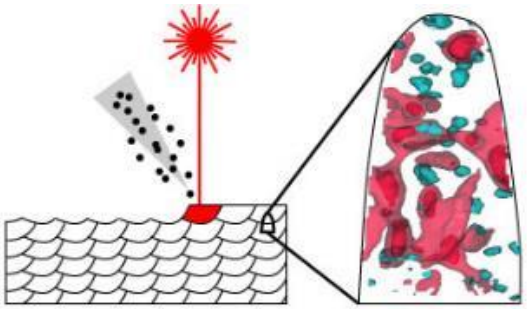
Small variations in powder composition lead to strong differences in part properties

Insights from an Al-Sc alloy and a Ni-base superalloy

Max-Planck-Institut für Eisenforschung GmbH



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2016-07-05

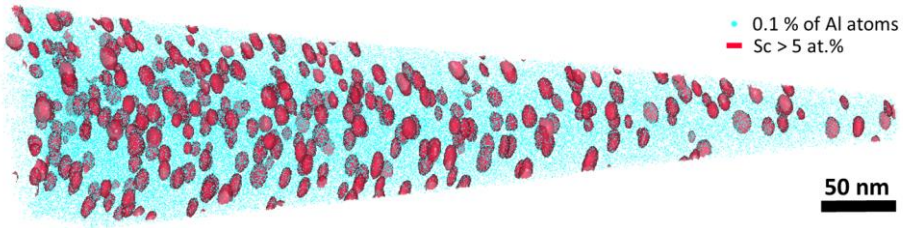
Department Microstructure Physics and Alloy Design

Alloys for Additive Manufacturing Group

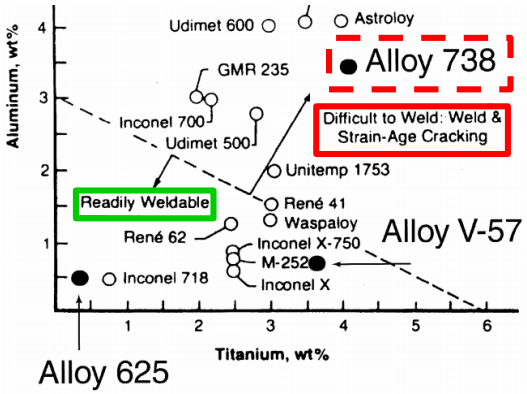
www.mpie.de/aam

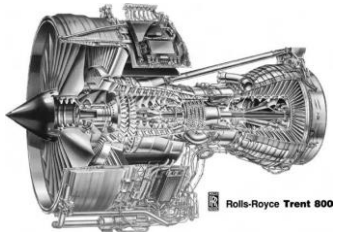
Outline

1. Strength variations in an Al-Sc alloy (Scalmalloy®):



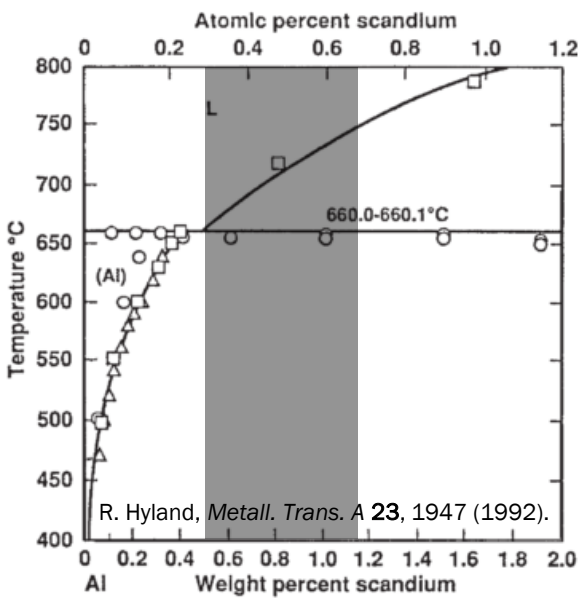
2. Hot cracking in a Nickel-base superalloy: Inconel 738LC

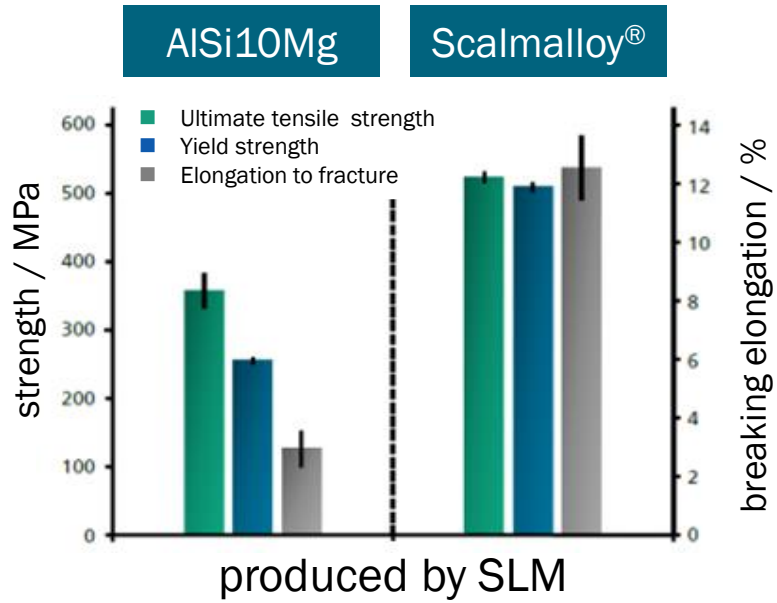


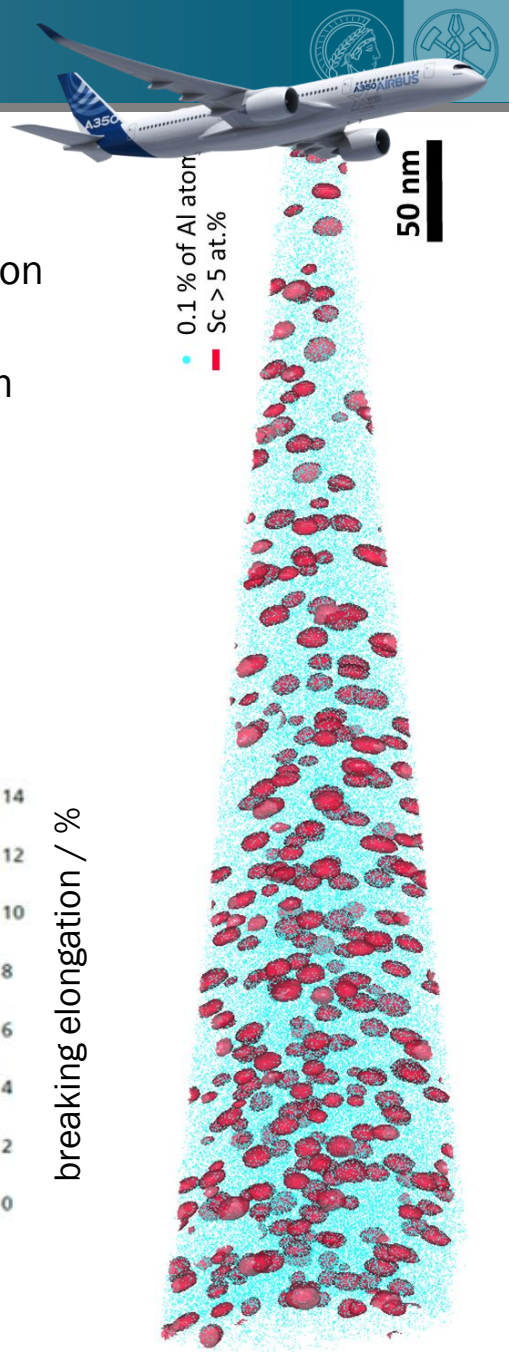


Aluminium-Scandium Alloys

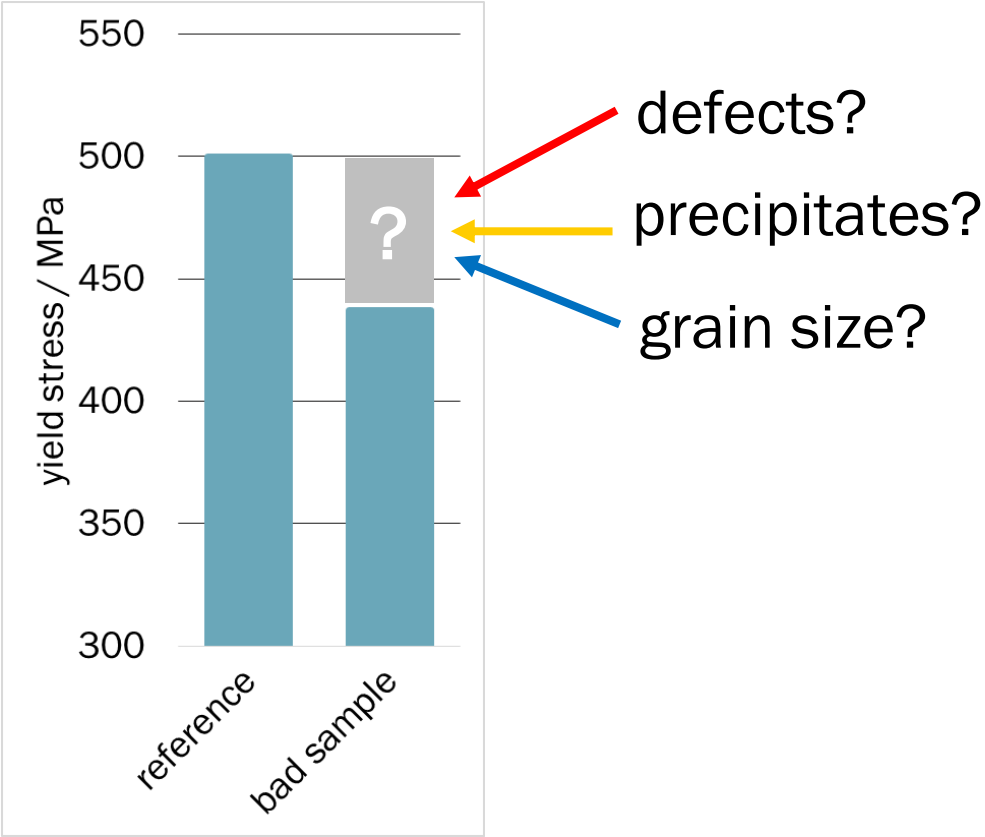
- **Strengthening** achieved **by precipitation** of $Al_3(Sc,Zr)$ nanoparticles upon **ageing heat treatment**
- Scalmalloy®: AlMgScZr-alloys with **hypereutectoid** content of scandium developed by Airbus
- **Rapid quenching** necessary to retain supersaturation:
  melt spinning + hot compaction or LAM can be employed







The problem – unexpected drop in strength



- Same powder composition
- Similar process parameters
- Different powder atomization supplier
- Different SLM-machine vendor

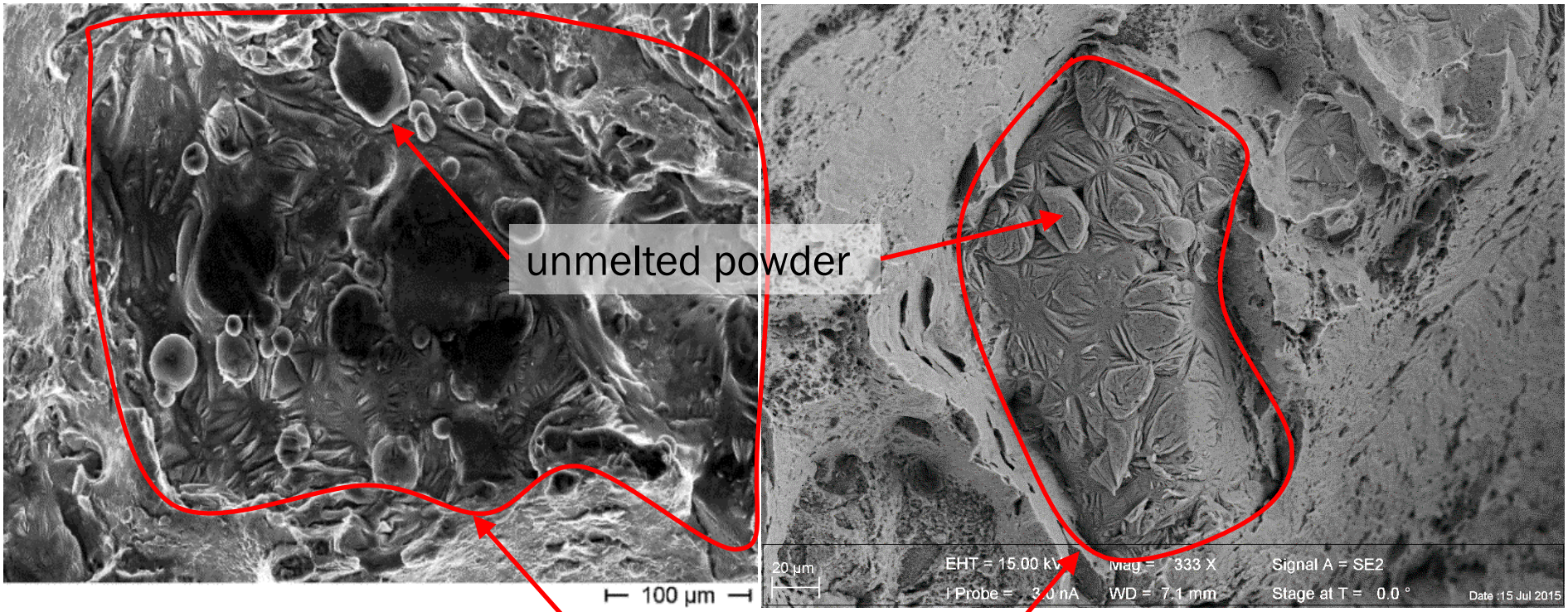
Sample overview – low ductility and strength?

SAMPLE	2	3	5
Powder atomization	VIGA	EIGA	EIGA
SLM machine	Concept Laser	Concept Laser	SLM Solutions
Post-heat treatment	HT	HT + HIP	HT
Yield strength / MPa	520	437	395
Ductility / %	14	17	7.4

EIGA: Electrode Inert Gas Atomization / VIGA: Vacuum Inert Gas Atomization
HT: Heat treatment (4 h@ 325°C)
HIP: Hot Isostatic Pressing (2h @ 325°C)

Low ductility – bonding defects on fracture surf.

As-produced	HIPed

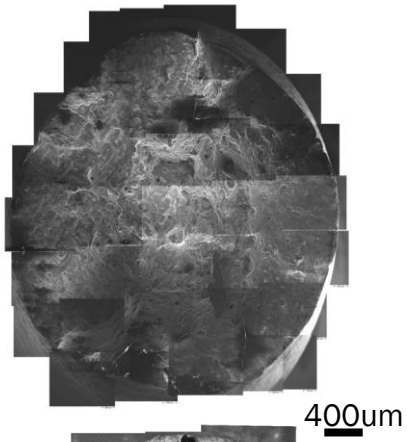


bonding defects

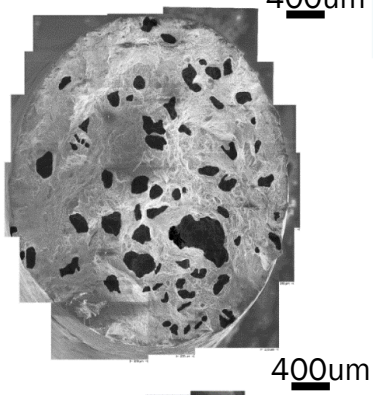
- Unmelted powder particles and bonding defects in samples produced by the SLM Solutions machine (samples 4 – 6)
- Bonding defects are not healed by HIPing!

Low ductility – bonding defects on fracture surf.

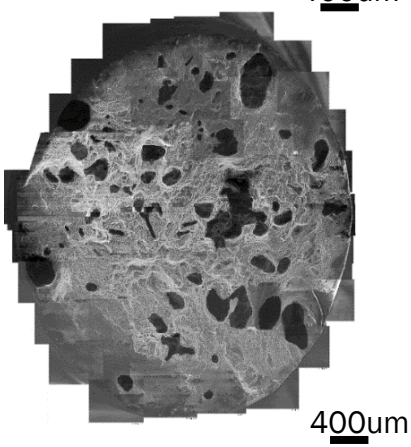
3



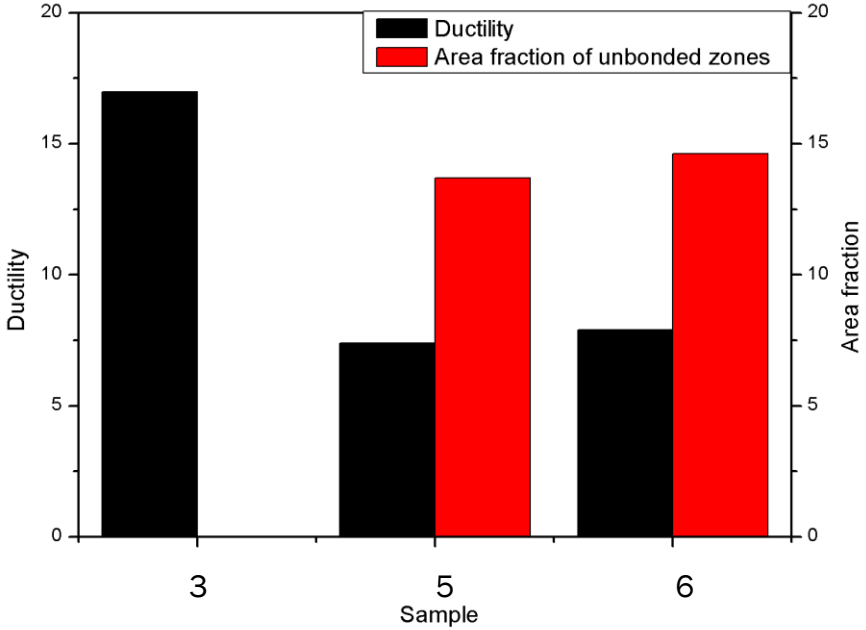
5



6

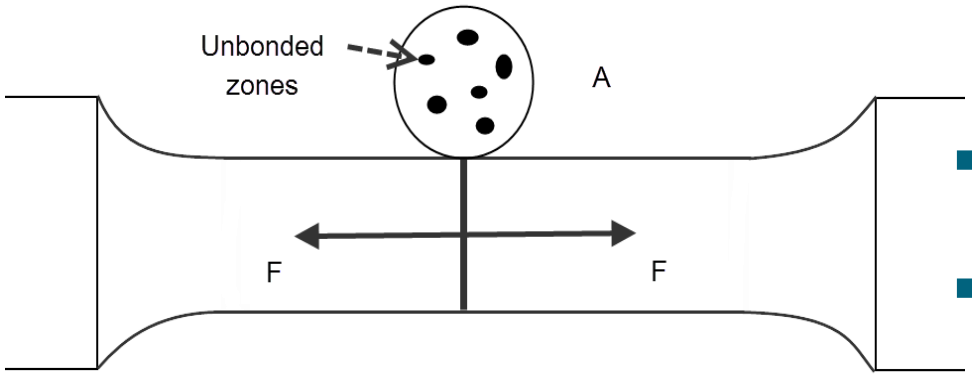


SAMPLE	3	5	6
Powder	EIGA	EIGA	EIGA
machine	Concept laser	SLM Solutions	SLM Solutions
heat treatment	HT + HIP	HT	HT + HIP
ductility	↑	↓	↓
yield strength	↓	↓	↓
Defect area fraction	Close to 0%	13.68%	14.62%



- Bonding defects are the reason for low ductility
- They cannot explain low yield strength (cf. sample 3)

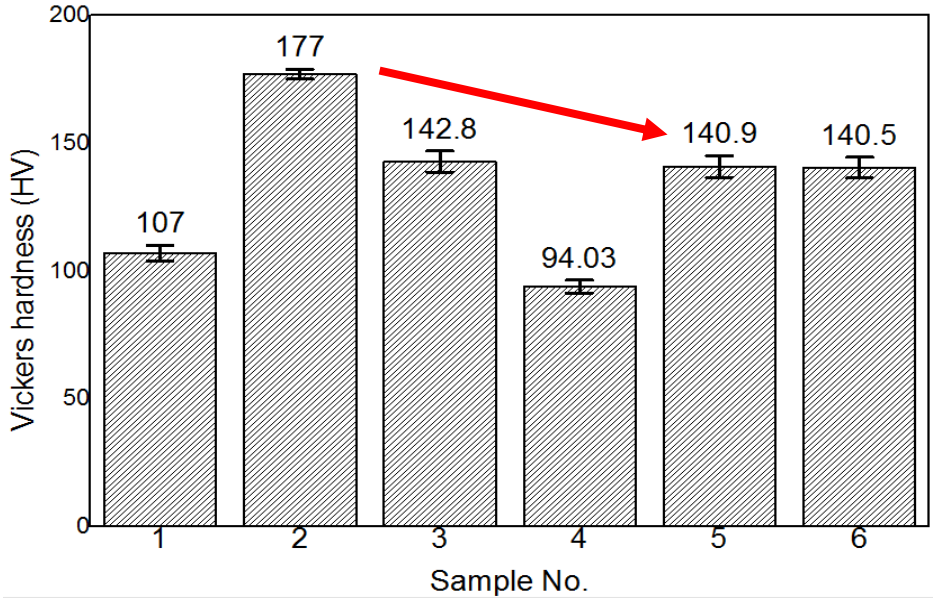
Influences of unbonded zones on yield stress



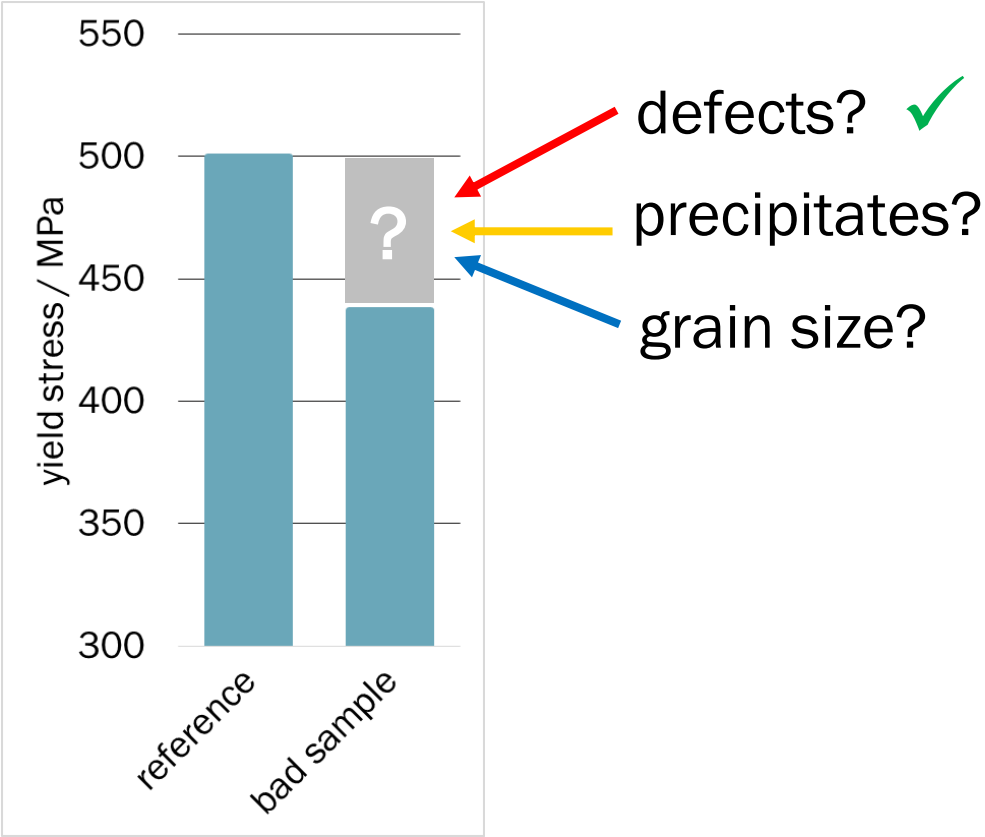
- Unbonded zones lead to a reduction of load-bearing area in cross section
- Decrease of **apparent** yield stress

But hardness measurement unaffected by bonding defects!

- bonding defects can't explain drop in hardness

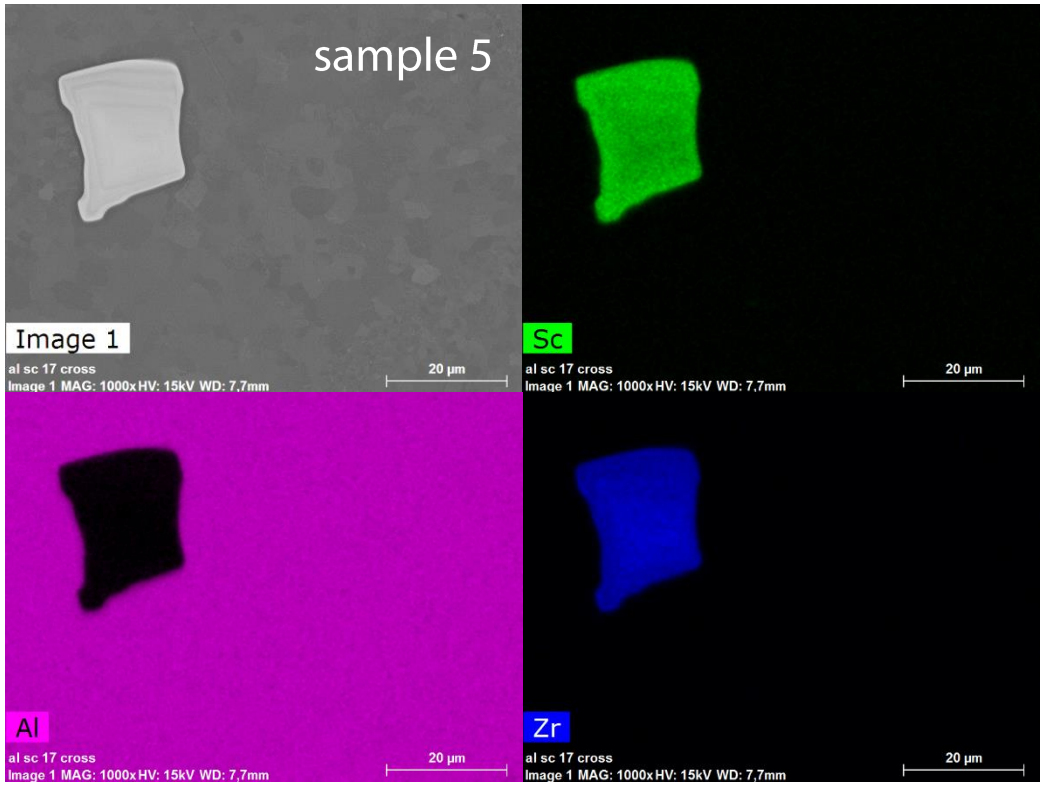


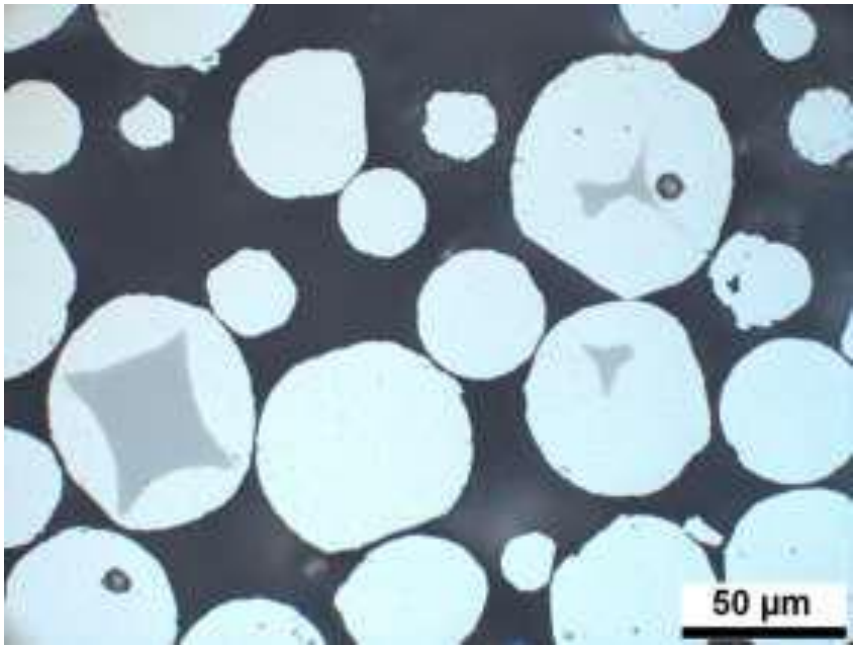
The problem – unexpected drop in strength



- Same powder composition
- Similar process parameters
- Different powder atomization supplier
- Different SLM-machine vendor

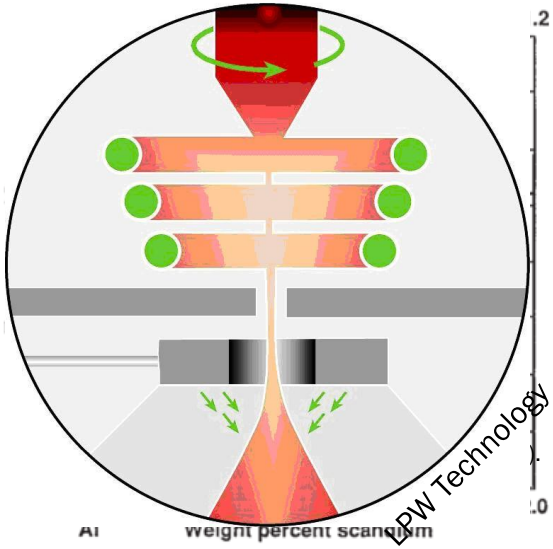
Coarse precipitates in AP-material and powder





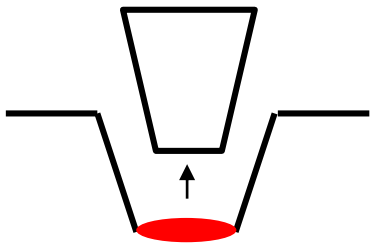
courtesy of Airbus Defense and Space

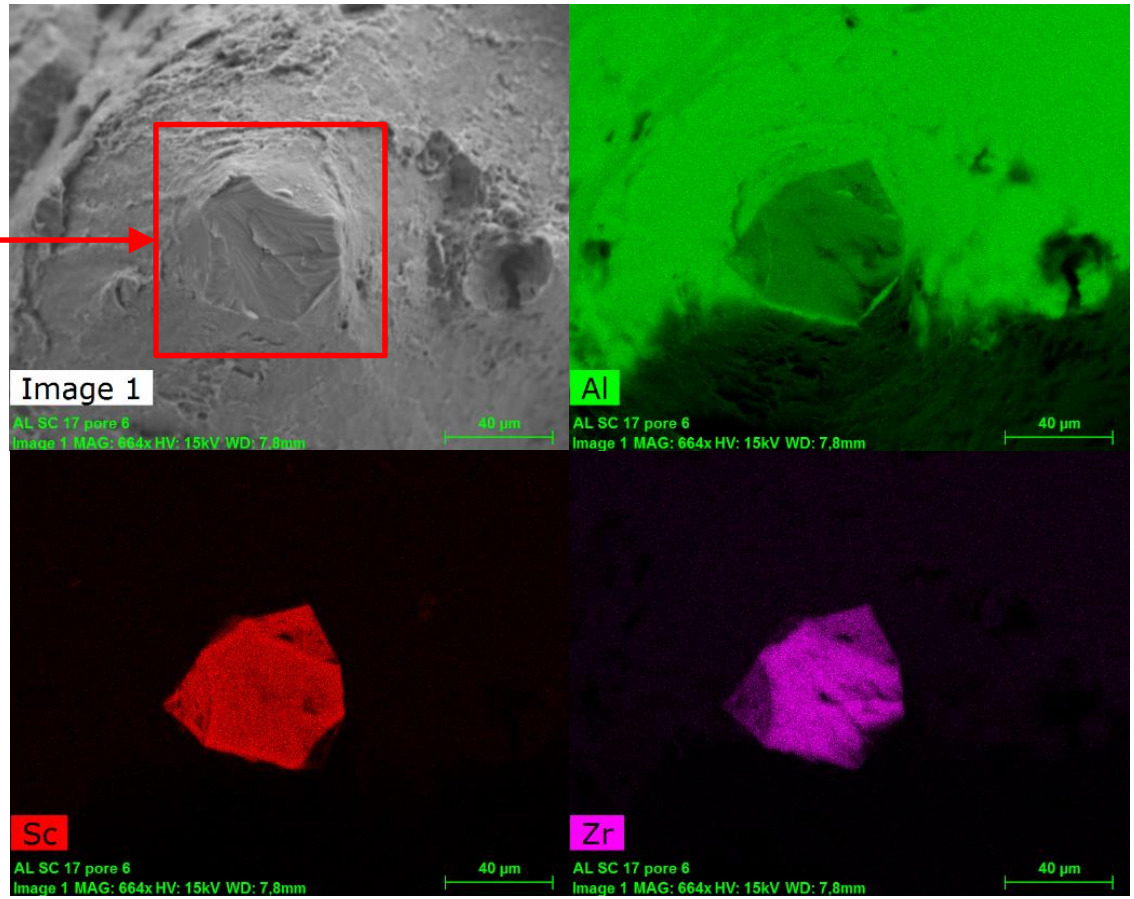
- Large precipitates (20-50 µm diameter) in the SLM-produced parts are identified by EDX as $Al_3(Sc,Zr)$
- They are **only** present in powder atomized by the EIGA process.
- Presumably the time in the liquid state is not long enough to dissolve them.



Large precipitates: low strength, but high ductility

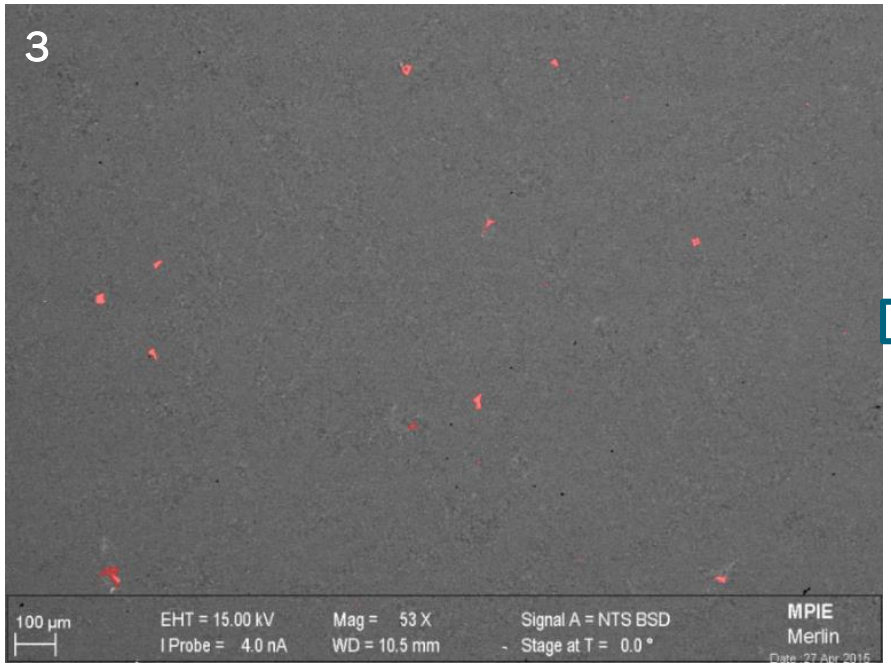
SAMPLE	2	3	5	6
Atomisation	VIGA	EIGA	EIGA	EIGA
Y. strength / MPa	520	437	395	410
Ductility/ %	14	17	7.4	7.9





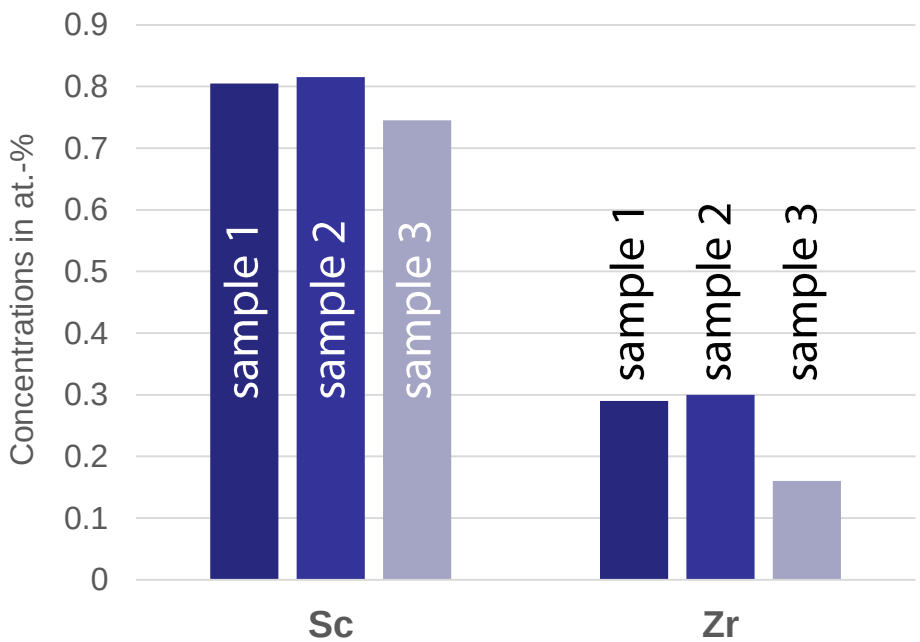
- Large precipitates apparently don't deteriorate ductility!
- Cup-and-cone fracture around large precipitates
- Maybe they decrease strength by "consuming" solute, which then cannot lead to nm-size precipitates?

Influences of unexpected precipitates



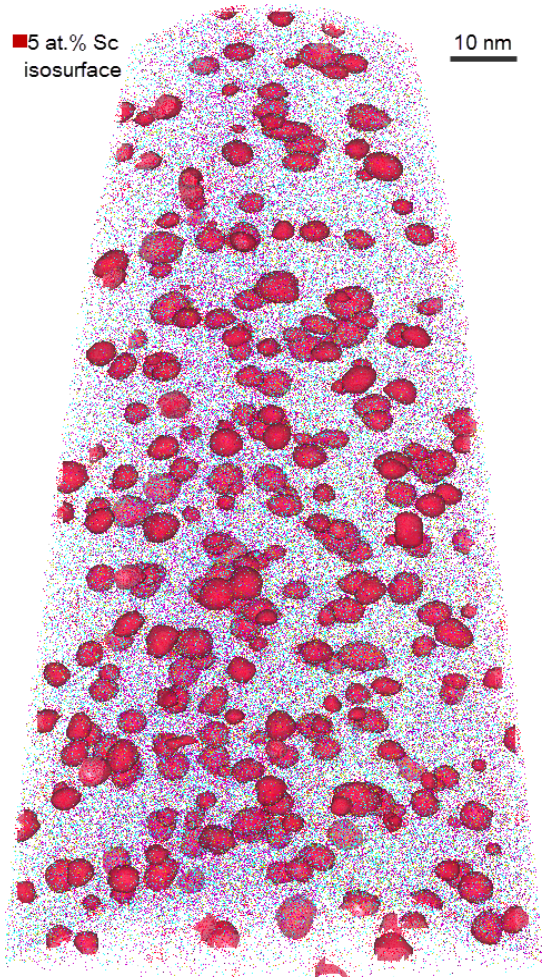
	Matrix	Precipitates
Area fraction	99.69%	0.31%

Elements	nominal composition	composition sample 3	change
Mg	4.50	4.52	+0.02
Al	93.87	94.02	+0.15
Si	0.17	0.17	0.00
Sc	0.66	0.57	-0.09
Mn	0.50	0.50	0.00
Zr	0.30	0.23	-0.07

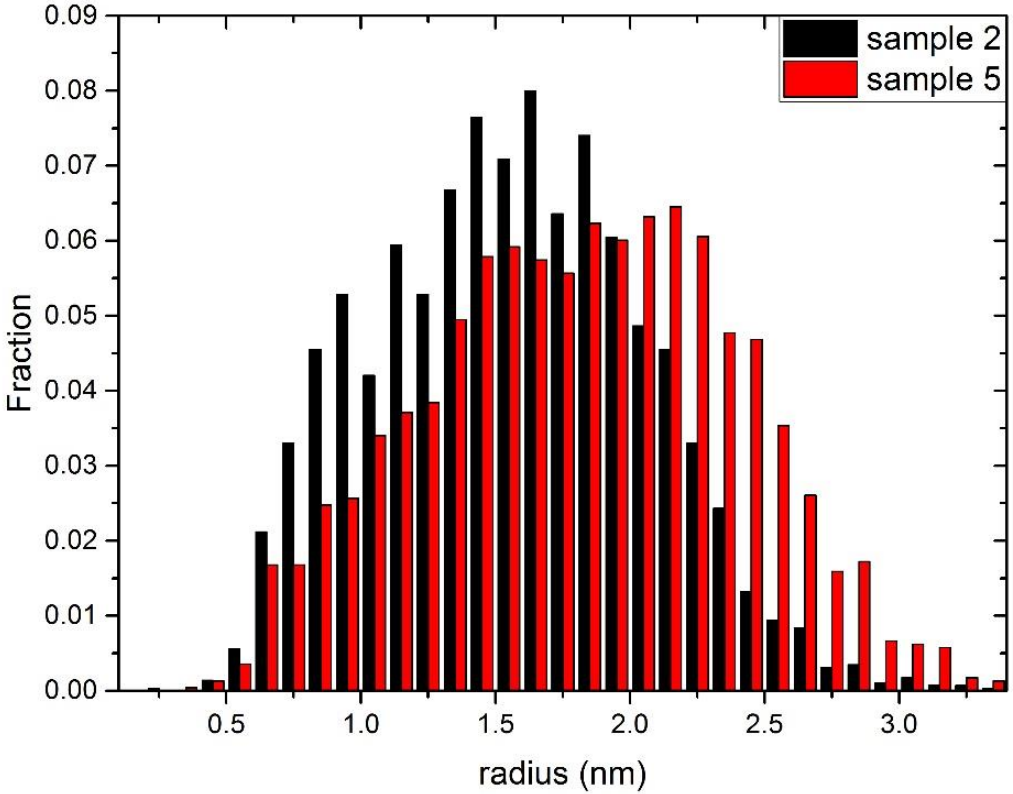


- Large precipitates only "consume" about 0.1% of Sc!
- Does this explain the remaining difference in yield strength?

Atom Probe Tomography of Al_3Sc



Sample No.	2	5
Number density ($\times 10^{23}/m^3$)	7.06	4.98
Average radius (nm)	1.57	1.80
volume fraction	~2%	~2%

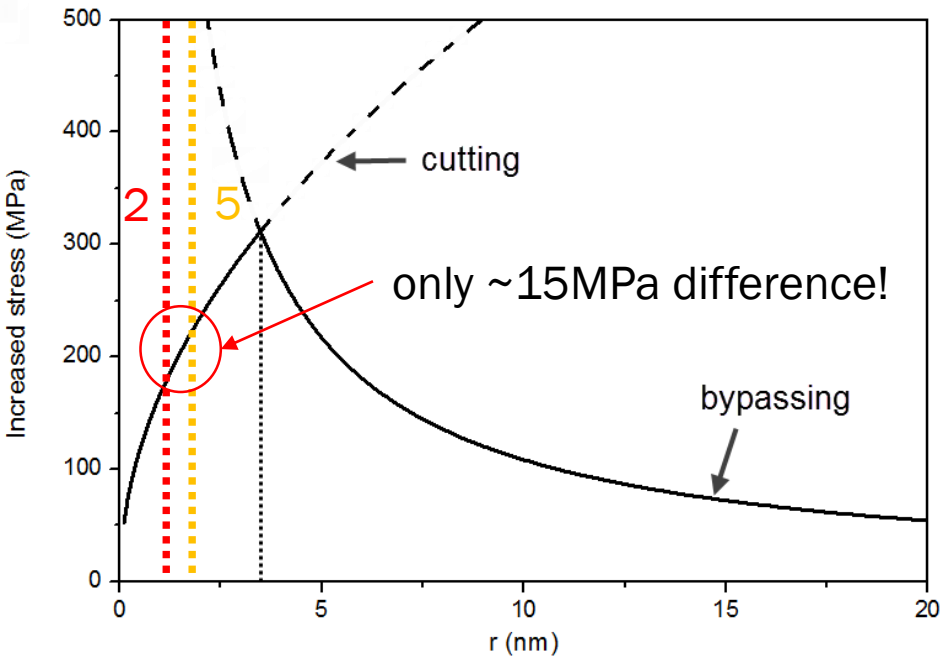


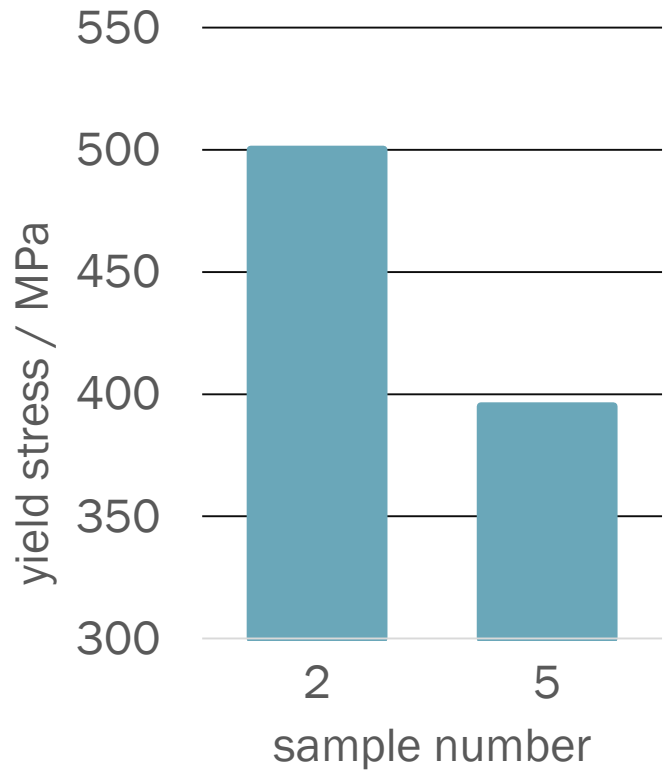
⇨ Only small influence of coarse precipitates on secondary precipitates.

Estimation of strengthening by Al_3Sc

Bypassing mechanism: $$\tau_{OR} = \frac{Gb\sqrt{f}}{r}$$

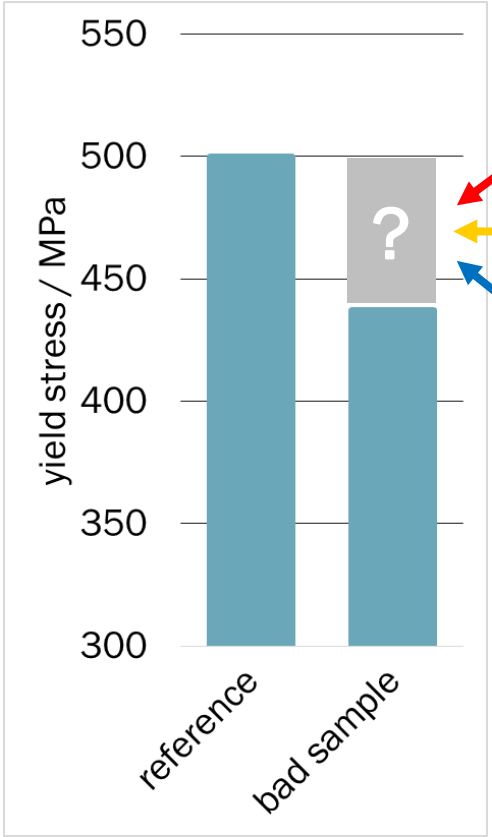
Cutting mechanism: $$\tau_c = \gamma^{3/2}\sqrt{f}\frac{\sqrt{r}}{\sqrt{6E_v}}$$





Precipitate strengthening follows cutting mechanism due to the size.
⇨ Sample 5 should be even a little stronger than sample 2!

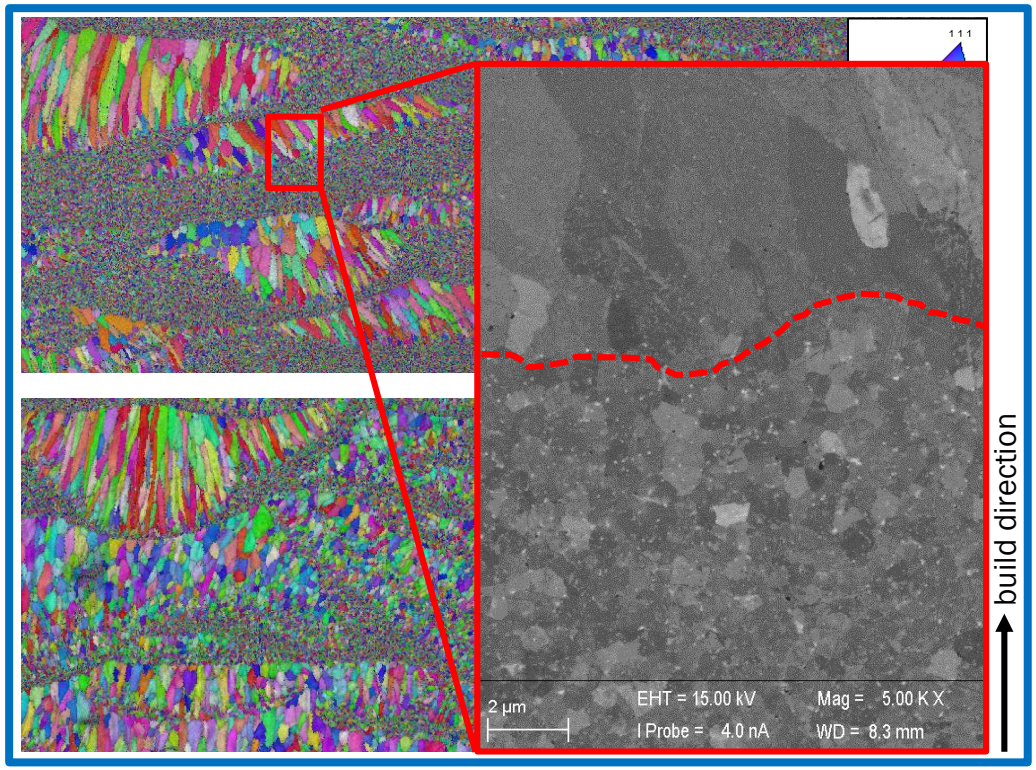
The problem – unexpected drop in strength



reduced cross sectional area due to bonding defects? ✓

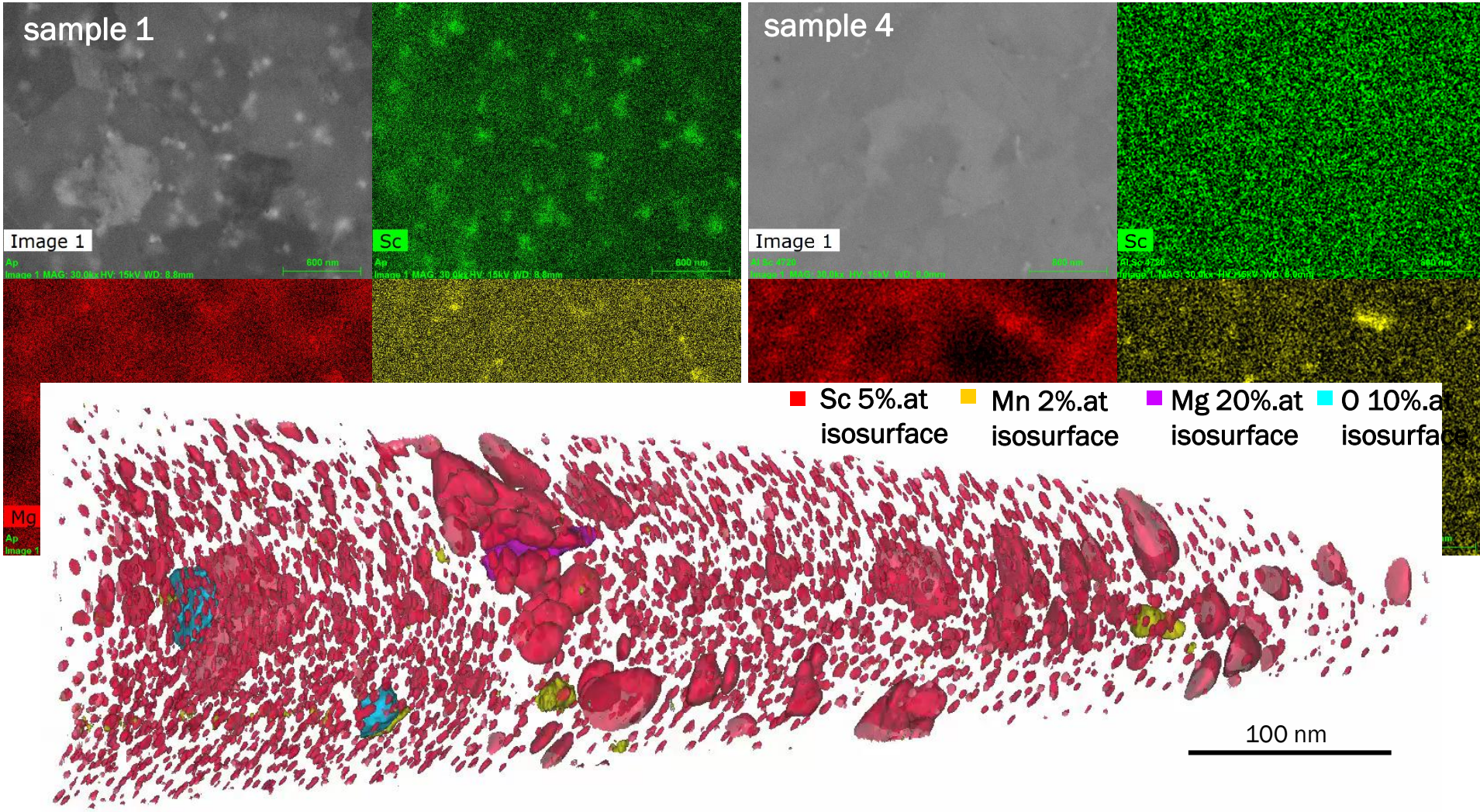
fewer nm-sized precipitates due to large precipitates? ✗

larger volume fraction of small, equiaxed grains



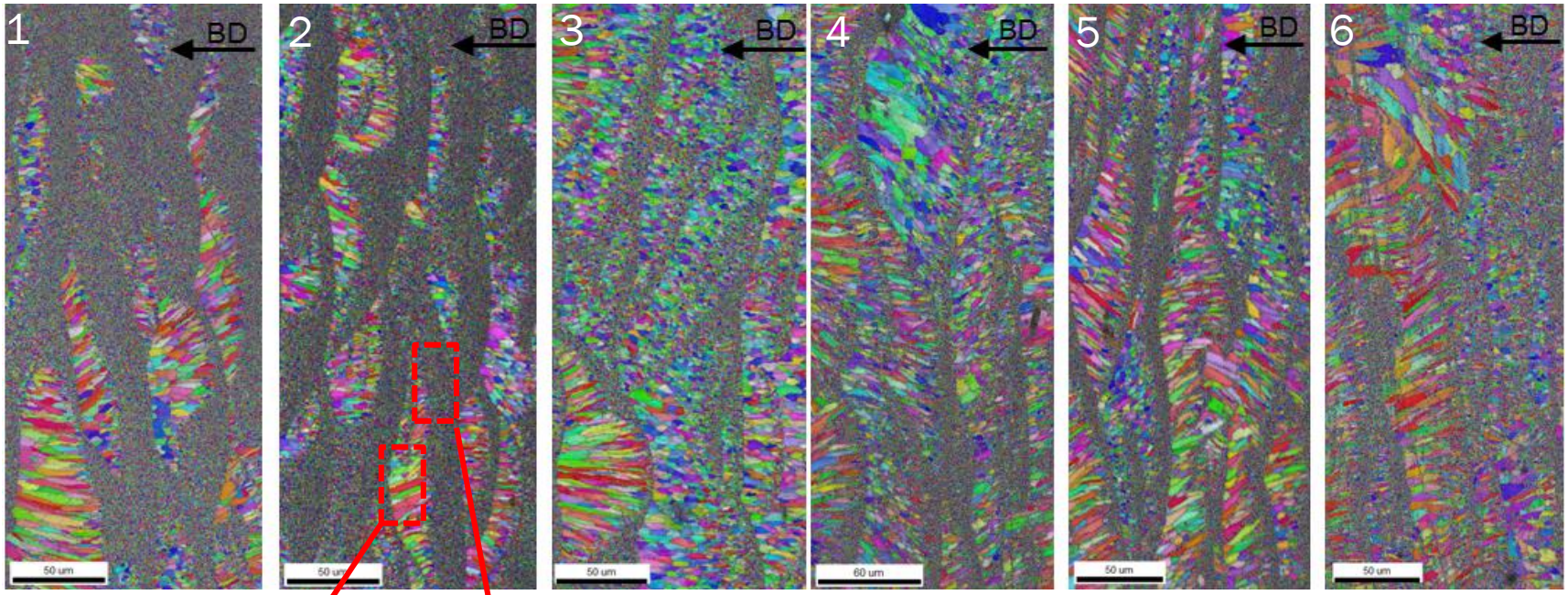
- Bonding defects and large ppts alone cannot explain drop in strength
- Strong difference in the fraction of small grains depends on the atomization process (!)

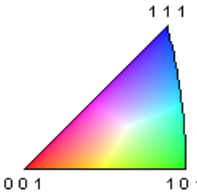
Grain refinement by intermediate precipitates?



➡ Different number of medium-sized precipitates (20-50 nm) – grain refinement during re-melting?

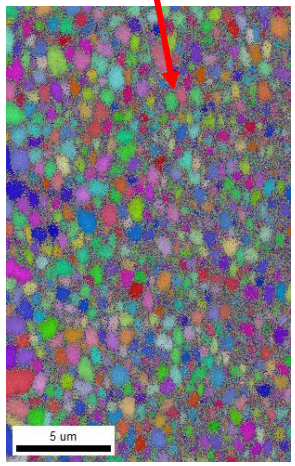
Different fractions of equiaxed grains



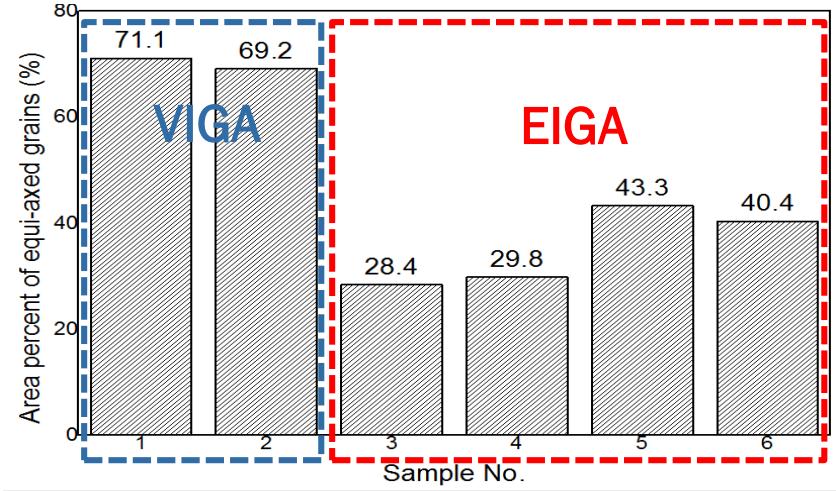


IPF (001)

Columnar grains

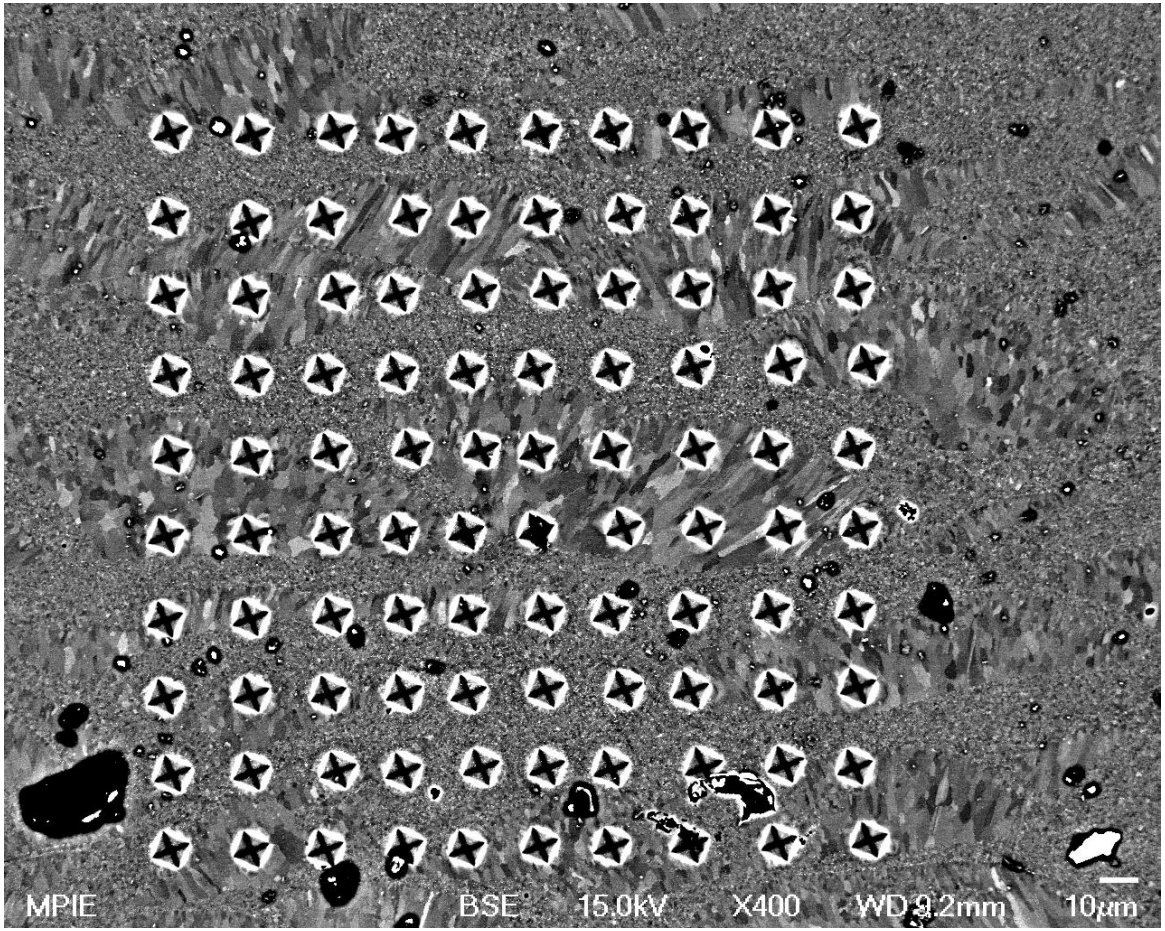


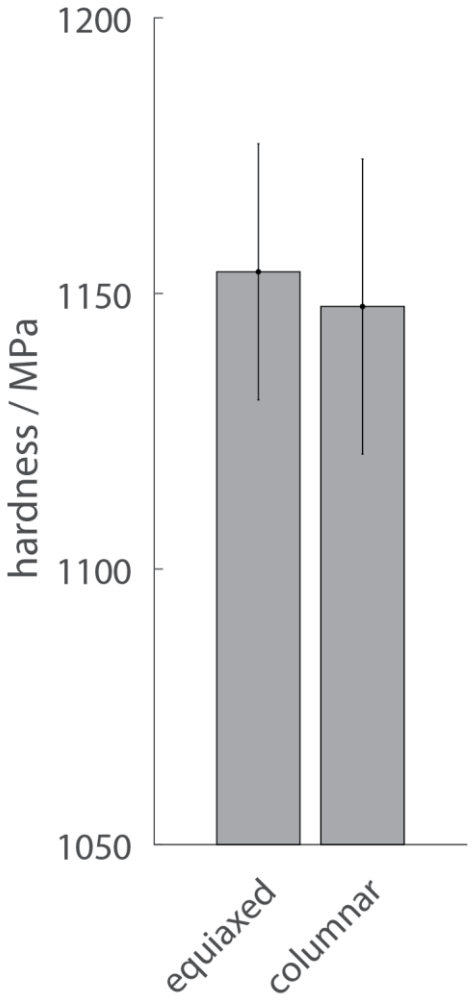
Equi-axed grains



➡ Area fraction of equiaxed grains depends on atomization method!

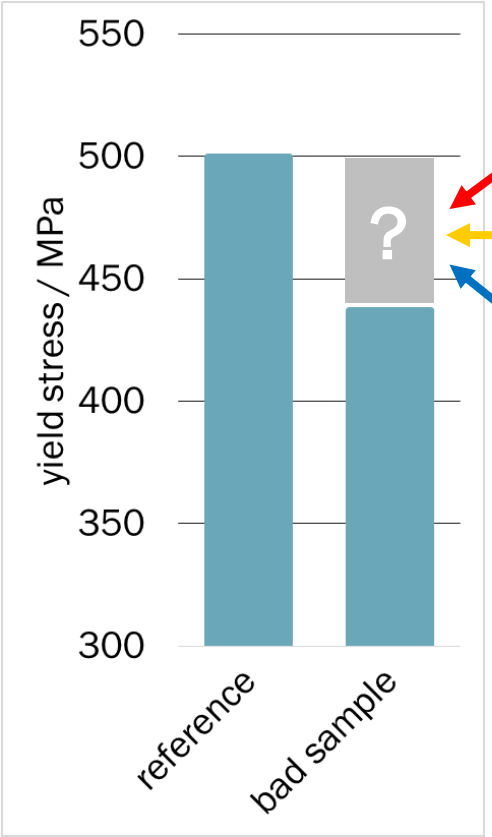
Micro-hardness measurements





➡ No significant influence of grain size on hardness (and hence strength)

The problem – unexpected drop in strength



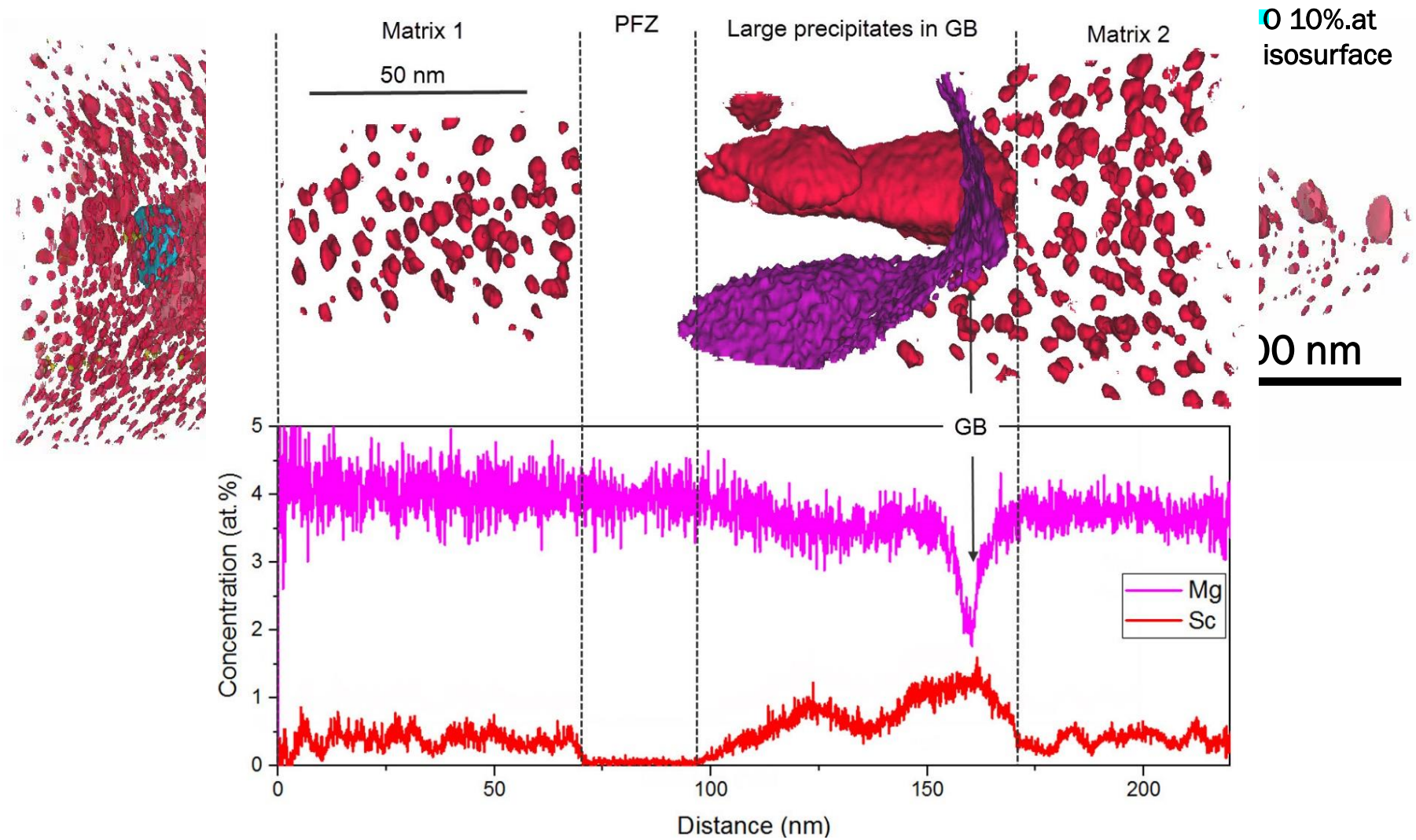
reduced cross sectional area due to bonding defects? ✓

fewer nm-sized precipitates due to large precipitates? ✗

larger volume fraction of small, equiaxed grains ✗

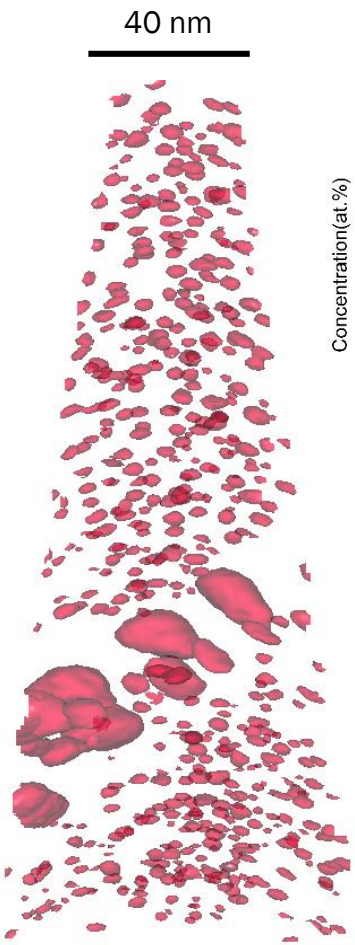
- Same powder composition
- Similar process parameters
- Different powder atomization supplier
- Different SLM-machine vendor

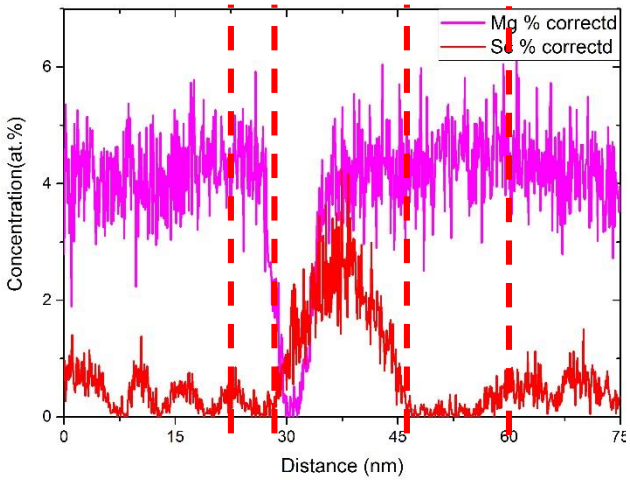
Observations of precipitates free zones

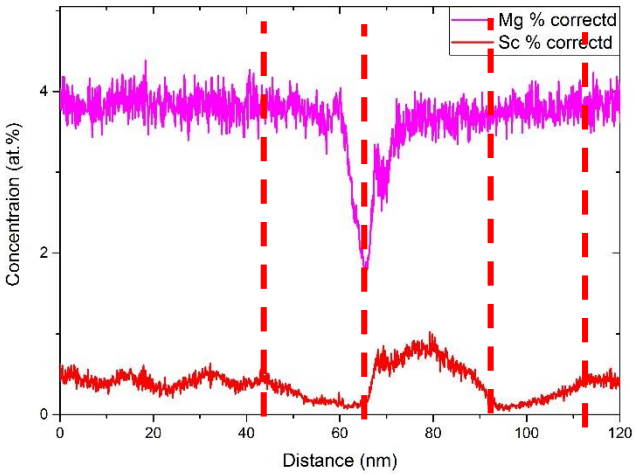


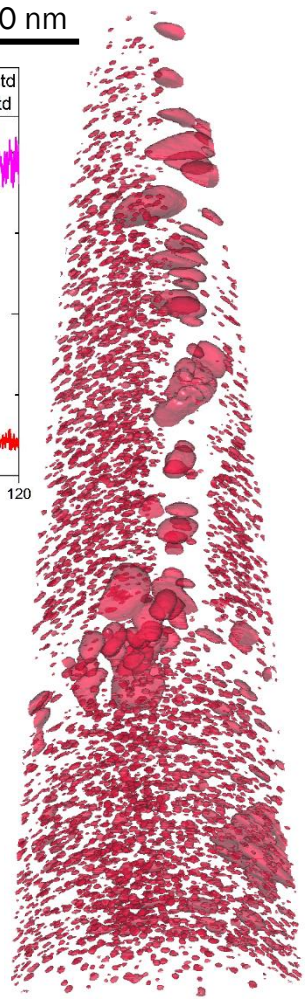
➡ Width of PFZs is significant compared to radius of small, equiaxed grains

Precipitates free zone







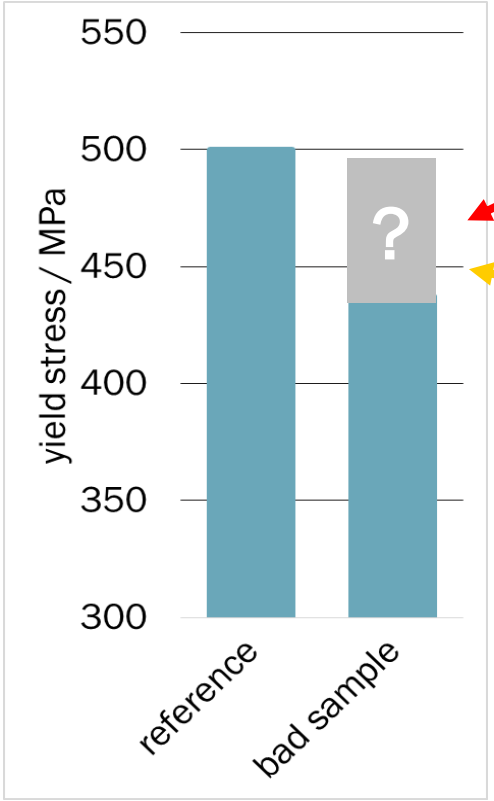


Sample No.	2	5
Total width / nm	38	70
Width of ppt / nm	18	28
Width of PFZ / nm	19	42

- Soft zone around PFZ may be 10 times wider than PFZ[1].
- Considering of the fine grain size (less than 1 µm), the PFZs may contribute significantly to low yield strength.

[1]Fourmeau et al., *Phil. Mag.*, **95** (2015) 3278

Small changes yield large differences



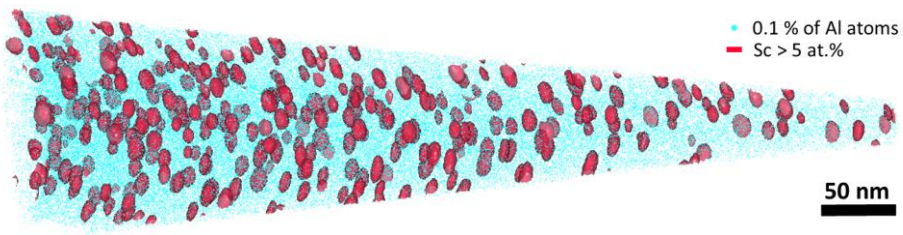
reduced cross sectional area due to bonding defects? ✓

wider PFZs may contribute to lower strength ✓

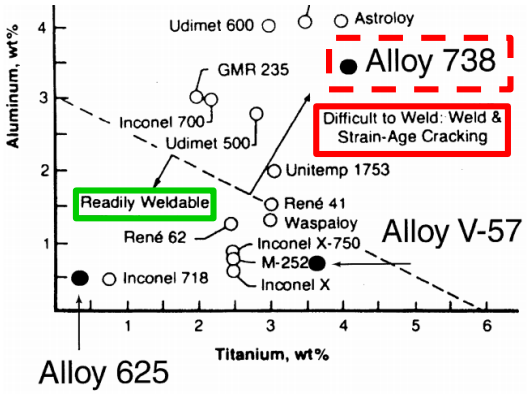
- Bonding defects due to poor process parameters explain difference in ductility and partially explain difference in strength
- Different atomization process leads to coarse precipitates, that in turn
  - bind Sc and Zr
  - lead to a lower number of intermediate-size precipitates
  - lead to a lower fraction of equiaxed grains
  - lead to wider PFZs

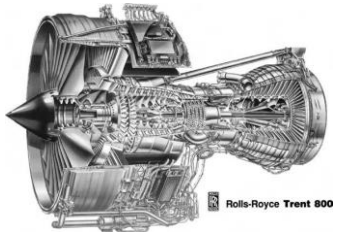
Outline

1. Strength variation in an Al-Sc alloy (Scalmalloy®):



2. Hot cracking in a Nickel-base superalloy: Inconel 738LC

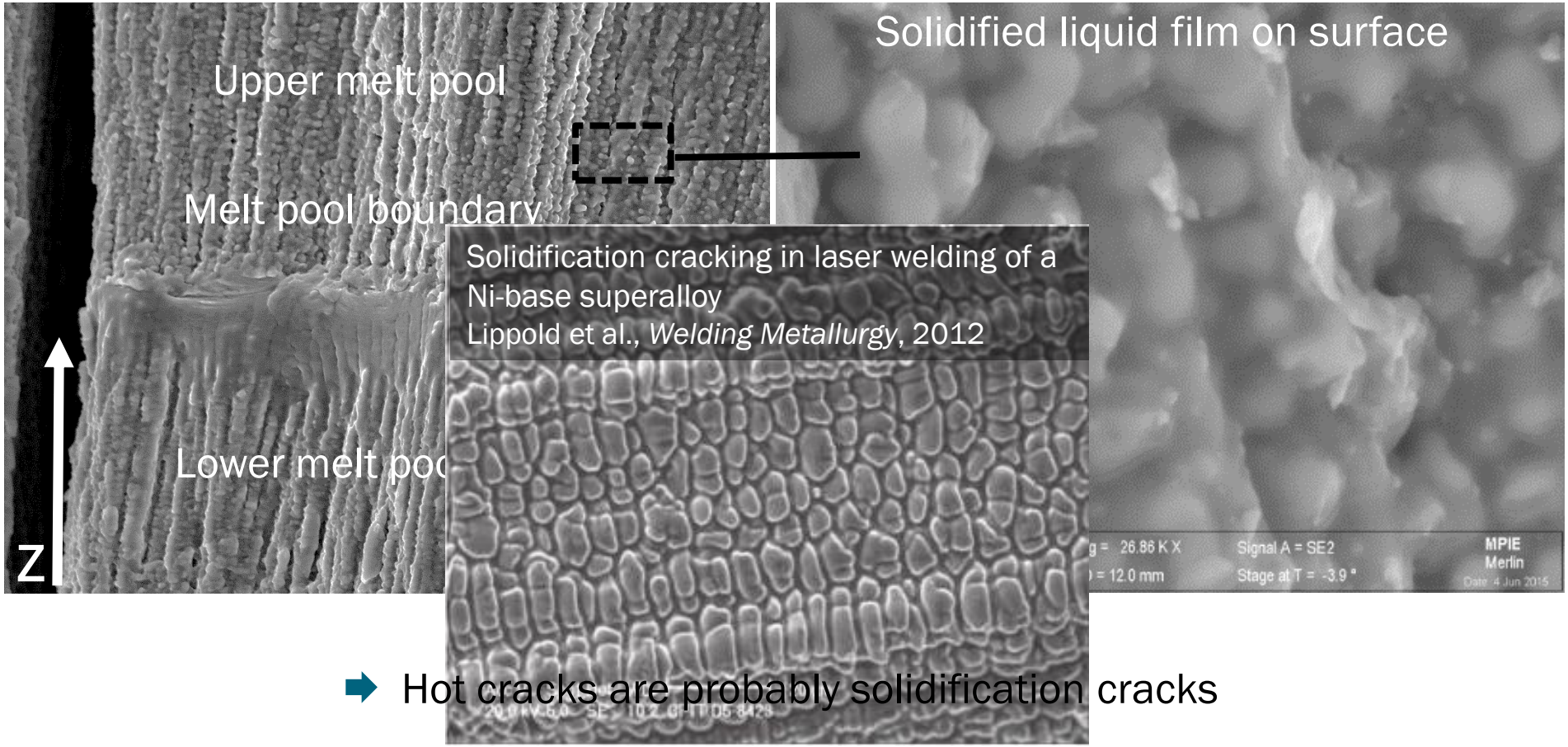




Problem: hot cracking in IN738LC

What type of cracking?

1. Liquation cracks? ✘ no low-melting phases
2. Strain age cracks? ✘ cracking without presence of γ'
3. Solidifcation cracks? ✓



Solidification cracking in laser welding of a Ni-base superalloy
Lippold et al., *Welding Metallurgy*, 2012

➡ Hot cracks are probably solidification cracks

Solidification cracking

Tensile stresses develop upon solidification and cooling shrinkage

⇩

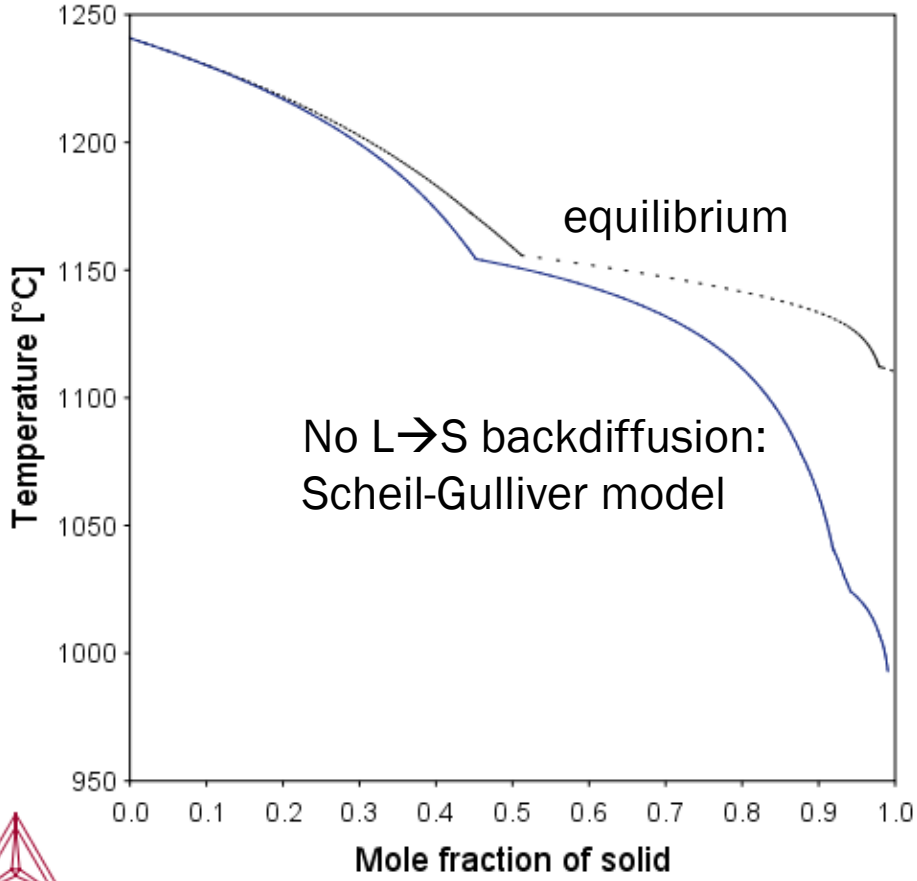
Liquid film persists to low temperatures

⇩

Liquid film ruptures

⇩

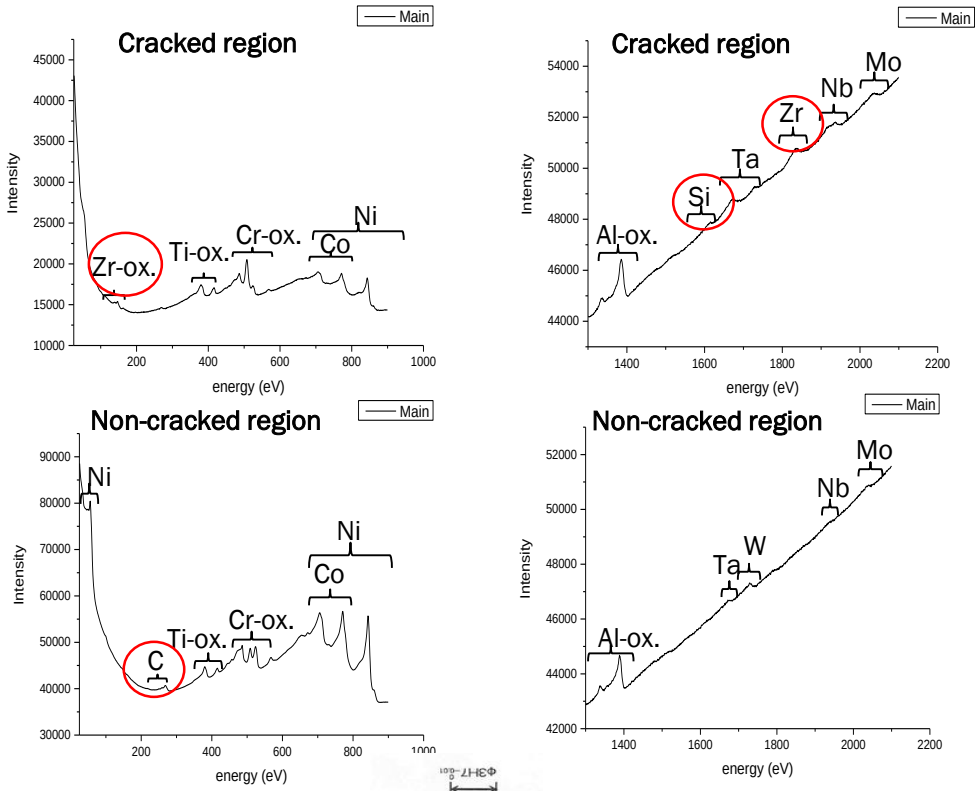
cracking

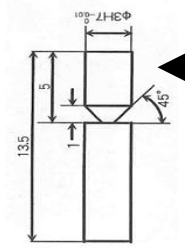


⇨ Microsegregation during solidification exacerbates the problem by lowering solidus temperature

Crack chemistry part I

Auger Electron Spectroscopy

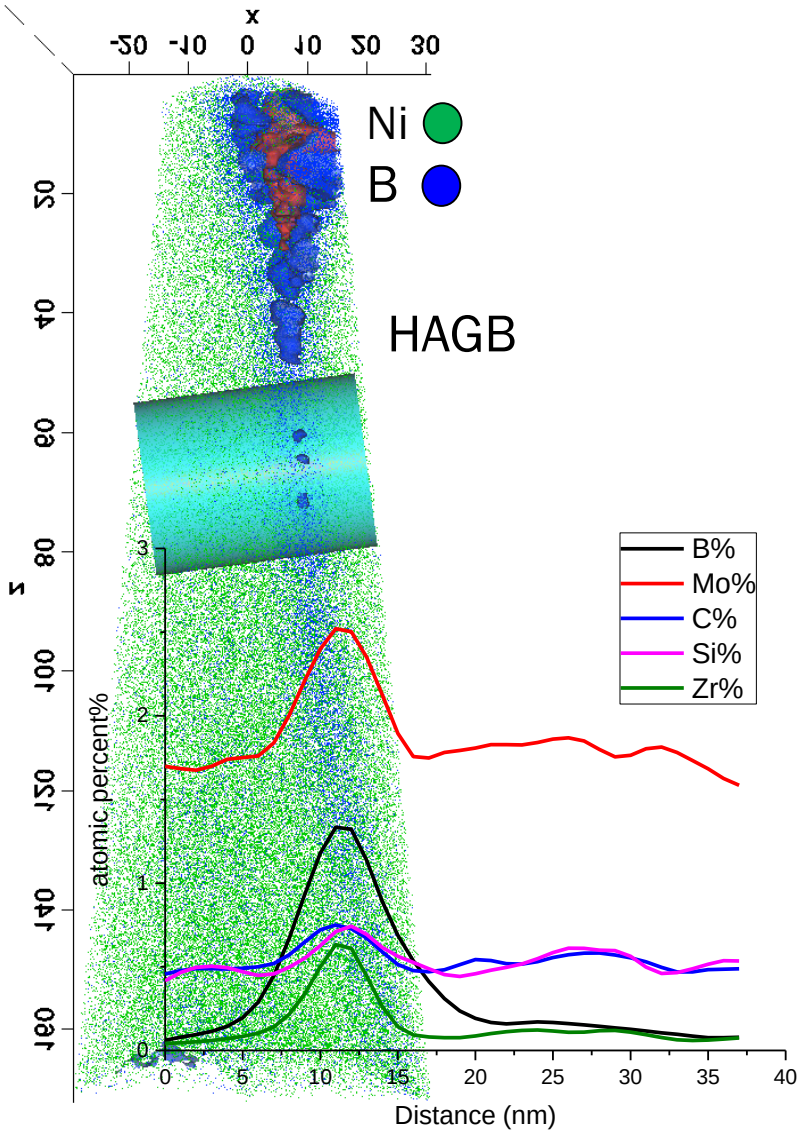




F

Impact in analysis chamber

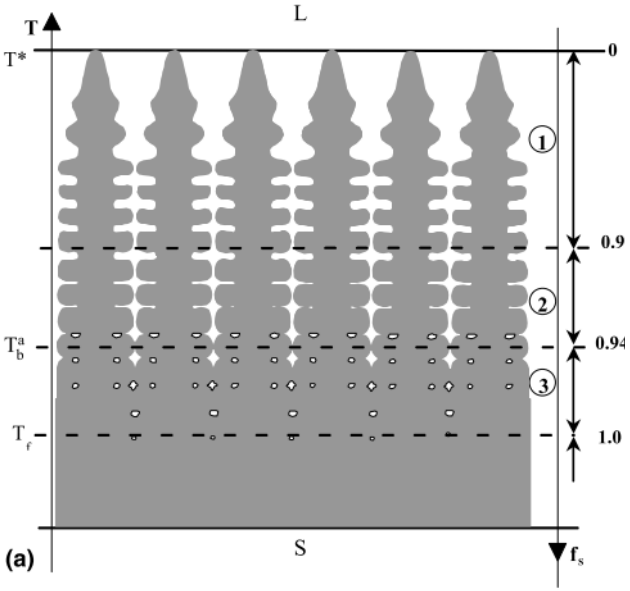
Atom Probe Tomography

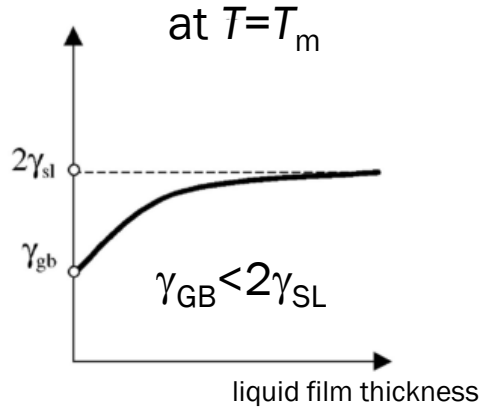


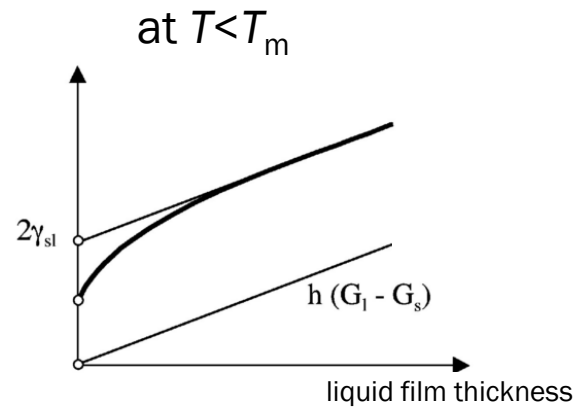
➡ Si and Zr enrichment on crack surface

Solidification cracking theory: Rappaz/Boettinger

attractive, i.e. LAGB

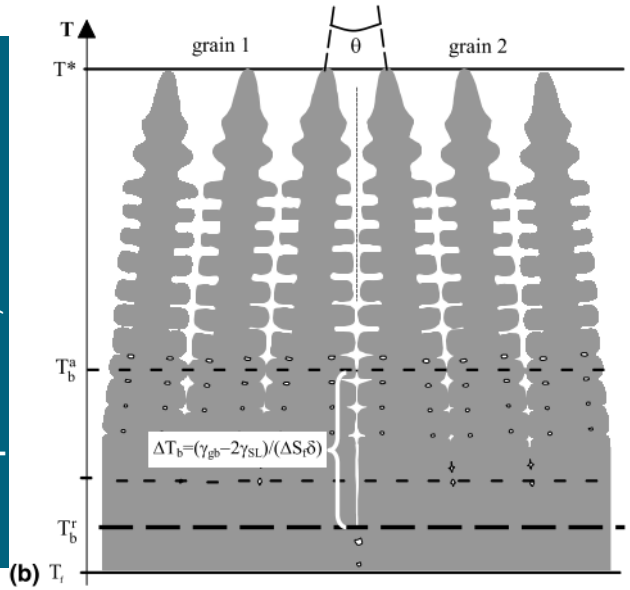


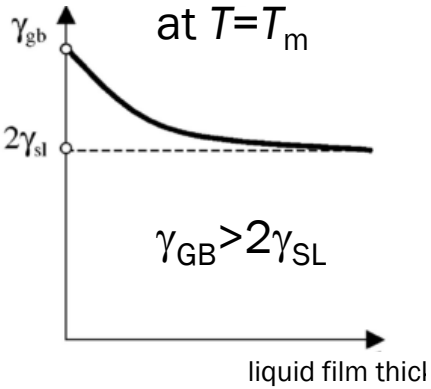


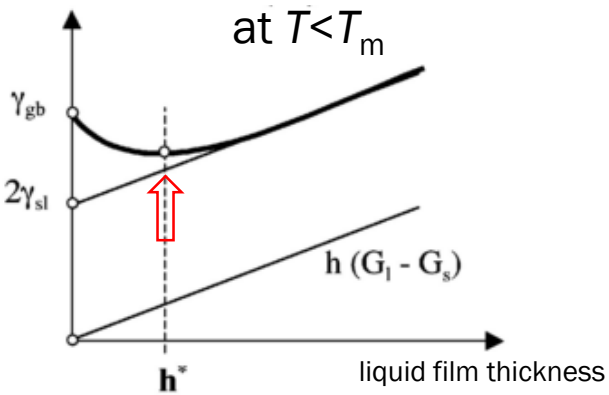


=> below melting point, liquid film is *un*stable

repulsive, i.e. HAGB





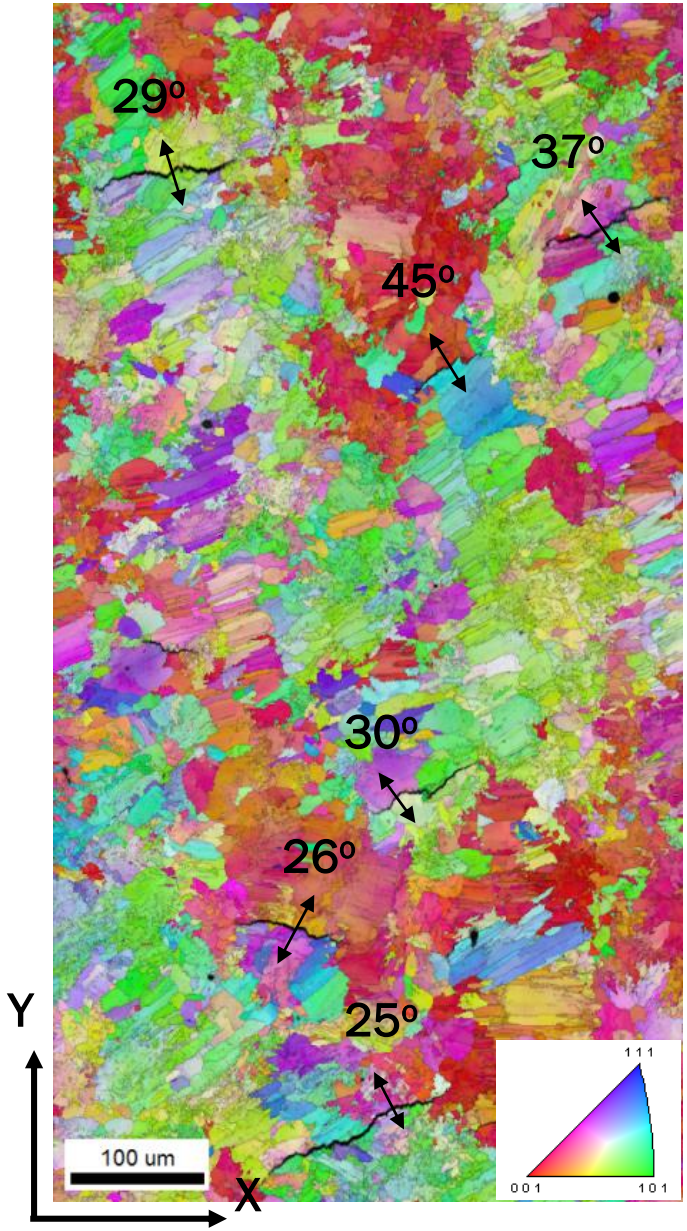


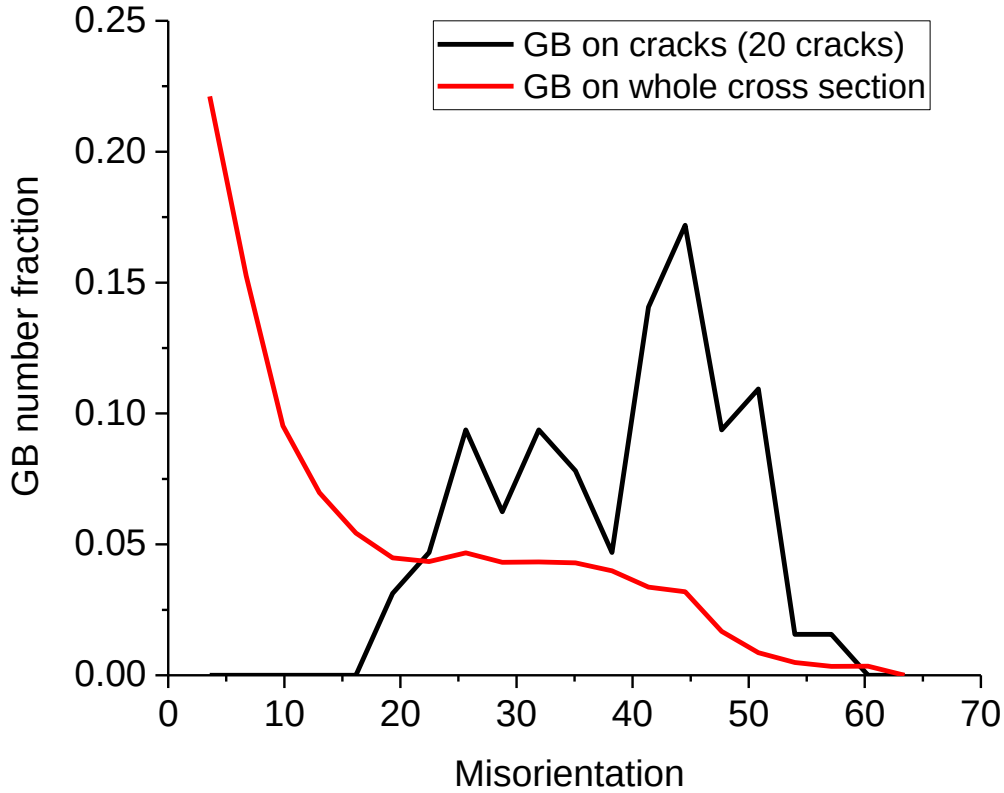
⇨ there is a *stable* liquid film thickness even below T_m

Undercooling to make liquid film unstable: $\Delta T_b = \frac{\gamma_{gb} - 2\gamma_{sl}}{\Delta s_f} \frac{1}{\delta}$

Rappaz et al., *Metall. Mater. Trans. A* **34**, 467 (2003).

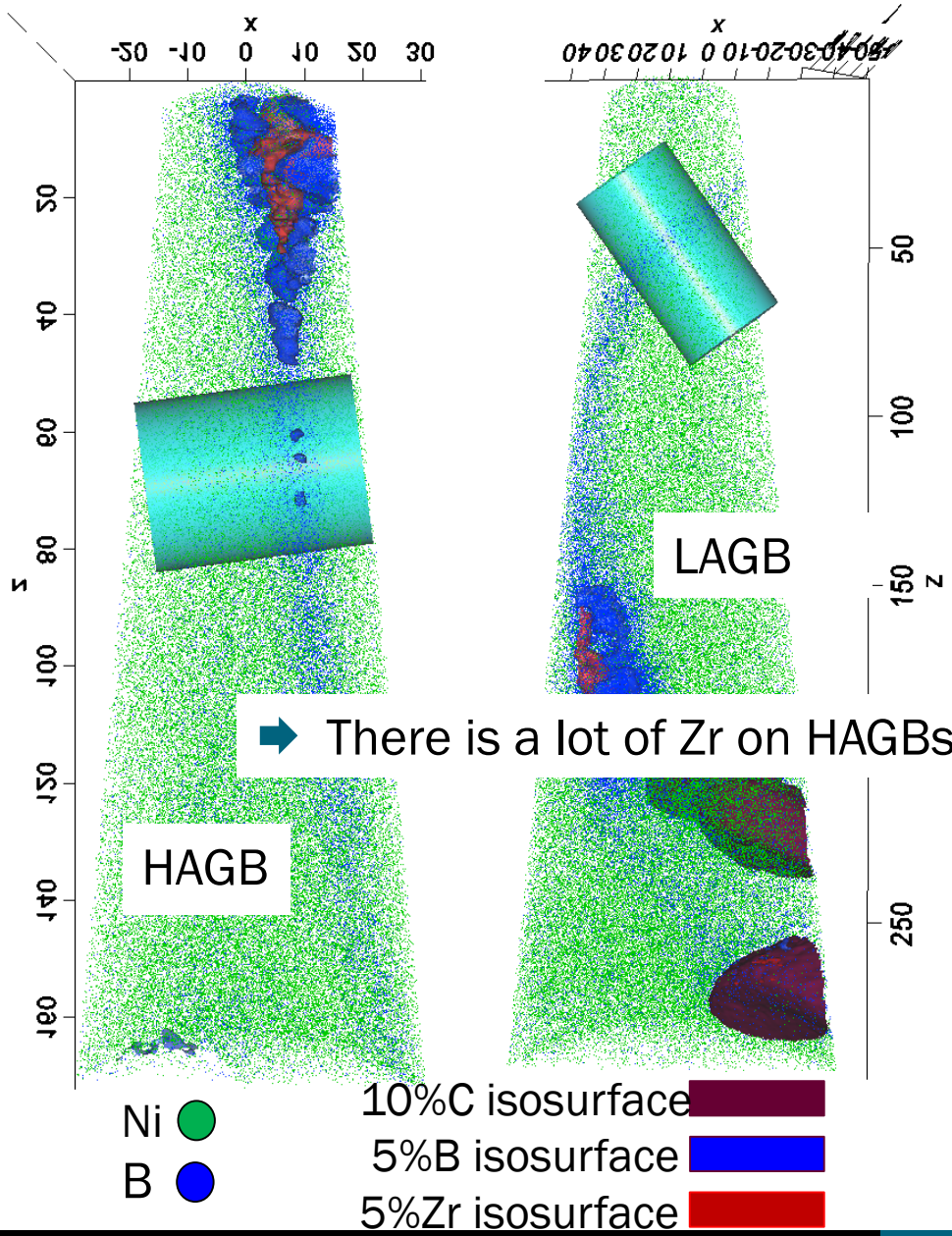
Cracked GB misorientation

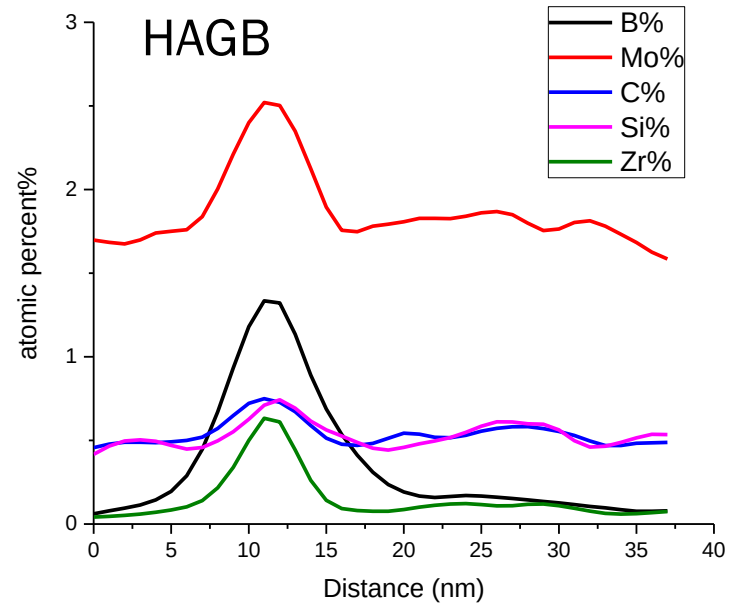


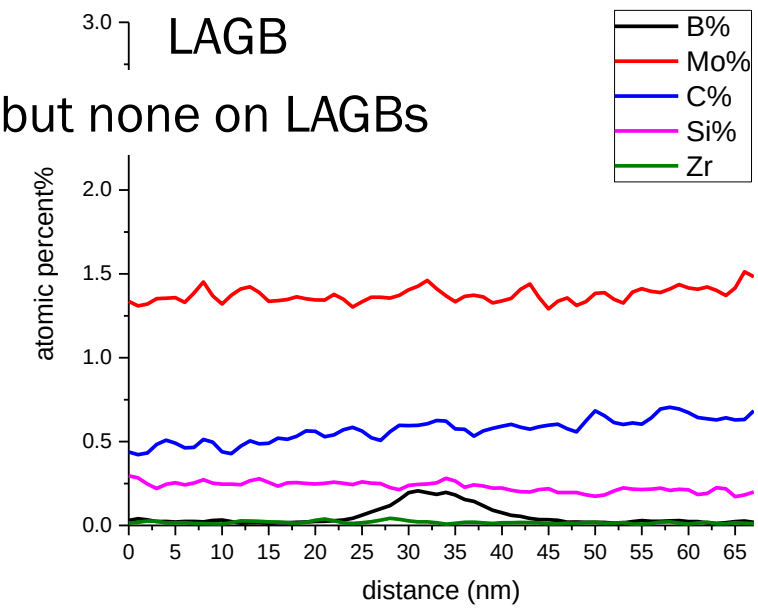


➡ Only HAGBs show cracking, as predicted by theory.

Crack chemistry part II







➡ There is a lot of Zr on HAGBs but none on LAGBs

Mystery: Why is Zr high on HAGBs?

M. Rappaz, A. Jacot, and W.J. Boettinger, *Metall. Mater. Trans. A* **34**, 467 (2003).

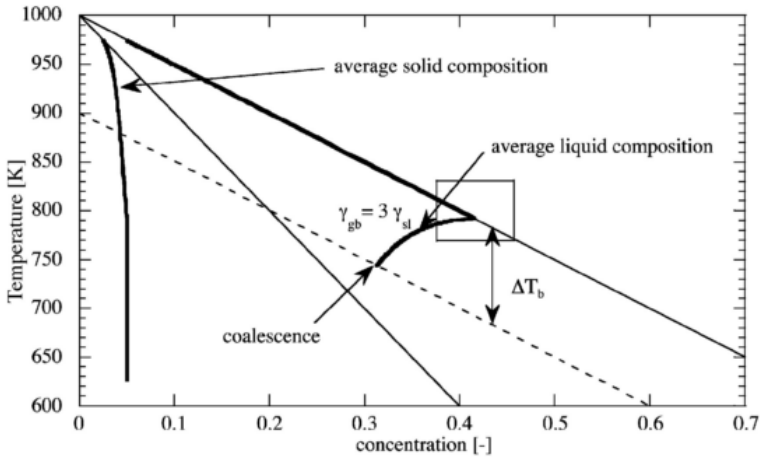


Fig. 7—Evolution of the average composition of solid and liquid as a function of temperature as predicted with the sharp interface model and the parameters of the left column of Table I ($D_s = 10^{-13}$ m^{2}s^{-1}, $\gamma_{sl} = 0.1$ Jm^{-2}, $\gamma_{gb} = 0.3$ Jm^{-2}, $\lambda/2 = 10$ μm, and $\delta = 1$ nm). The dashed line corresponds to the coalescence line.

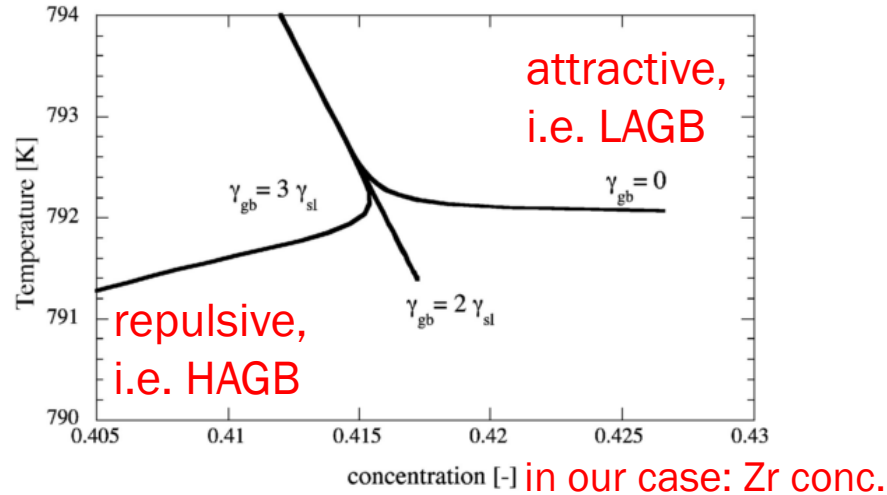
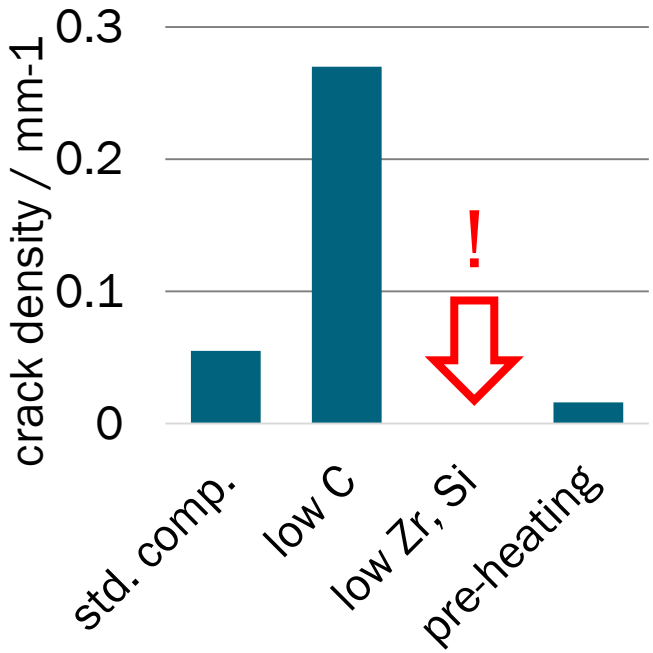


Fig. 8—Influence of the grain-boundary energy on the deviation from the liquidus line during the last stage of solidification, as predicted with the sharp interface model and the parameters of the left column of Table I for three types of interfaces: attractive ($\gamma_{gb} = 0$), neutral ($\gamma_{gb} = 2\gamma_{sl}$), and repulsive ($\gamma_{gb} = 3\gamma_{sl}$).

➡ Model that correctly predicts misorientation-dependence of cracking predicts **opposite** behaviour of Zr µ-segregation

Samples with low Zr/Si content?

Experiments



Thermocalc Simulation (TTNi8)

	high Zr,Si		low Zr, Si
Zr concentration	0.06 wt.-%		0.02 wt.-%
Si concentration	0.07 wt.-%		0.01 wt.-%
liquidus	1346°C	+2K →	1348°C
solidus – equilibrium	1267°C	+15K →	1282°C
solidus – Scheil	1058°C	+25K →	1083°C

- Cracking vanishes **completely** when Zr & Si are reduced (0.04% difference!)
- Cracking intensifies when C is reduced (probably due to less binding of Zr&Si in carbides)
- Solidus temperature increases by ~25K when excluding Zr, supporting *µ*segregation as reason for solidification cracking

Conclusions

Small variations in

- powder composition,
- powder atomization,
- process parameters,
- post-processing

can lead to large changes in

- defect density,
- precipitation,
- grain size,
- yield strength and ductility.

Two examples:

- Unexpected drop of yield strength in an Al-Sc alloy
- Elimination of hot cracking in a Ni-base superalloy by Zr & Si reduction

Thank you for your attention.

E. A. Jägle, Z. Sheng, L. Lu, L. Wu, J. Risse, A. Weisheit, D. Raabe, *JOM,* **68**, 943 (2016)
E. A. Jägle, L. Wu, F. Palm, D. Raabe, *in preparation*
E. A. Jägle, L. Lu, J. Risse, D. Raabe, *in preparation*
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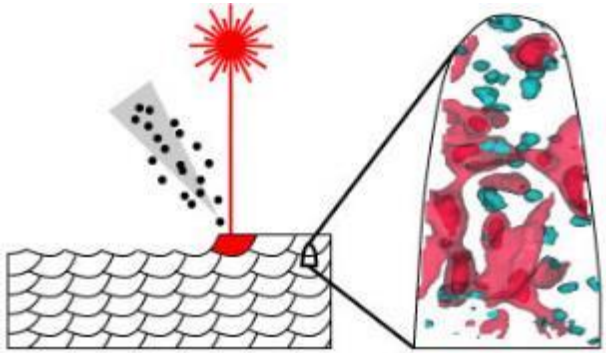

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