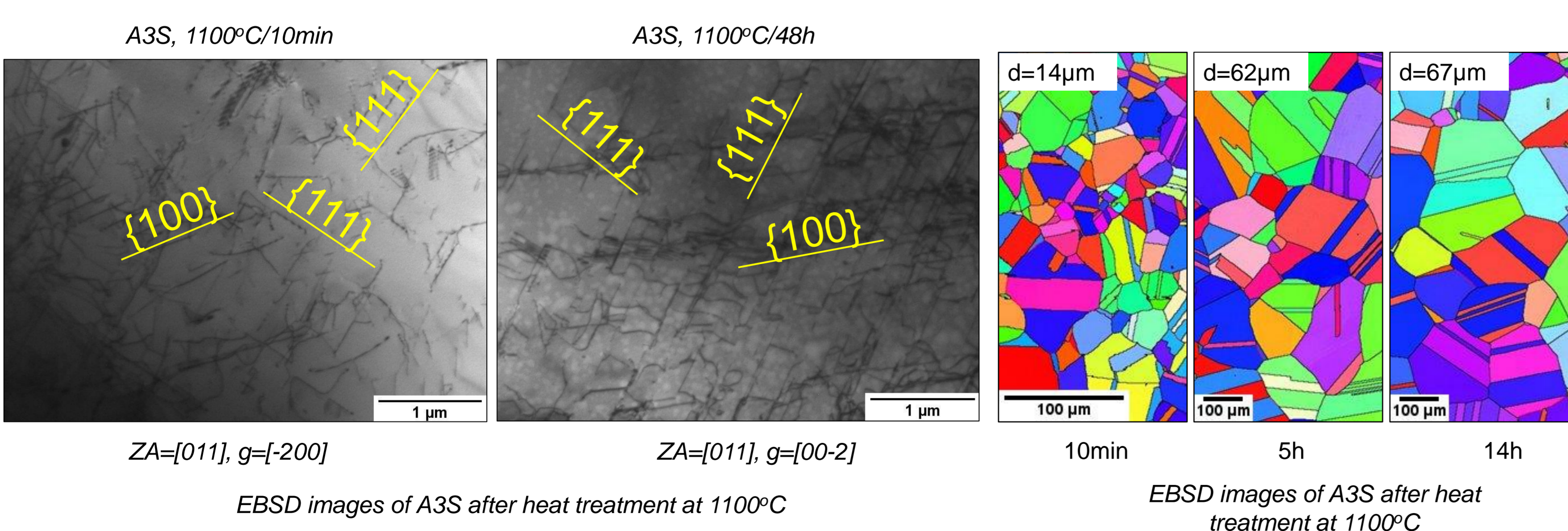
Recovery and recrystallization phenomena

- Nanostructure is stable until $\sim 700^\circ\text{C}$, when the alloy loses its high strength properties
- Even after long heat treatment at 600°C (1 month), grains with dislocations cells are still present



TEM bright field images of A3S annealed at: a) $600^\circ\text{C}/48\text{h}$, b-c) $600^\circ\text{C}/1\text{ month}$

- High density of dislocations even after high T annealing ($1000, 1100^\circ\text{C}$)
- Dislocations traces in characteristic planes: $\{111\}$ and $\{100\}$

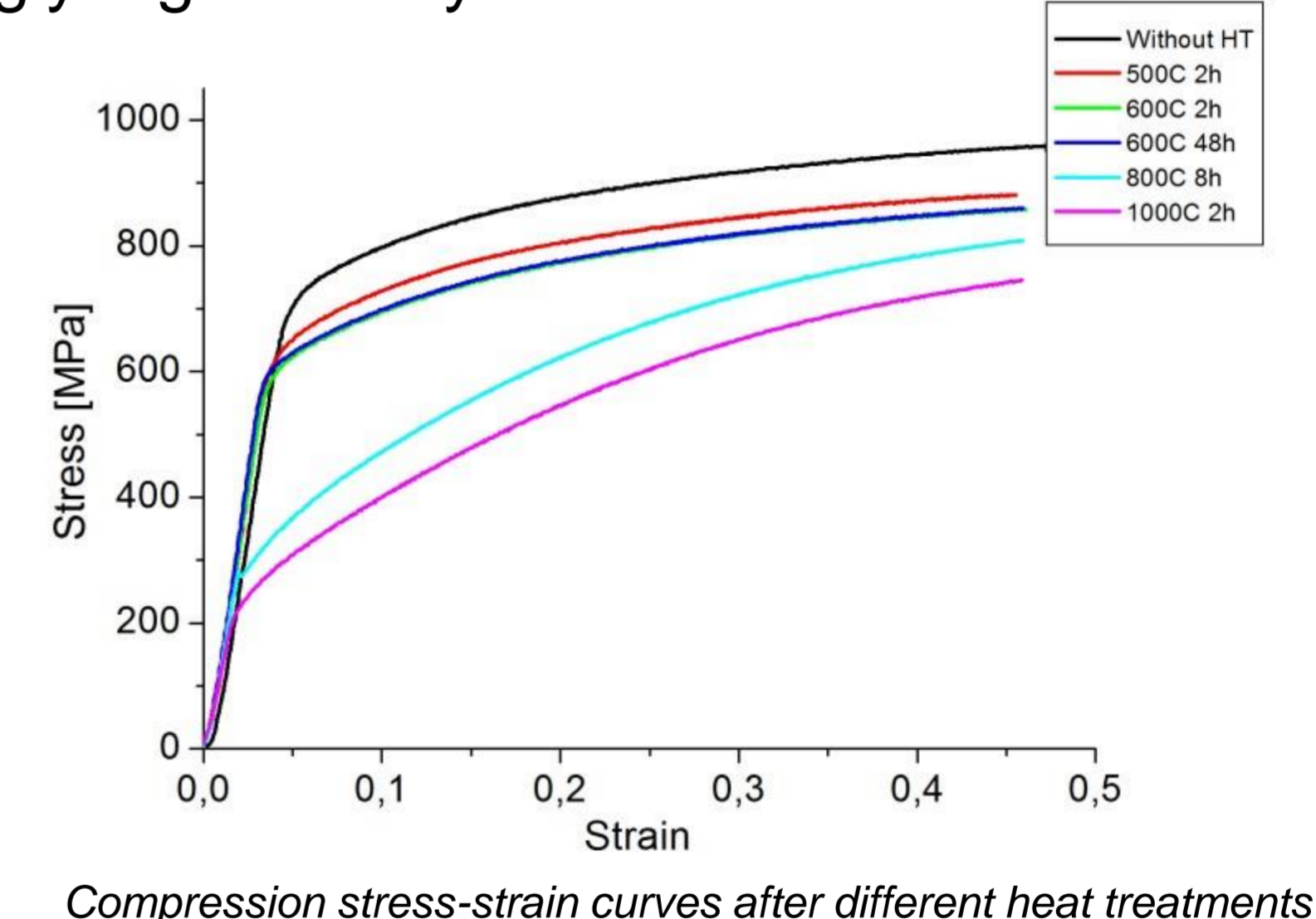


EBSD images of A3S after heat treatment at 1100°C

EBSD images of A3S after heat treatment at 1100°C

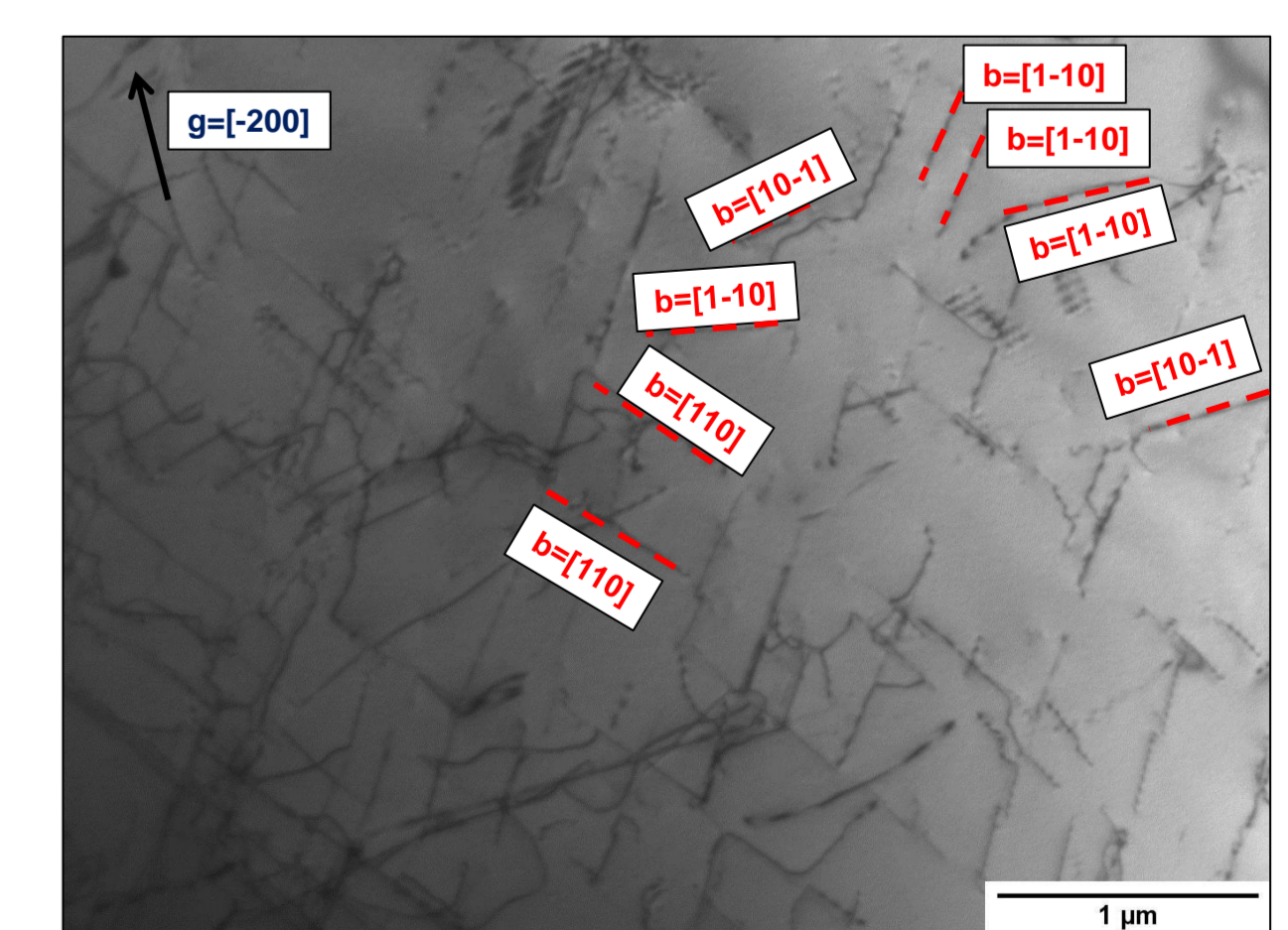
- Series of heat treatments at 1100°C revealed no recrystallization phenomena
- Only recovery occurs at high temperatures
- Dislocations locking may be due to nanofluctuations of composition

- After $800^\circ\text{C}/8\text{h}$ annealing there are no substructures, grains with HAGB of few μmeters
- Surprisingly high density of dislocations



Compression stress-strain curves after different heat treatments

- Typical Burger vectors of type $\langle 110 \rangle$ were observed
- Common for FCC metals



TEM bright field image of A3S/ $1100^\circ\text{C}/10\text{ min}$

CONCLUSIONS

- New original composition A3S® was developed starting from equiatomic CoCrFeMnNi high-entropy alloy
- Comparing to reference composition, **much higher (+150MPa) yield strength** is achieved with **similar elongation**
- Easy formation of **nanostuctures** after hot forging
- Stable UFG structure until $\sim 700^\circ\text{C}$
- Untypical phenomena of recovery/recrystallization:
 - grains free of dislocations after low T annealing
 - high density of dislocations after high T annealing