X-ray CT for microstructure characterization of light metals

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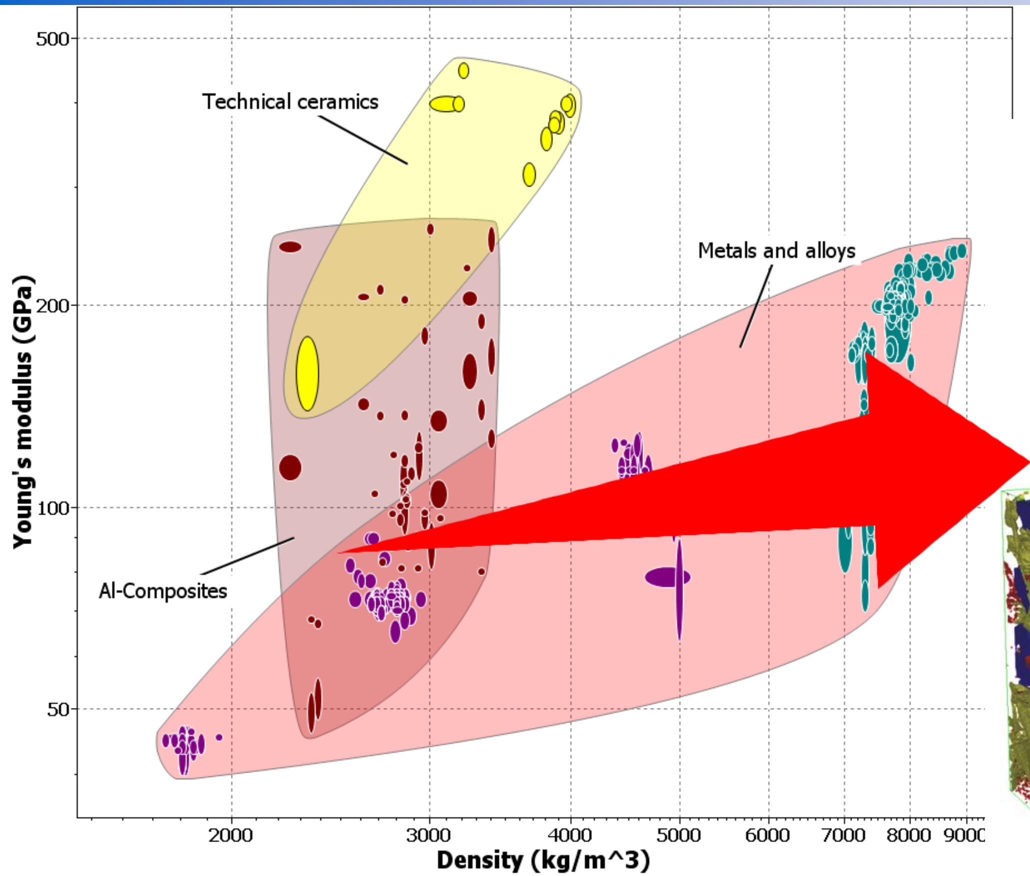
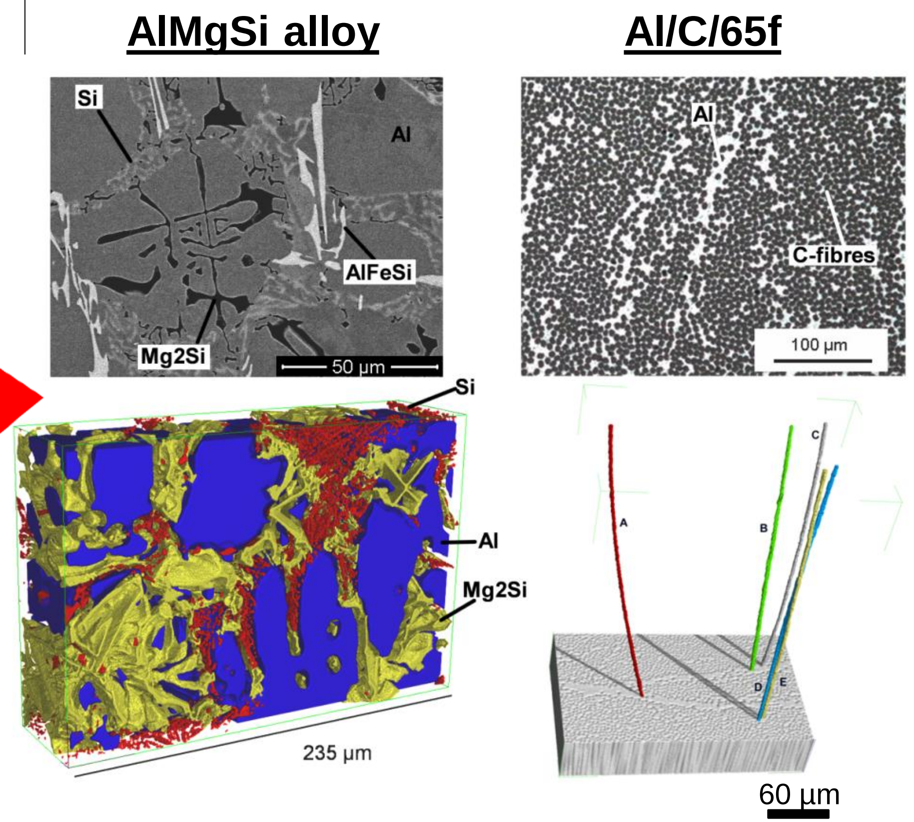
Properties of multiphase materials





In multiphase materials → **Properties = f [prop., vol.fract., distr., shape, size, interconnectivity, contiguity, ...]**

Architecture

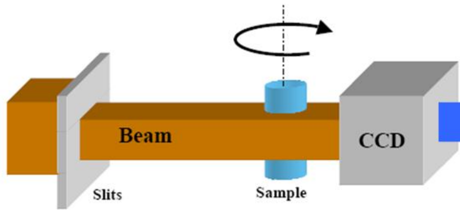
X-ray -based tomography

Lab-XCT

High resolution
Non-destructive
Larger volumes
Reveal Si and Al
Time resolution

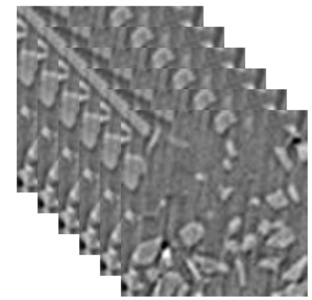
Synchrotron Tomography

1- **Acquisition** of n **projections** (radiographies) taken at n angles



- **Absorption** and/or **phase** contrast imaging
- Phase retrieval → **Holotomography**

2- **Reconstruction** of the slices from n projections using, e.g, a filtered backprojection algorithm



-Reconstruction algorithms: filtered backprojection, algebraic techniques, …
- Filtering may be necessary

3- **Stacking** of the reconstructed 2D slices → 3D Image

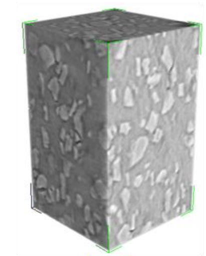


Image Improvement (filtering)

Segmentation (phase discrimination)

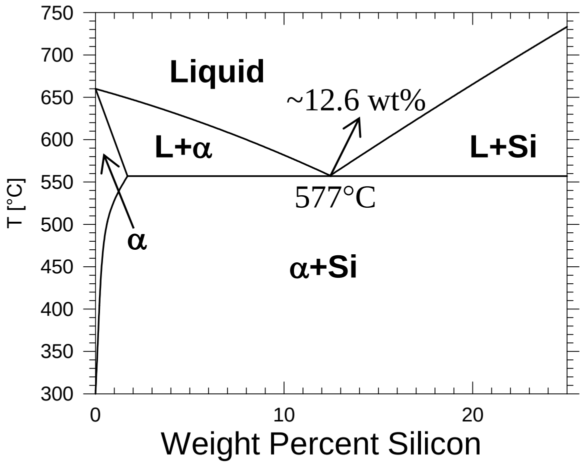
Analysis

Why Synchrotron Tomography for metallurgical investigations?

- Coherence → phase contrast → e.g. holotomography
- High Brilliance → real time (in situ) experiments, ultra fast acquisition times
- Tunability → posibility of using monochromatic beam, use of the most suitable energy for a certain experiment/material
- Combination with other techniques available at the beamline (e.g. diffraction)

Microstructure of cast Al-Si alloys

Al-Si system

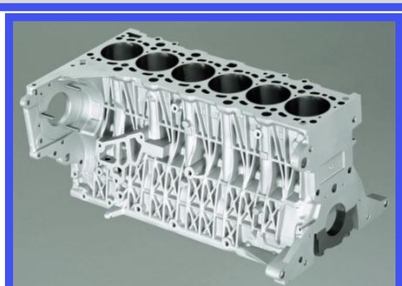


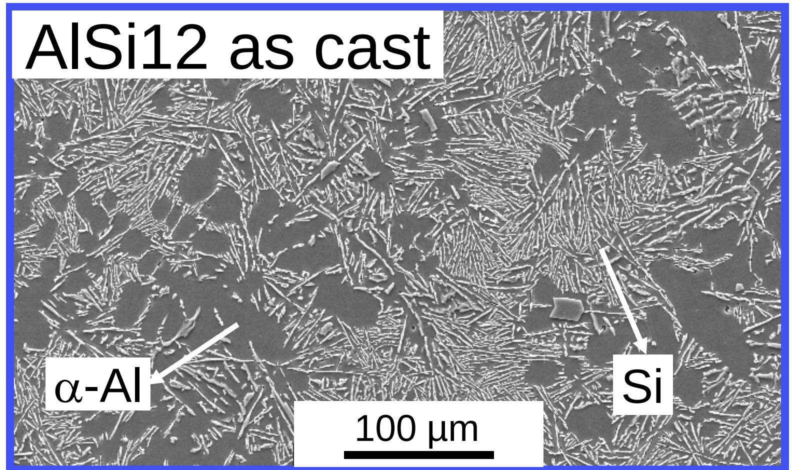
ρ_{Al}= 2.7 g/cm³
ρ_{Si}=2.3 g/cm³

$E_{Al(RT)}$=70 GPa
$E_{Al(300°C)}$=50GPa
$E_{Si(RT-300°C)}$=150 GPa

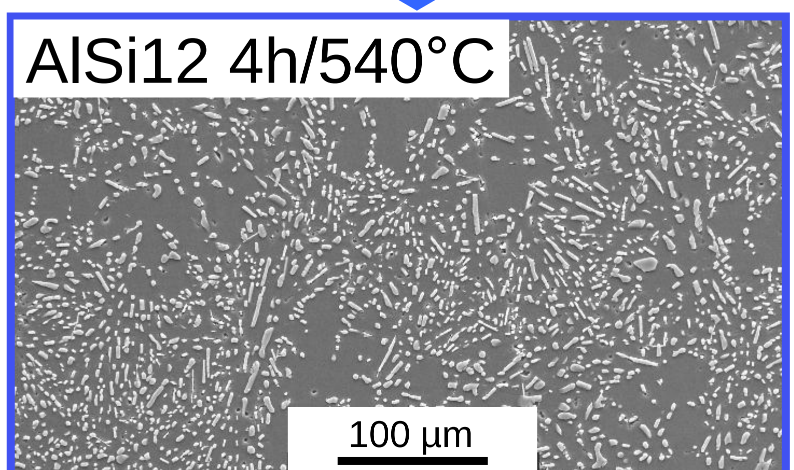
σ_{02Al} ~ 50 MPa
σ_{02Si} ~ 160-180MPa

$CTE_{Al(RT-300°C)}$ ~ 24 ppm/K
$CTE_{Si(RT-300°C)}$~ 3ppm/K





4h
540°C



Deep etching of Al

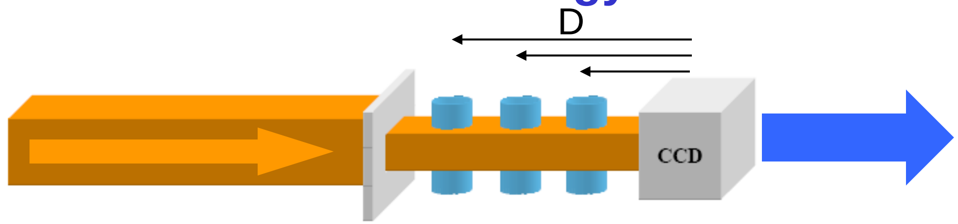
Eutectic Si can be highly interconnected

J. Bell, W. Winegrad, Nature 5006 (1965) 177

Eutectic Si spheroidises→ gets rounder and disintegrates

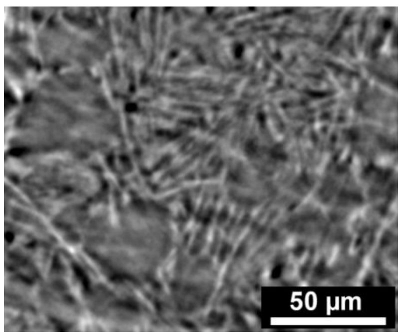
Microstructure of cast Al-Si alloys

ID19 – ESRF / Energy = 29 keV / voxel size = (0.3 µm)³

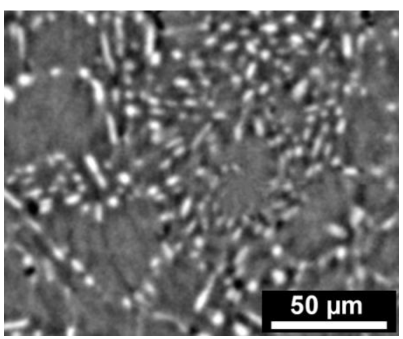


Phase contrast tomography e.g.holotomography using three distances

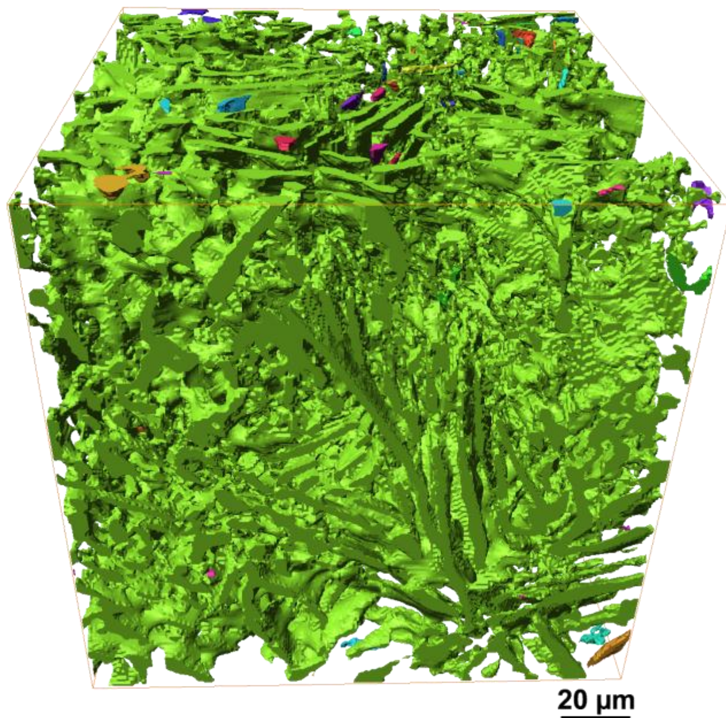
Resolution ~ 1µm
Non-destructive (same sample!)
Larger volumes than FIB



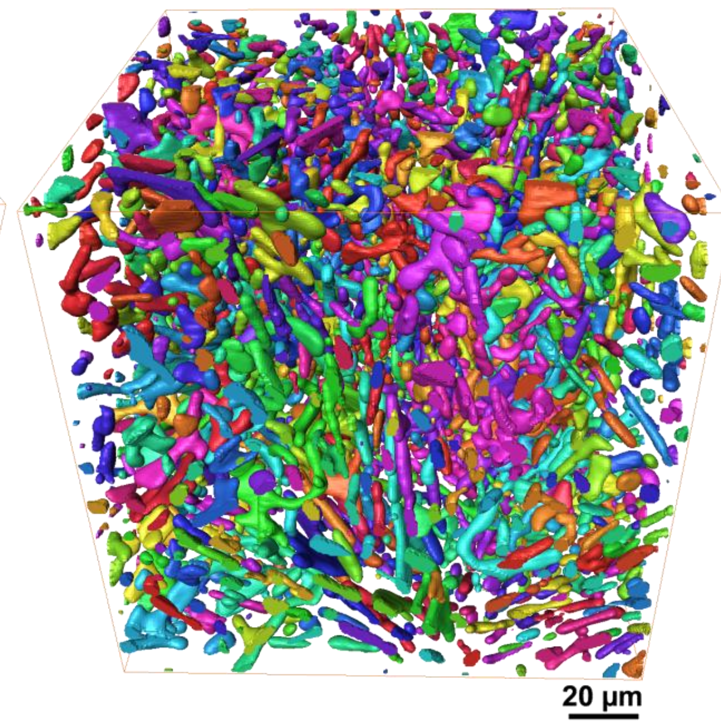
as cast



540°C / 4h



as cast

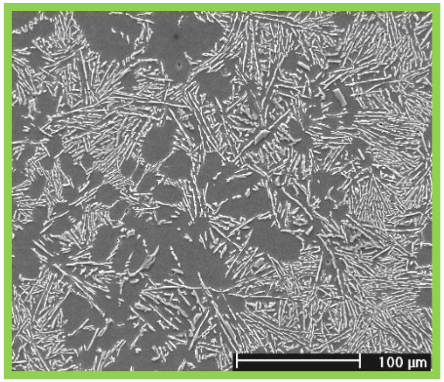


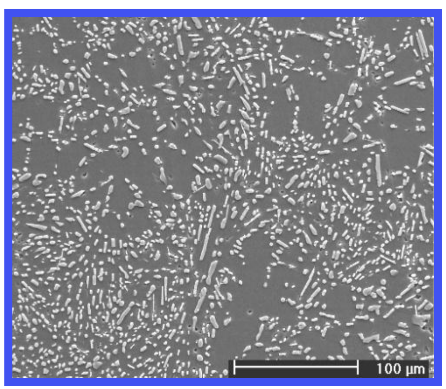
540°C / 4h

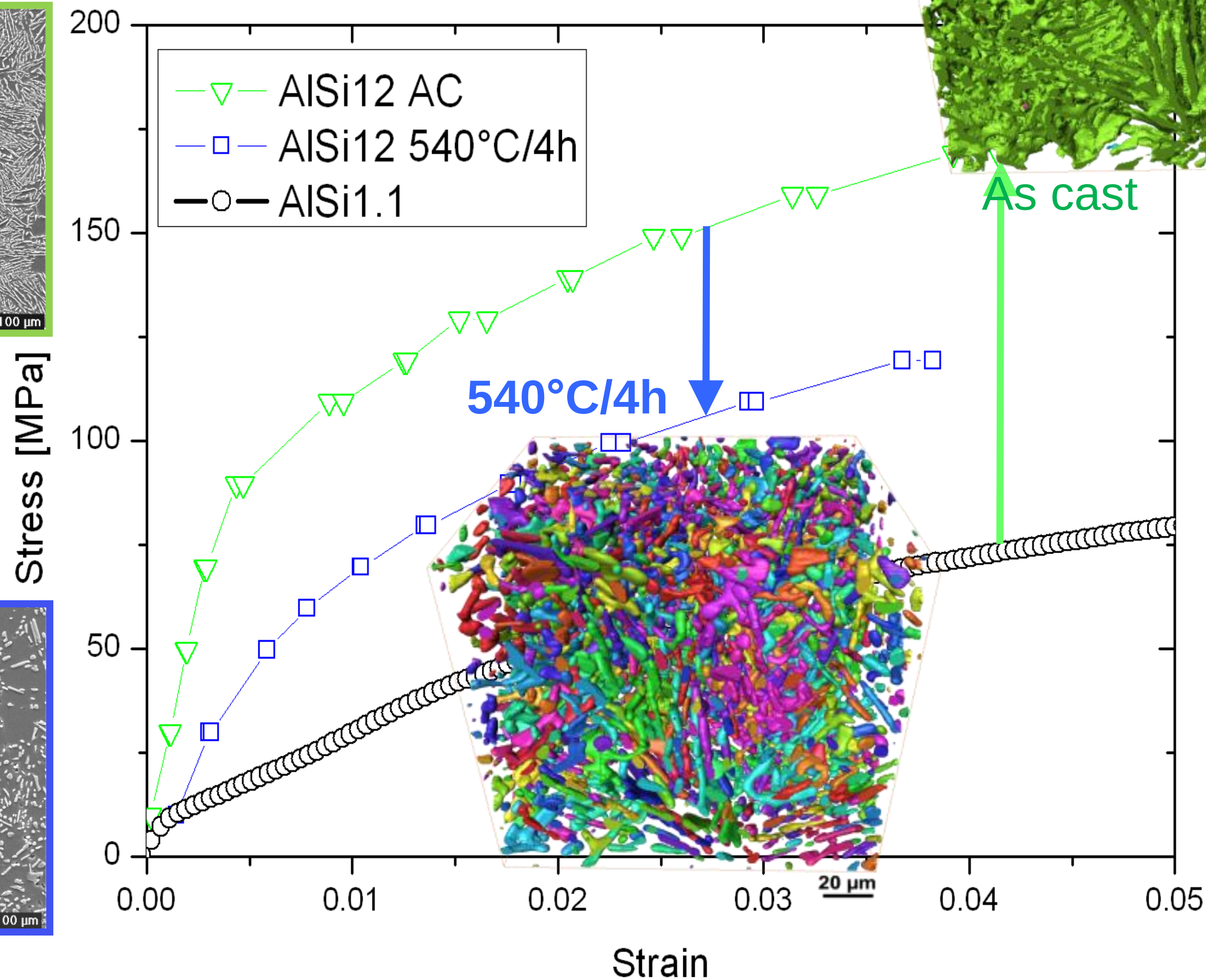
Requena, Garcés, Rodríguez, Pirling, Cloetens, AdvEngMat 11 (12) (2009)

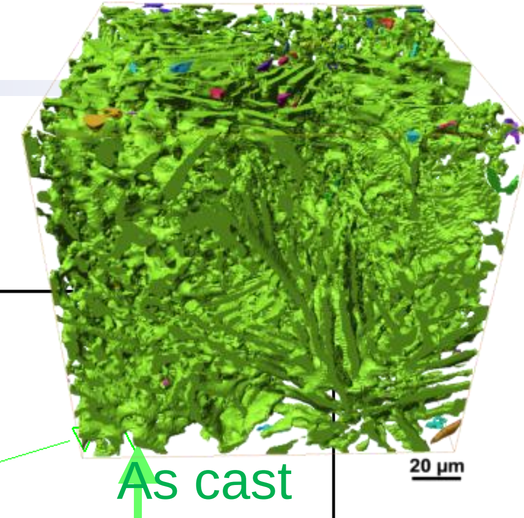
Strength of cast Al-Si alloys

Compression tests (RT)



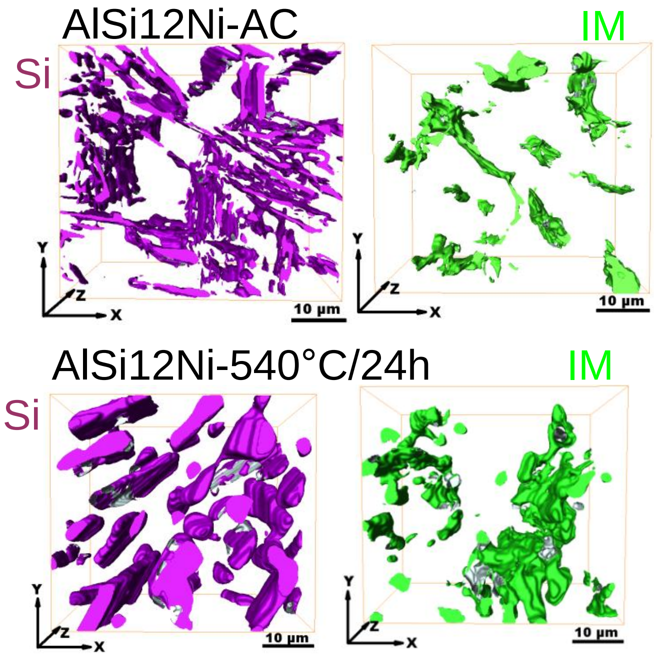






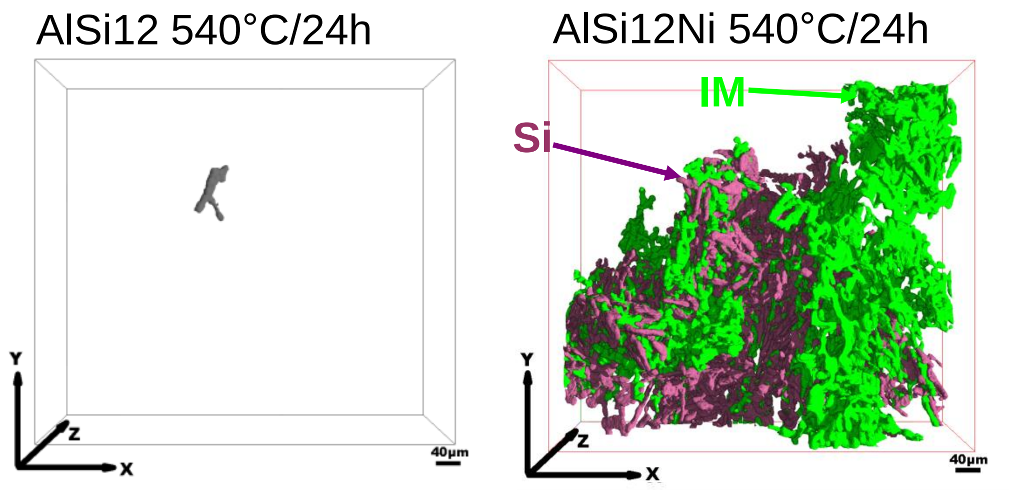
Requena, Garcés, Rodríguez, Pirling, Cloetens, AdvEngMat 11 (12) (2009)

Light Optical Tomography
~ (0.1x0.1x0.5) µm³



Synchrotron Tomography (0.3x0.3x0.3) µm³

Largest Si particle after 540°C/24h

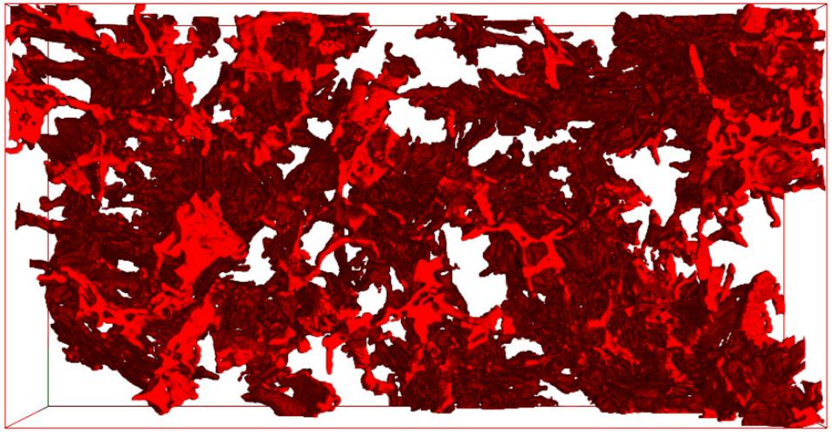


-Total volume fraction of aluminides ~ 8vol%
-High degree of interconnectivity and contiguity after 24h/540°C

Asghar, Requena, Degischer, Cloetens – Acta Materialia 57 (2009)

Synchrotron Tomography (0.3x0.3x0.3) µm³

(vol=290x170x88 µm³)



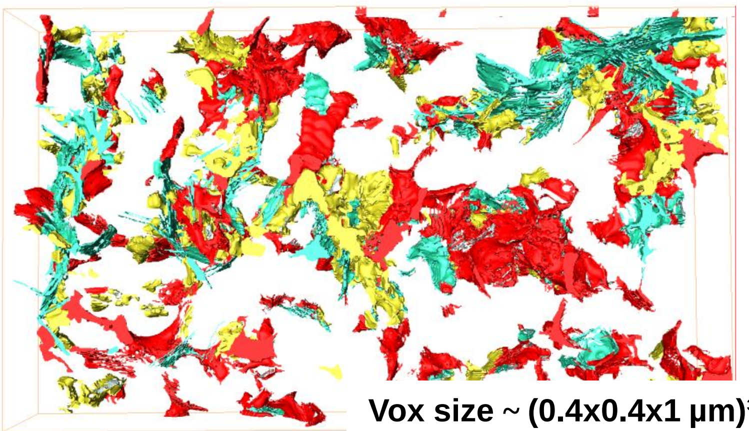
Asghar, Requena, Boller – Acta Materialia 59 (2011)

AlSi10Cu5NiFe

- Al_2Cu
- Al_3CuNi
- $Al_8FeMg_3Si_6$ + $Al_{15}(FeMn)_3Si_2$

Light Optical Tomography+EDX

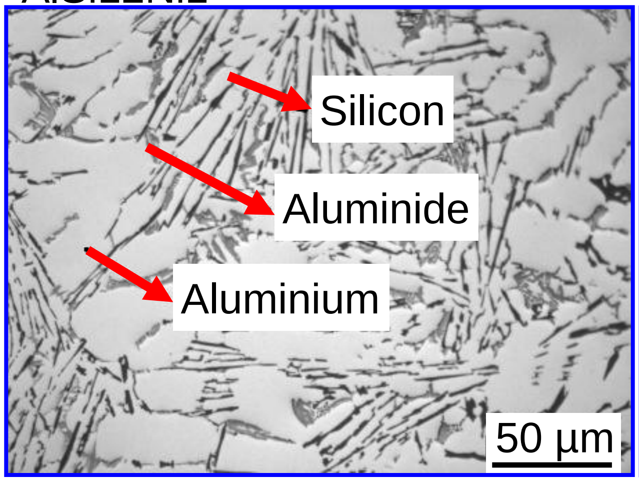
(vol=290x170x43µm³)



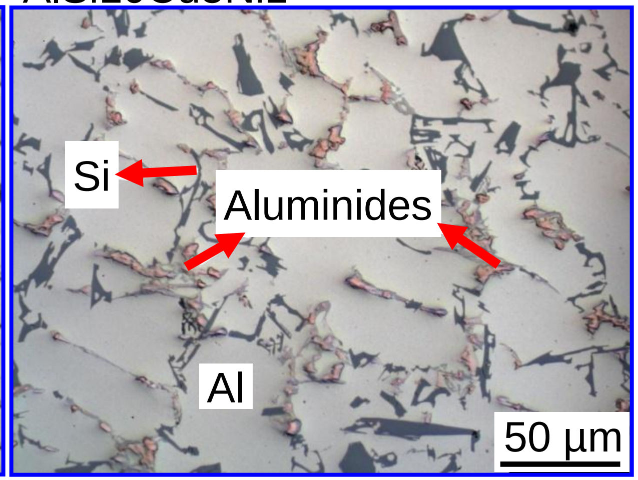
Asghar, Requena, Boller – Prakt Met 47 (2010)

Addition of Cu, Ni, Fe to Al-Si alloys to form stable aluminides

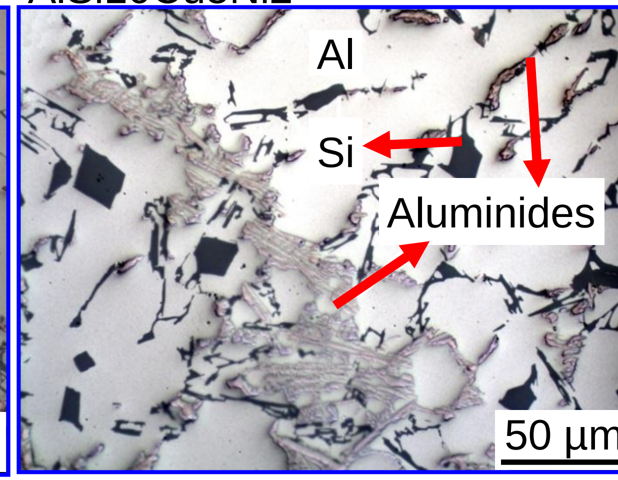
AlSi12Ni1



AlSi10Cu5Ni1



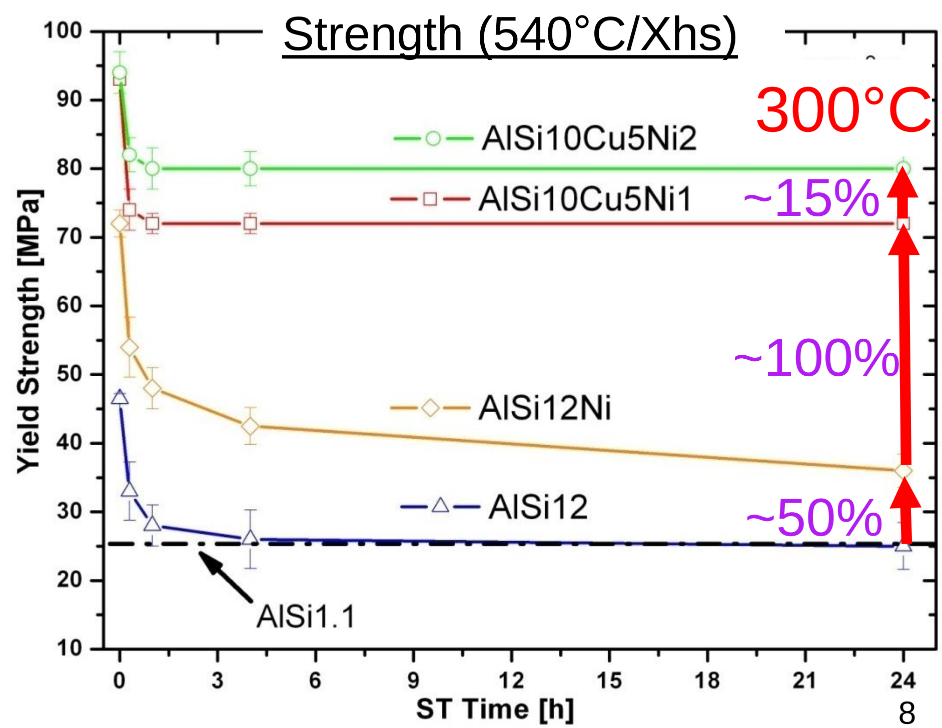
AlSi10Cu5Ni2



Composite-like behaviour:

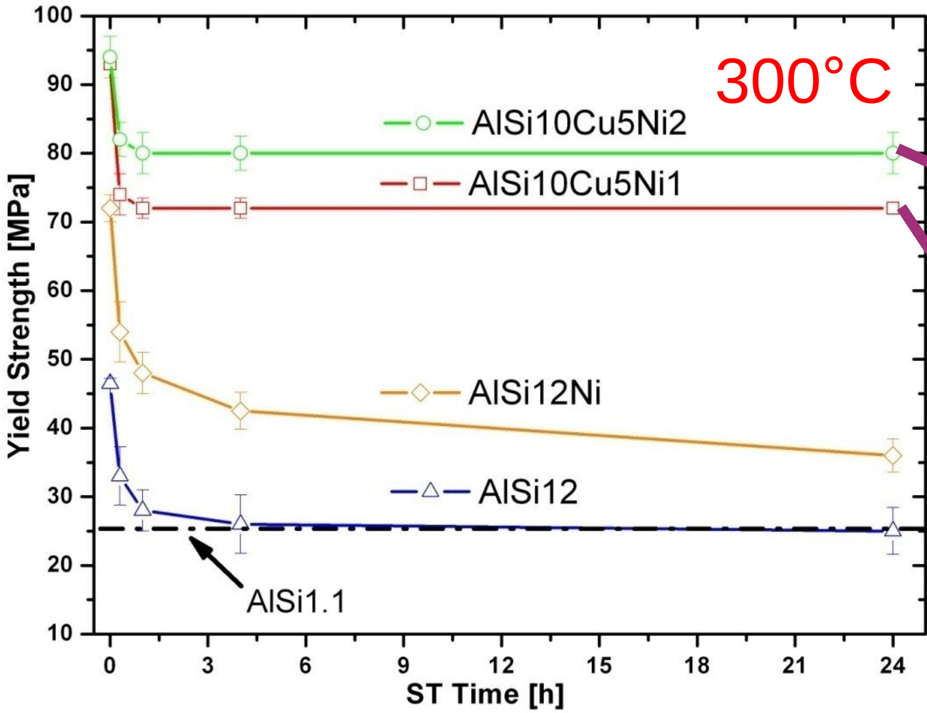
-Al → matrix (low strength but ductile)
- Eutectic Si
- different Aluminides

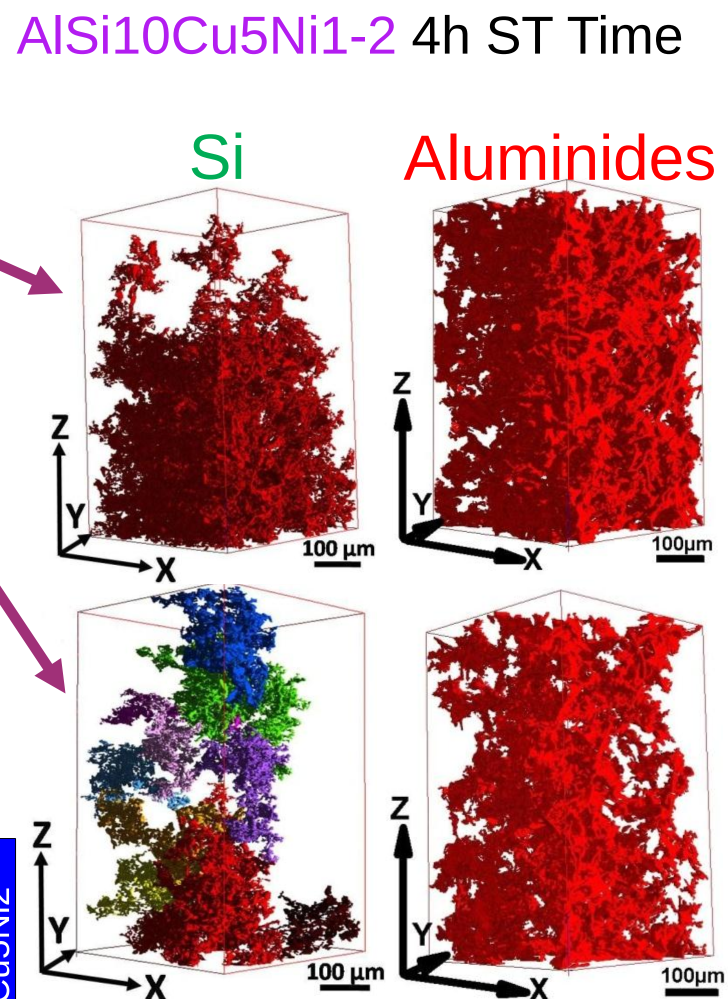
Reinforcement (high strength and E)

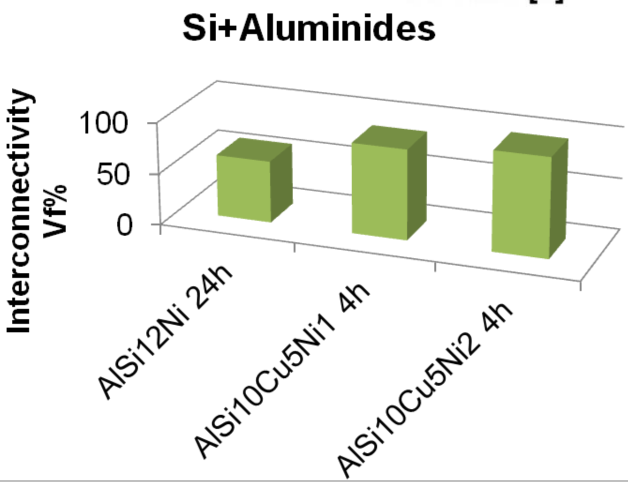


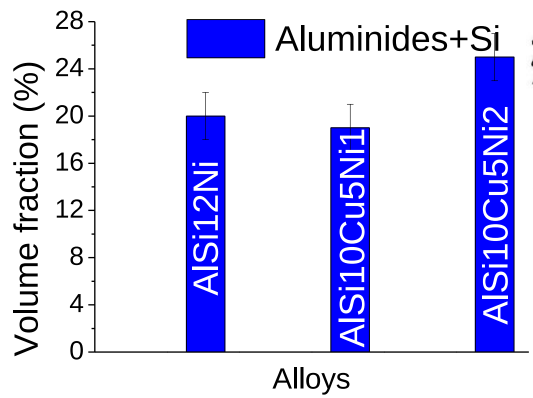
Asghar, Requena, Kubel – Mat Sci Eng A 527 (2010)
Asghar, Requena, Boller – Acta Materialia 59 (2011)

Strength of cast Al-Si alloys







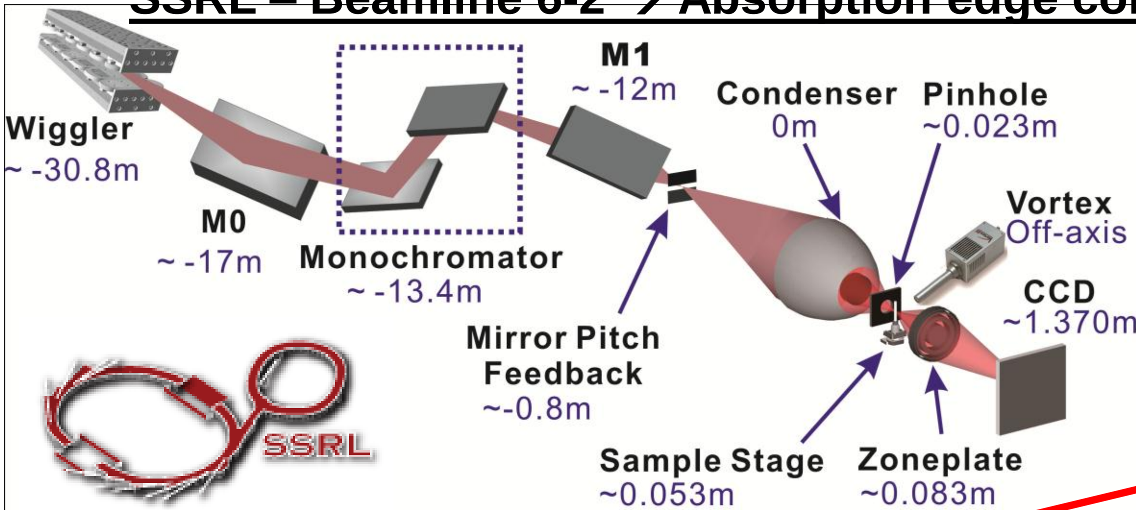


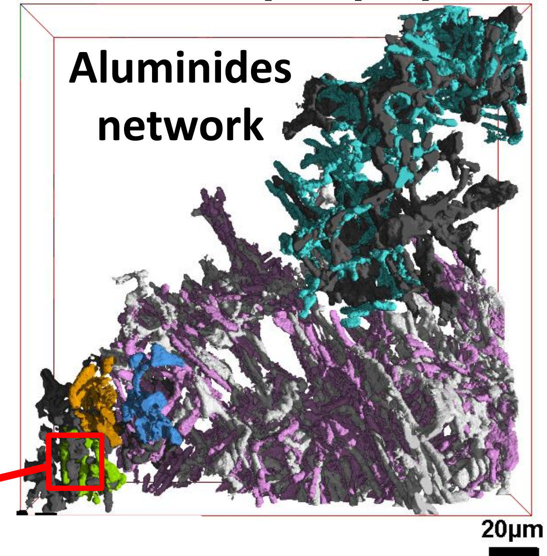
Asghar, Requena, Boller– Acta Materialia 59 (2011)

3D Elemental sensitive imaging

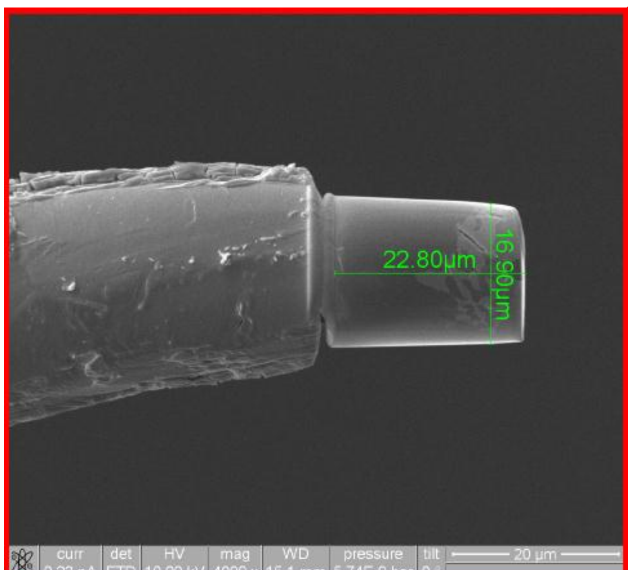
ESRF ID19
vox = $(0.3\ \mu m)^3$

SSRL – Beamline 6-2 → Absorption edge contrast

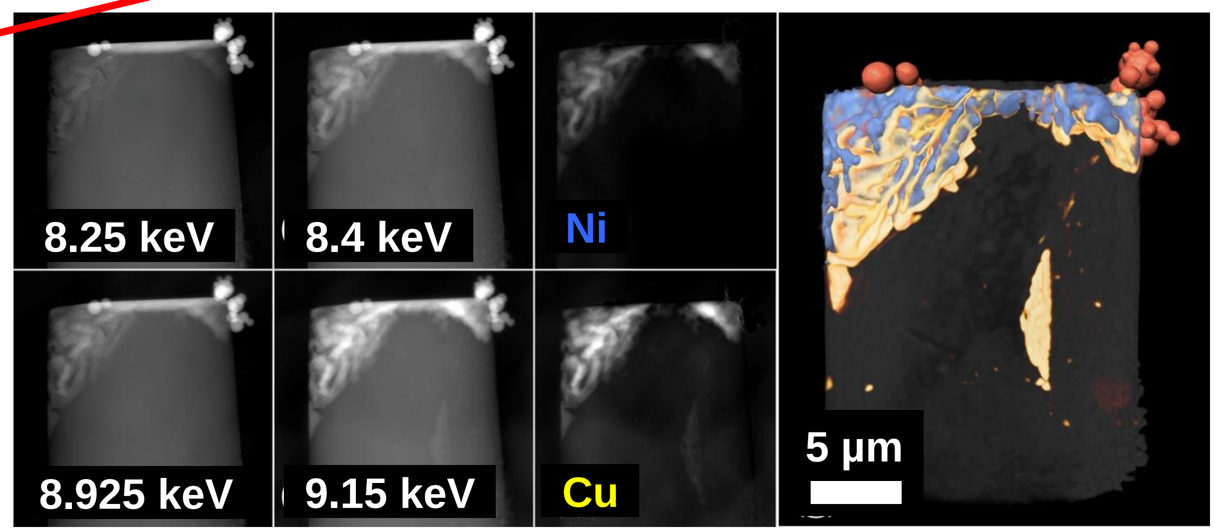




Sample prepared by FIB



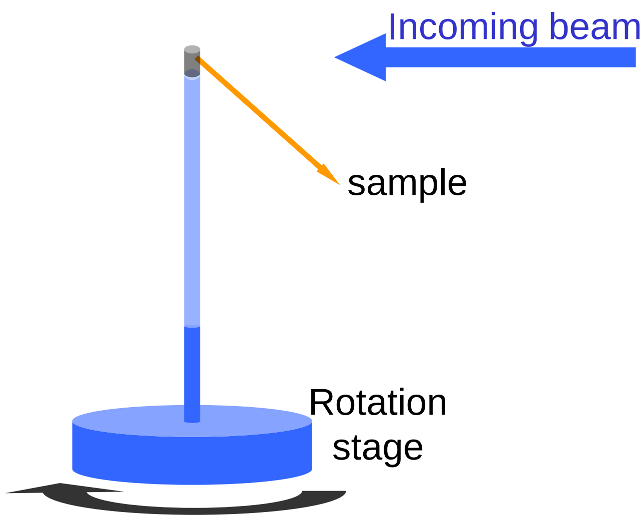
Cu-Ni Aluminide

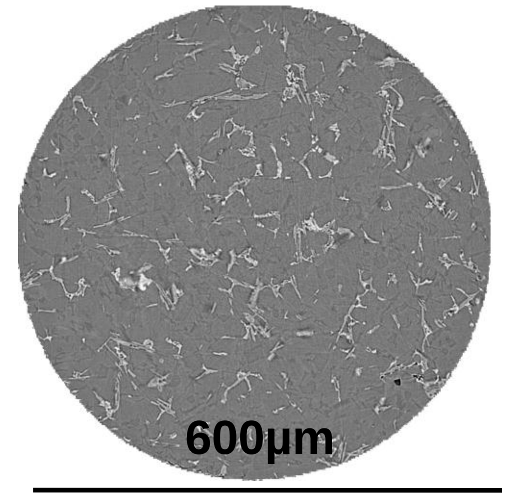


Vox size ~ $(24nm)^3$
Resol.~ 60nm

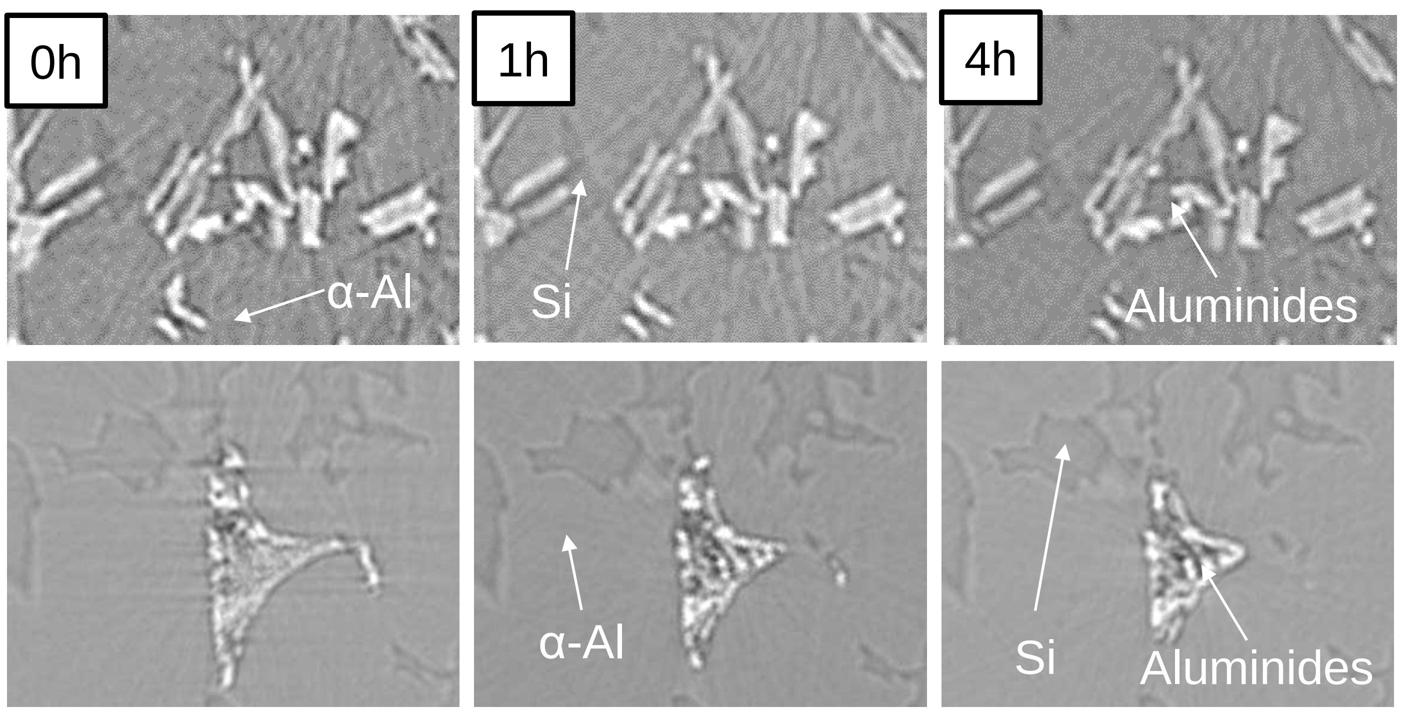
Liu, Mairer, Requena et al. Anal.&Bional.Chemistry, 2012

„In situ“ solution treatment of Al-Si alloys





(0.28 µm)³/voxel



AlSi12Cu5Ni2

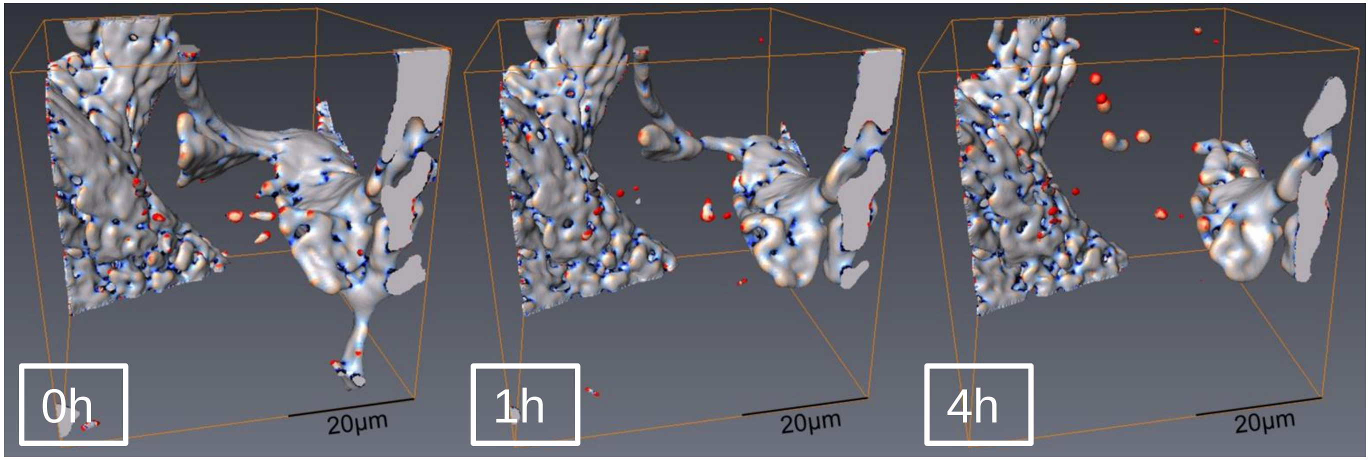
AlSi17Cu4

50µm

„In situ“ solution treatment of Al-Si alloys

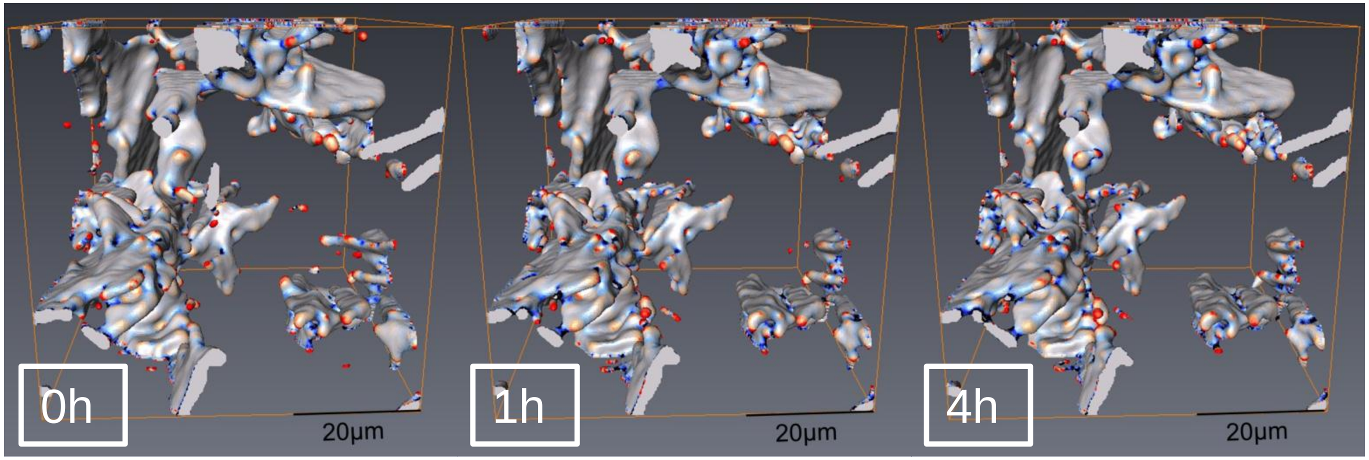
Stability of aluminides and their morphology after exposure 500°C

Hypereutectic alloy AlSi17Cu4



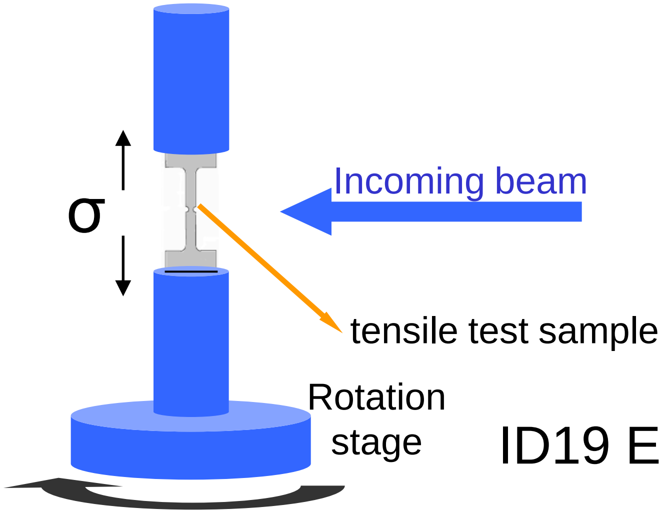
- Dissolution of Al_2Cu
- Spheroidisation

AlSi12Cu5Ni2 piston alloy



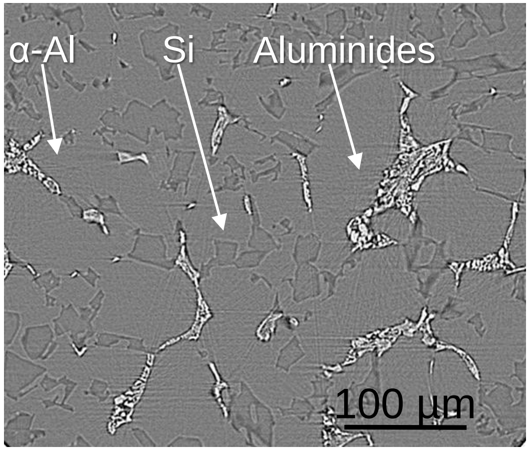
- Network of Aluminides stable during ST at 500°C

In situ tensile test of Al-Si alloys

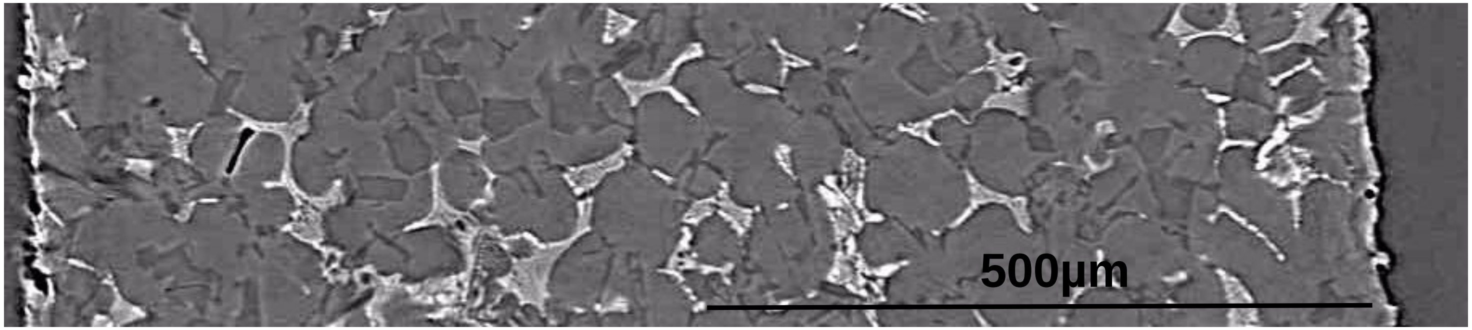


ID19 ESRF

AlSi17Cu4 alloy

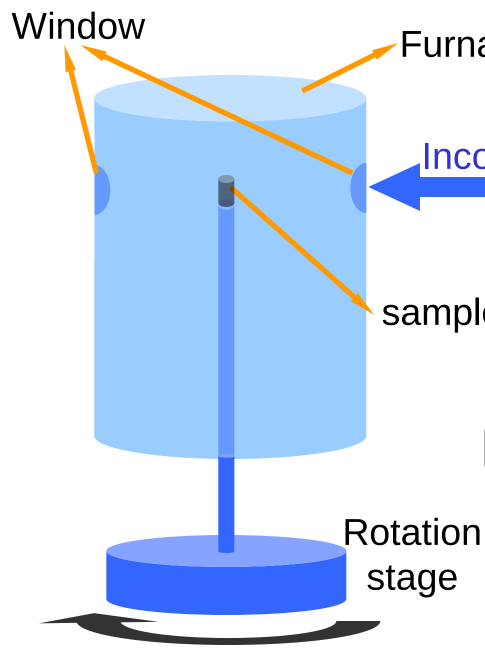


(1.4µm)³/voxel, 10s/tomo



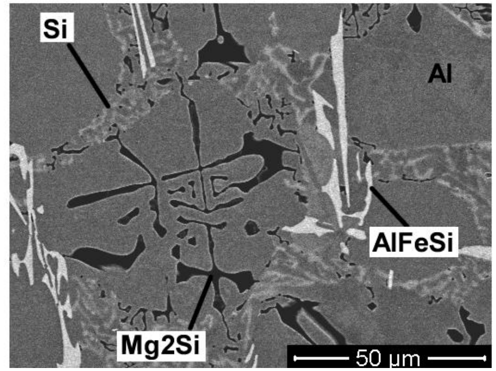
Formation and growth of cracks and pores

In situ solidification of Al-Si alloys



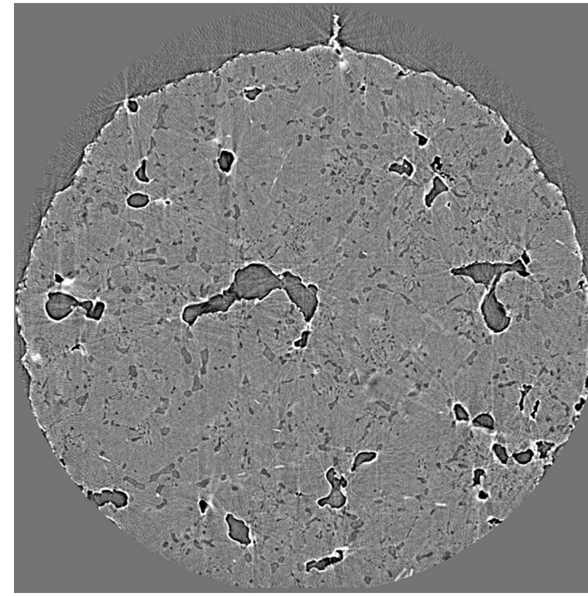
ID19-ID15
ESRF

AlMg5Si8 alloy

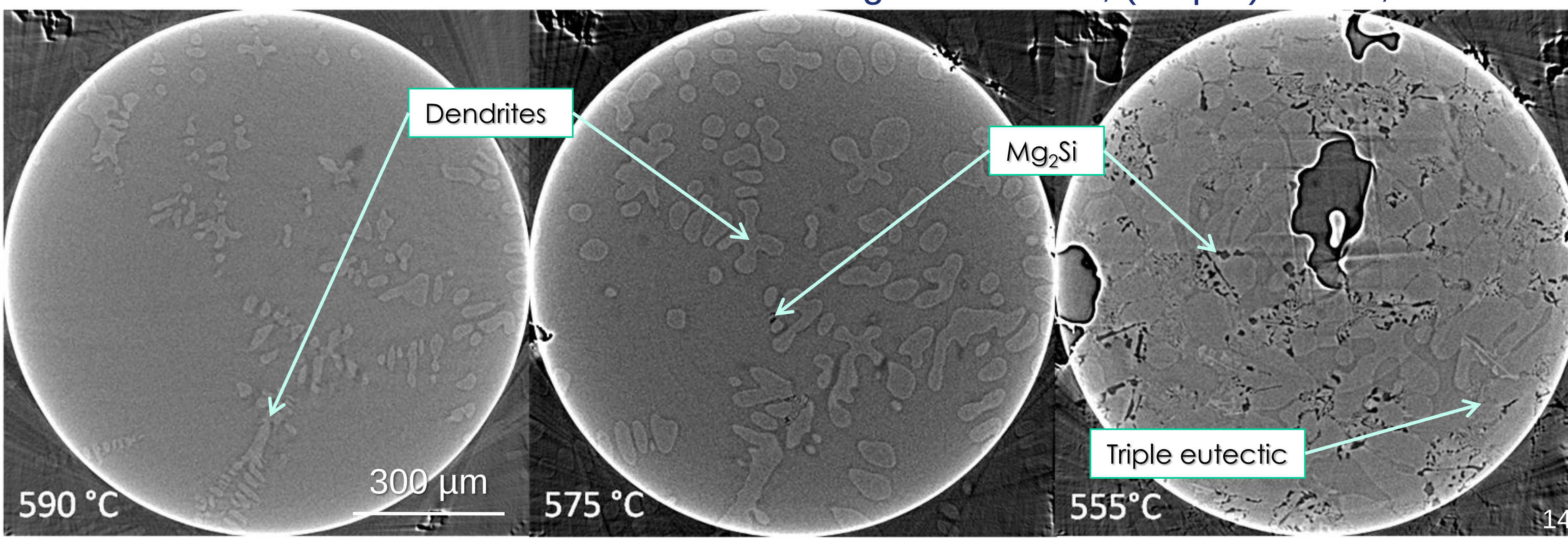


α-Al → α-Al/Mg_2Si eutectic → α-Al/Mg_2Si/Si triple eutectic

Tolnai, Requena, et al.– Acta Mater (2012)

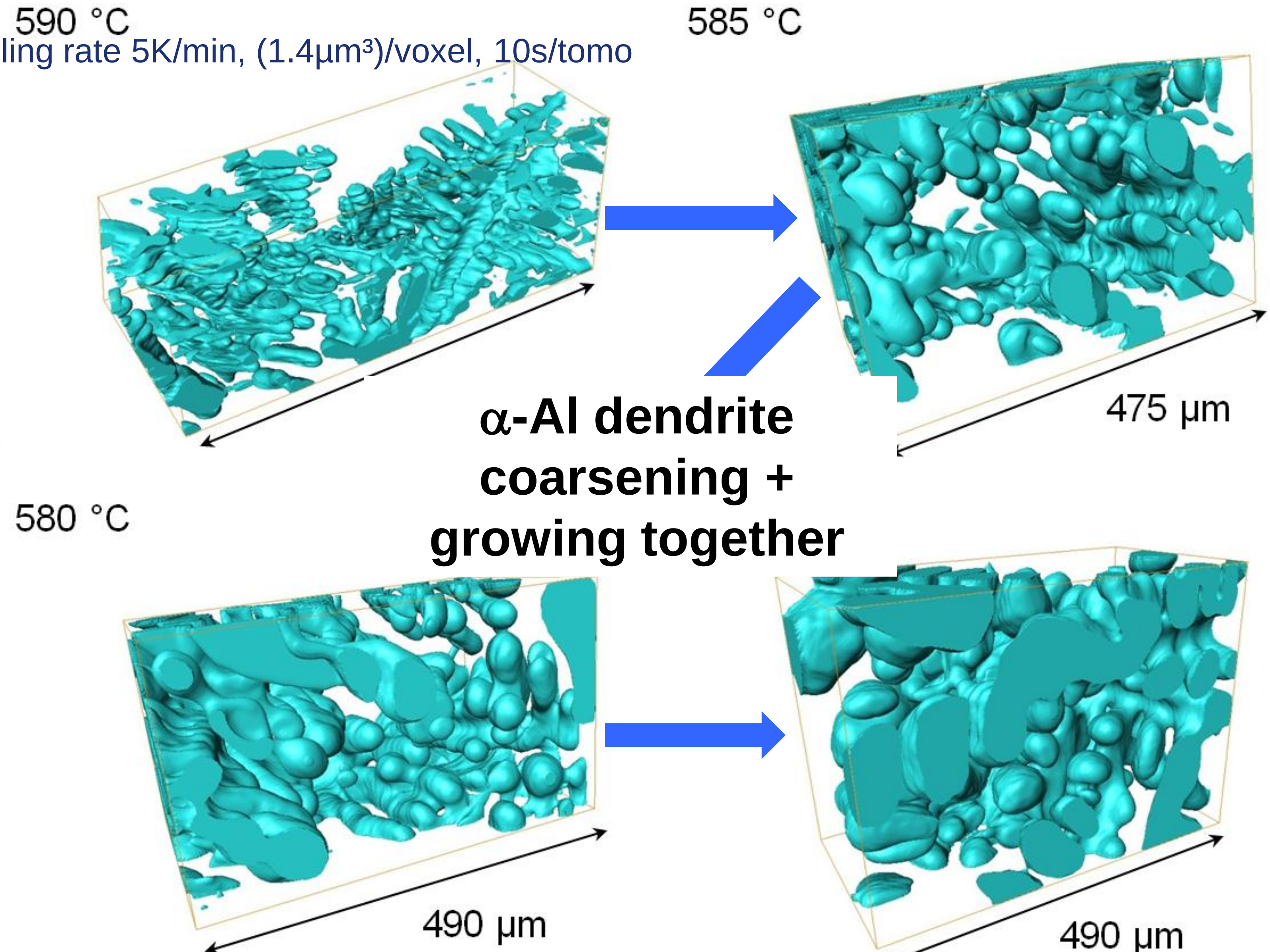


cooling rate 5K/min, (1.4µm)³/voxel, 10s/tomo



Evolution of α-Al dendrites with Temp

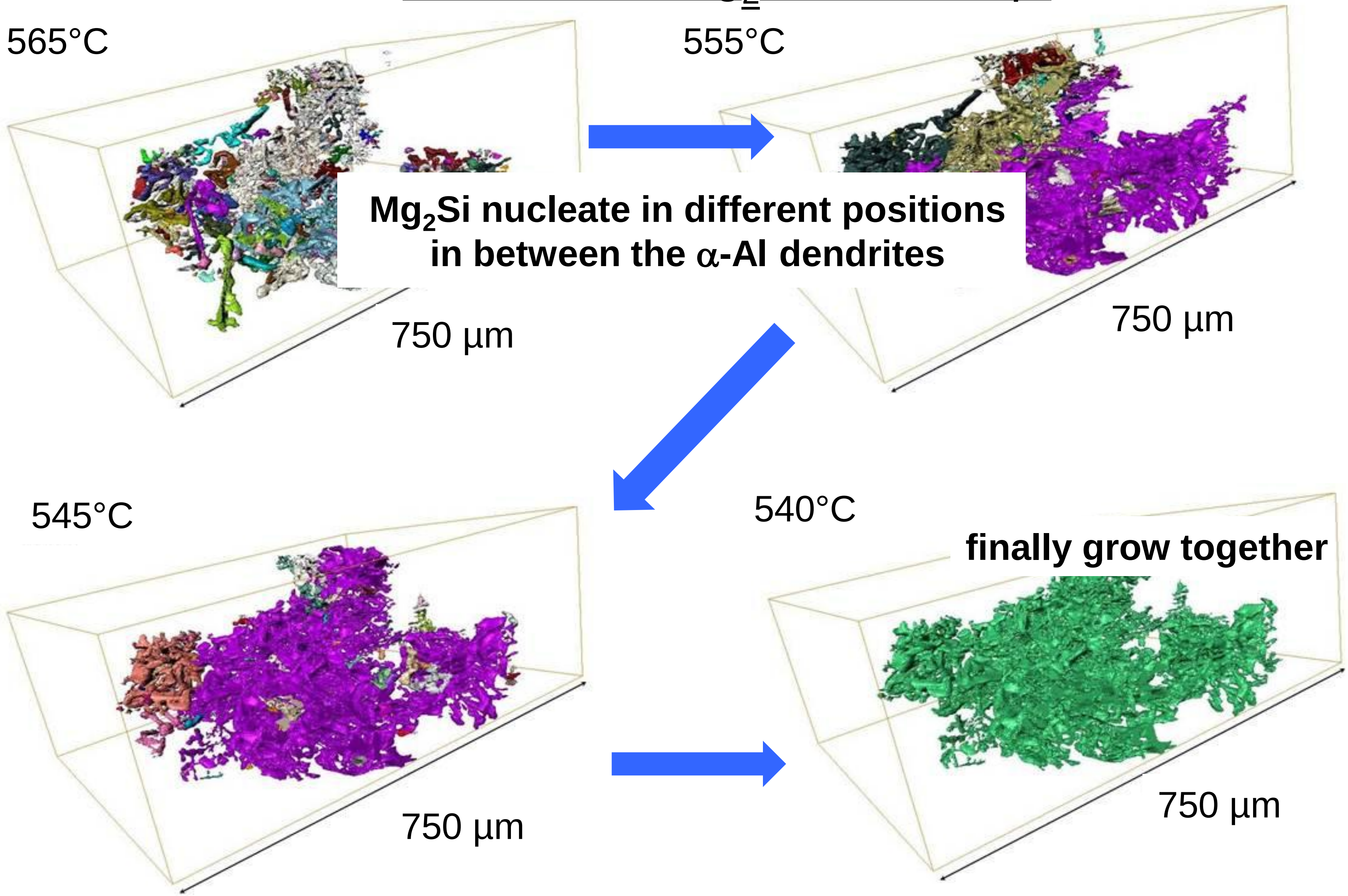
cooling rate 5K/min, (1.4µm³)/voxel, 10s/tomo



Tolnai, Townsend, Requena, Lendvai, Degischer– Acta Mater (2012)

In situ solidification of Al-Si alloys

Evolution of Mg_2Si with Temp



Tolnai, Townsend, Requena, Lendvai, Degischer– Acta Mater (2012)

Summary

2D characterization is sometimes not enough → 3D characterization of heterogeneous materials is necessary for cases where:

-**The connectivity of the phases and/or contiguity between phases must be analysed. These two parameters can play a decisive role on the mechanical properties of heterogeneous lightweight materials.**

-**The physical sectioning of the material would affect its internal state (e.g. distribution of internal stresses during loading, thermal cycling, etc.)**

-**New in situ characterization methods have opened new opportunities to help to understand dynamic processes.**

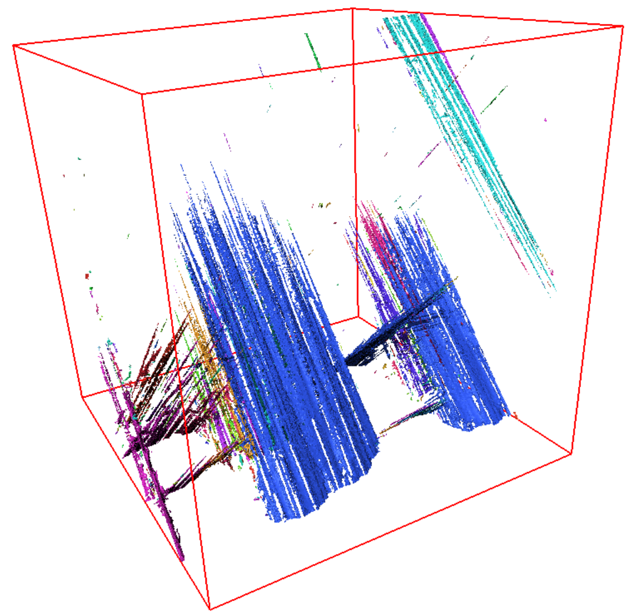
Acknowledgements

Vienna Group:, Pere Barriobero Vila, David Canelo Yubero, (Hans Peter Degischer), Ricardo Fernández Gutiérrez, Robert Koos, (Georg Fiedler), Marta Rodríguez Hortalá, (Zahid Asghar), Johannes Jonke, (Maya Jaber), Edith Asiemo, Christian Zaruba

External: G. Garcés (CENIM-Madrid), D. Tolnai (HZG-Germany), F. Sket (IMDEA-Madrid), L. Salvo (INP-Grenoble), E. Maire, J. Adriane, C. Landron (INSA-Lyon), C. Poletti, Fernando Warchomicka (TU-Graz), Michael Schöbel (TUM-FRMII)

Beamline Scientists

Elodie Boller (ESRF-ID19)
Peter Cloetens (ESRF-ID19, ID22)
Marco Di Michiel (ESRF-ID15)
Mario Scheel (ESRF-ID15)
Thomas Buslaps (ESRF-ID15)
Thilo Pirling (ILL-Salsa)
Florian Meirer (SSRL-6-2)
Andreas Stark (DESY)
Norbert Schell (P07-DESY)



In Situ study of damage in a CFR-Polymer

M. Rodríguez Hortalá, G. Requena, F. Sket

ZERSTÖRUNGSFREIE
PRÜFUNG UND
TOMOGRAFIE

nemak

BÖHLER

KOLBENSCHMIDT

THANK YOU!