Ni-base Superalloy Single Crystals

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Fundamental Aspects of Materials Science and Engineering (FAMSE)

Objective of this lecture:

We want to understand in which field high temperature materials are needed and how they are produces. We will focus in the Brightman process. The microstructure of superalloys must be understood and how it leads to good mechanical properties at high temperatures.

(1) Which applications depend on high temperature materials
(2) The role of SFB Transregio 103
(3) Solidification process of single crystal materials
(4) Microstructure of superalloys
(5) Dislocation movement in the γ/γ'-Microstructure

Text books/recommended reading

- G.W. Meetham, The Development of Gas Turbine Materials, Applied Science Publishers, London, 1981.
- M. McLean, Directionally Solidified Materials for High Temperature Service. The Metal Society, London, 1983.
- M. Durand-Charre, The Microstructure of Superalloys, CRC Press, 1997.
- T.M. Pollock, R.D. Field, Dislocations and High-Temperature Plastic Deformation of Superalloy Single Crystals, in: F.R.N. Nabarro and M.S. Duesbery (Eds.), Dislocations in Solids, Elsevier Sciences, 2002, pp. 566–568.
- R.C. Reed, The Superalloys: Fundamentals and Applications, Cambridge University Press, Cambridge, 2006.
- many others

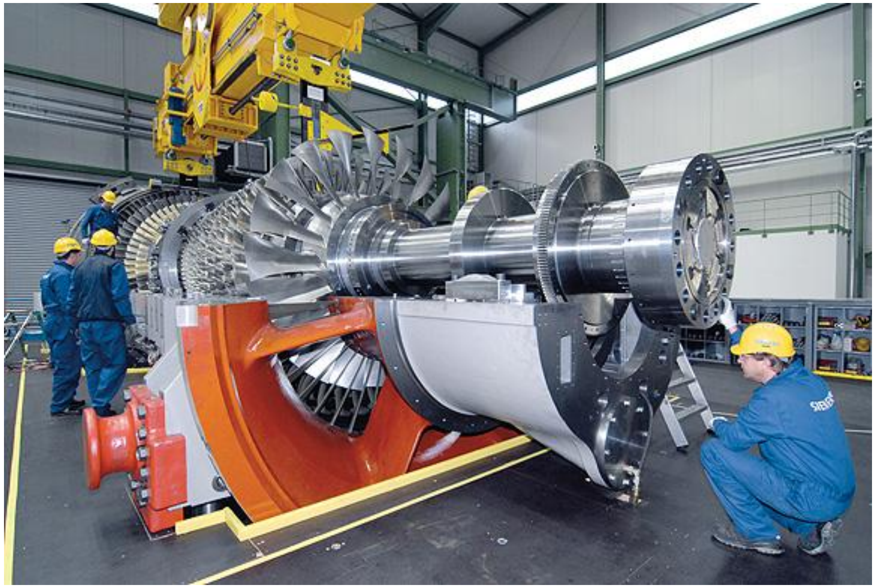
Ni-base superalloy single crystals are used to make first stage blades for gas turbines operating and aero engines and in power plants

Key materials for air traffic and energy supply

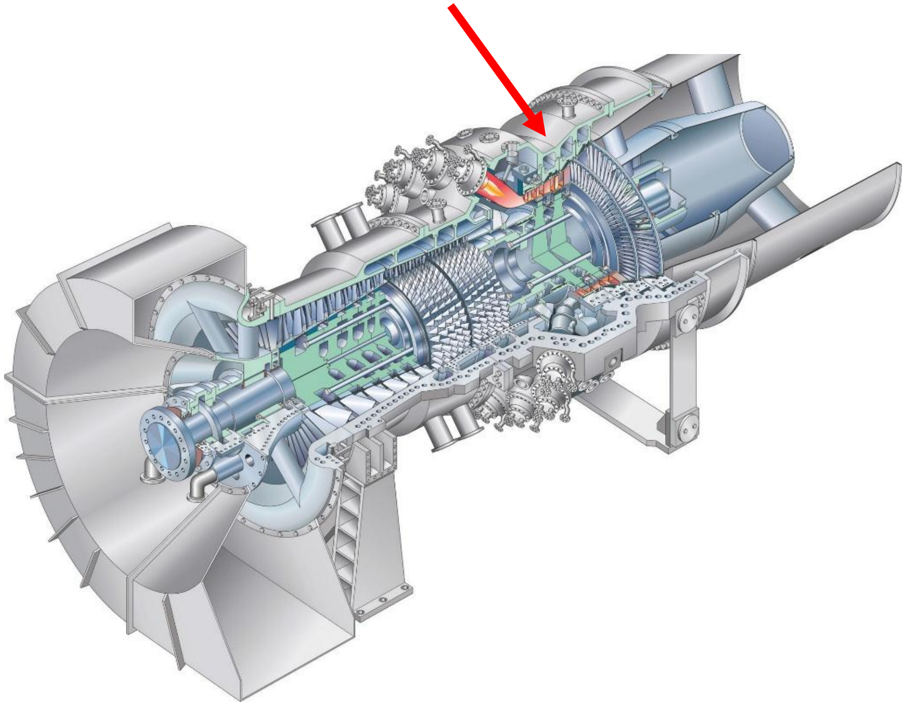
Airbus A380
Source: Pressefotos MTU München

Kraftwerk Irsching
Source: Pressefotos Siemens Mülheim an der Ruhr

Solarturm Jülich
Source: DLR



SGT5-8000H
50 Hz, 400 MW, 60,7 %

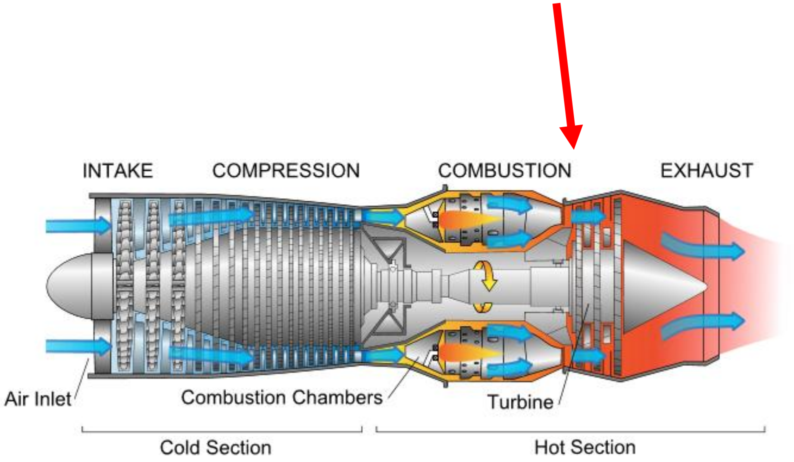


Gas turbine (energy)

Source: Pressefotos Siemens Mülheim an der Ruhr

Airbus A380
Source: Pressefotos MTU München

LM 2500+
30 MW
5000 rpm



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Section summary – turbine blades

Ni-base single crystal superalloys are used to make first stage blades for gas turbines in power plants and aero engines.

There is a permanent driving force for increased thermal efficiency, this is why first stage blades operate at extremely high temperatures (1000 °C). There is interest to increase performance, safe fuel and to make better use of fossil ressources. There is also interest in replacing expensive alloy elements without loosing performance (like creep strength). And there is an interest in having components with long exploitable service lives.

SFB/TR 103

SFB/Transregio 103 on Single Crystal Superalloys (SX)

scale bridging modelling
(atoms, phases, dislocations, constitutive equations)

scale bridging characterisation
(SEM, TEM, atom probe)

SFB/Transregio 103
SUPERALLOY SINGLE CRYSTALS

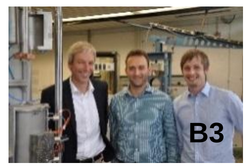
processing and manufacturing
(melting, casting, heat treatements)

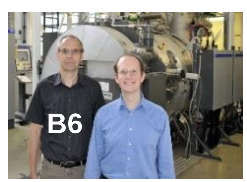
mechanical and thermodynamic/kinetic property assessement
(elastic constants, creep, thermal fatigue, rafting, ...)

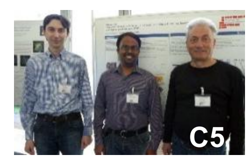
SFB/Transregio 103
SUPERALLOY SINGLE CRYSTALS

I: 2012 - 2015

Structure: blocks, projects and people







May 2012, Erlangen

December 2012, Bochum

May 2013, Erlangen

SFB/Transregio 103

SUPERALLOY SINGLE CRYSTALS

I: 2012 - 2015

Interaction weeks

February 2015, Grainau

December 2013, Bochum

May 2014, Erlangen

December 2014, Bochum

June 2014, Giens

SFB/Transregio 103
SUPERALLOY SINGLE CRYSTALS

May 2014, Erlangen

I: 2012 – 2015
Cross sectional groups

Ni-base SX

Mechanical properties

Scale bridging modelling

Co-base SX

SFB/Transregio 103
SUPERALLOY SINGLE CRYSTALS

May 2014, Erlangen

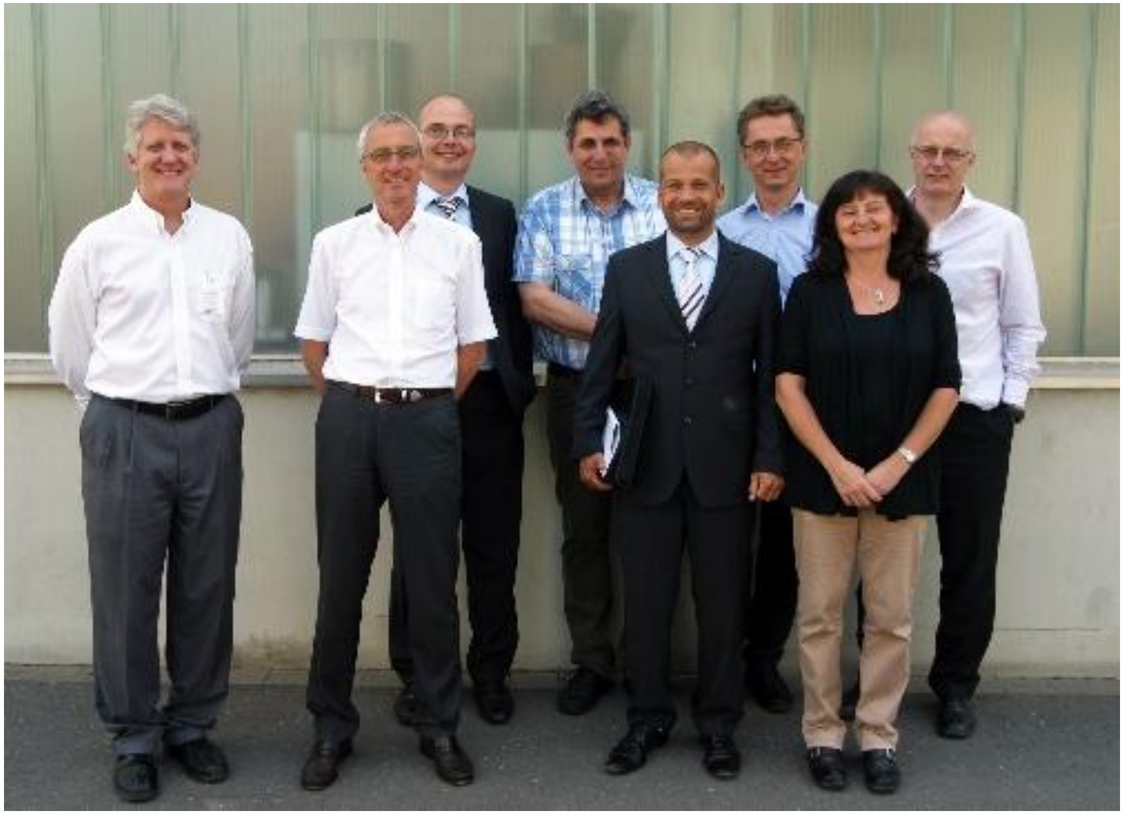
I: 2012 - 2015

Technical academic advisory board (TAAB)

O. Lüsebrink, Siemens Energy

G. Eggeler, RUB

J. Gabel, MTU Aeroengines



R. Reed, Univ. Oxford

T. Pollock, UCSB

M. Mills, OSU

R.F. Singer, FAU

T. Wagner, Doncasters

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Section summary – SFB/TR 103

The SFB/TR 103 is a collaborative research center which focusses on the scientific basis for a new generation of single crystal superalloys.

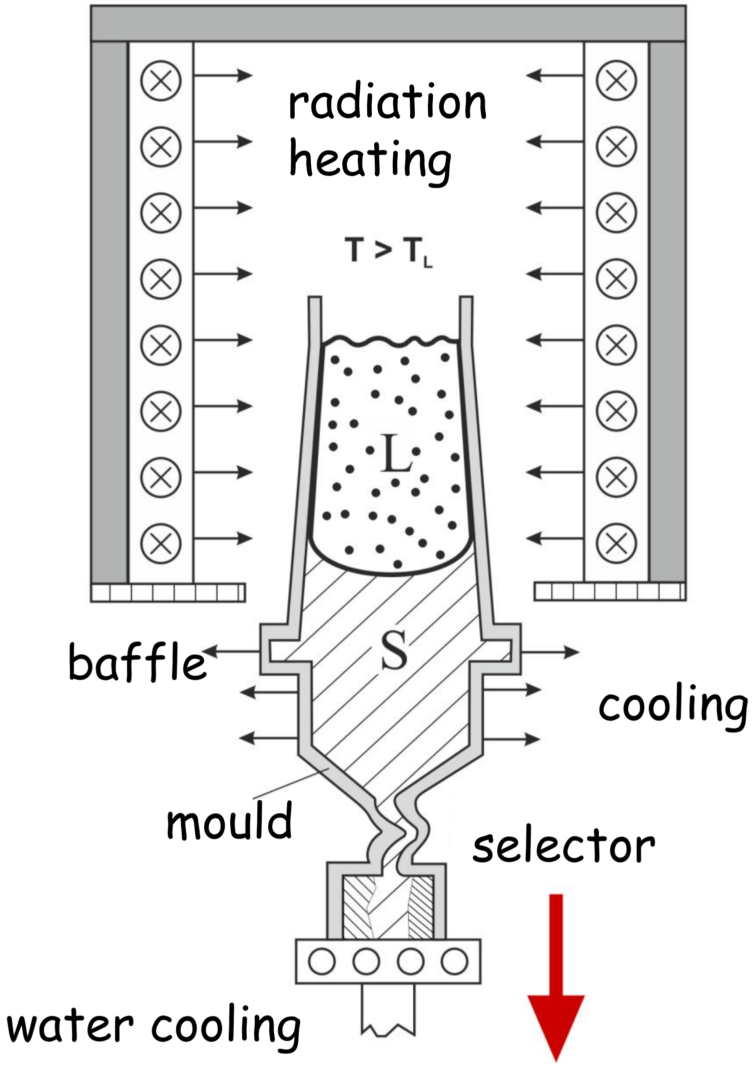
The two universities RUB Bochum and FAU Erlangen/Nürnberg join forces together with MPIE (Düsseldorf), DLR (Köln) and FZ Jülich.

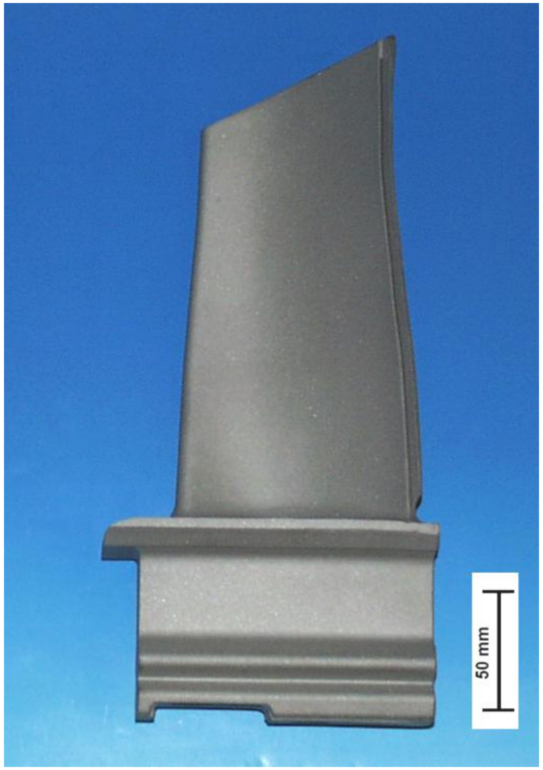
The SFB/TR 103 started work in 2012. It is now in its second funding period.

All members of SFB/TR 103 meet two times a year. There are cross sectional groups which help to initiate collaboration between the 20 individual projects of SFB/TR 103.

Ni-base superalloy single crystals are produced by a directional solidification process, followed by a multiple step heat treatment

Bridgman solidification process



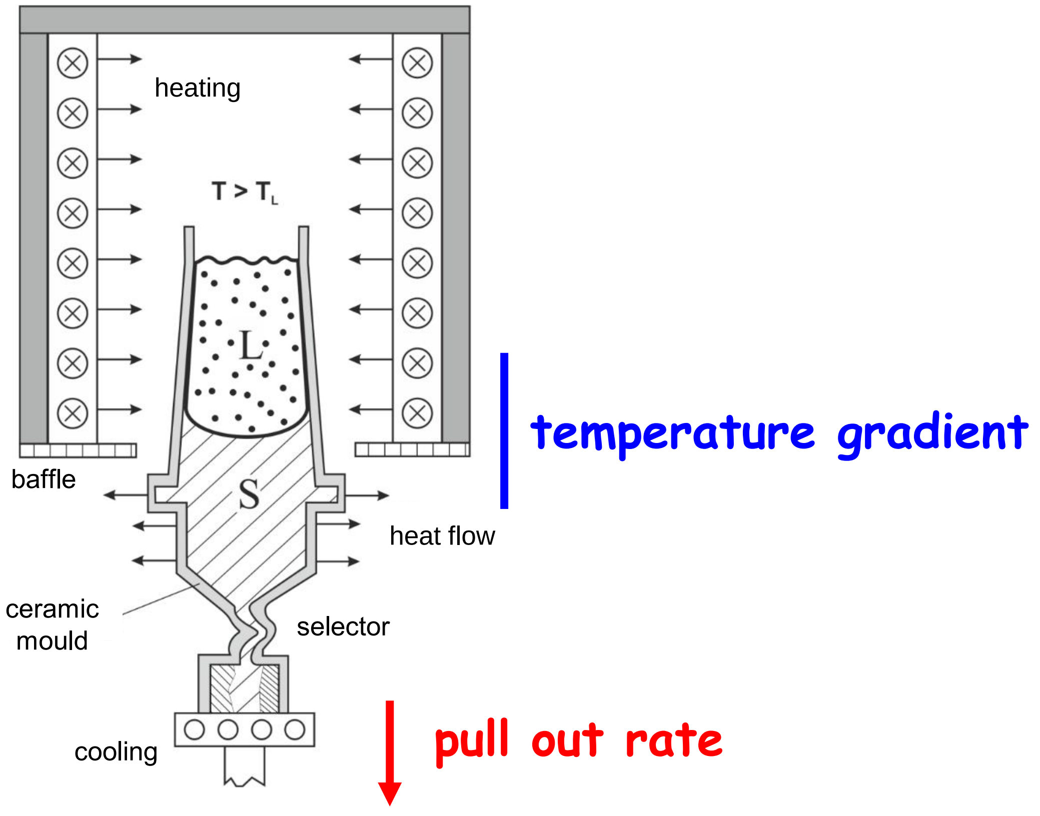


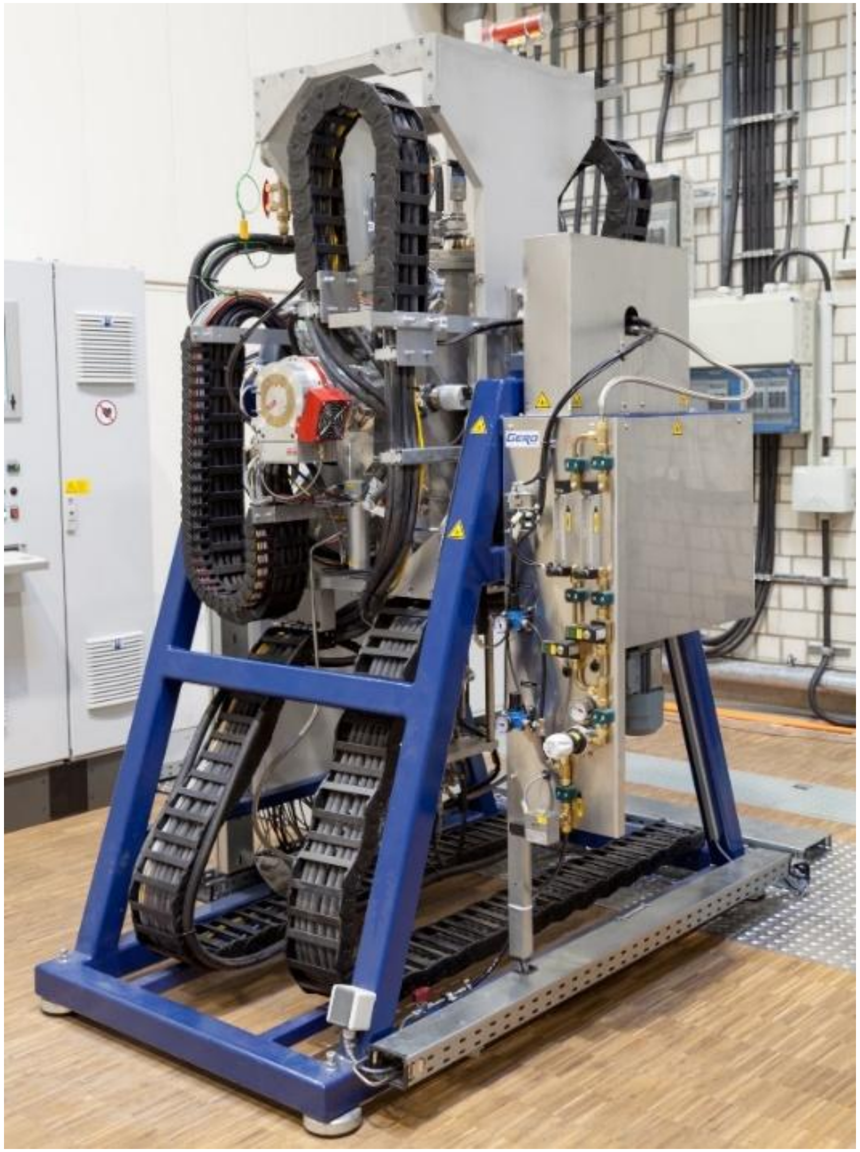
Source: Lehrstuhl WTM FAU (R. Singer),
University of Erlangen

(our partner in SFB/TR 103)

Critical Parameters

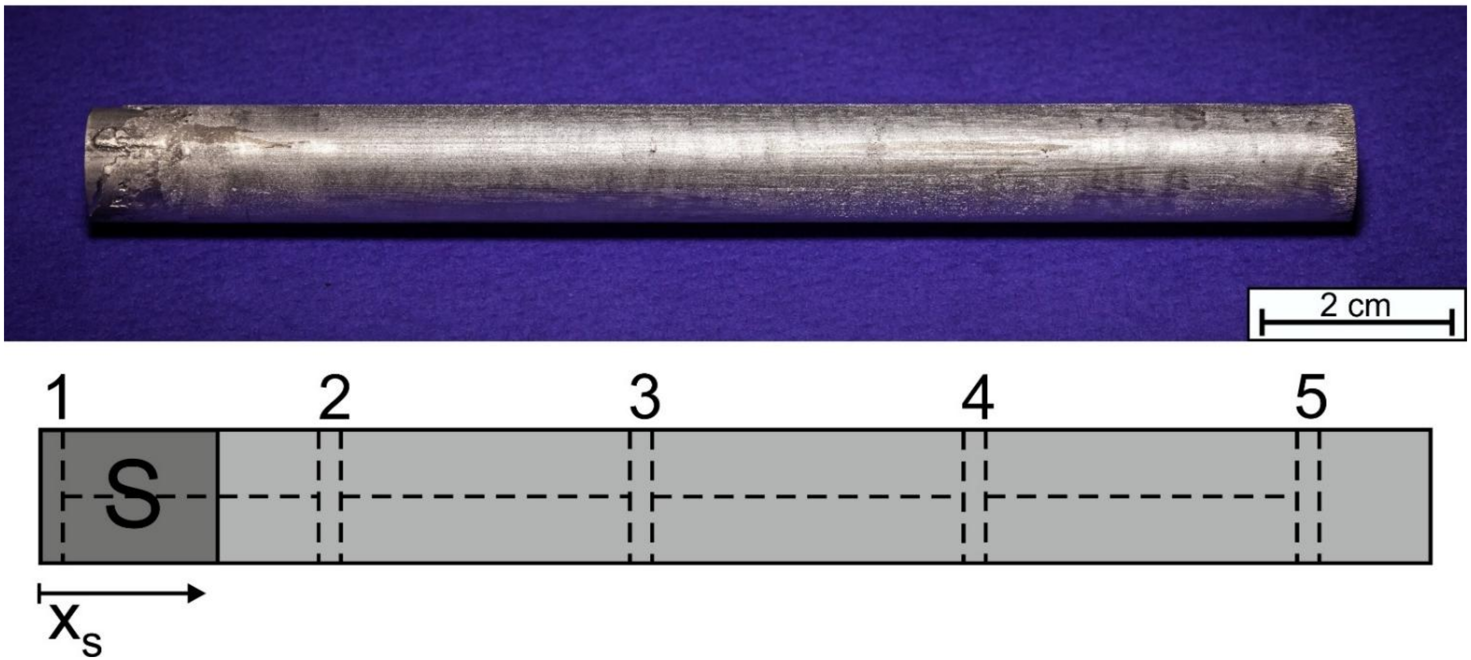
LWW Lehrstuhl Werkstoffwissenschaft
materials science and engineering

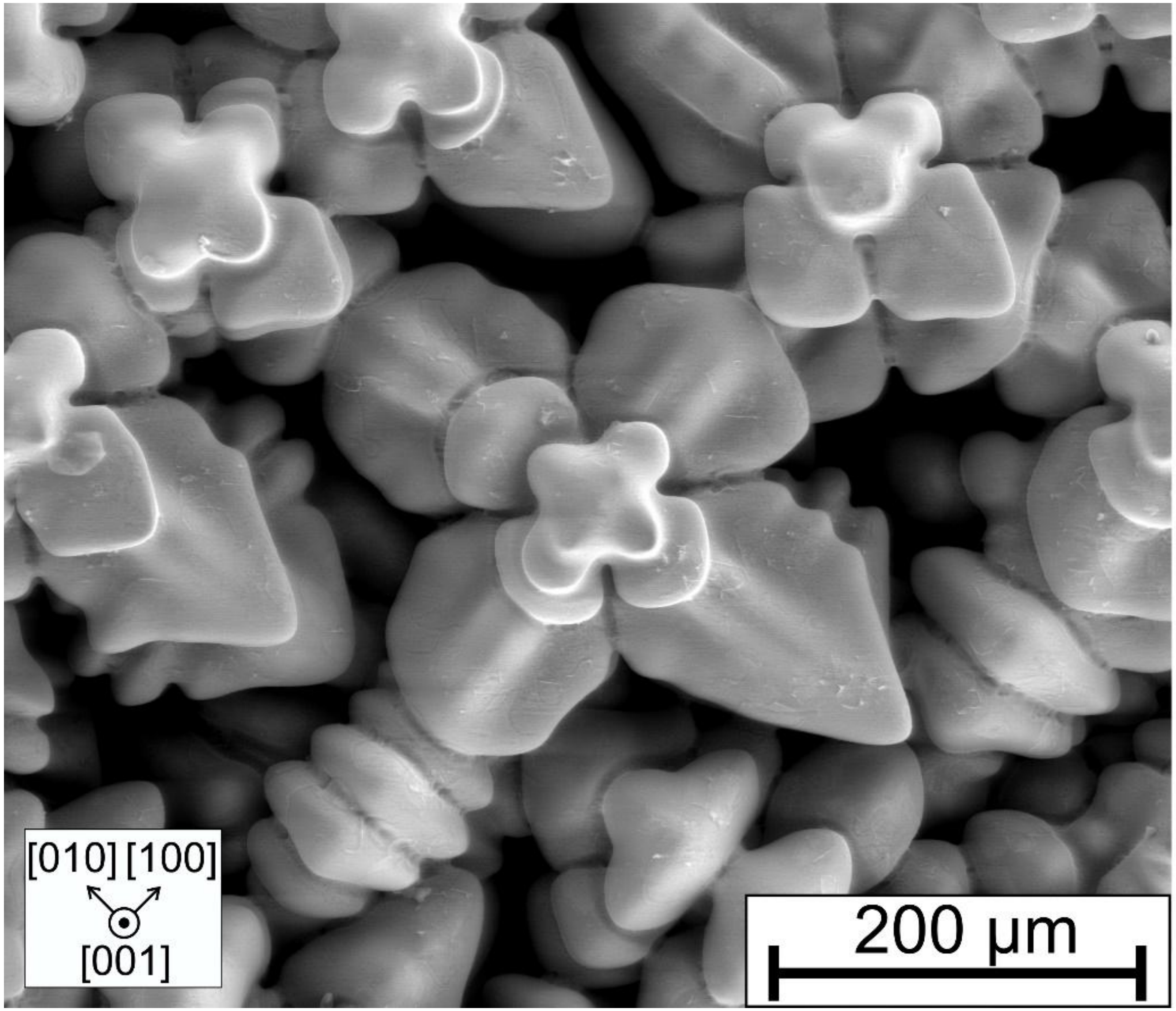


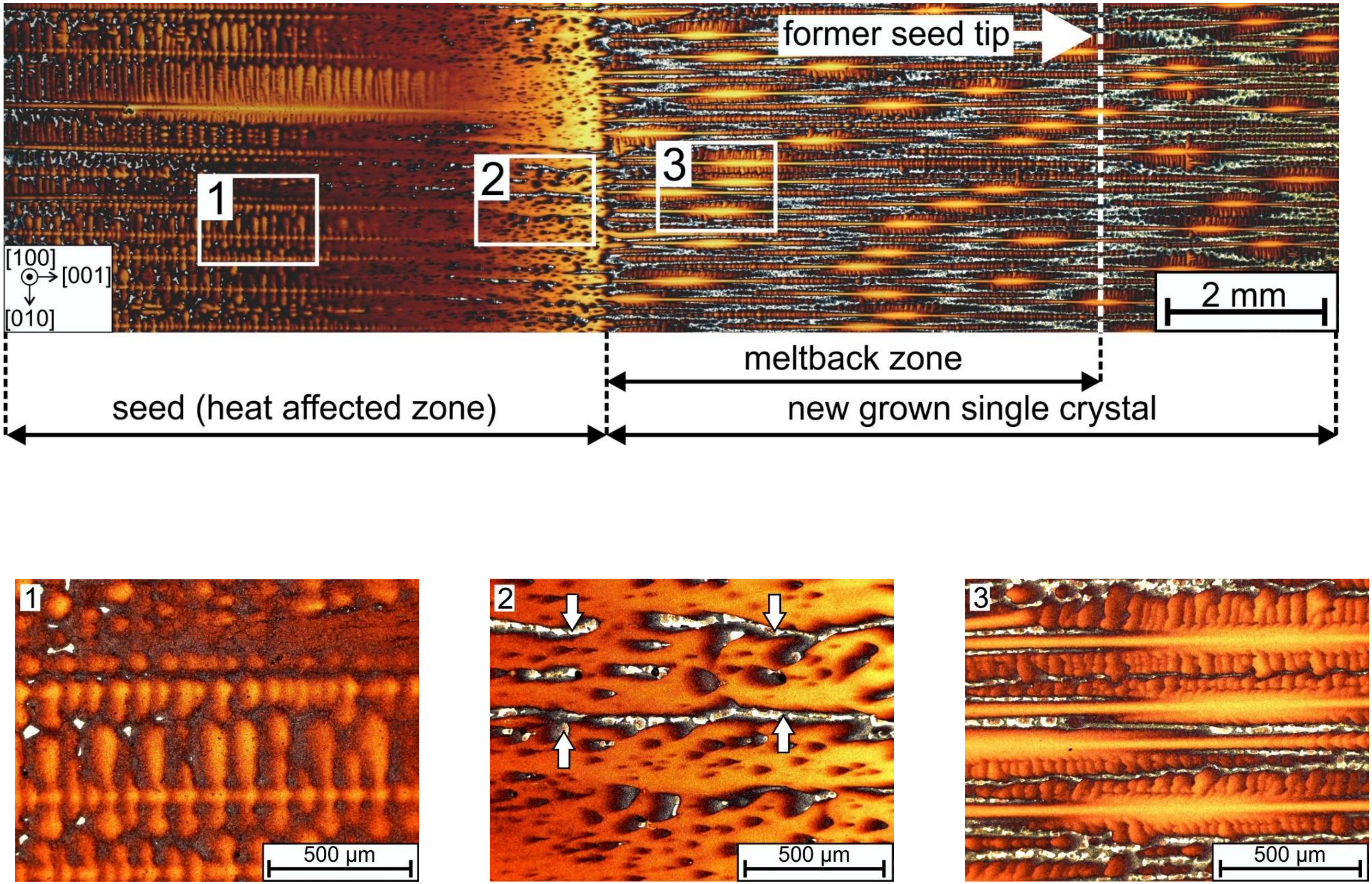


our Bridgman furnace
at Intstitute for Materials

We use a seed technique




[010] [100]
[001]
200 µm



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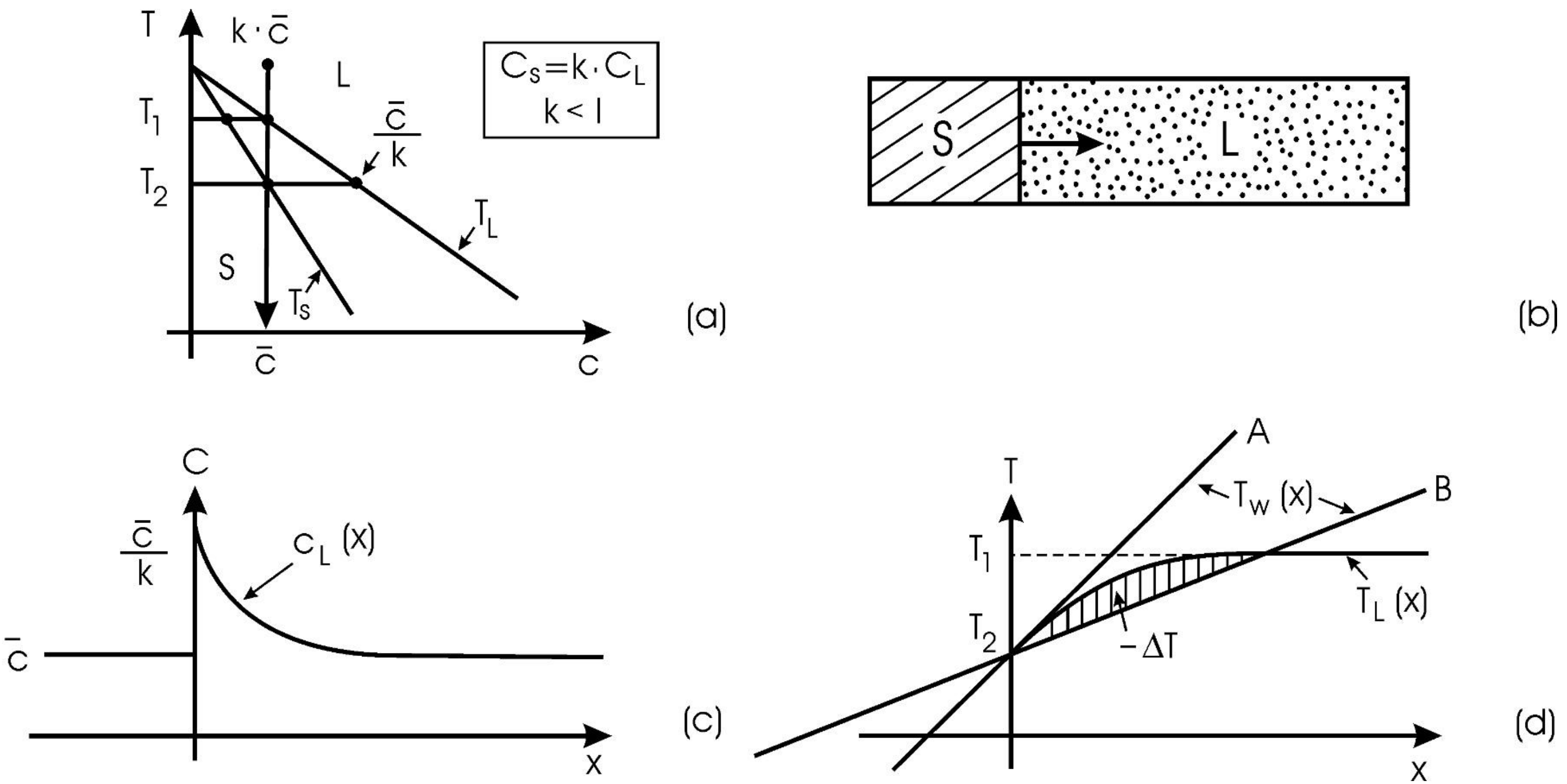
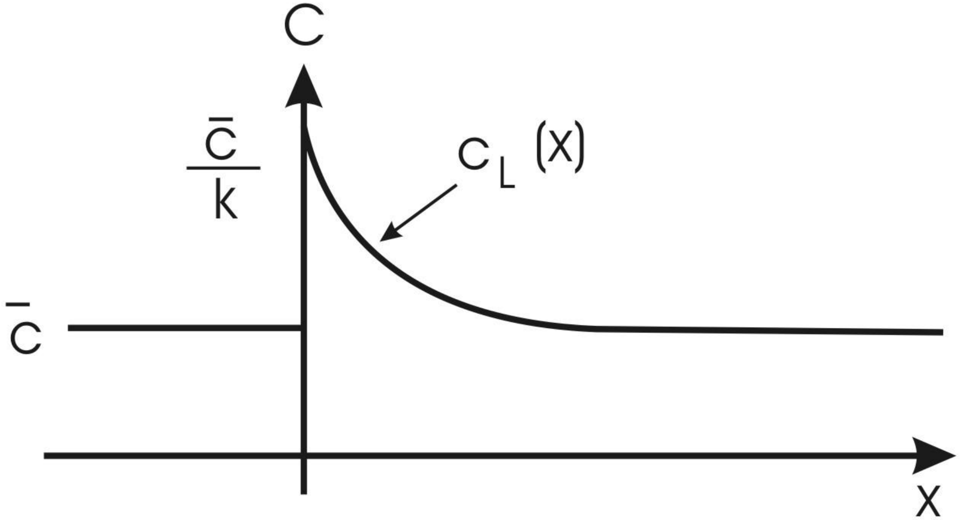
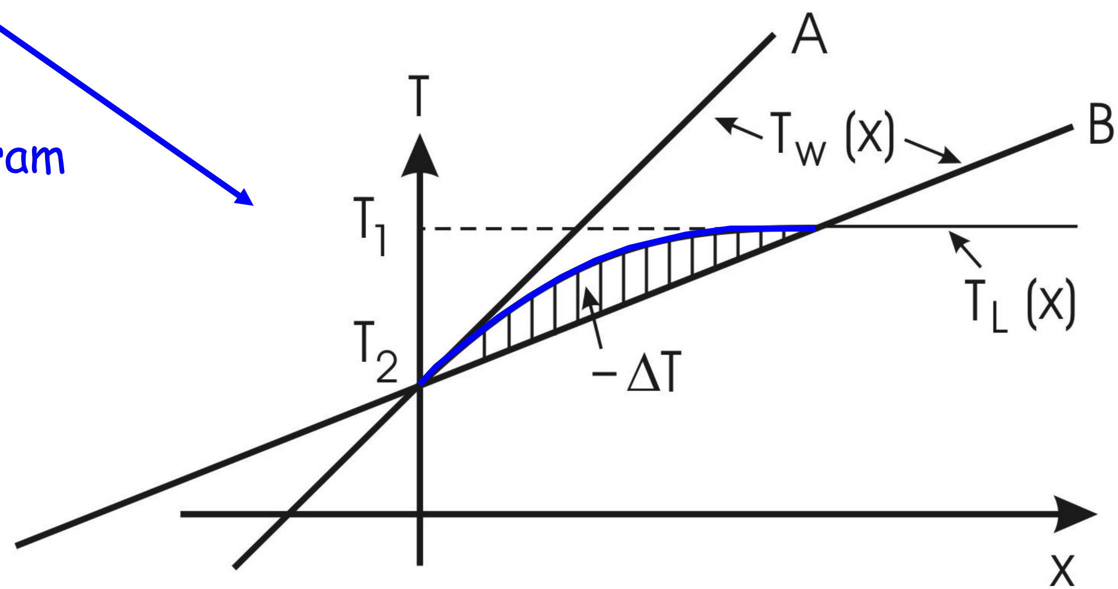
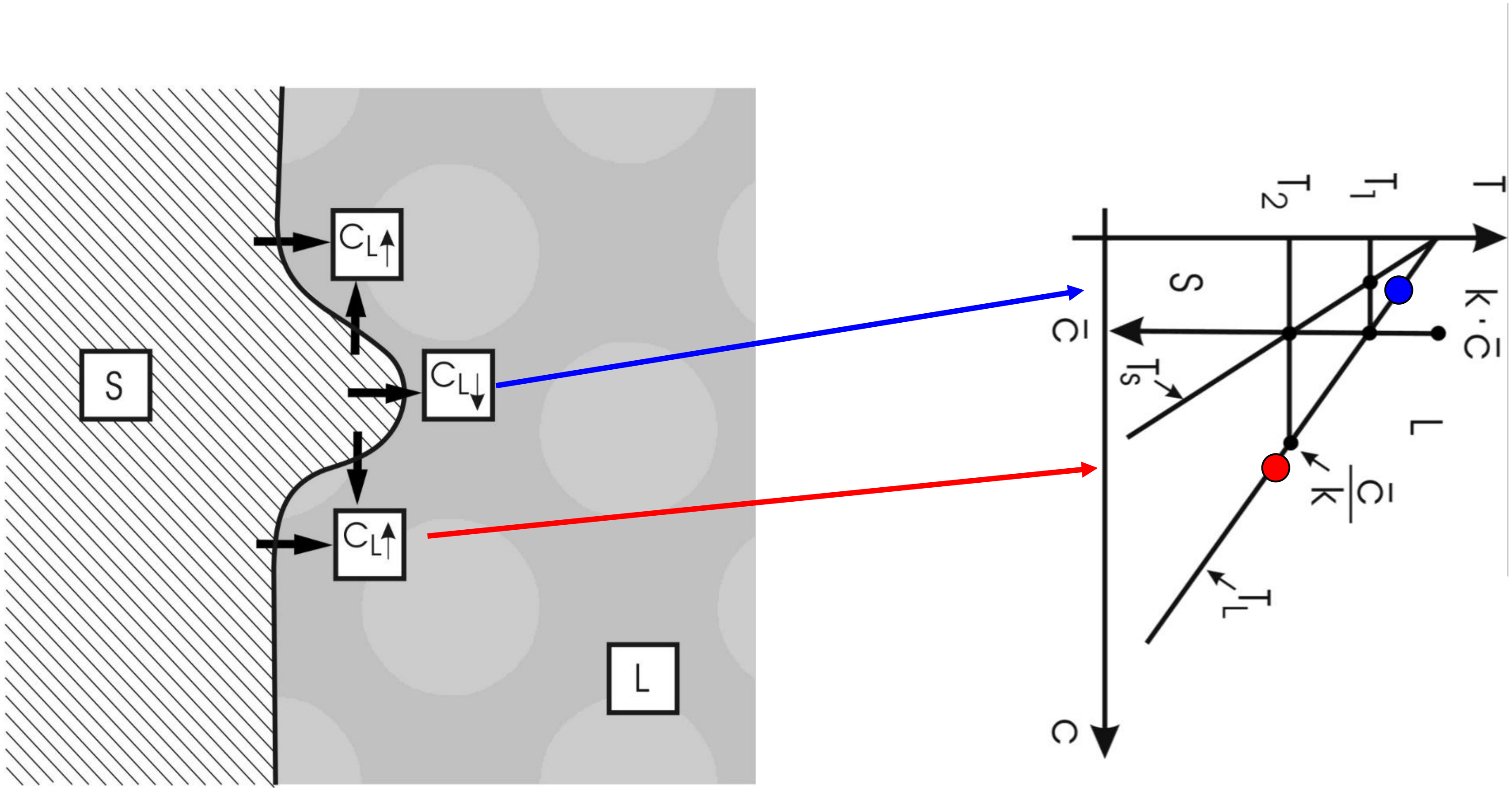
Reminder: Constitutional undercooling





from c(x) with phase diagram
-> T(x)

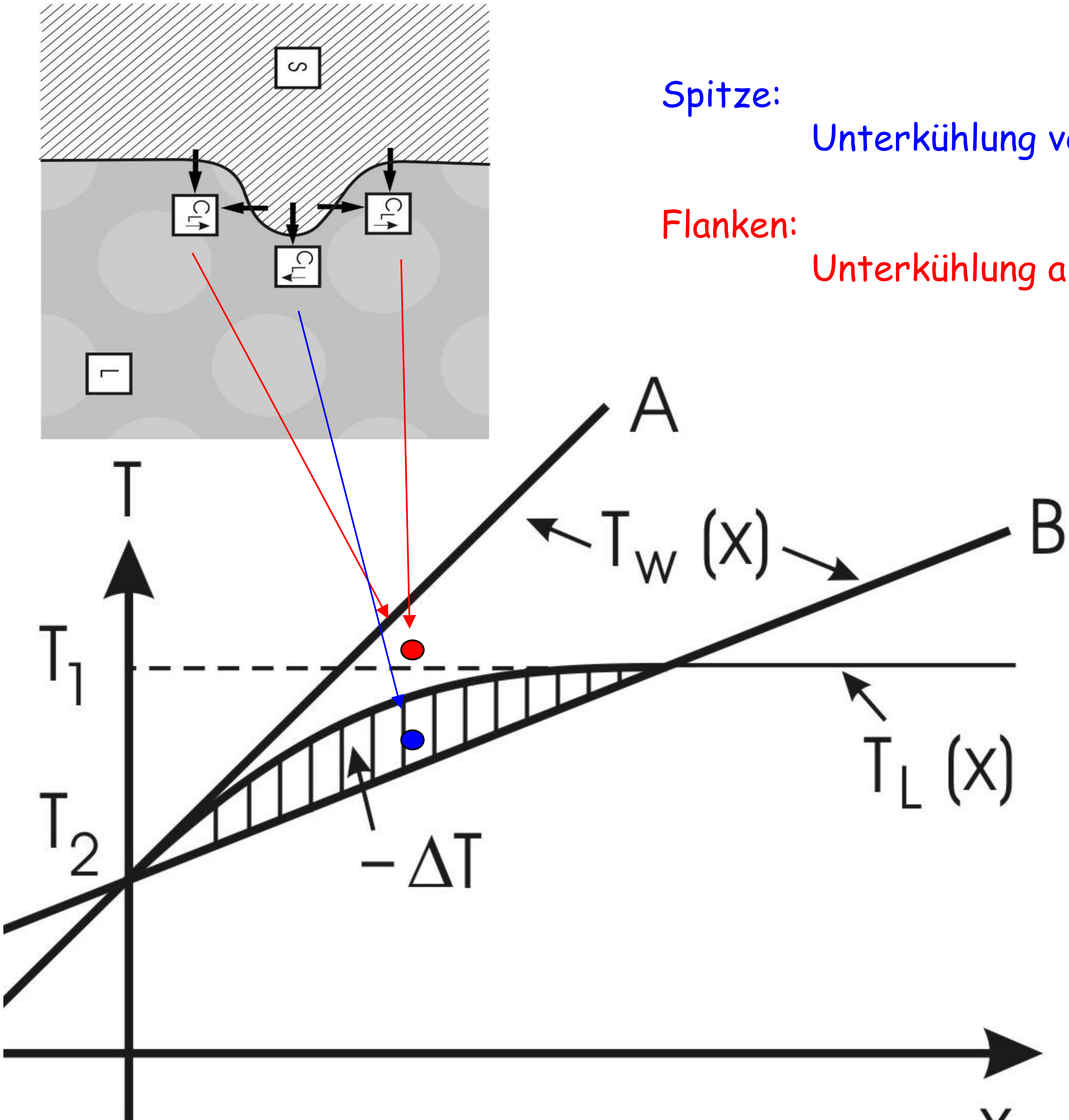


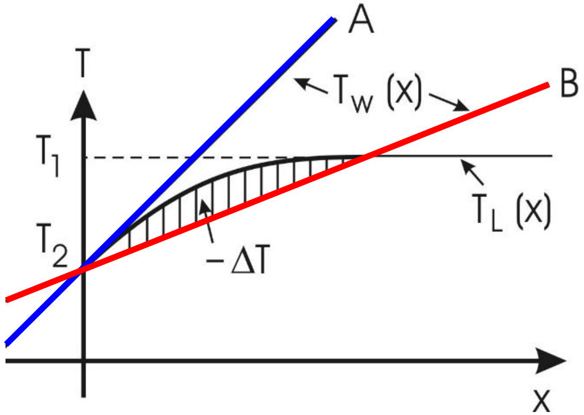


S
L
$C_L\uparrow$
$C_L\downarrow$
$C_L\uparrow$
T
T_1
T_2
S
L
$k \cdot \bar{C}$
$\bar{C}$
T_S
$\frac{\bar{C}}{k}$
T_L
C

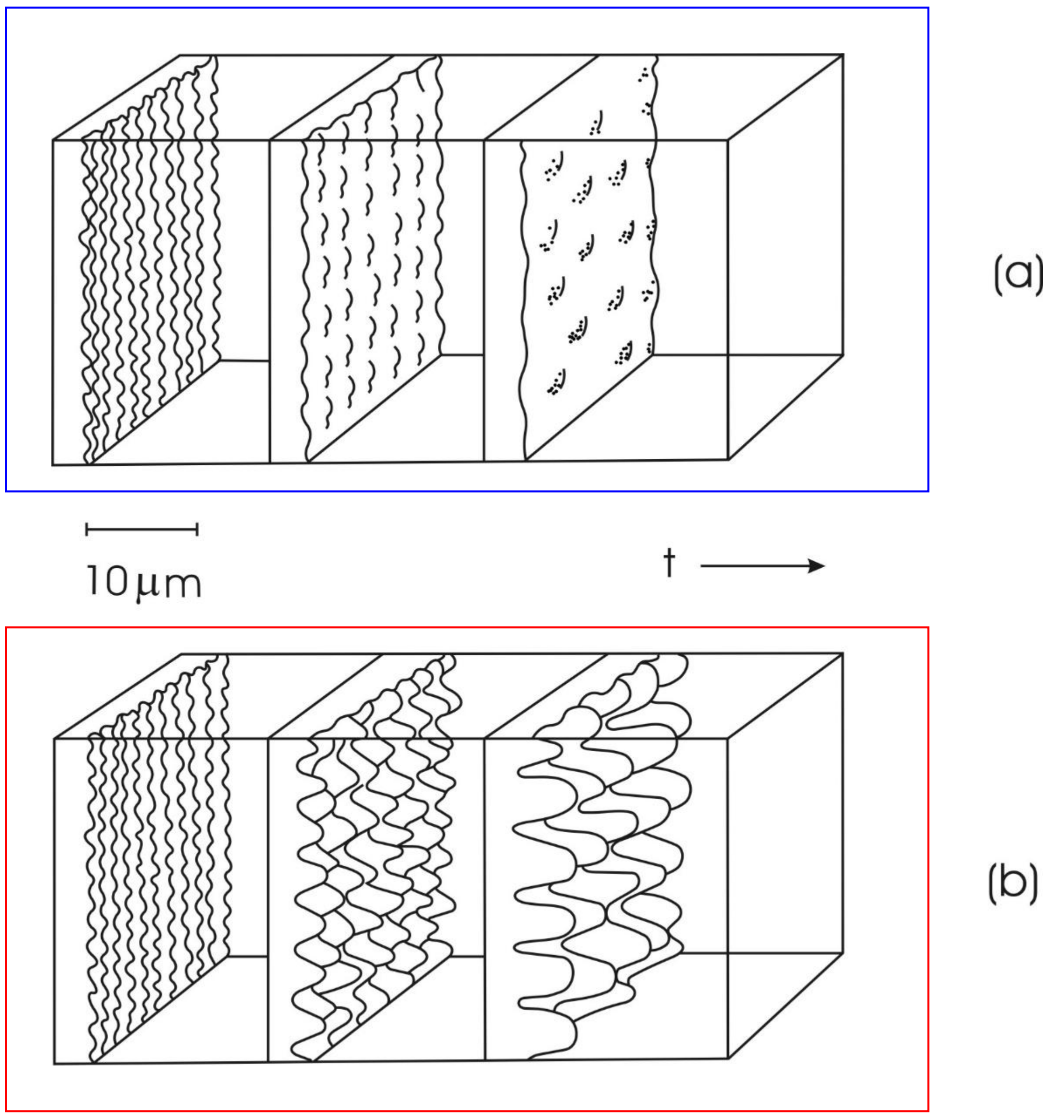
Spitze:
Unterkühlung verschärft

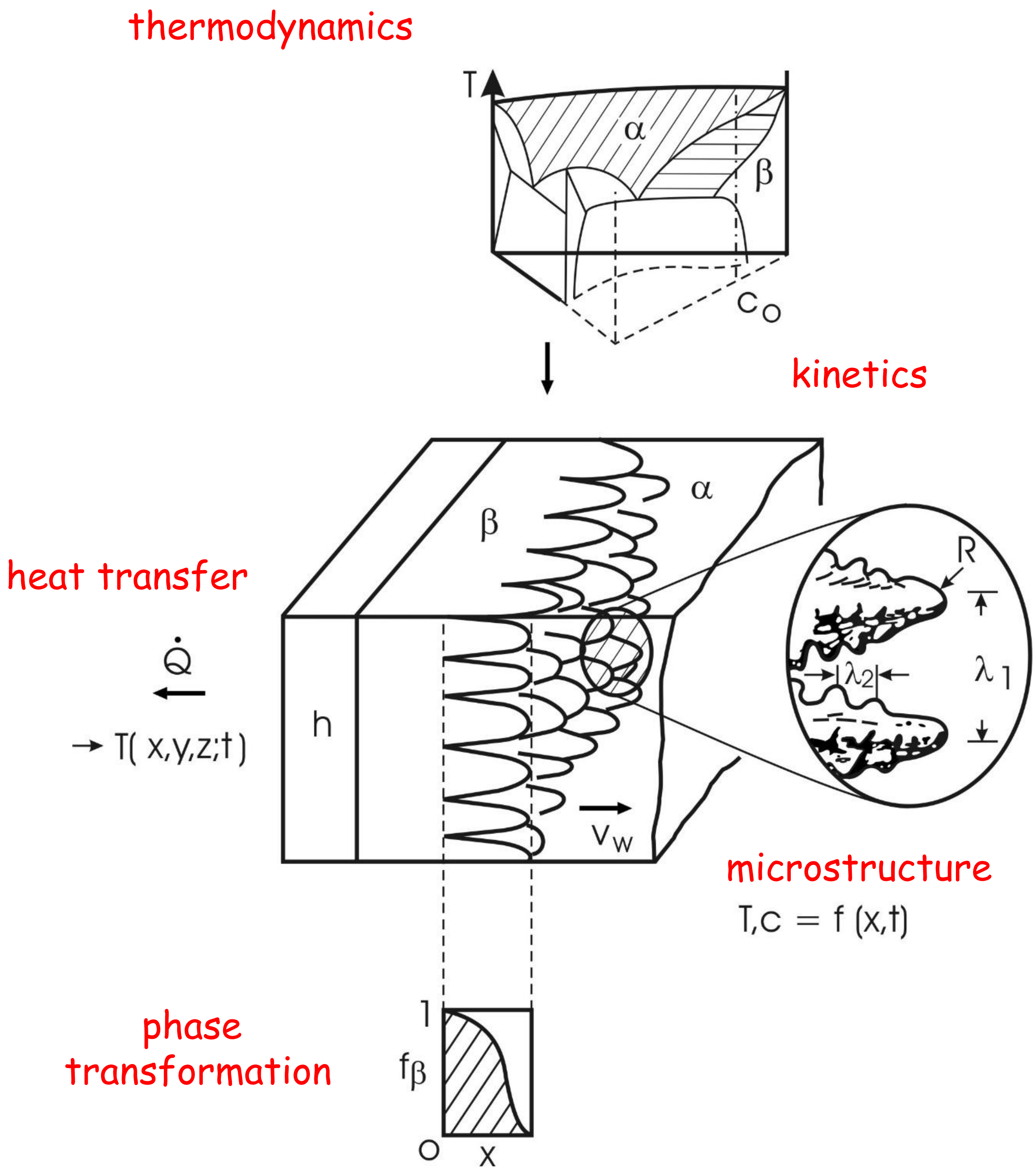
Flanken:
Unterkühlung abgebaut





flat and irregular
interfaces




thermodynamics
T
α
β
c_o
kinetics
α
β
heat transfer
$\dot{Q}$
→ T(x,y,z;t)
h
R
λ_1
λ_2
v_w
microstructure
T,c = f (x,t)
phase
transformation
1
f_β
o
x

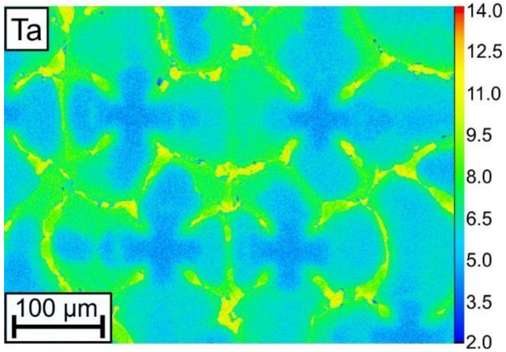
Cast microstructure - large scale heterogeneity

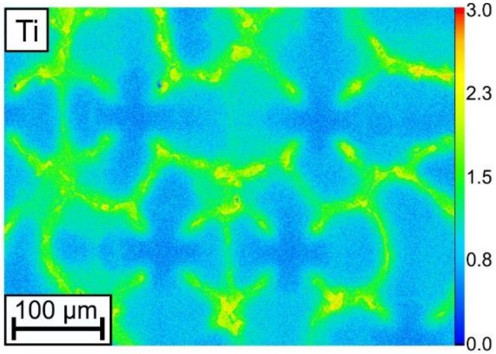
DENDRITES / INTERDENDRITIC REGIONS

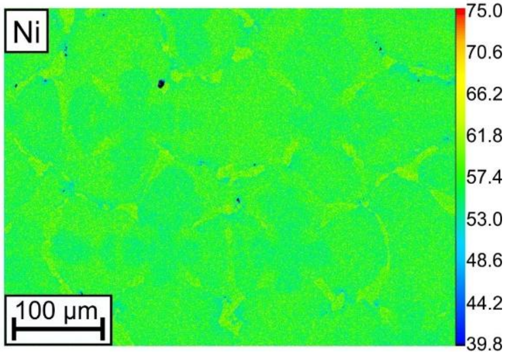
Superalloy SX

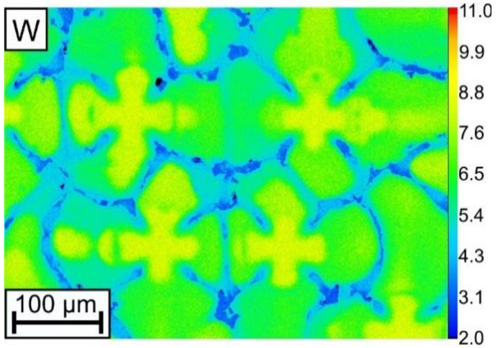
Complicated alloy compostions in wt.% (example - average)

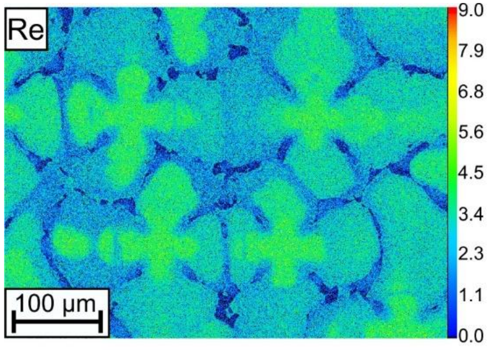
elements	Al	Co	Cr	Hf	Mo	Re	Ta	Ti	W	Ni
ERBO/1	5.8	9.3	6.2	0.1	0.6	2.9	6.9	1.0	6.3	bal.





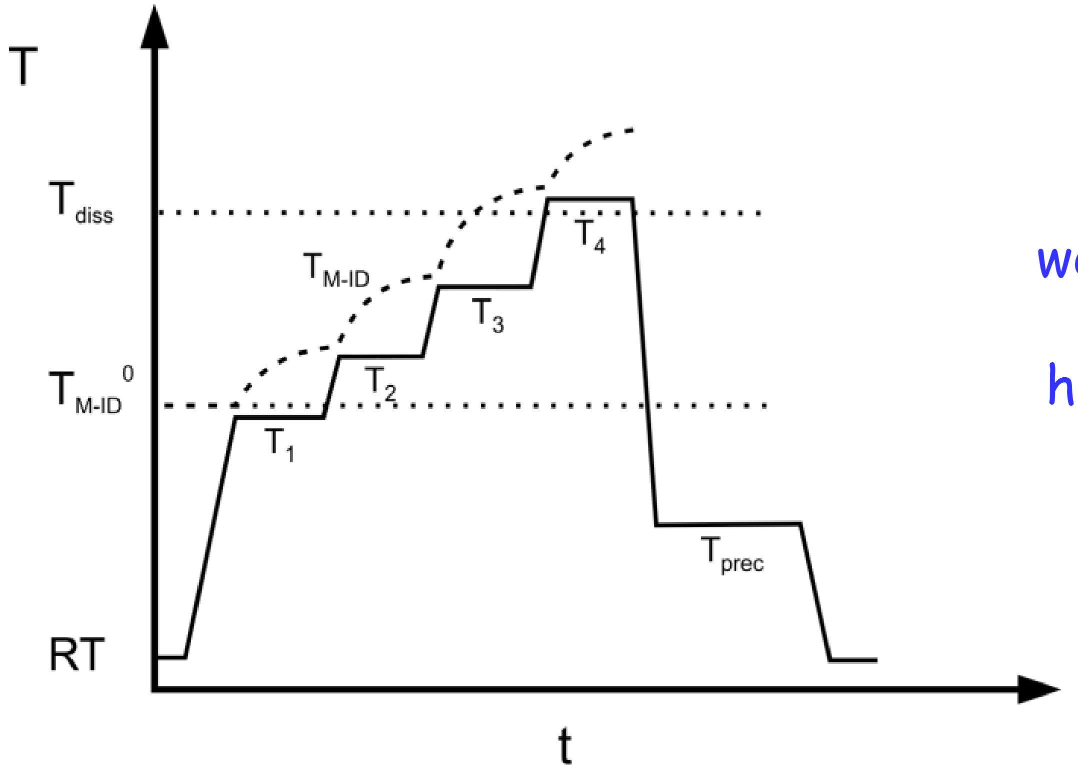




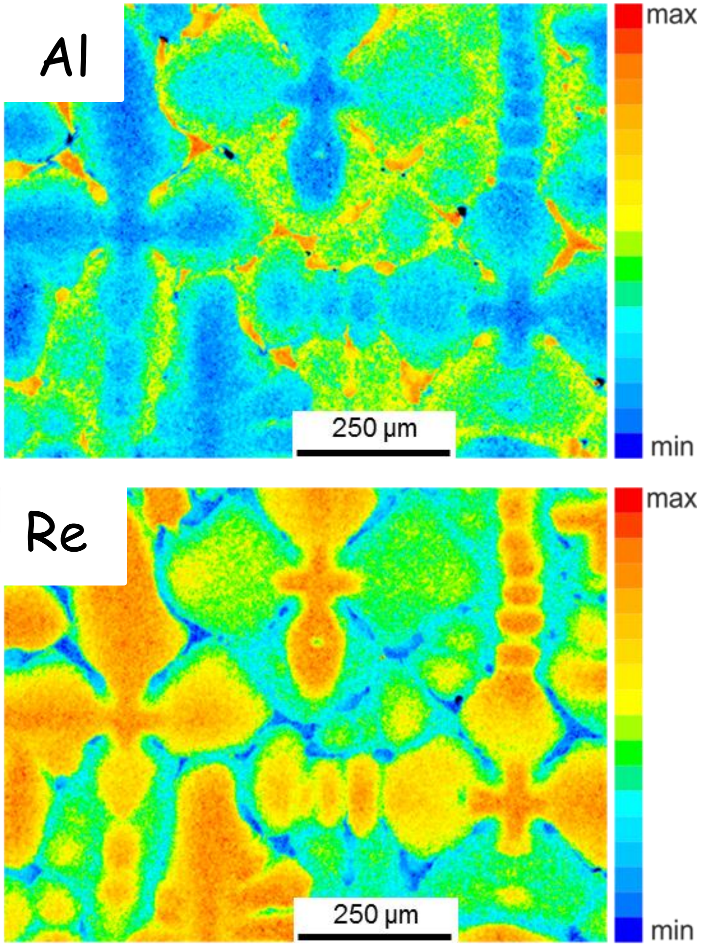


Partitioning

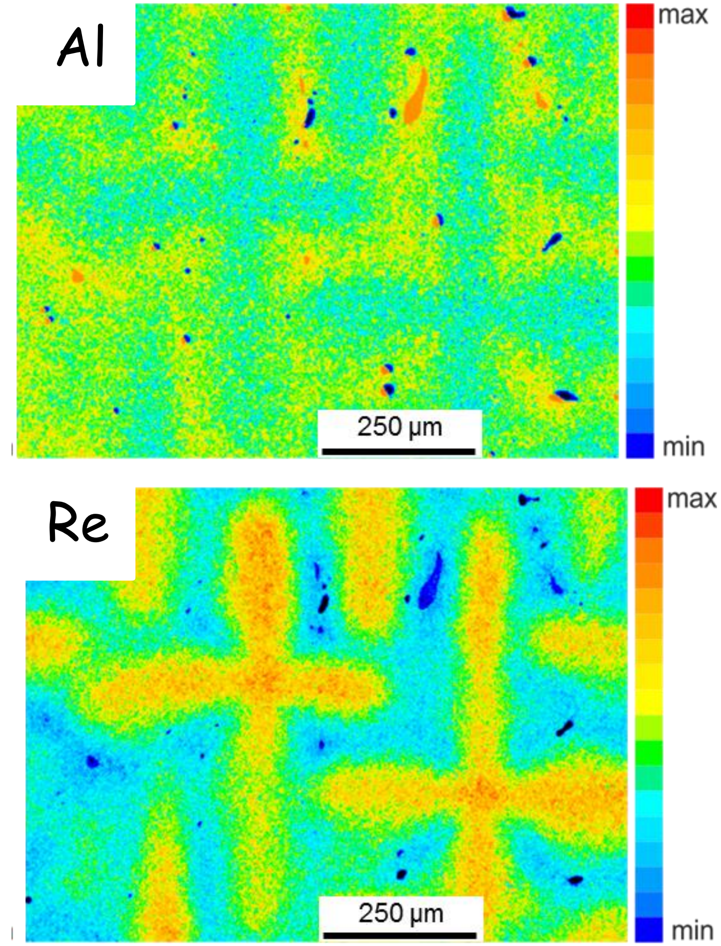
Homogenization



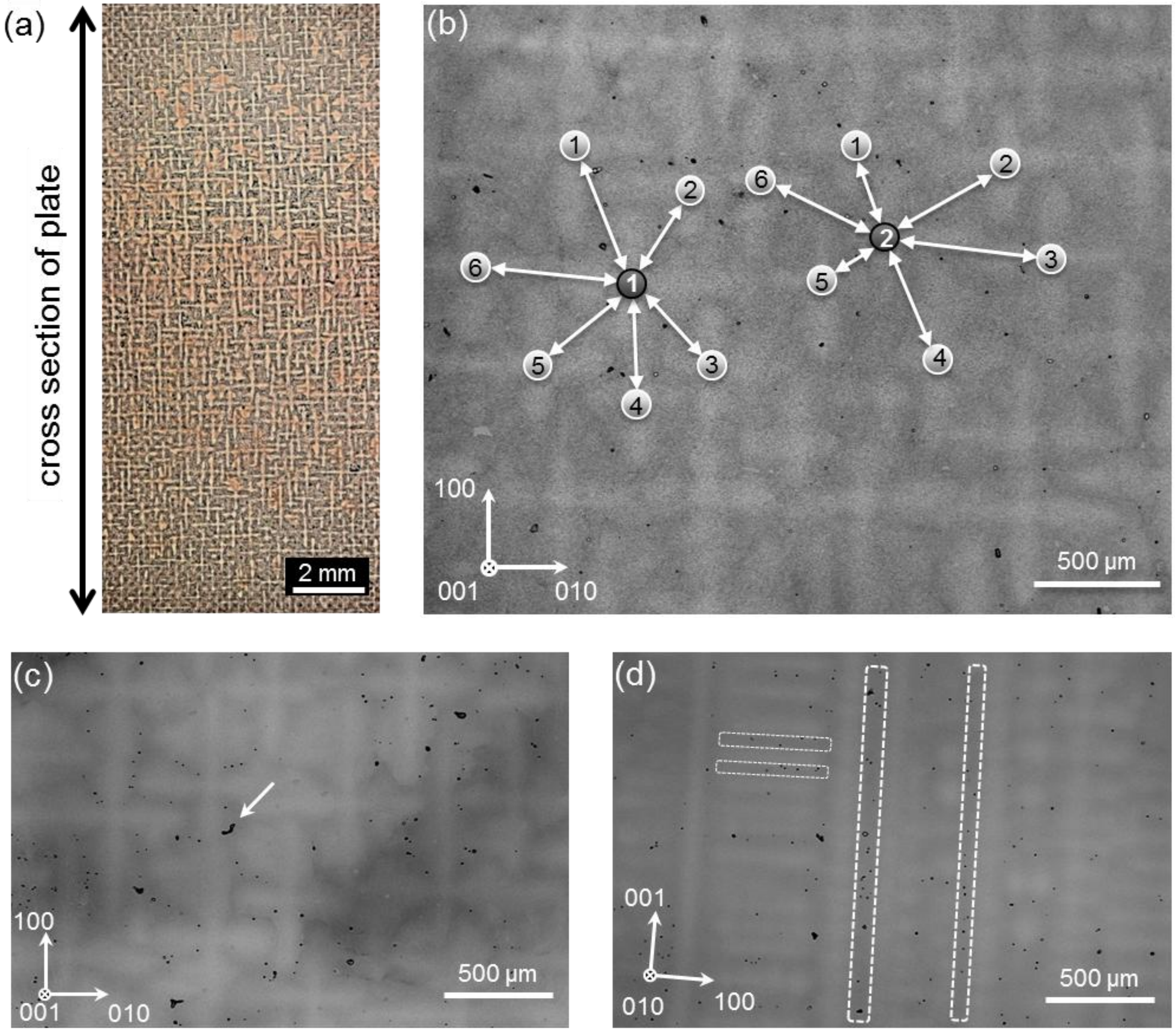
well thought out
multiple step
heat treatment



as cast



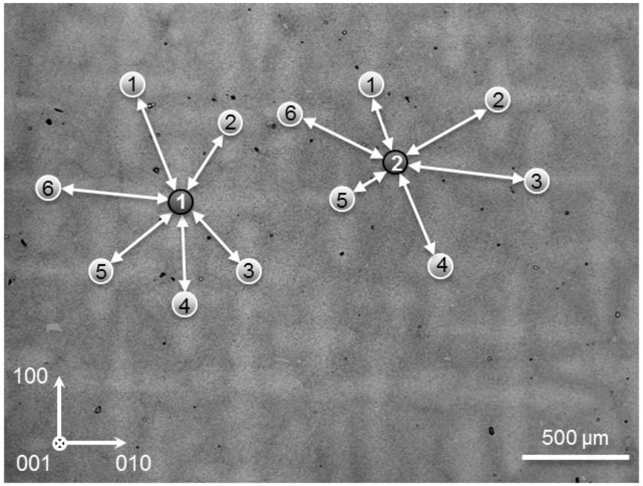
after homogenization

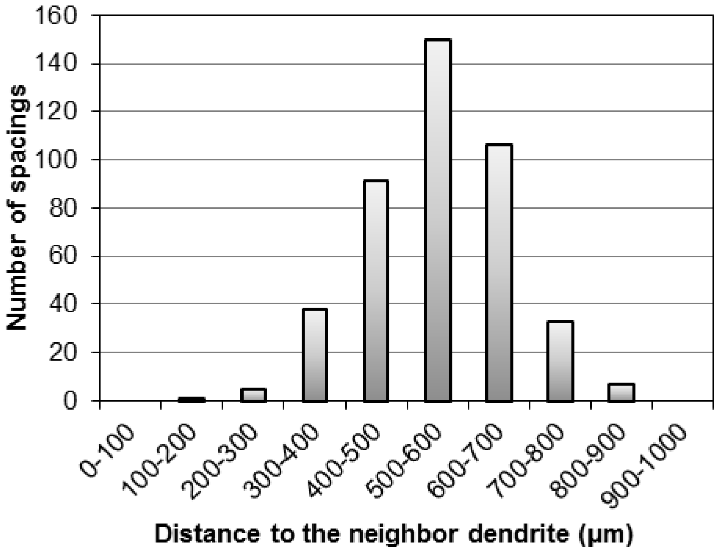


(a,b) dendrites

(c,d) cast micro porosity

Distribution of dendrite spacings





Recent study on microstructure of Ni-base SX

ADVANCED ENGINEERING MATERIALS

FULL PAPER

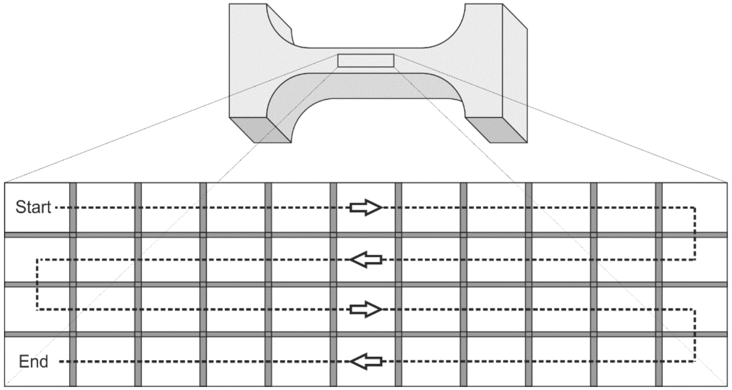
DOI: 10.1002/adem.201400136

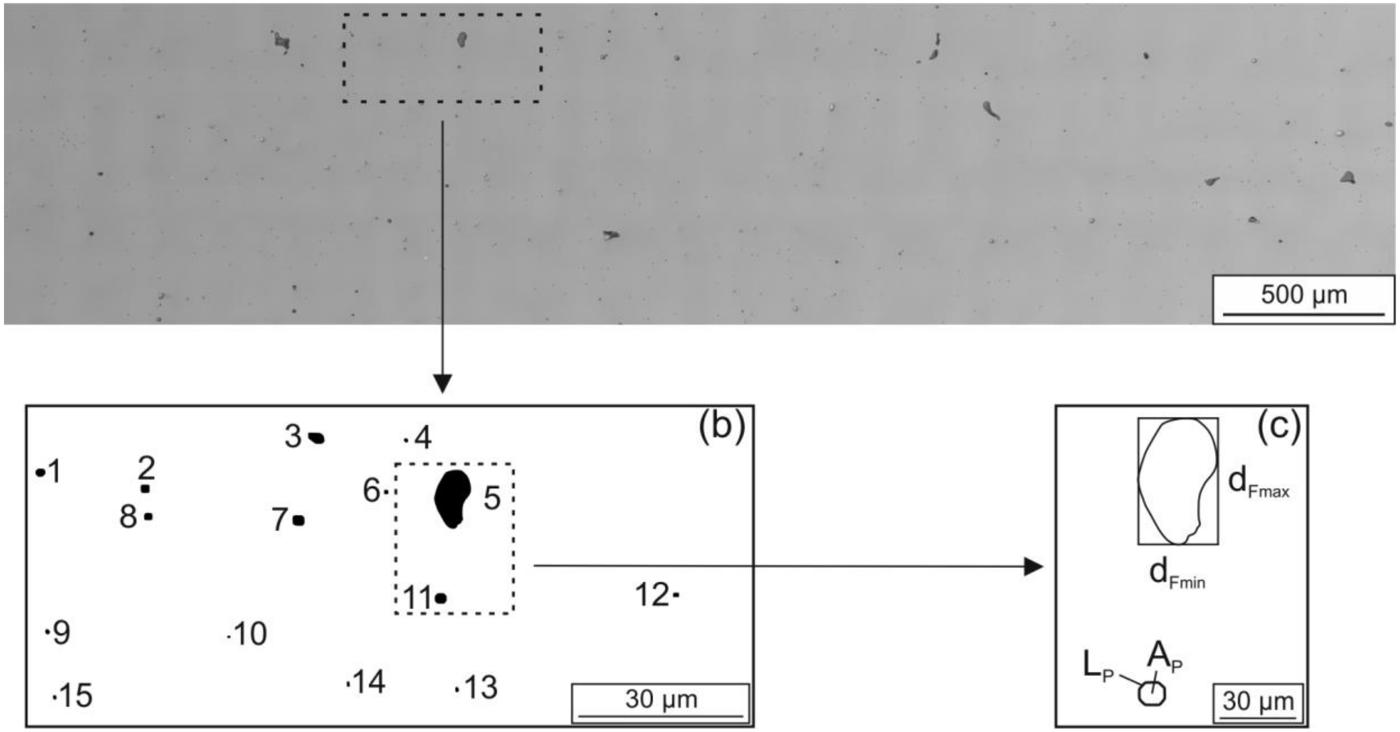
Advanced Scale Bridging Microstructure Analysis of Single Crystal Ni-Base Superalloys**

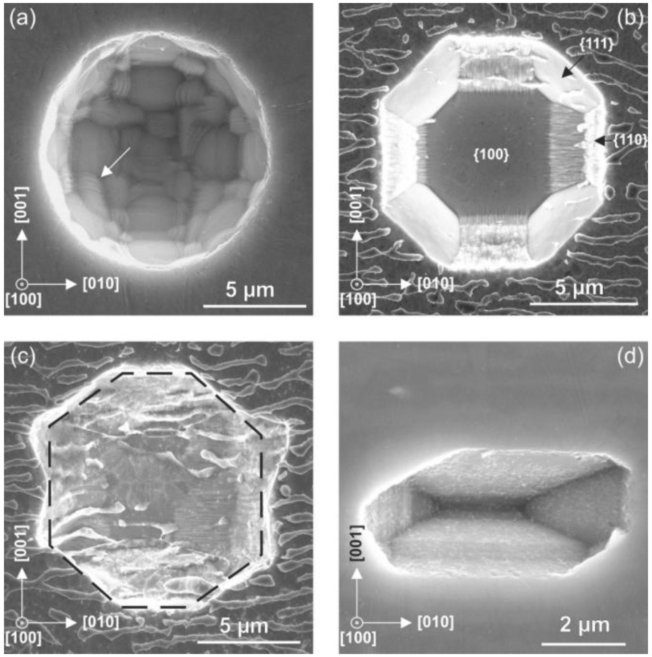
By *Alireza B. Parsa,* Philip Wollgramm, Hinrich Buck, Christoph Somsen, Aleksander Kostka, Ivan Povstugar, Pyuck-Pa Choi, Dierk Raabe, Antonin Dlouhy, Julian Müller, Erdmann Spiecker, Kathrin Demtroder, Jürgen Schreuer, Klaus Neuking* and *Gunther Eggeler*

ADVANCED ENGINEERING MATERIALS **2014**,
DOI: 10.1002/adem.201400136

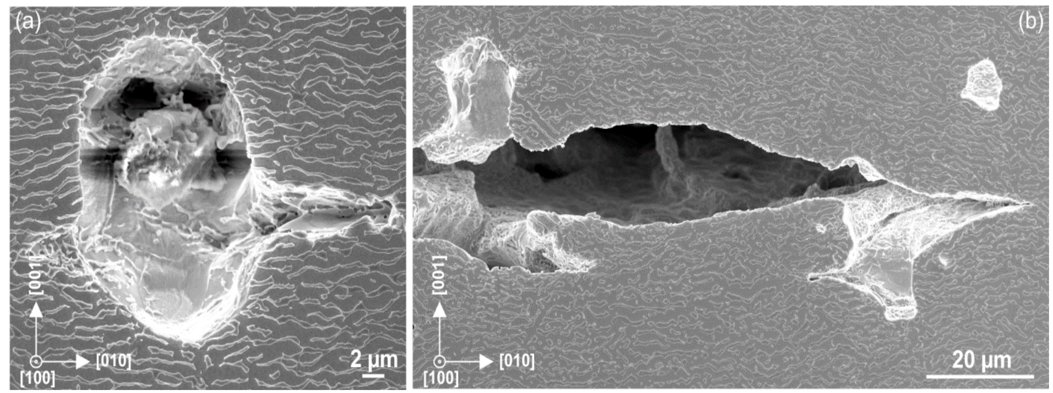
Quantitative metallography: cast pores, heat treatement pores and creep pores







shape / shape factor	a (circle)	a (octagon)	a (square)	2 a (rectangle)
f_F	1	0.9	0.7	0.5
f_P	3.14 (π)	3.0	2.8	2.7



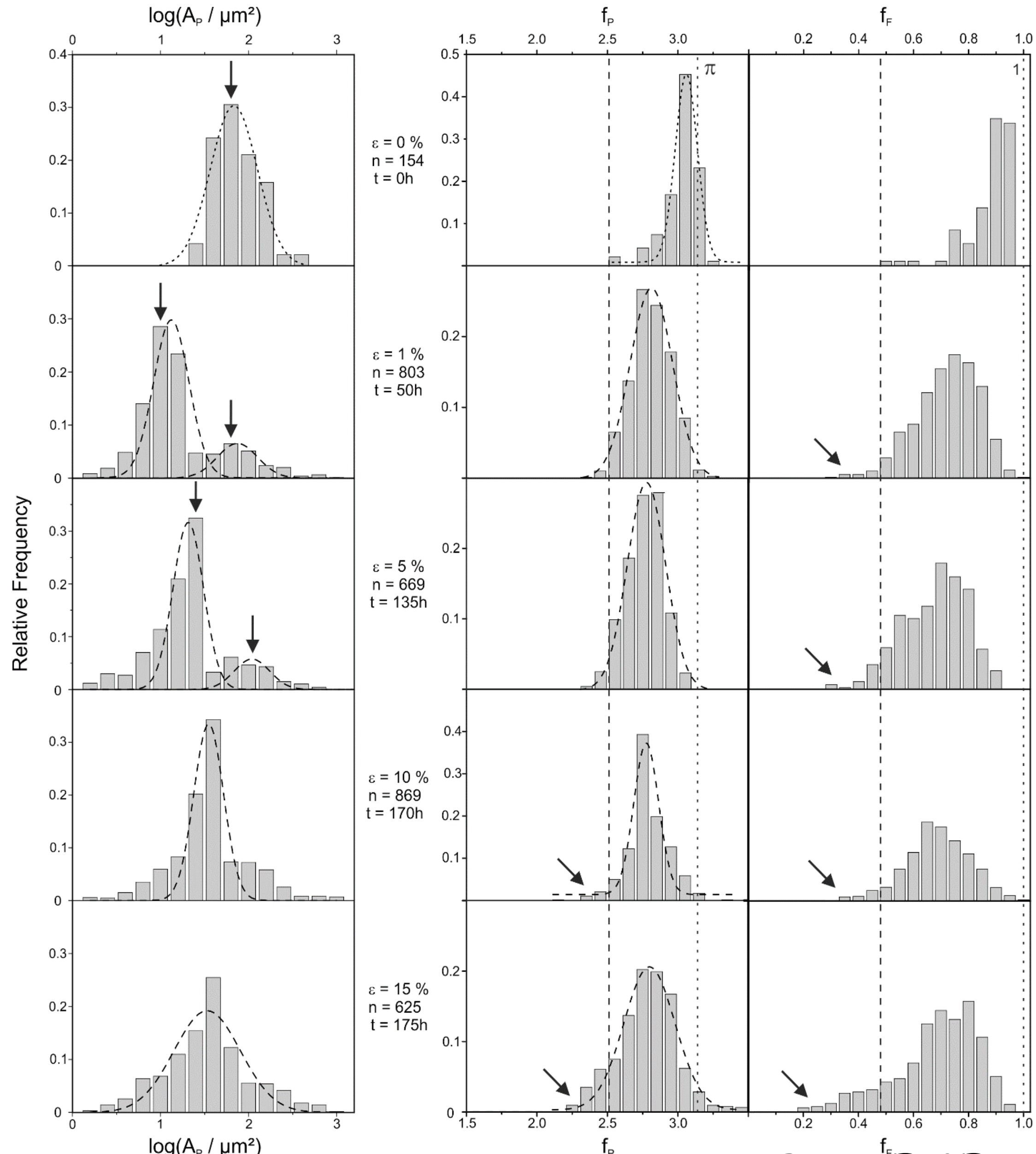
We can measure pore sizes (diameters, projected areas) and pore shapes. Quantitative metallography. Stereology.

Initially: only larger cast pores

New additional small creep pores appear

All pores grow

Shape factors indicate more elongated shapes: microcracks



Recent study on porosity in Ni-base SX

Mat.-wiss. u. Werkstofftech. **2015**, *46*, No. 6 **DOI** 10.1002/mawe.201500379

A quantitative metallographic assessment of the evolution of porosity during processing and creep in single crystal Ni-base super alloys

Eine quantitative metallographische Abschätzung der Porenentwicklung bei der Herstellung und beim Kriechen von einkristallinen Nickelbasis-Superlegierungen

H. Buck[1], P. Wollgramm[1], A. B. Parsa[1], G. Eggeler[1]

Section summary - cast microstructure

SX turbine blades are cast components.

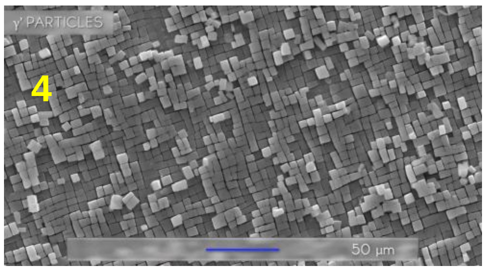
The elementary processes which occur during melting, casting and heat treatment are of utmost importance.

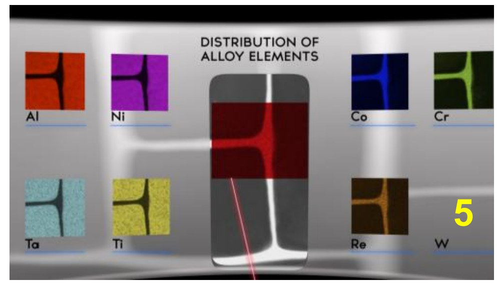
Bridgman solidification results in the formation of dendrites and interdendritic regions. These can be recognized in the microstructure and represent a large scale (0,2 mm) microstructural heterogeneity.

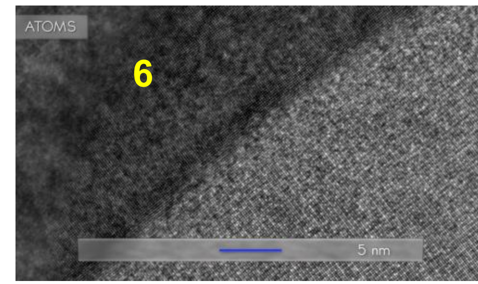
The cast microstructure accounts for chemical heterogeneity and for the presence of pores.

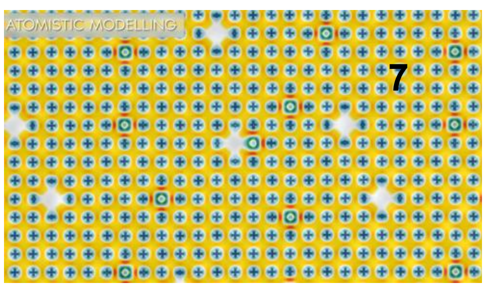
Microstructure after heat treatment-
fine scale heterogeneity

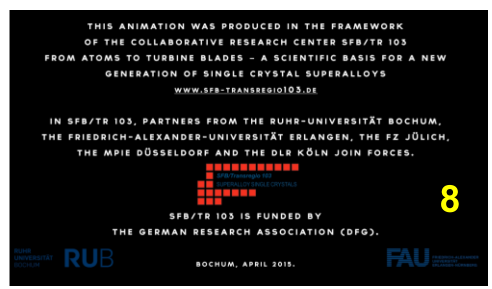
γ-CHANNELS / γ' CUBES







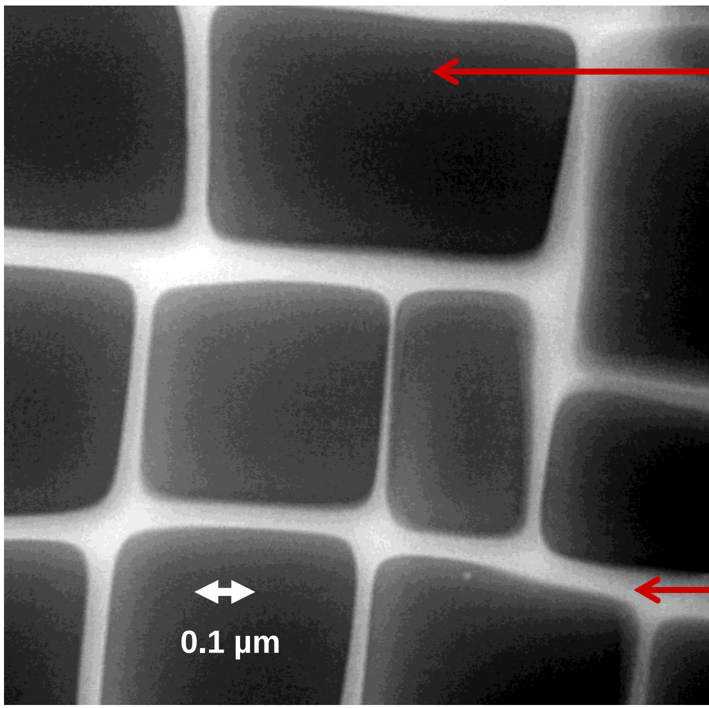




movie: https://www.youtube.com/watch?v=wYHch5QIWTQ

Two phases: γ-phase (thin channels) and / γ'-phase (cuboidal particles)

(1) The two phases are coherent, the atoms of the two phases occupy one common lattice. But the lattice constants are not the same. There is a crystallographic misfit. This causes local strains and stresses.

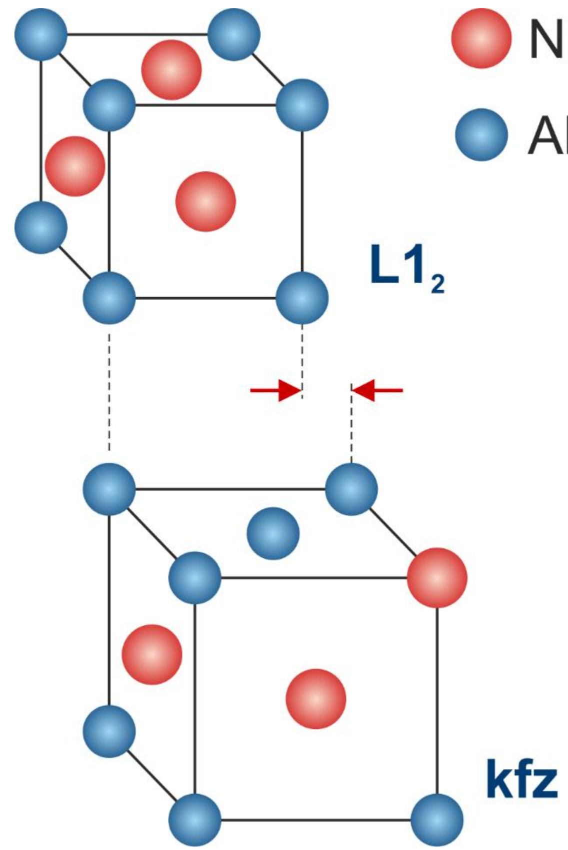


γ' - cubes

80 vol.-%

γ - channel

20 vol.-%



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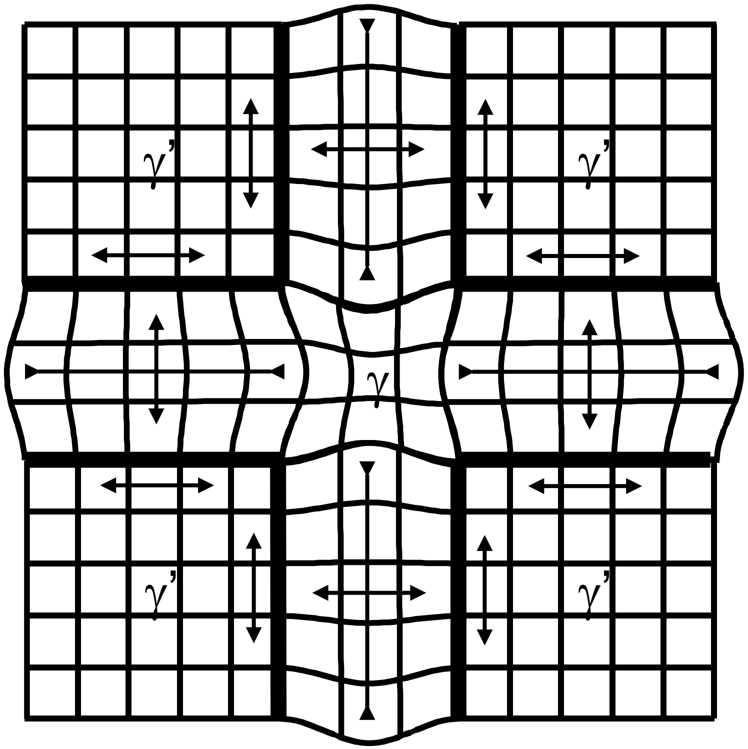
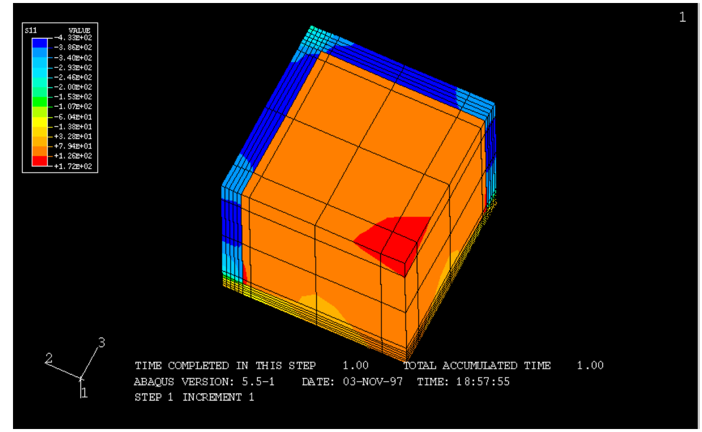
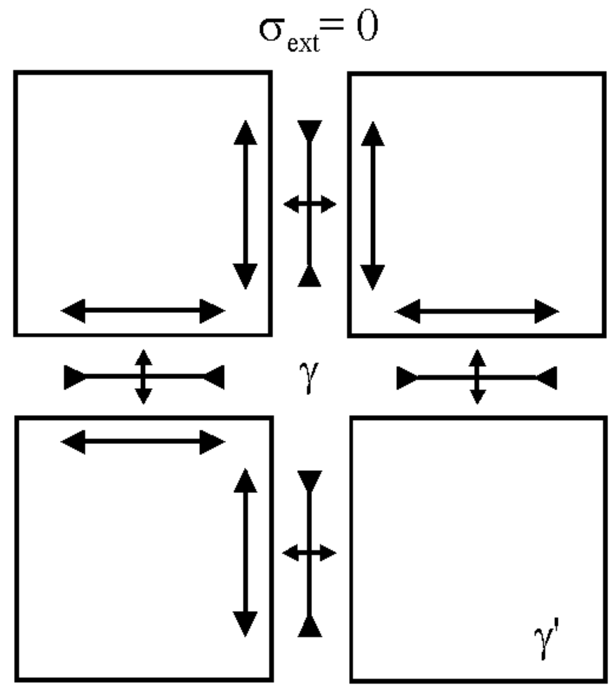
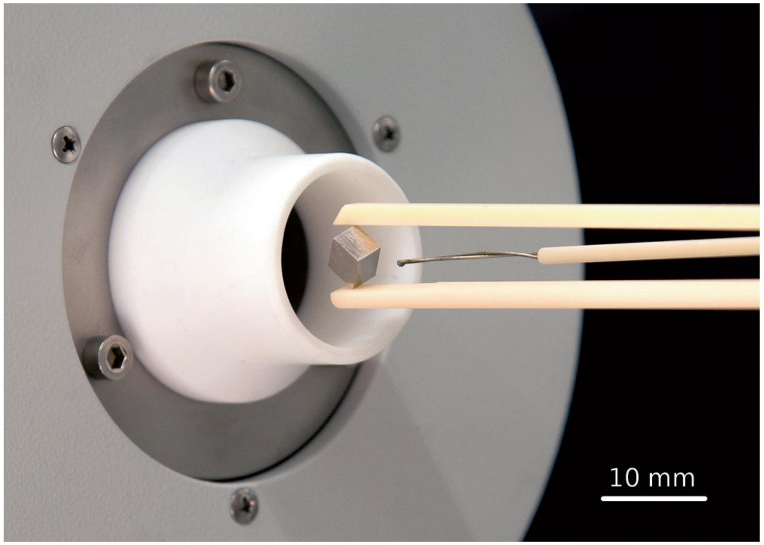




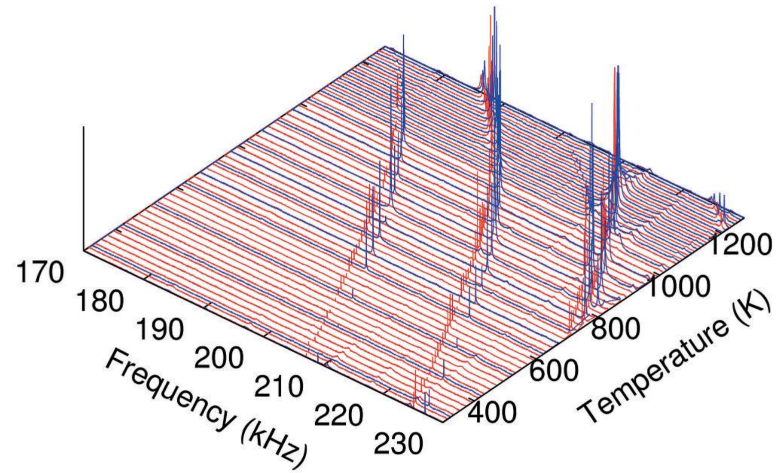


$$\delta = 2 \cdot \frac{a_{\gamma'} - a_{\gamma}}{a_{\gamma'} + a_{\gamma}}$$

Misfit δ. $a_{\gamma'}$ and a_{γ} are the lattice constants of the γ and the γ' phase. δ represents a strain. Typical value for Ni-base superalloys at 1000°C: δ = - 0.001. We think of it as a strain. Times E gives a stress!



a)



b)

Mat.-wiss. u. Werkstofftech. **2015**, *46*, No. 6

DOI 10.1002/mawe.201500406

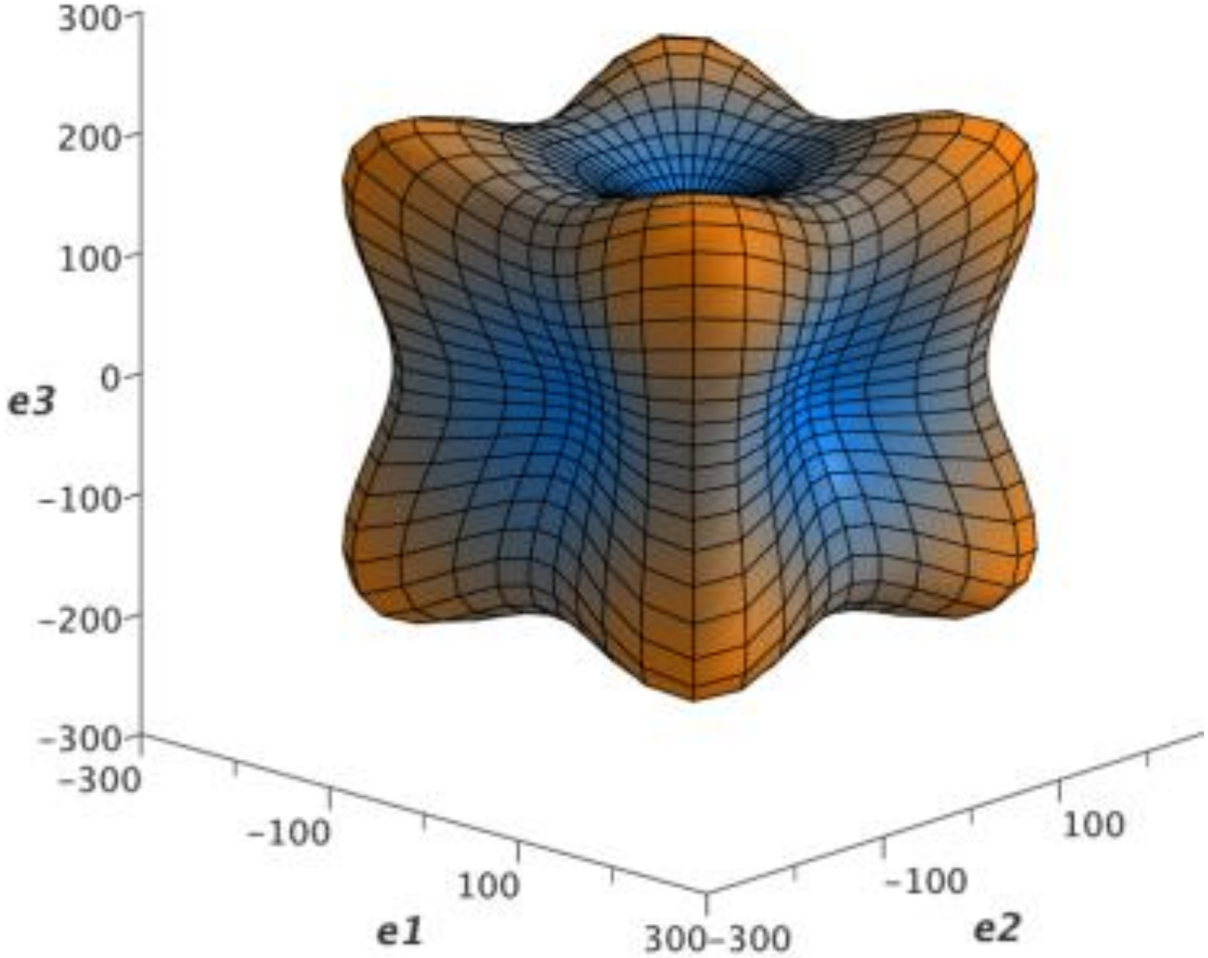
Influence of microstructure on macroscopic elastic properties and thermal expansion of nickel-base superalloys ERBO/1 and LEK94

Zum Einfluss der Mikrostruktur auf die elastischen Eigenschaften und auf die thermische Ausdehnung der einkristallinen Superlegierungen ERBO/1 und LEK94

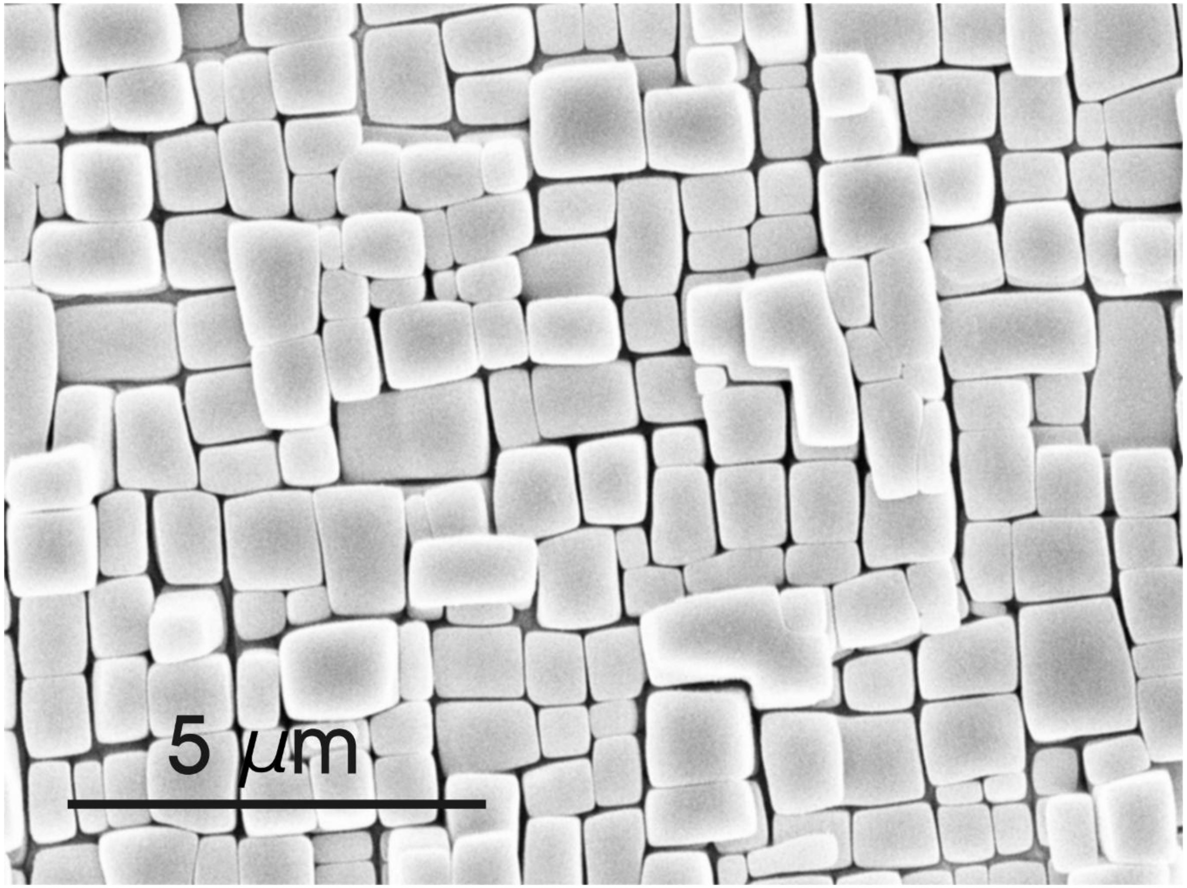
K. Demtröder[1], G. Eggeler[2], J. Schreuer[1]

FAMSE–GEII–46

(2) Single crystal superalloys are not elastically isotropic. The elastic modulus E shows minima in <100>-directions.



As a result of (1) & (2) γ’-particles form as cubes. This minimizes the elastic strain energy.



Section summary – γ/γ'-microstructure

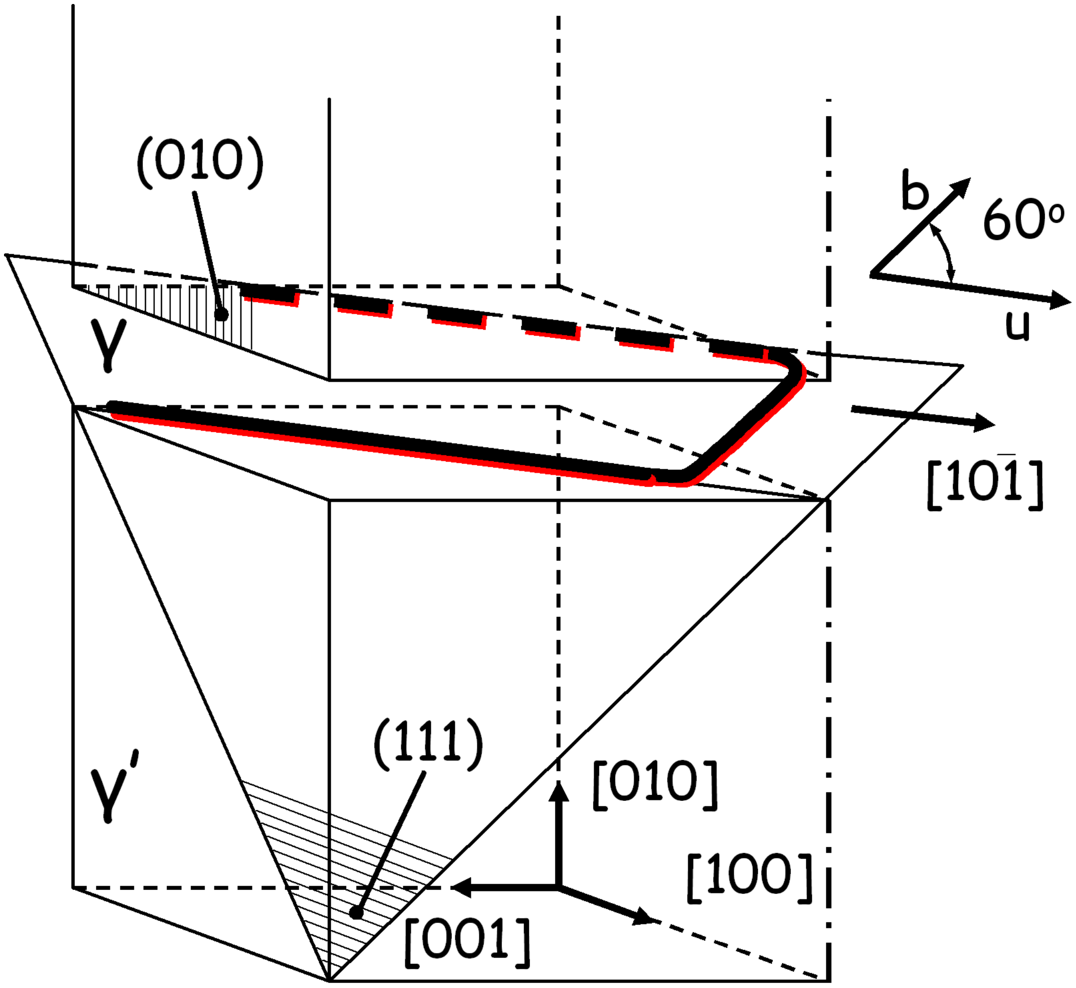
SX have the very well known γ/γ'-microstructure. This represents a small scale microstructural heterogeneity.

γ'-cubes (edge length: 0,5 µm, ordered L12 phase, volume fraction: 78 %) are separated by thin γ-channels (width: 0,1 µm, fcc, volume fraction: 22 %).

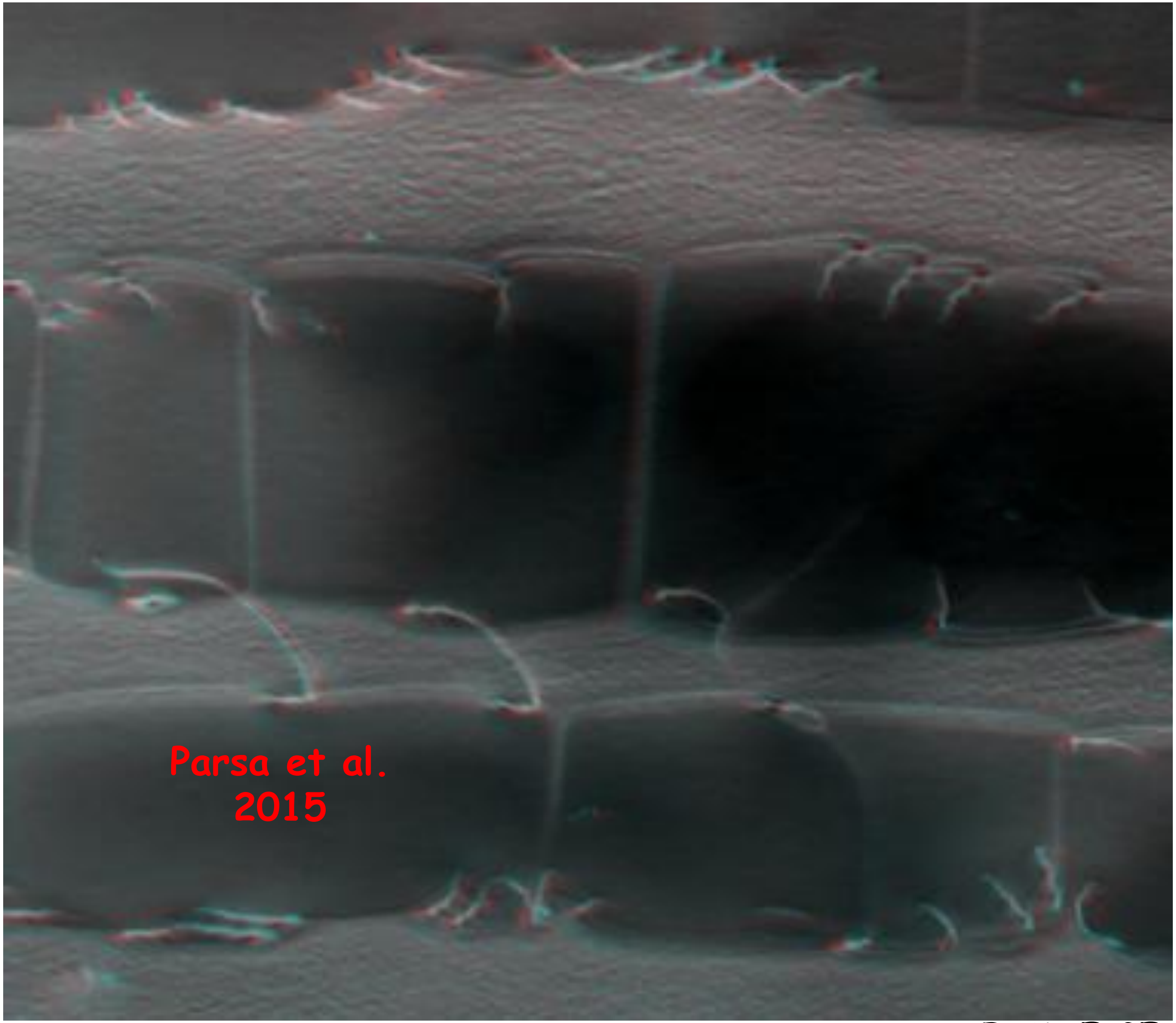
The atoms of the two phases occupy one common lattice. The lattice constants, however, are not the same. This results in a misfit which is associated with high internal stresses.

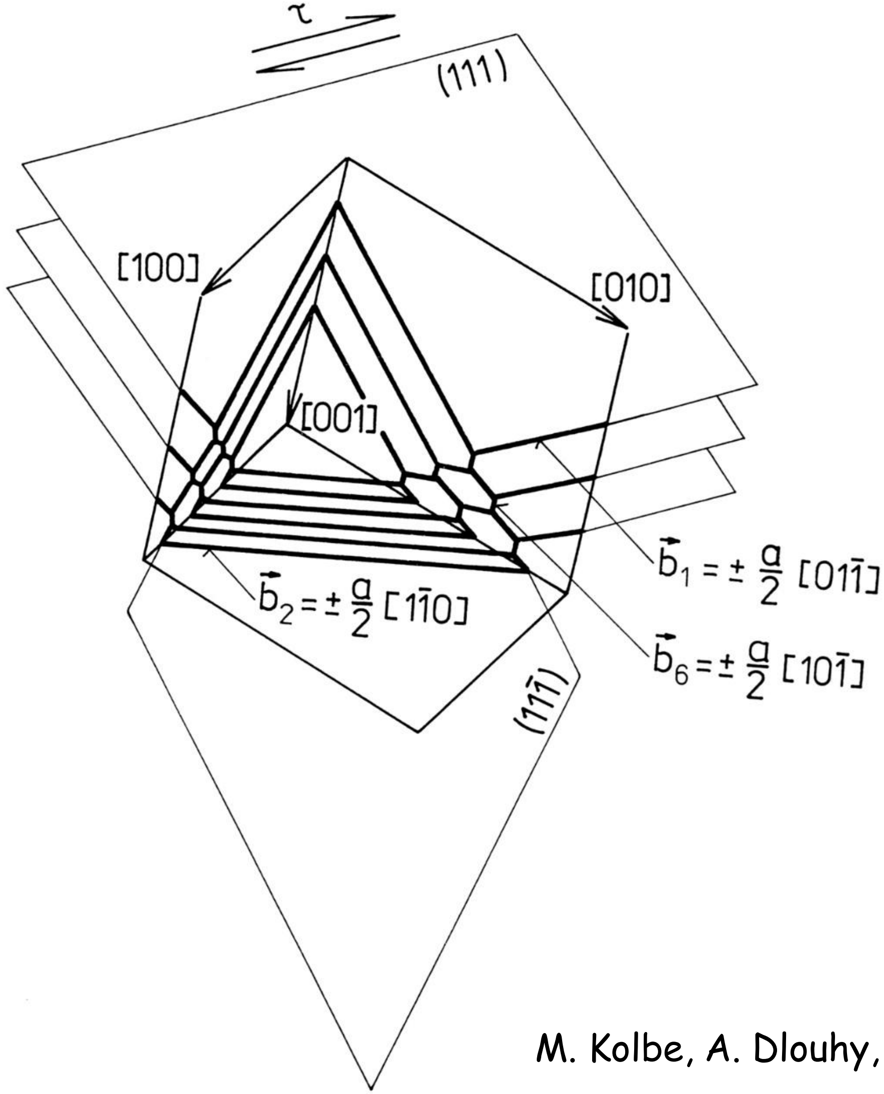
Microstructural parameters like γ'-cube size can scatter a lot (e.g. 0,2 – 1 µm. The same holds for γ'-channel width. Average quantities are not always helpful. Local situations must be interpreted regarding the local conditions.

Dislocations in γ/γ'-microstructures

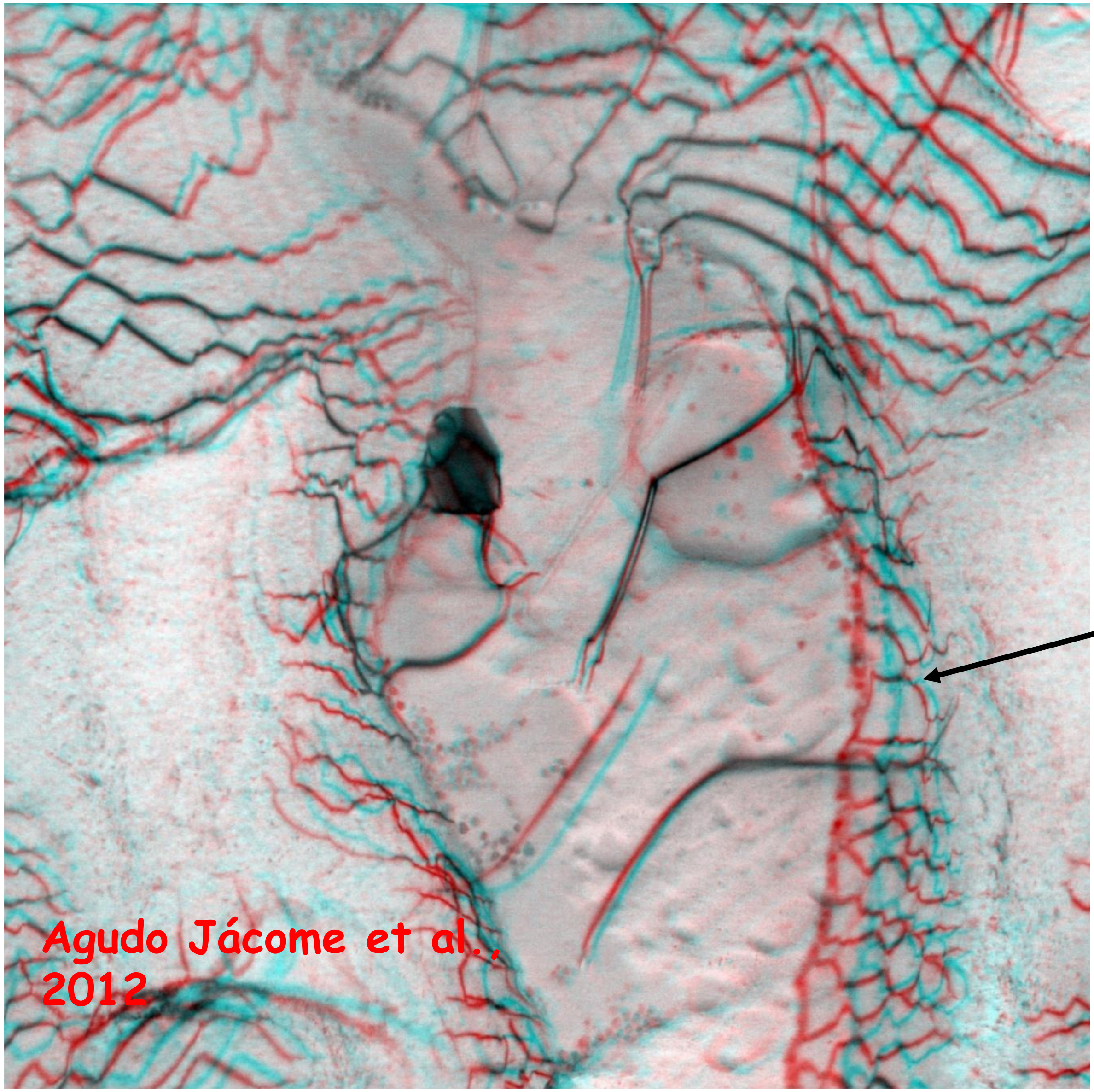


M. Probst-Hein, A. Dlouhy, G. Eggeler, Acta Metall., 1999


Parsa et al.
2015

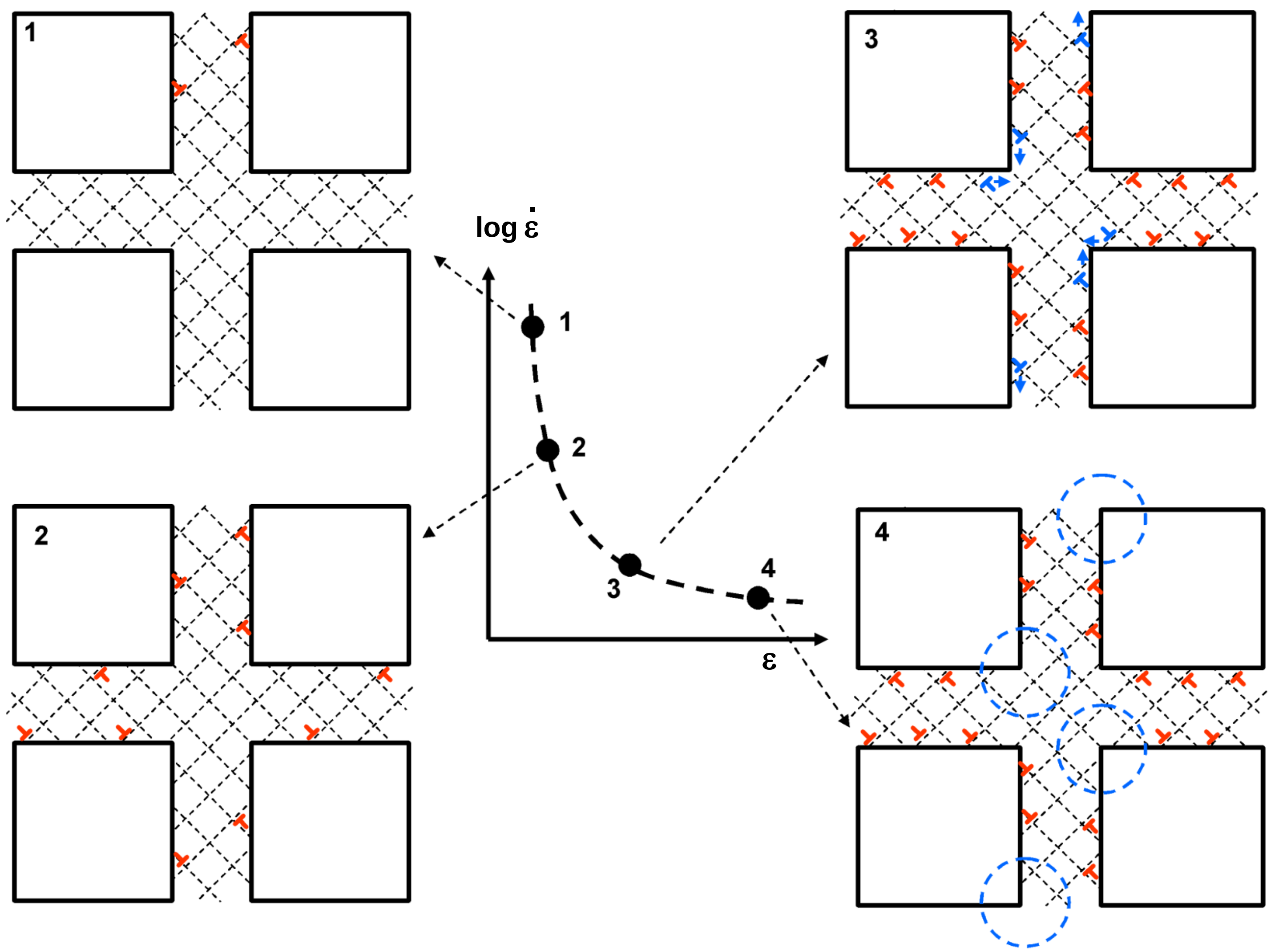


M. Kolbe, A. Dlouhy, G. Eggeler, Mat. Sci. Eng. A, 1998

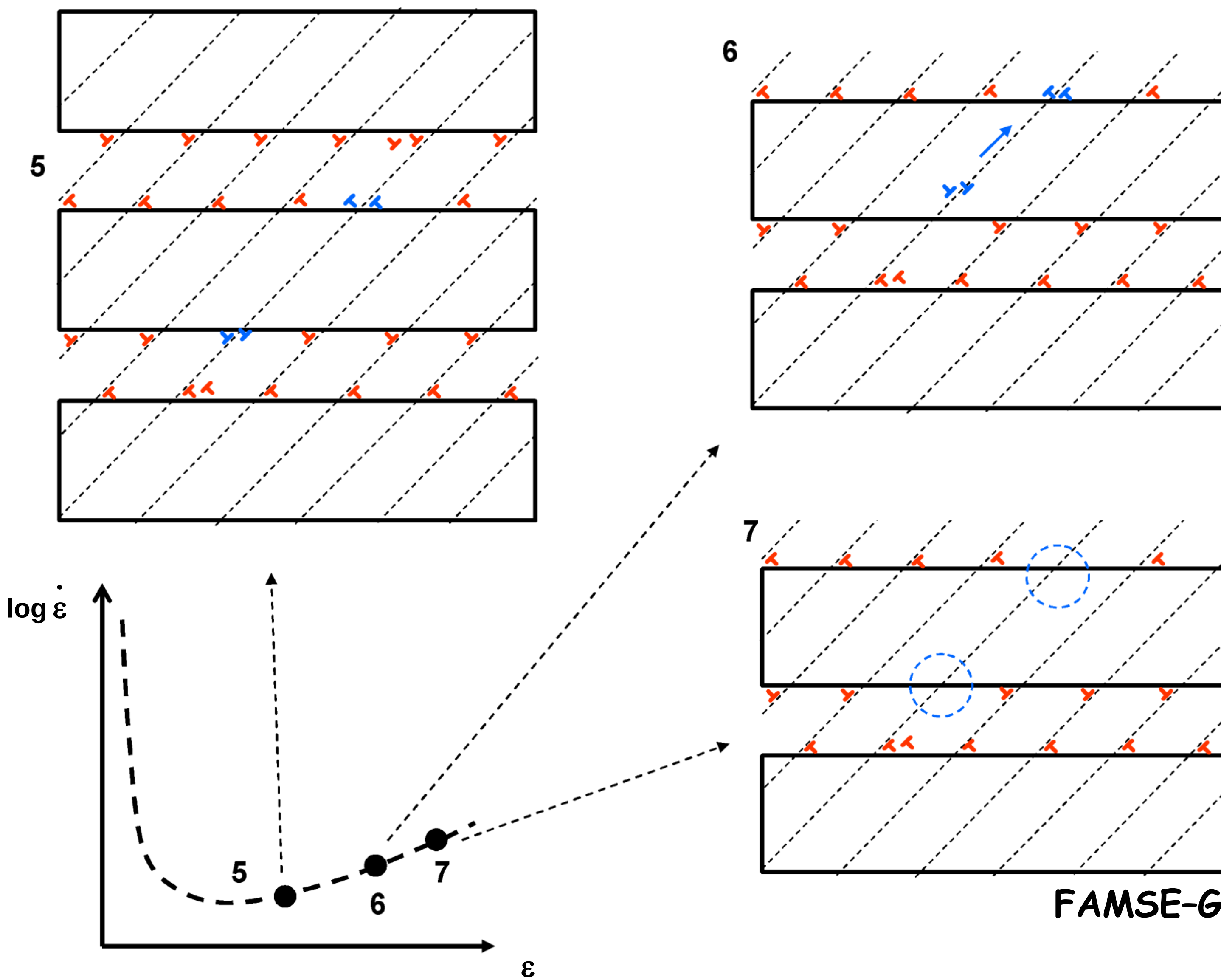


networks

early stages of high temperature and low stress creep

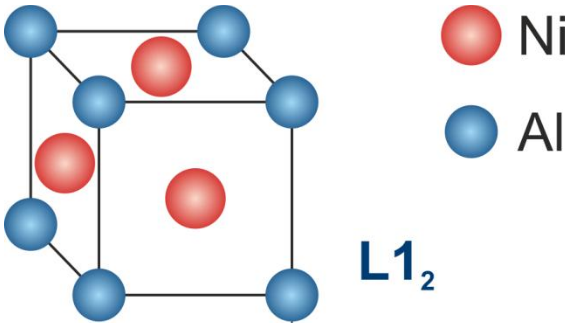


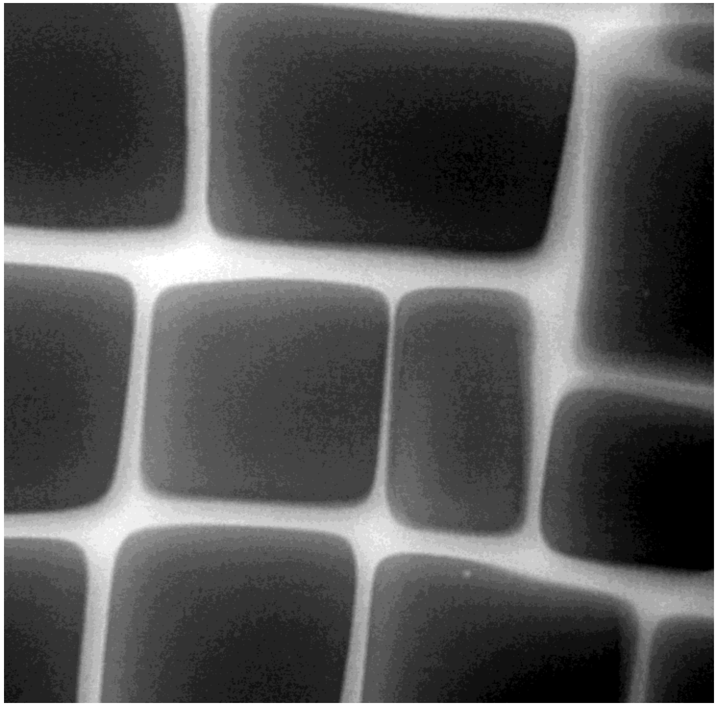
later stages of high temperature and low stress creep



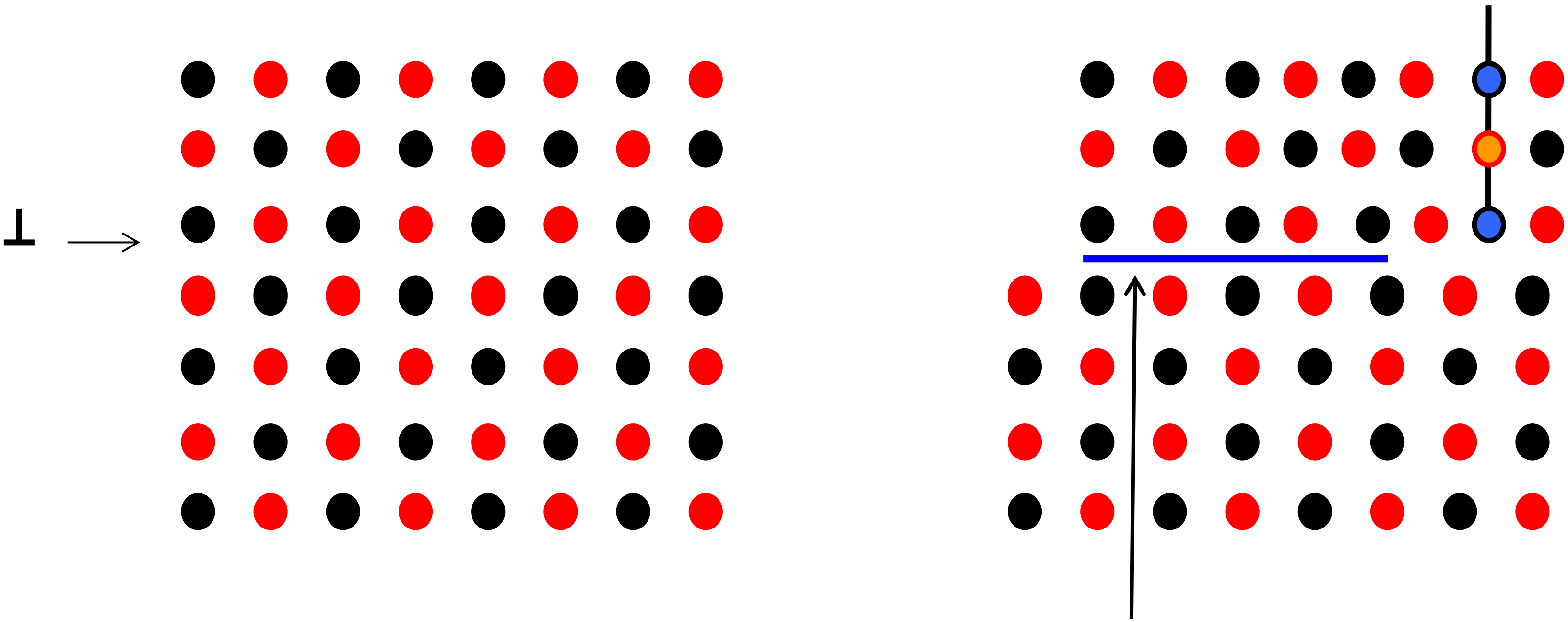
FAMSE-GEII-56

The γ'-phase is an ordered phase.



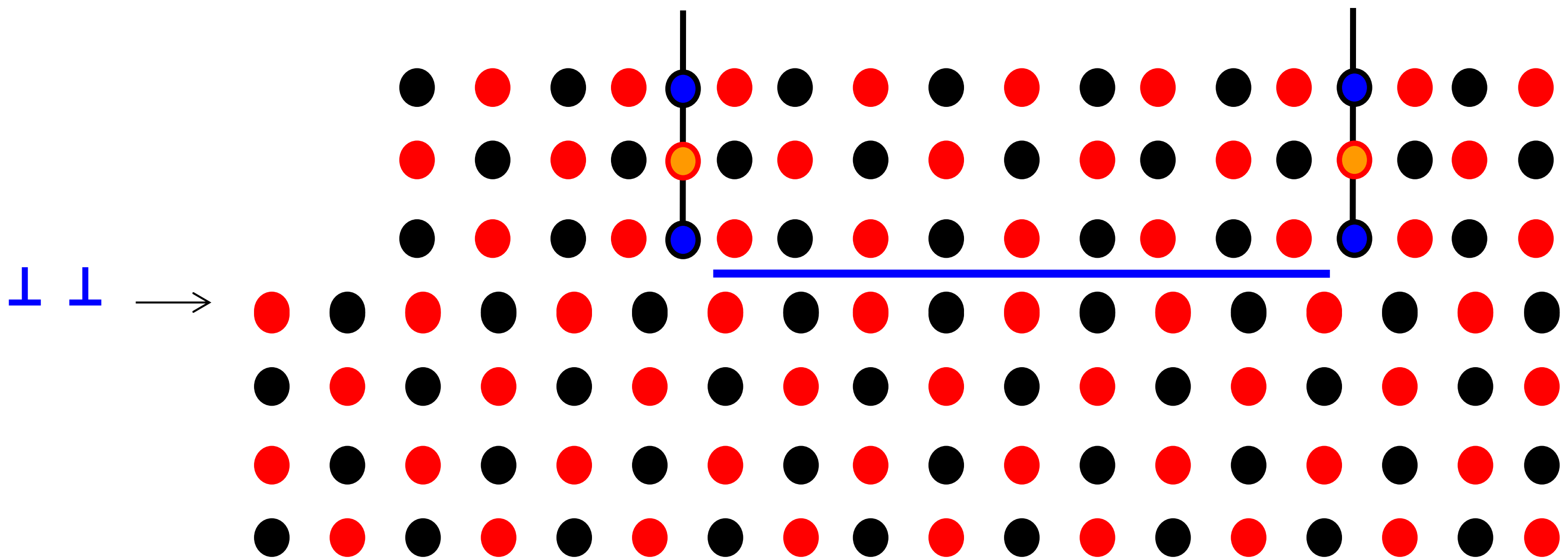


When a dislocation cuts in, the order is affected!

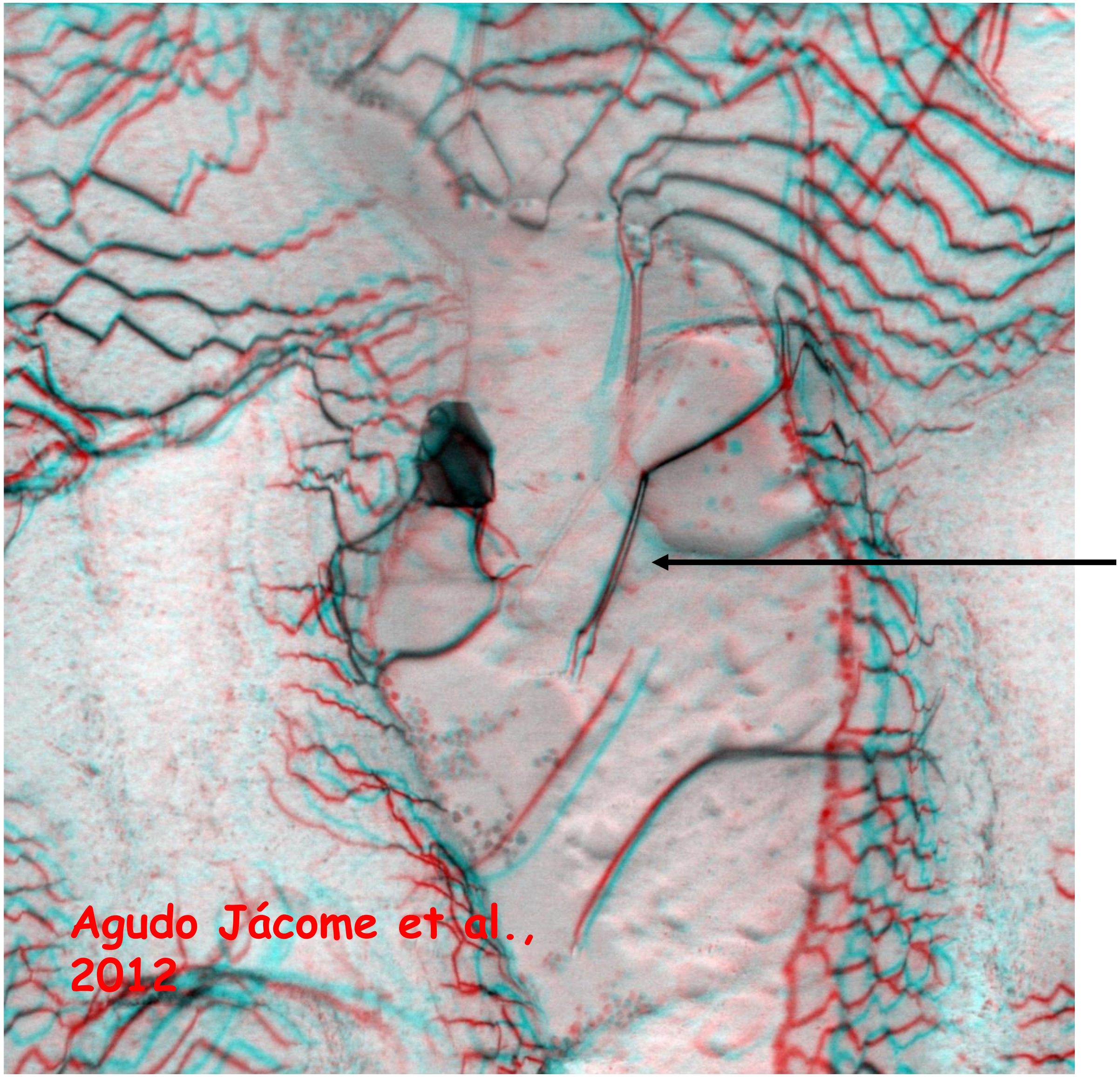


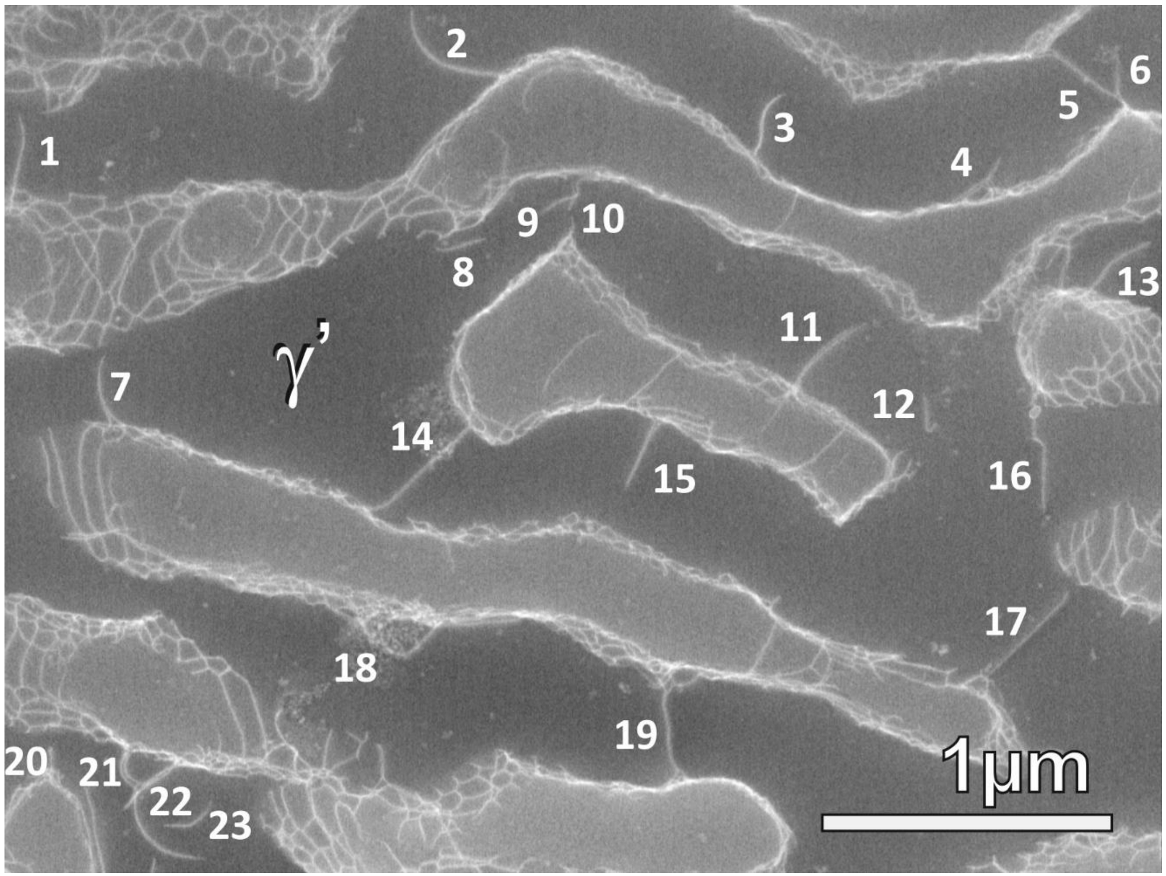
We create an antiphase boundary (APB), which costs energy.

Result: Pairwise cutting.



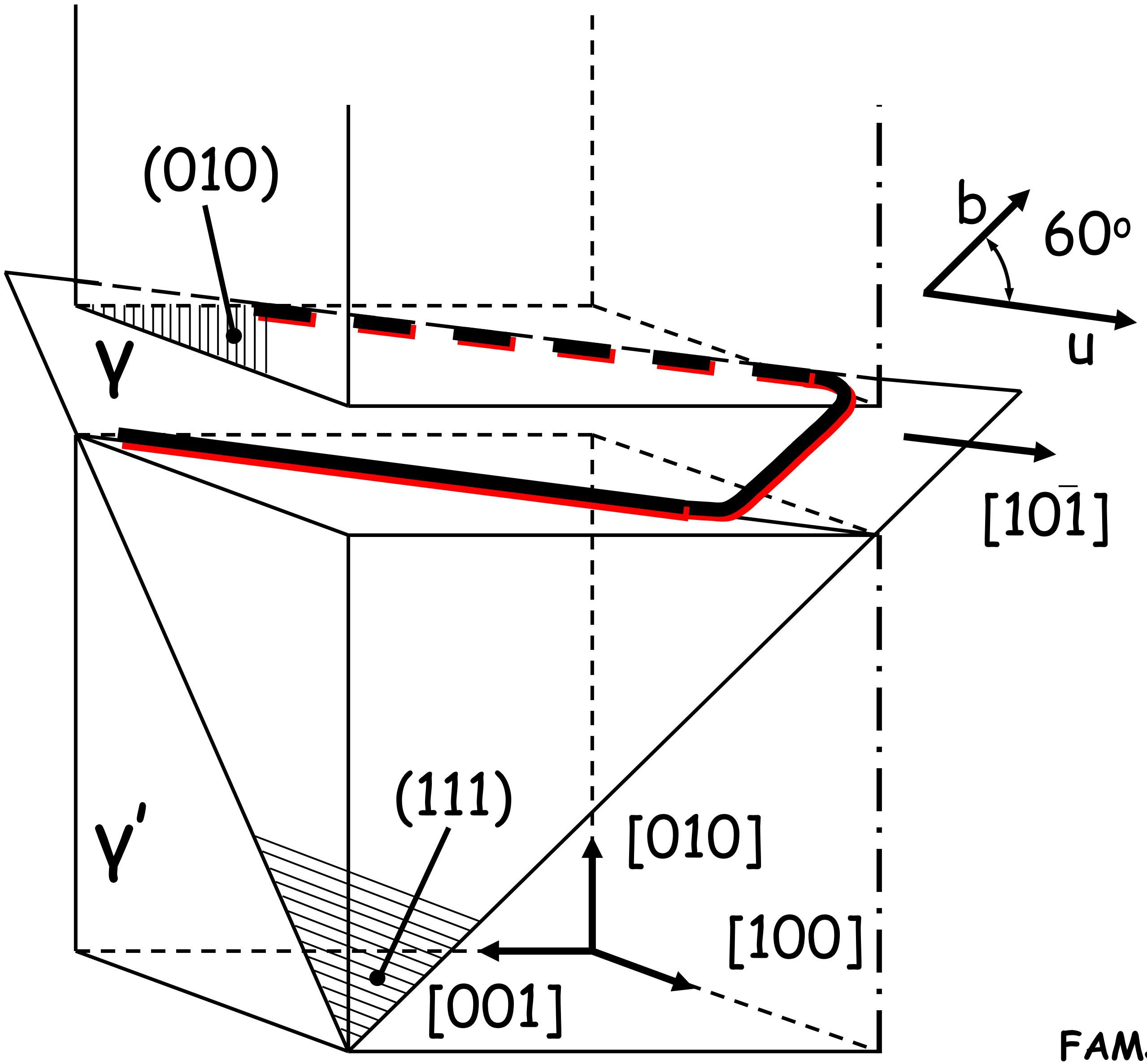
SX language: a superdislocation, consisting of two ordinary dislocations, shears the γ' - phase.


Agudo Jácome et al.,
2012
cutting

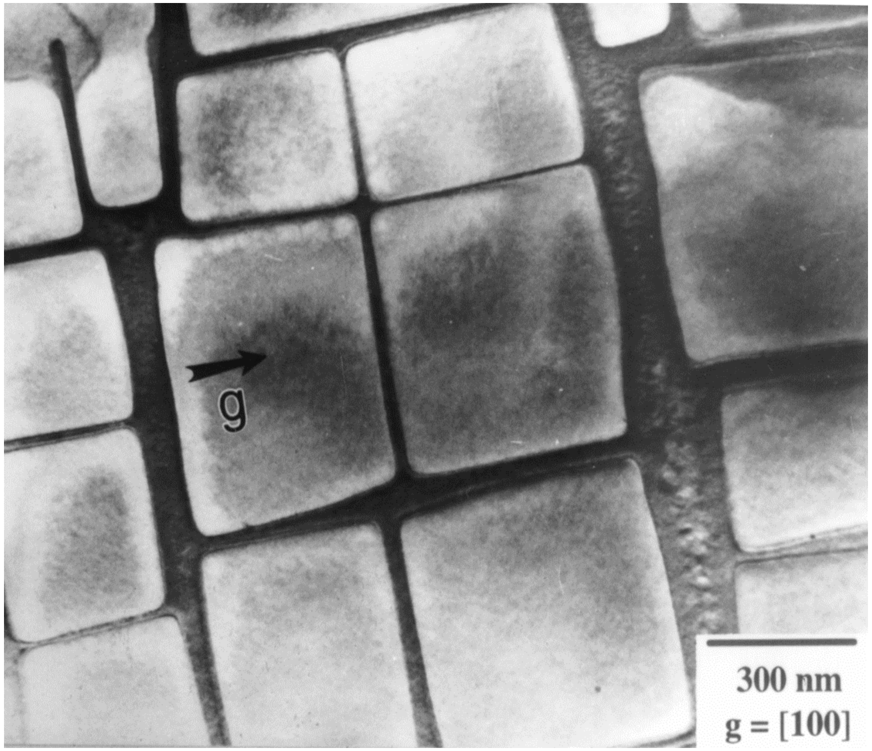

1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
γ'
1µm

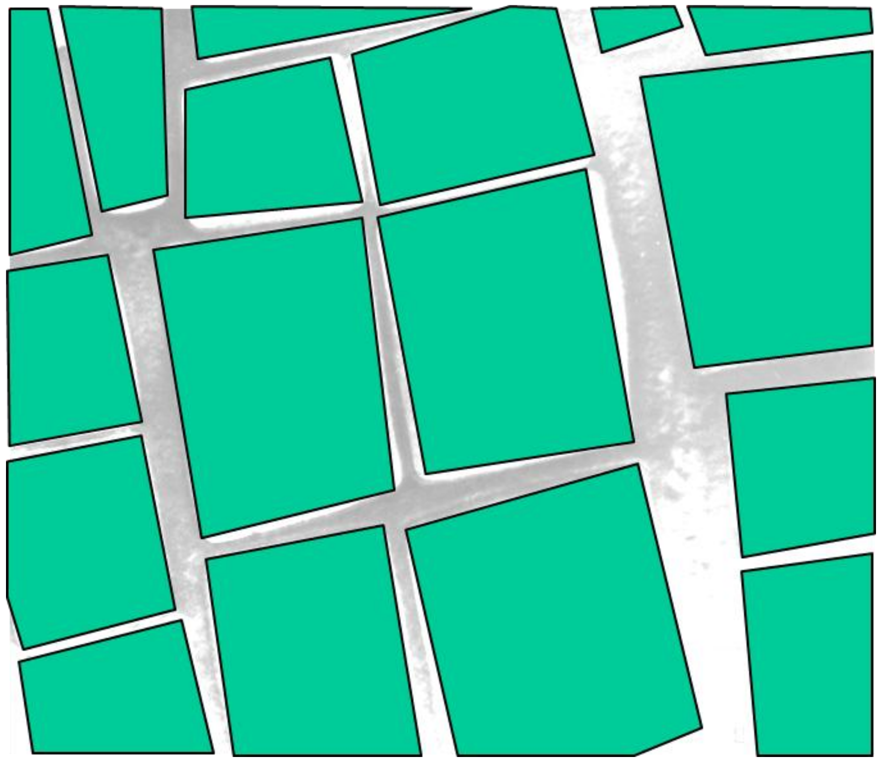
What can we learn from a calculation of Peach Köhler forces?

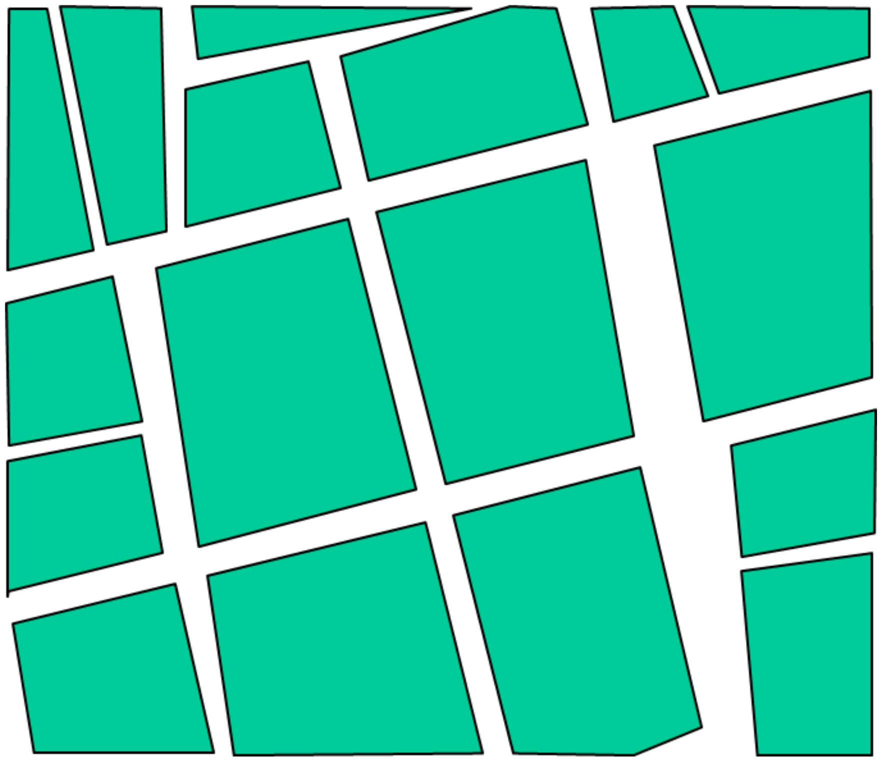
How a dislocation enters a γ-channel by glide, geometry:



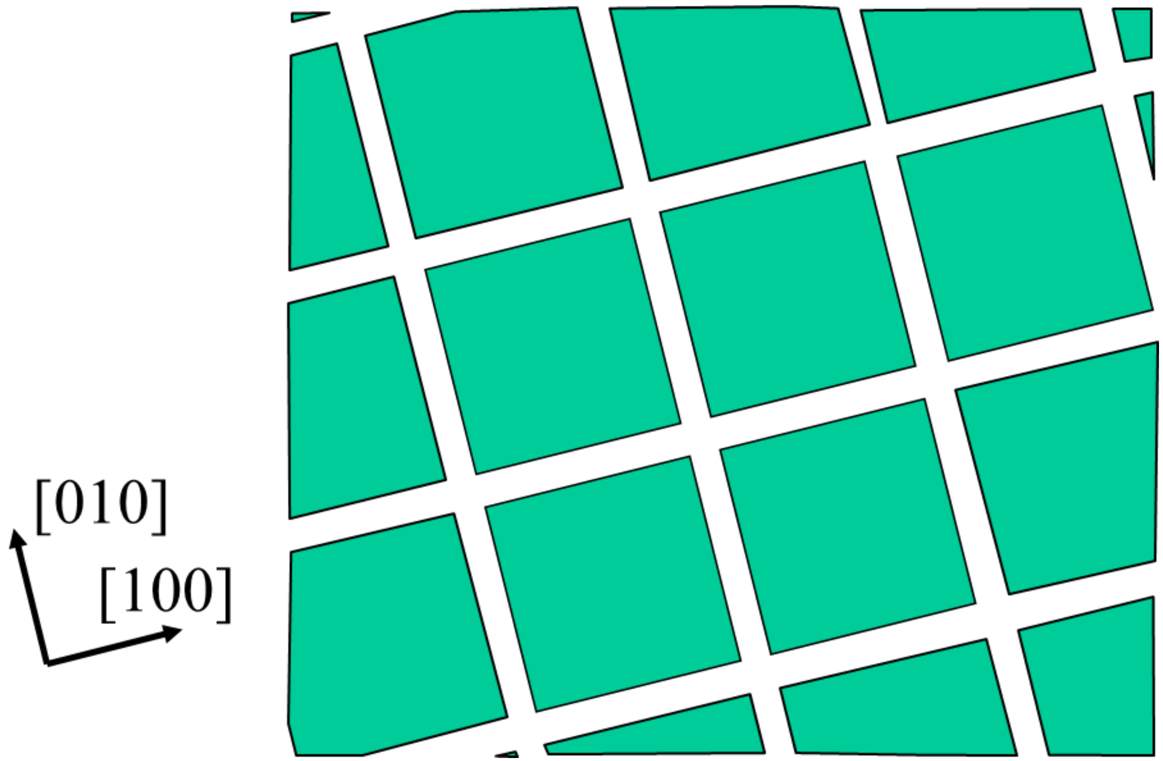
How do we get to this view?

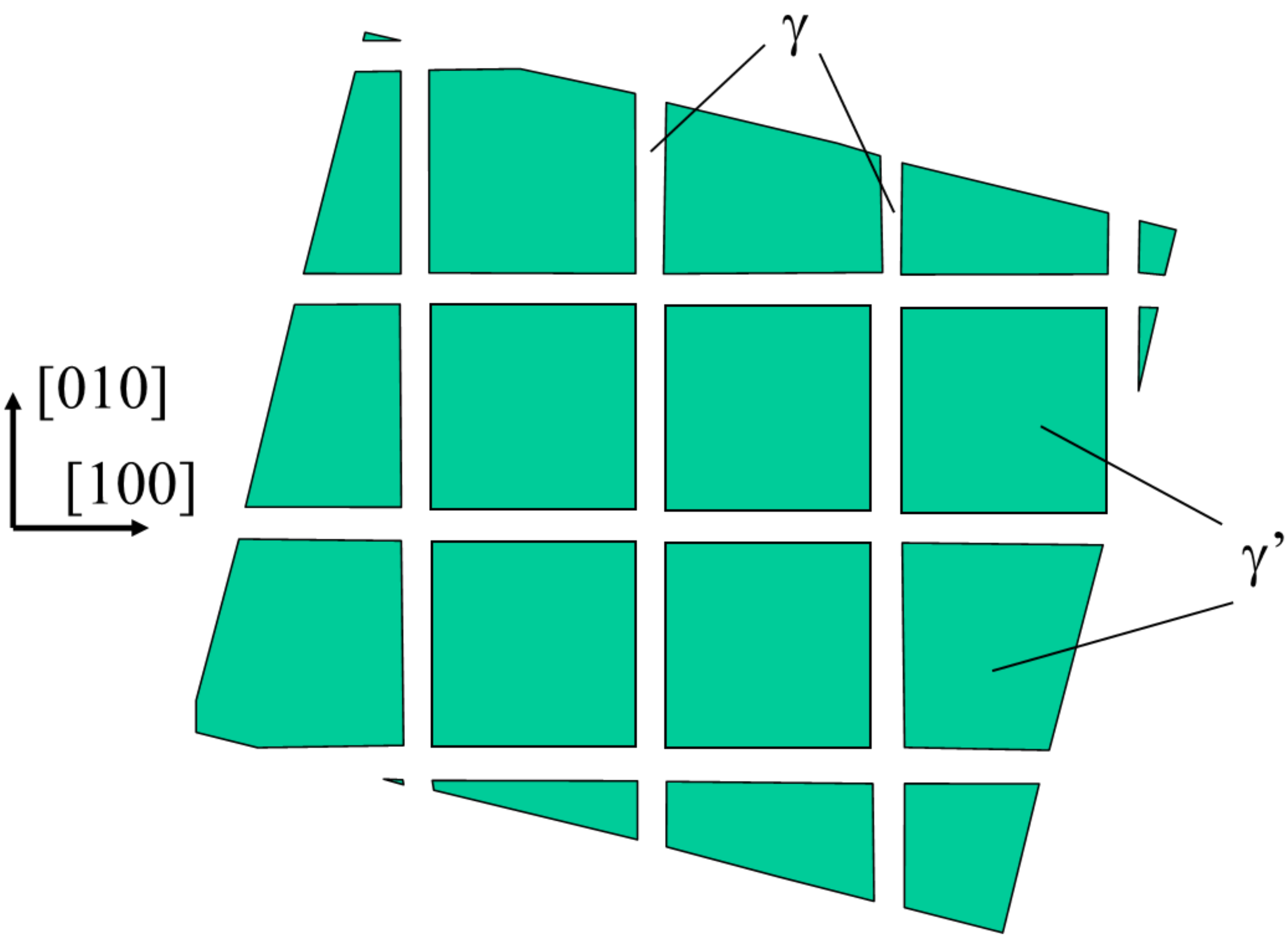


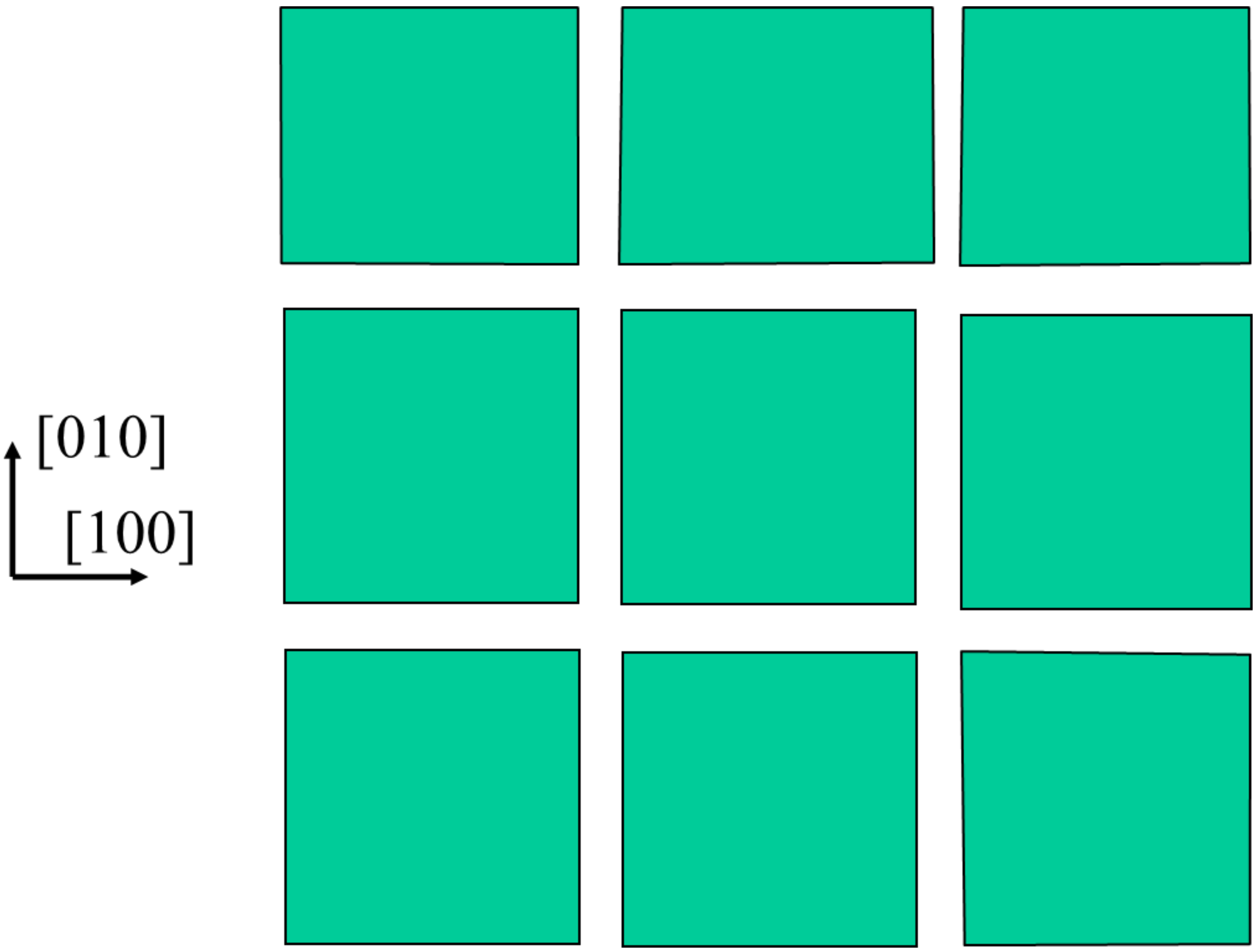


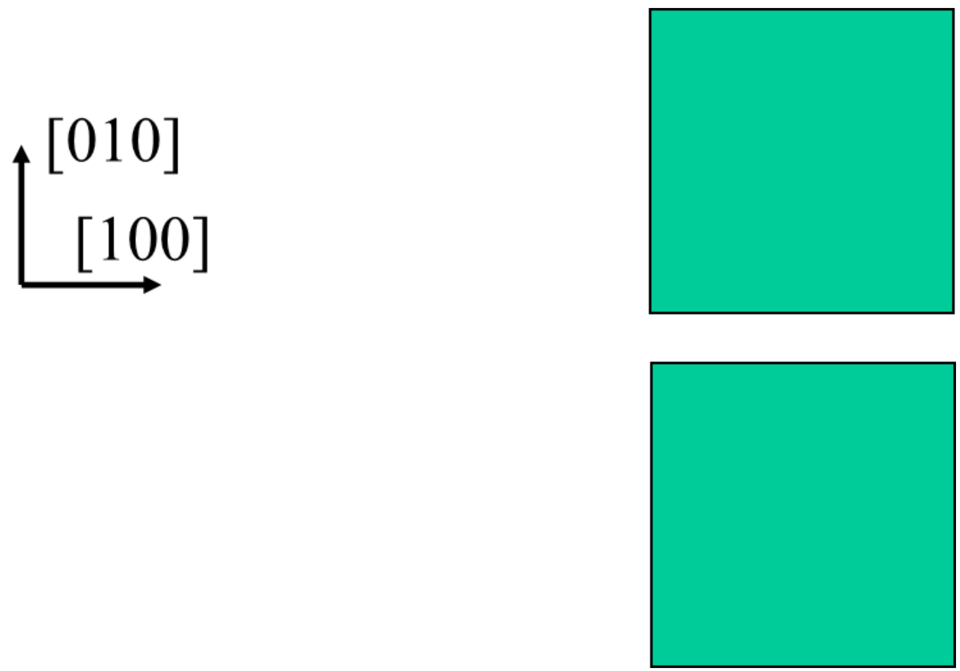


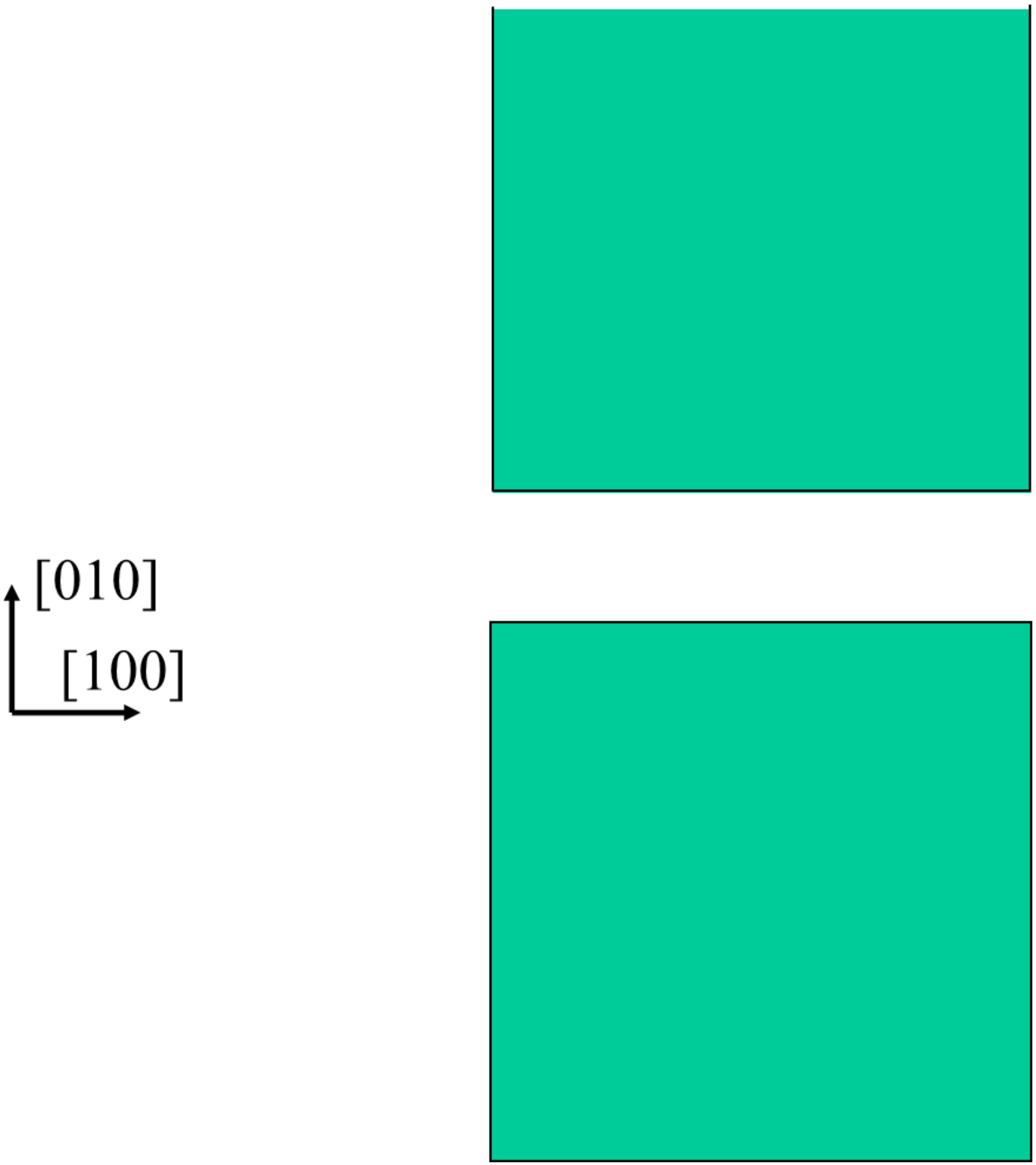
FAMSE-GEII-66

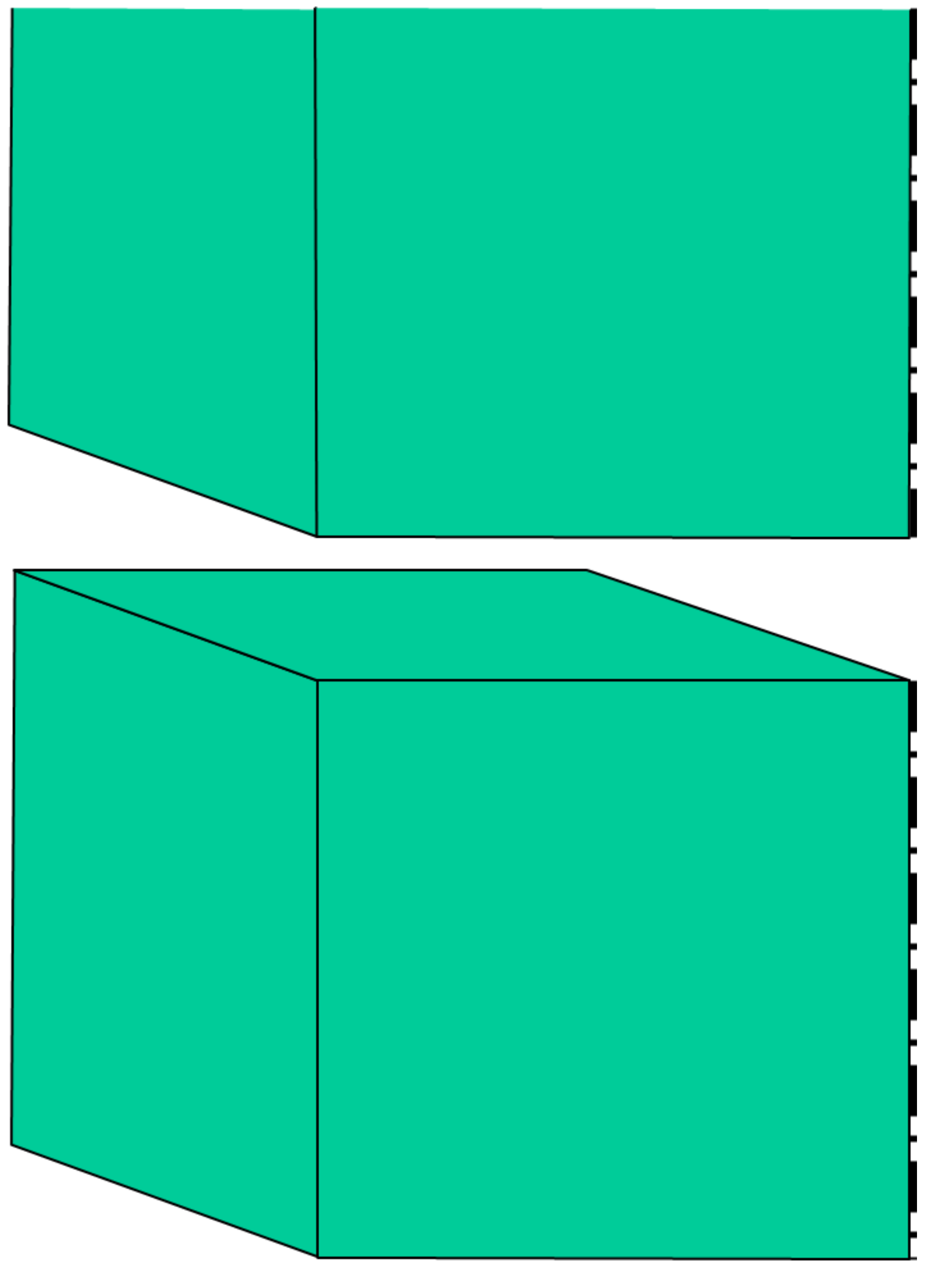

[010]
[100]


γ
[010]
[100]
γ'


[010]
[100]

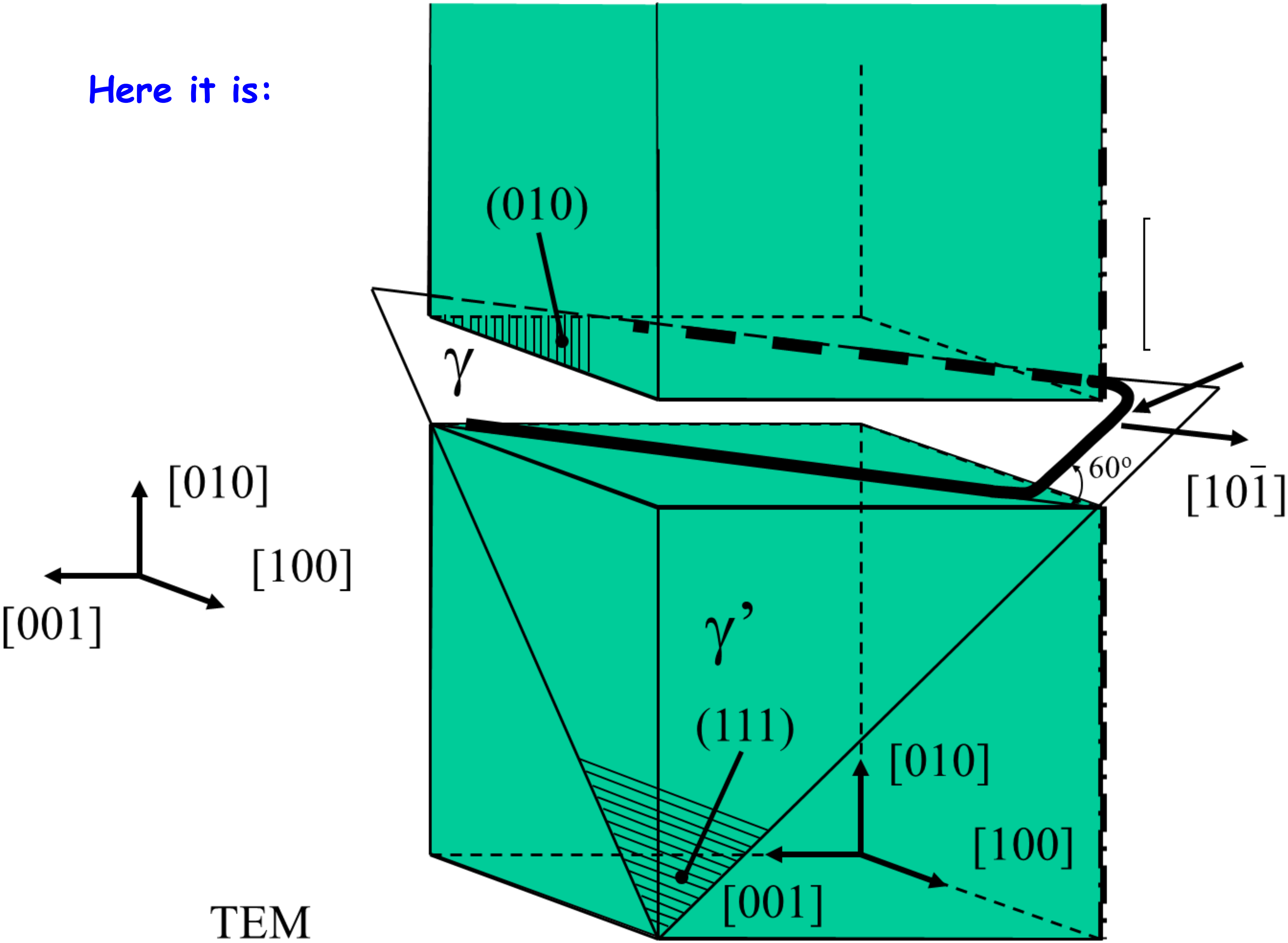

[010]
[100]

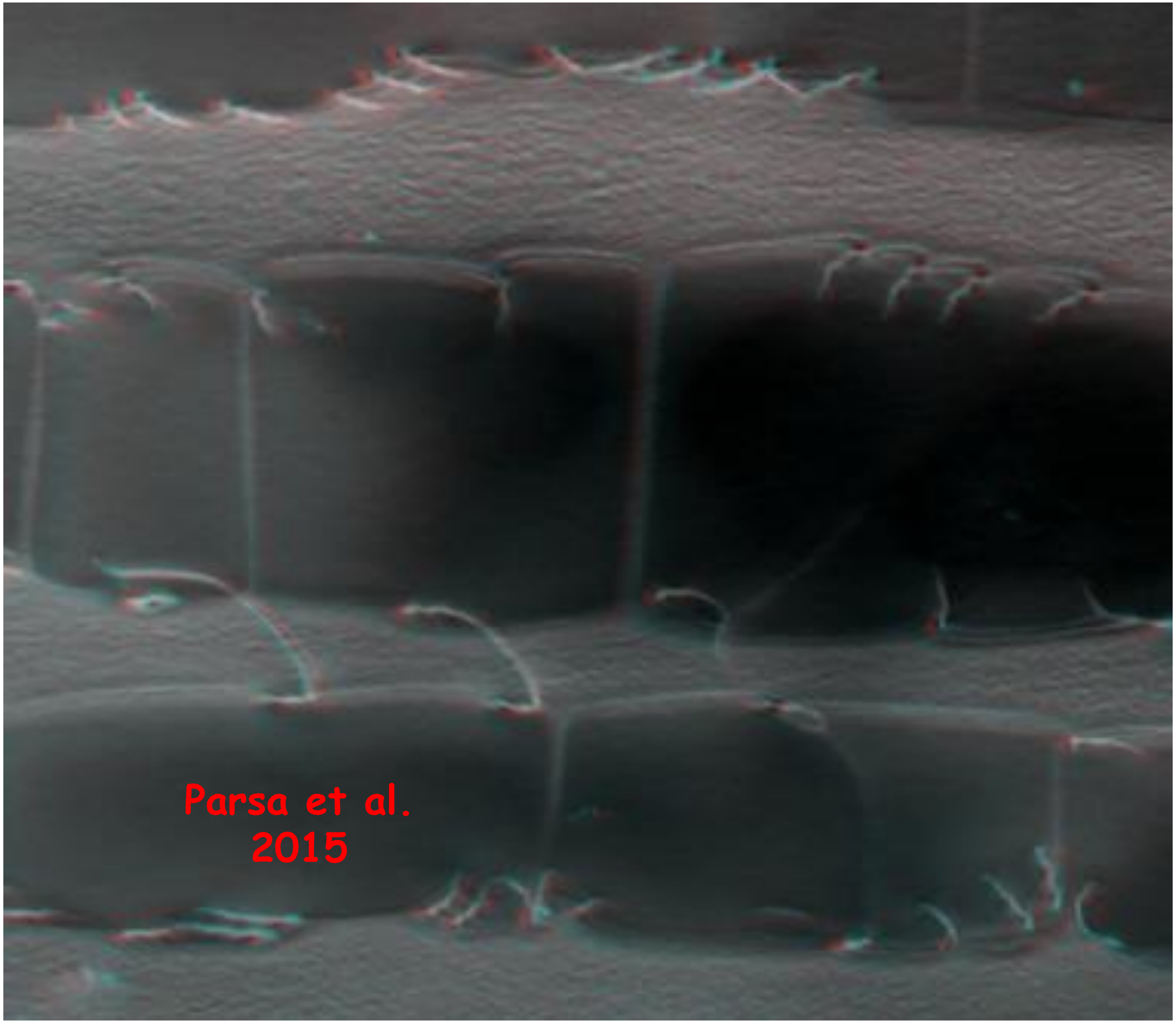

[010]
[100]

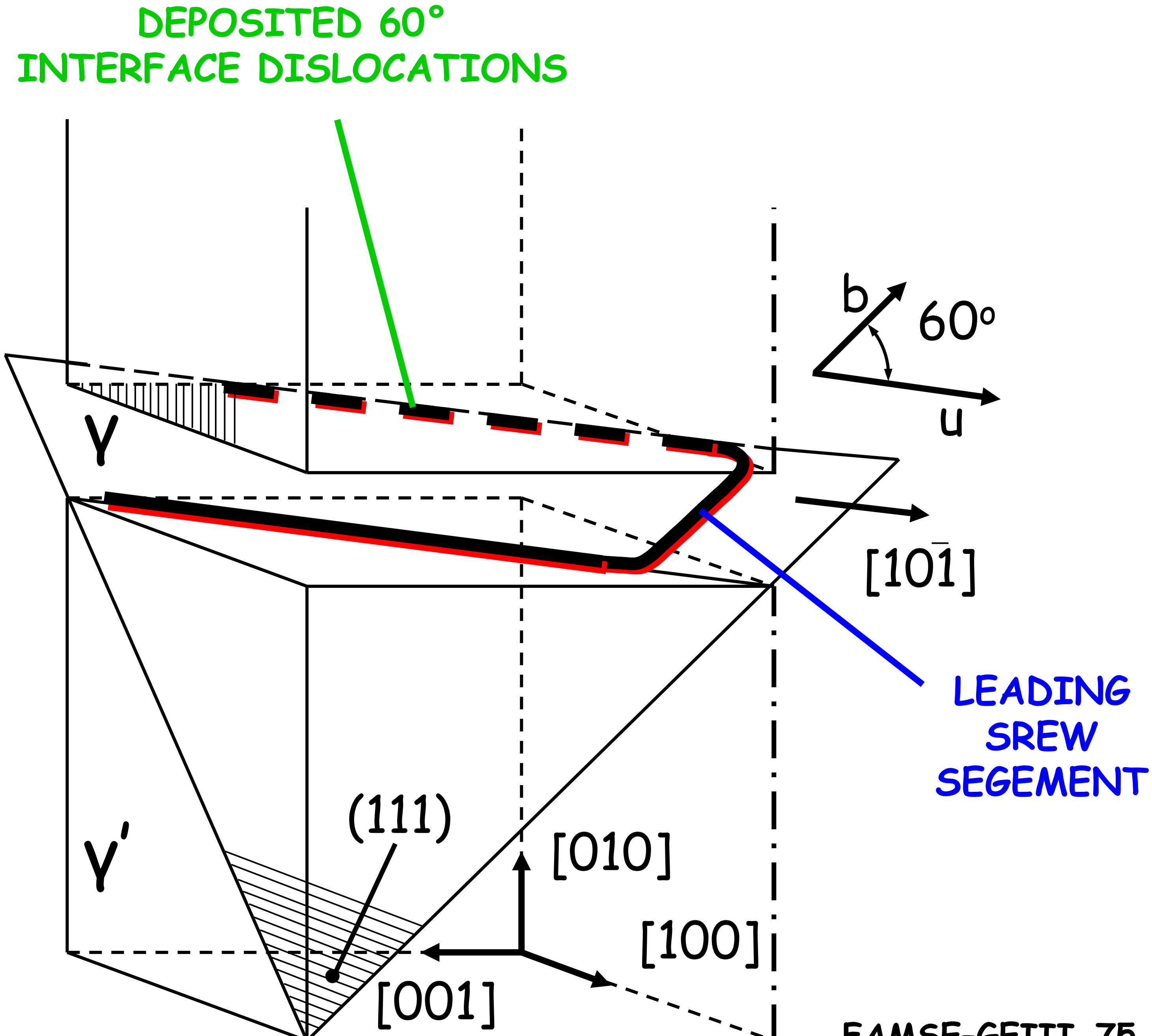


FAMSE-GEII-72

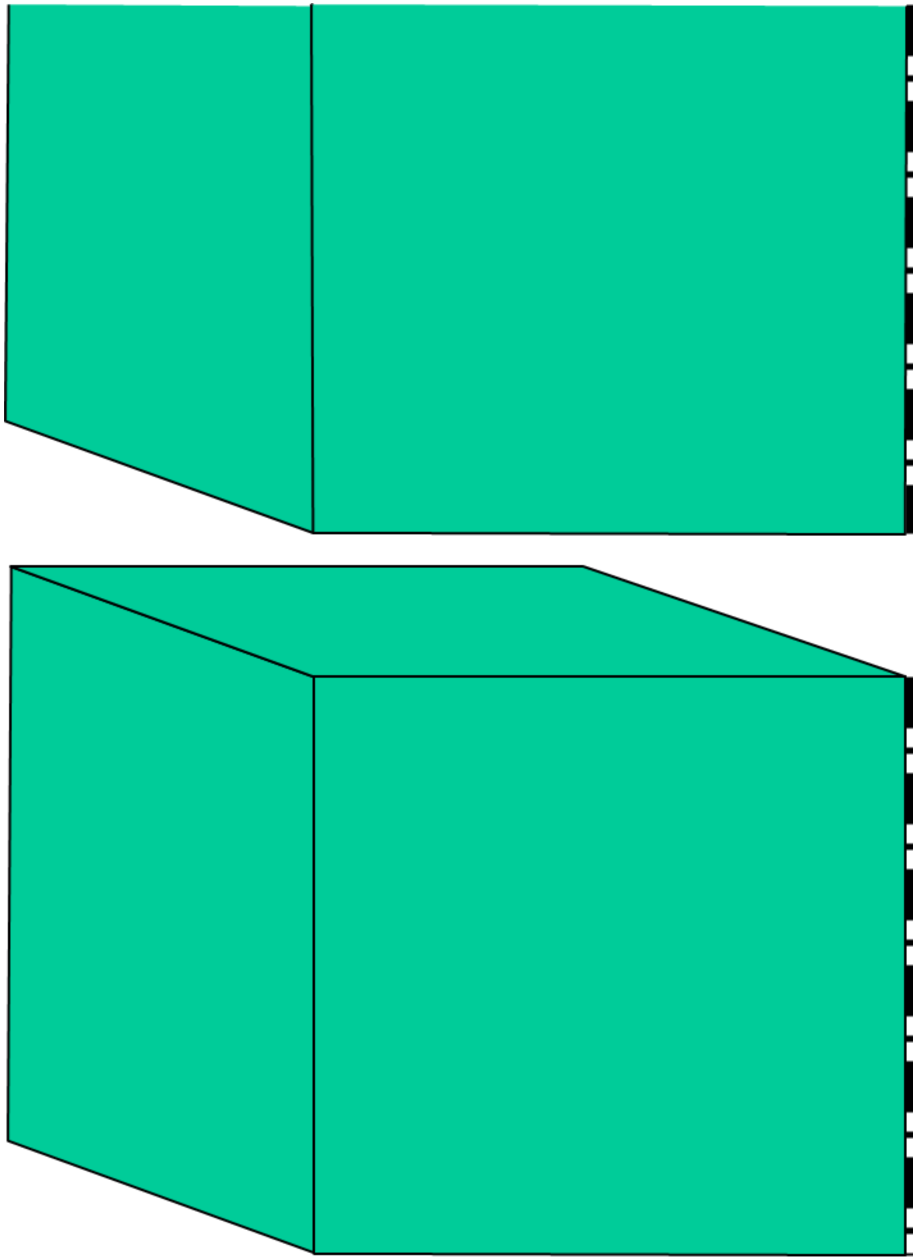
Here it is:



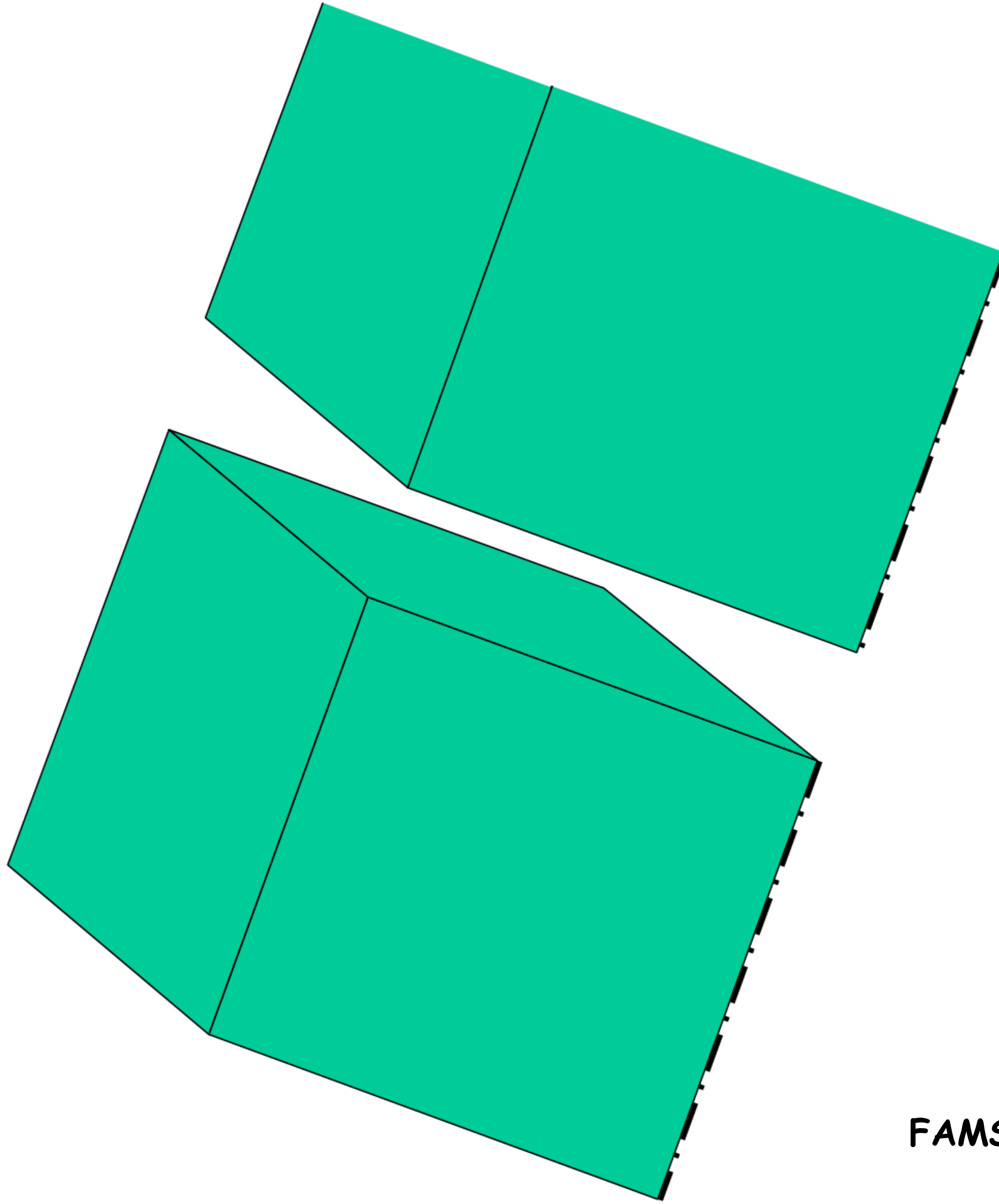

Parsa et al.
2015



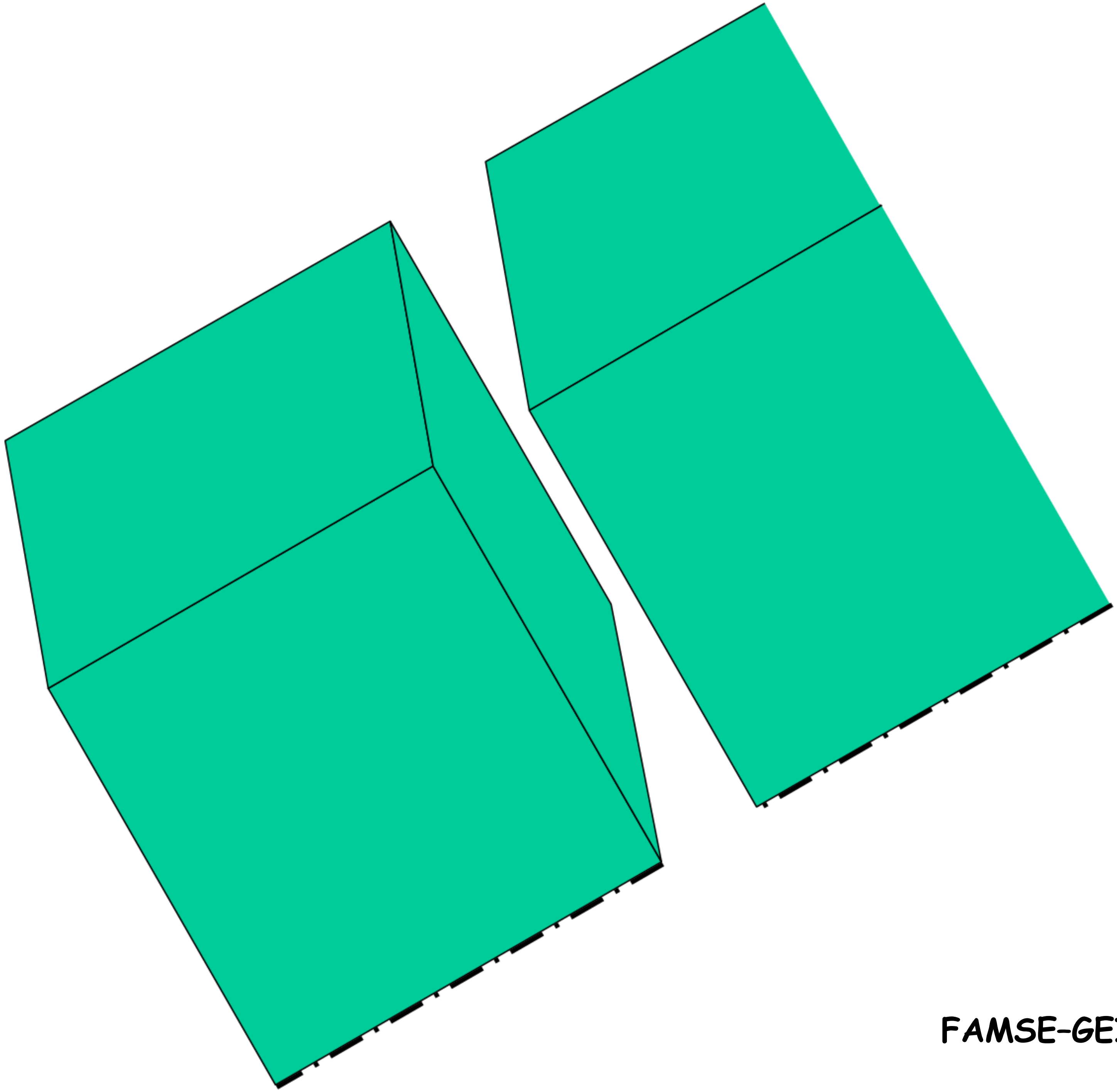
FAMSE-GEIII-75



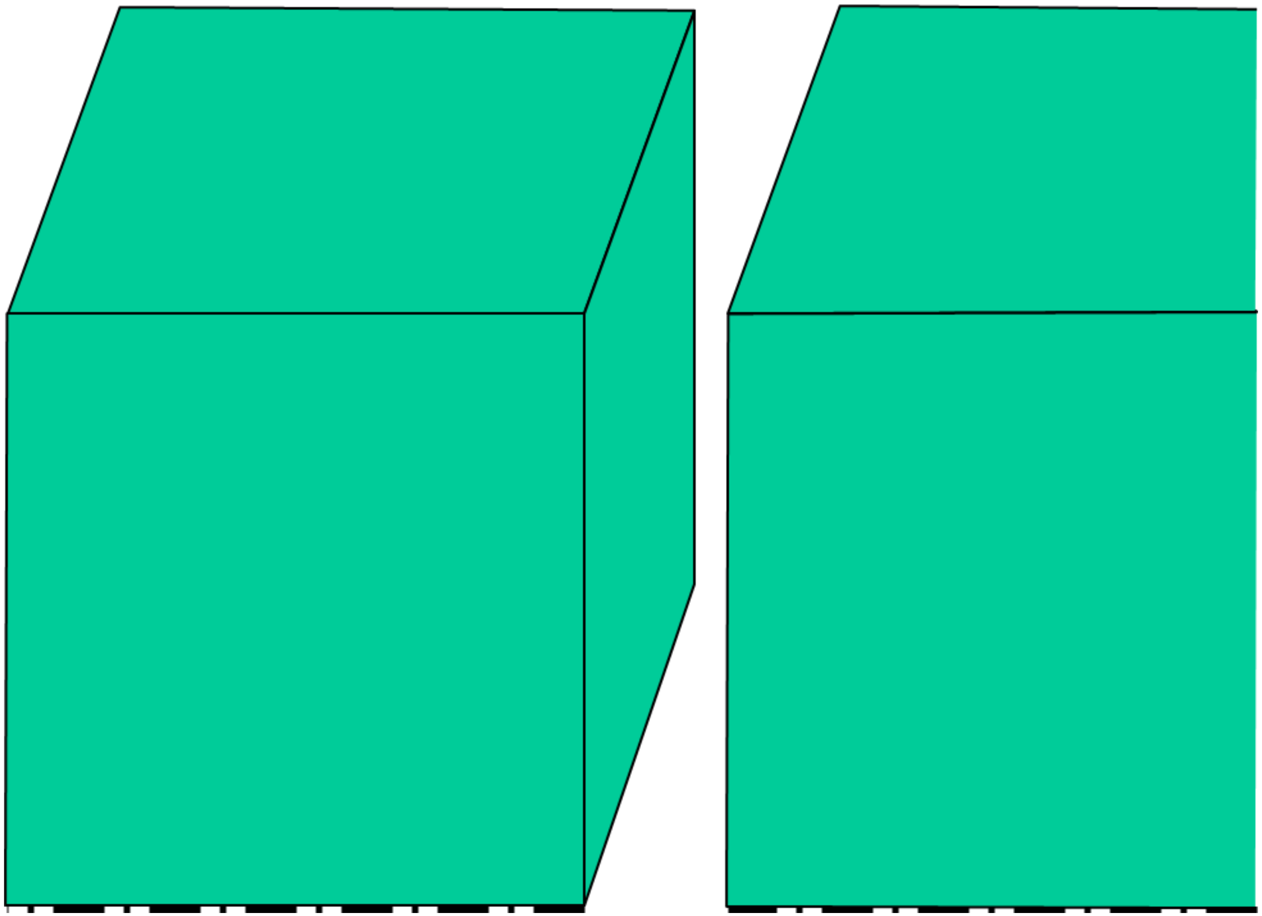
FAMSE-GEII-76

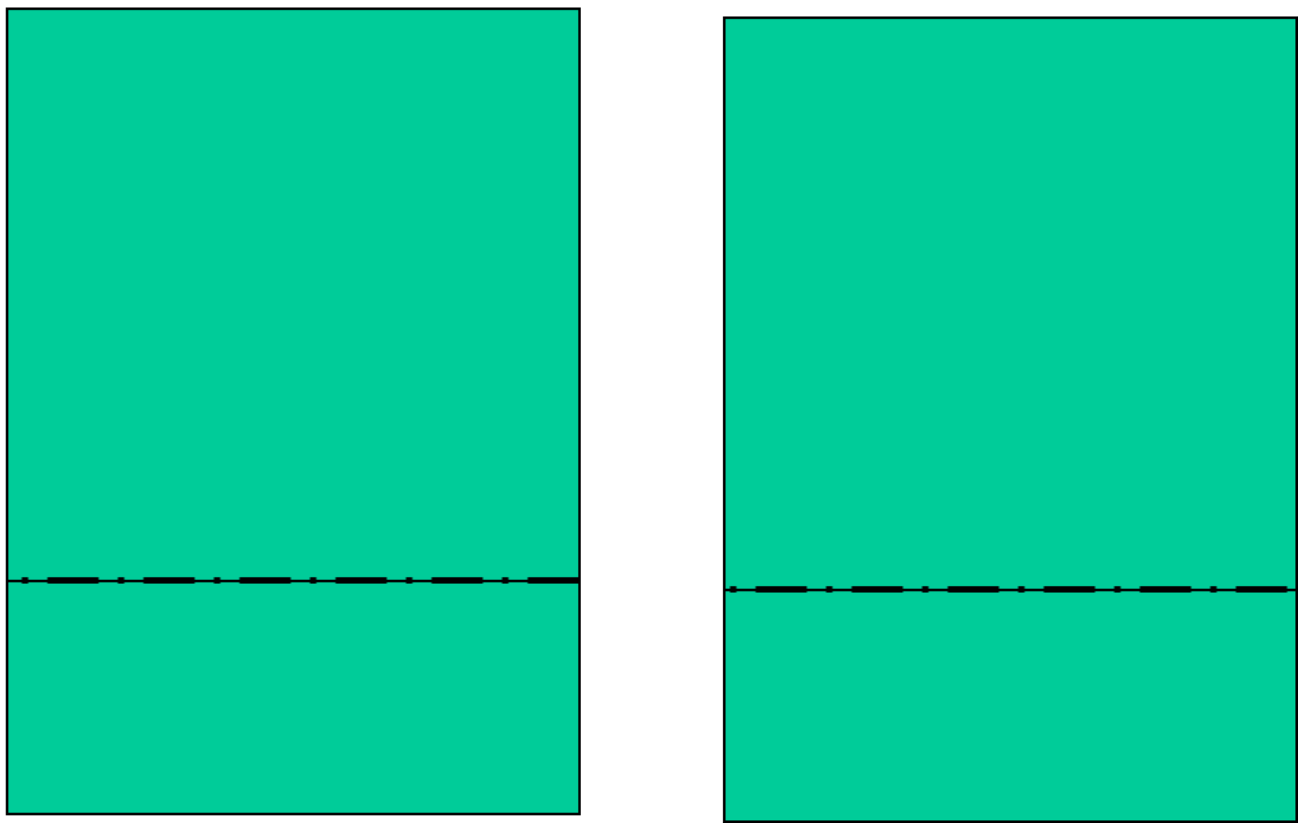


FAMSE-GEII-77

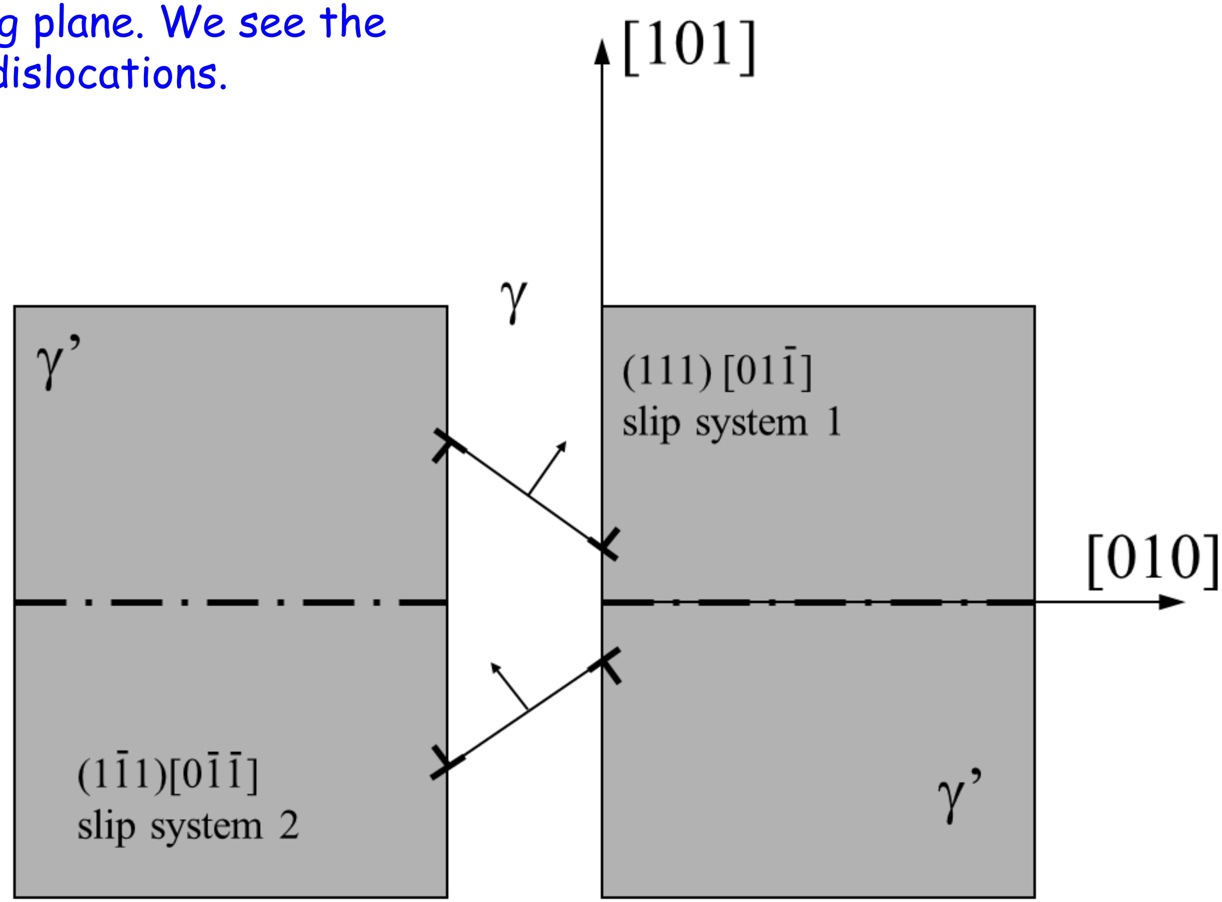


FAMSE-GEII-78



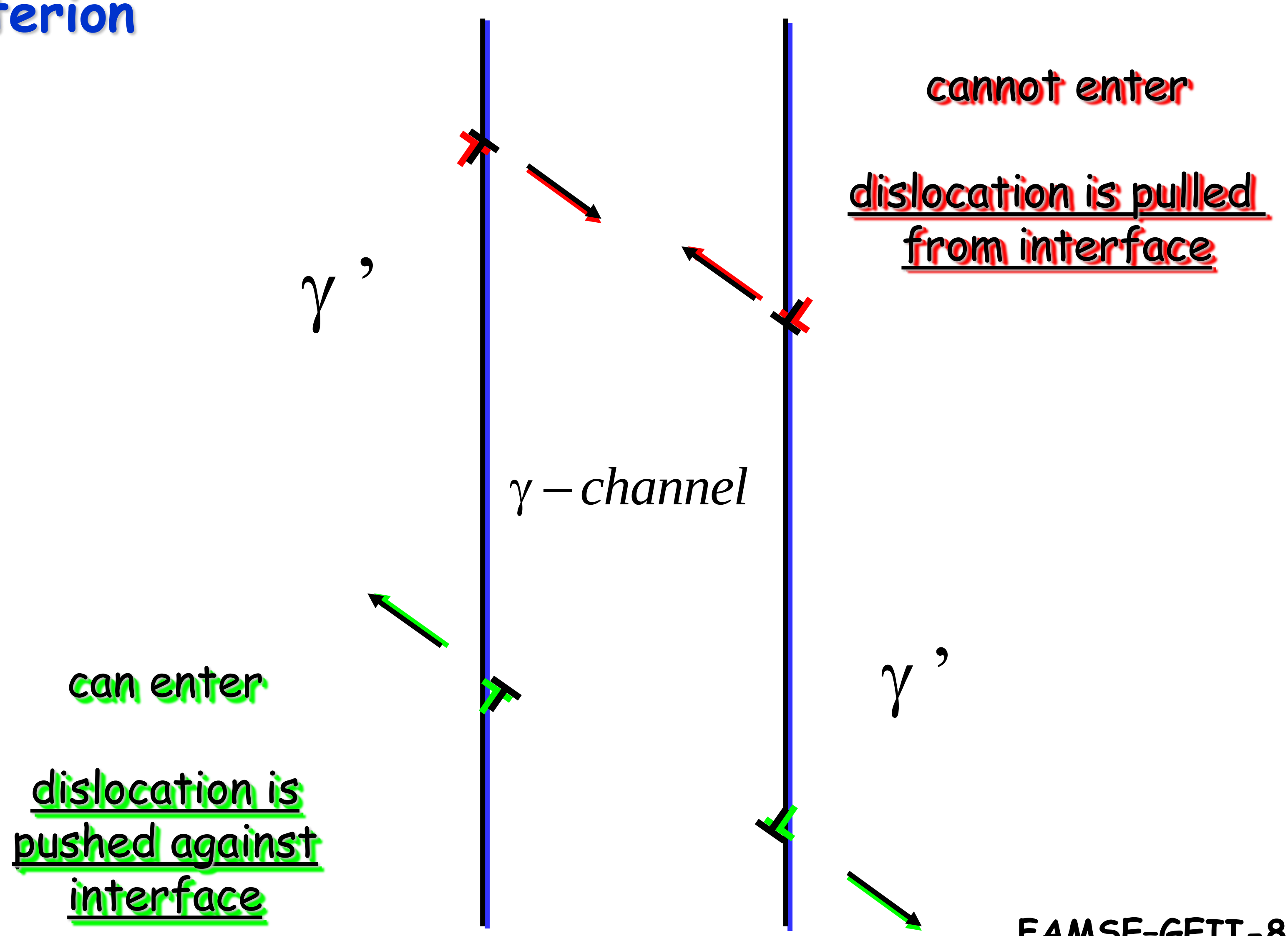


The leading screw segments moves into the drawing plane. We see the deposited 60° dislocations.

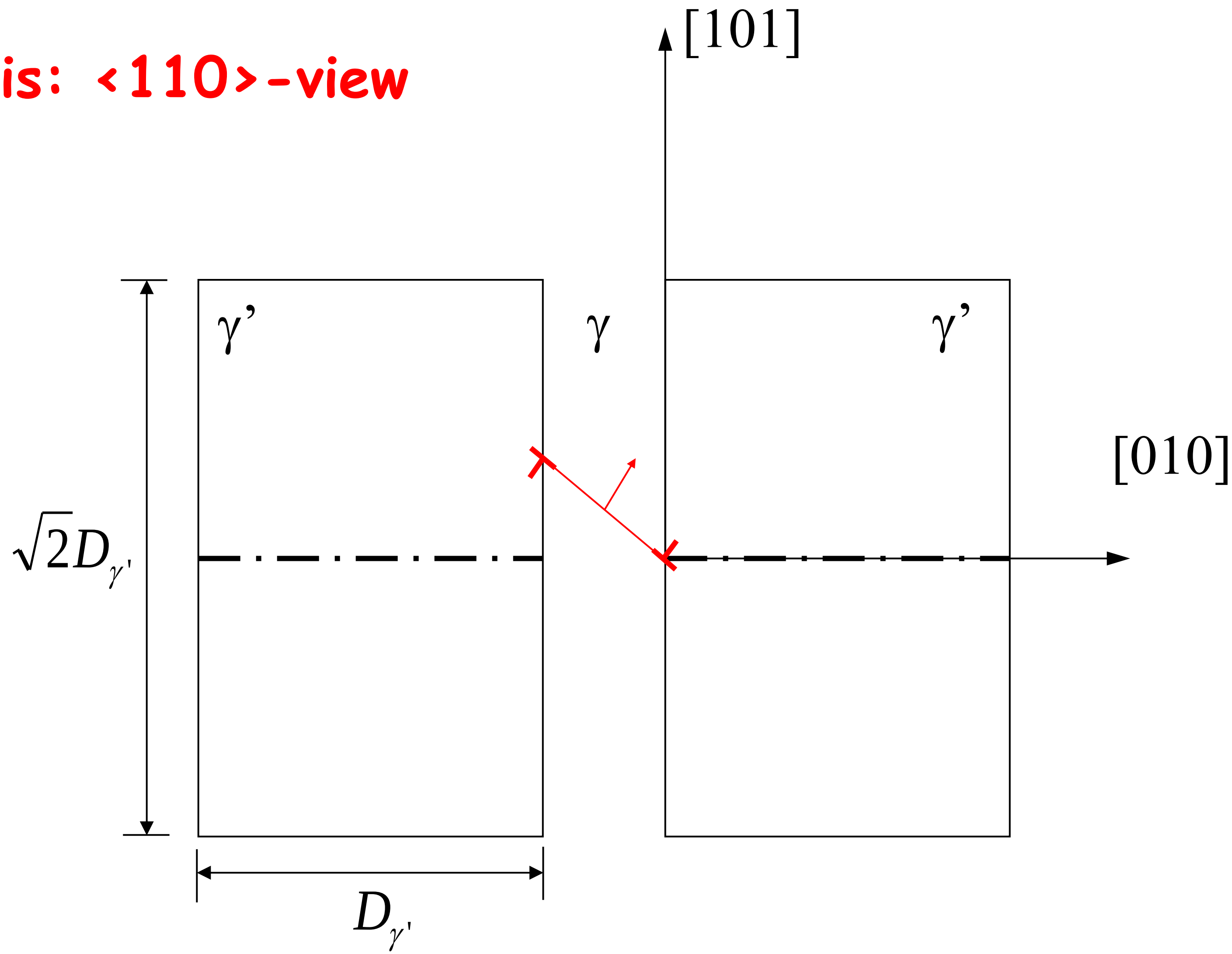


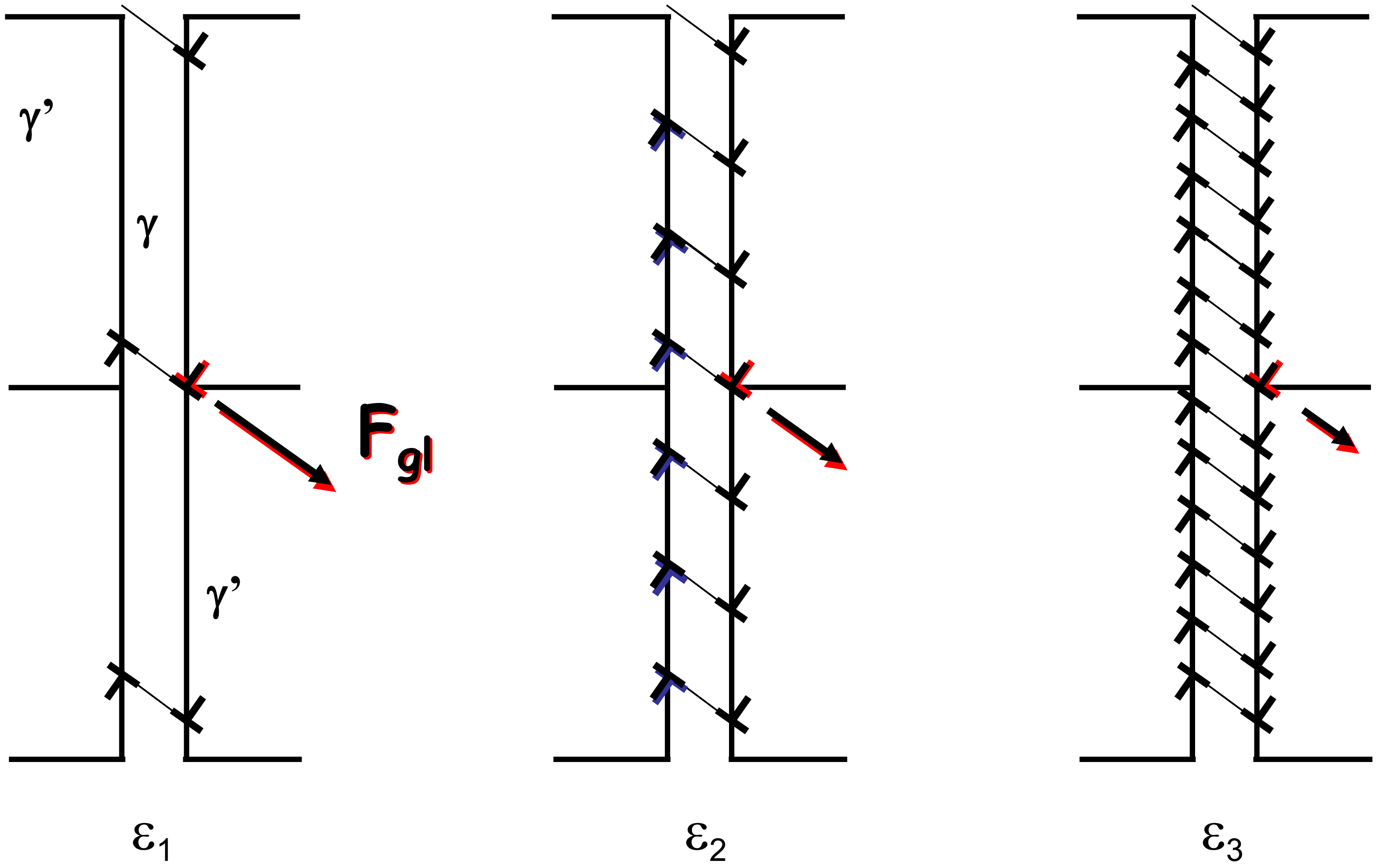
A leading screw segment deposits a 60° dislocation dipole. These are two dislocations of opposite sign on both sides of the γ-channel.

Peach Koehler force enter criterion



analysis: <110>-view





empty **increased** **full**

Calculation of Peach Koehler force (text book formula):

$$F_{gl}/L = \frac{\left[(b\cdot\sigma)\times\xi\right]\cdot\left[\xi\times(b\times\xi)\right]}{\left|b\times\xi\right|}$$

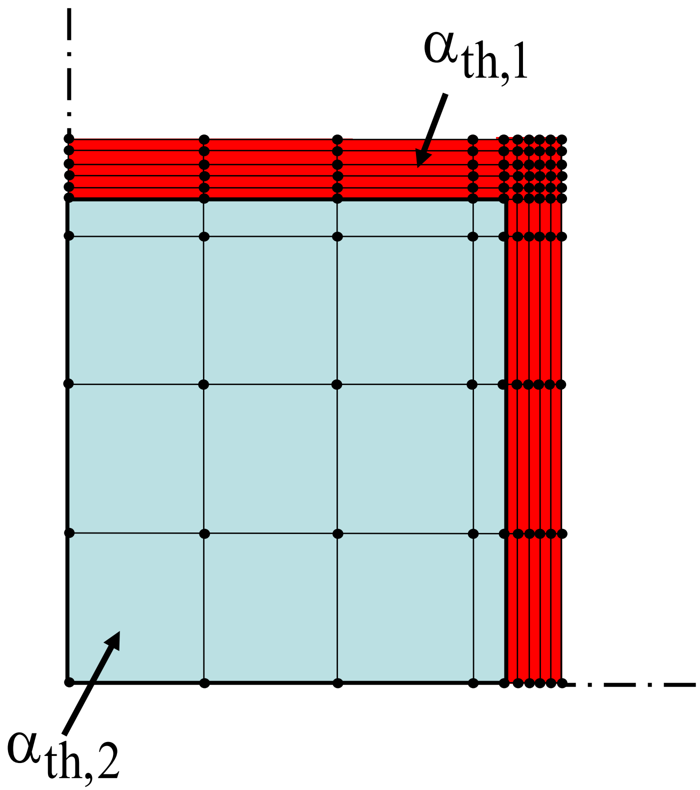
$$\sigma = \sigma_{creep} + \sigma_{misfit} + \sigma_{dislocations}$$

ext. load

FEM

Cottrell formulas for dislocation arrays

Misfit stresses - (artificial) heating in computer:

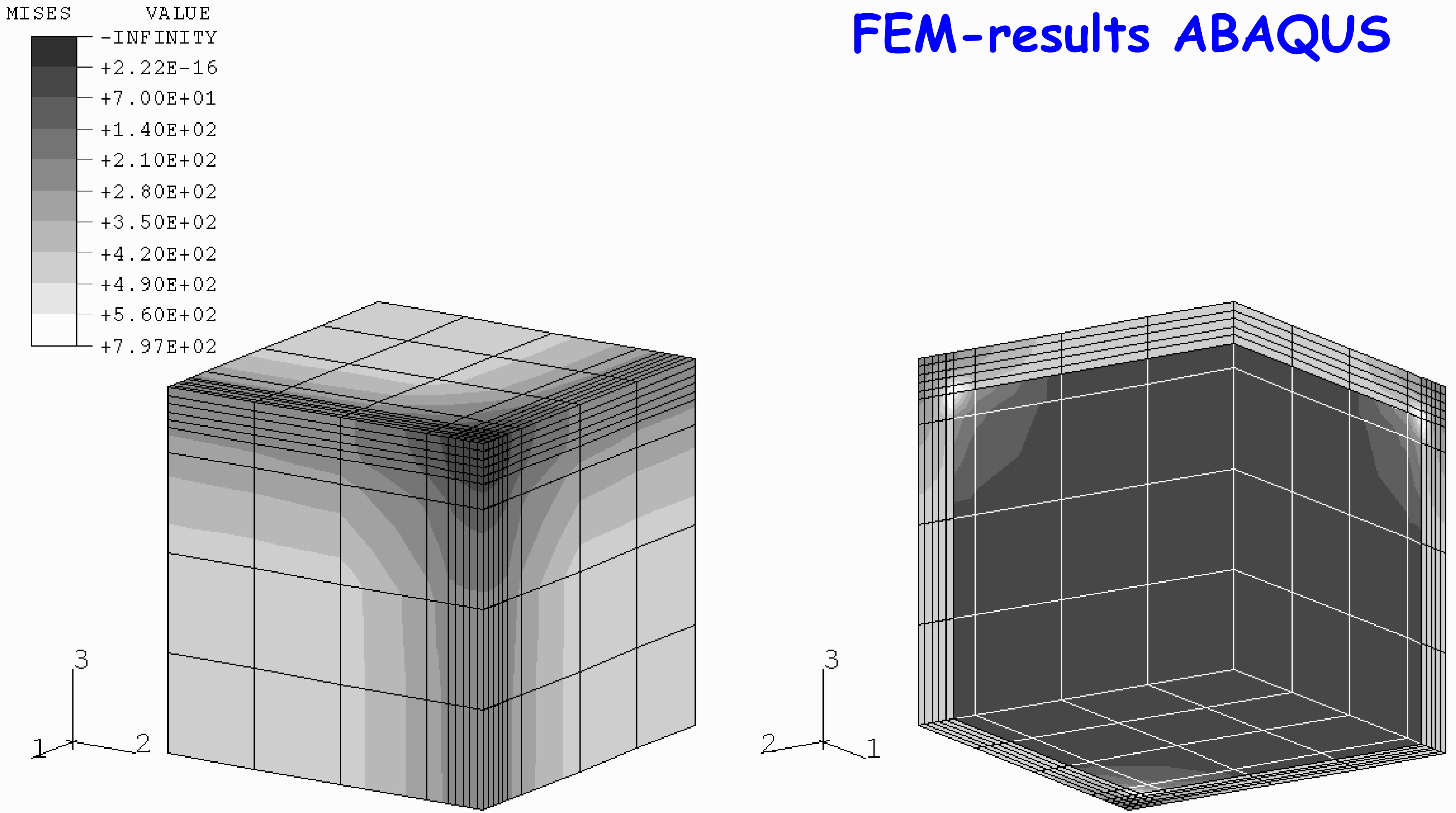


T_0 :
$d_1 = d_2$

$\longrightarrow$ kT

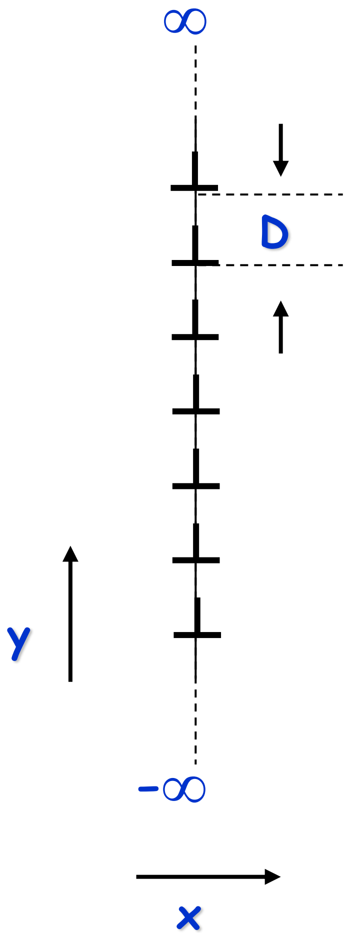
$T_0 + \Delta T$:
$d_1 = d_\gamma \ \wedge \ d_2 = d_\gamma$,

FEM-results ABAQUS



view: [3 2 1]

view: [-3 -2 -1]



stresses from channel dislocations

(1) add up all individual contributions:

$$\sigma_{\perp - xy} = \frac{G\,b}{2\,\pi(1-\nu)} \cdot \sum_{n=-\infty}^{\infty} \frac{x\,(x^2 - (y - nD)^2)}{(x^2 + (y - nD)^2)^2}$$

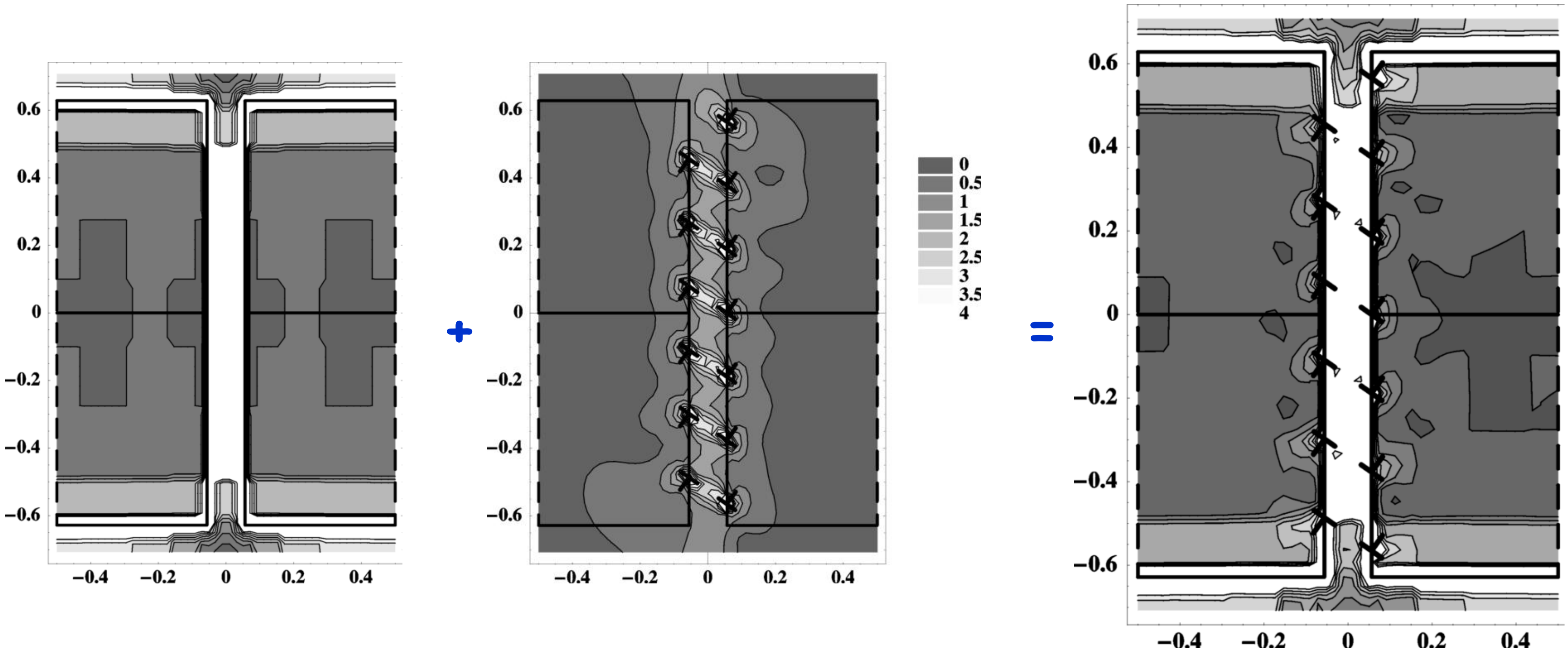
(2) analytical solution for networks (Cottrell):

$$\sigma_{\perp - xy} = \sigma_0 \cdot 2\,\pi\,X \cdot \left(\cosh 2\pi X \cos 2\pi Y - 1\right)$$

with:

$$\sigma_0 = \frac{G \cdot b}{2\,D \cdot (1-\nu) \cdot (\cosh 2\pi X - \cos 2\pi Y)^2}$$

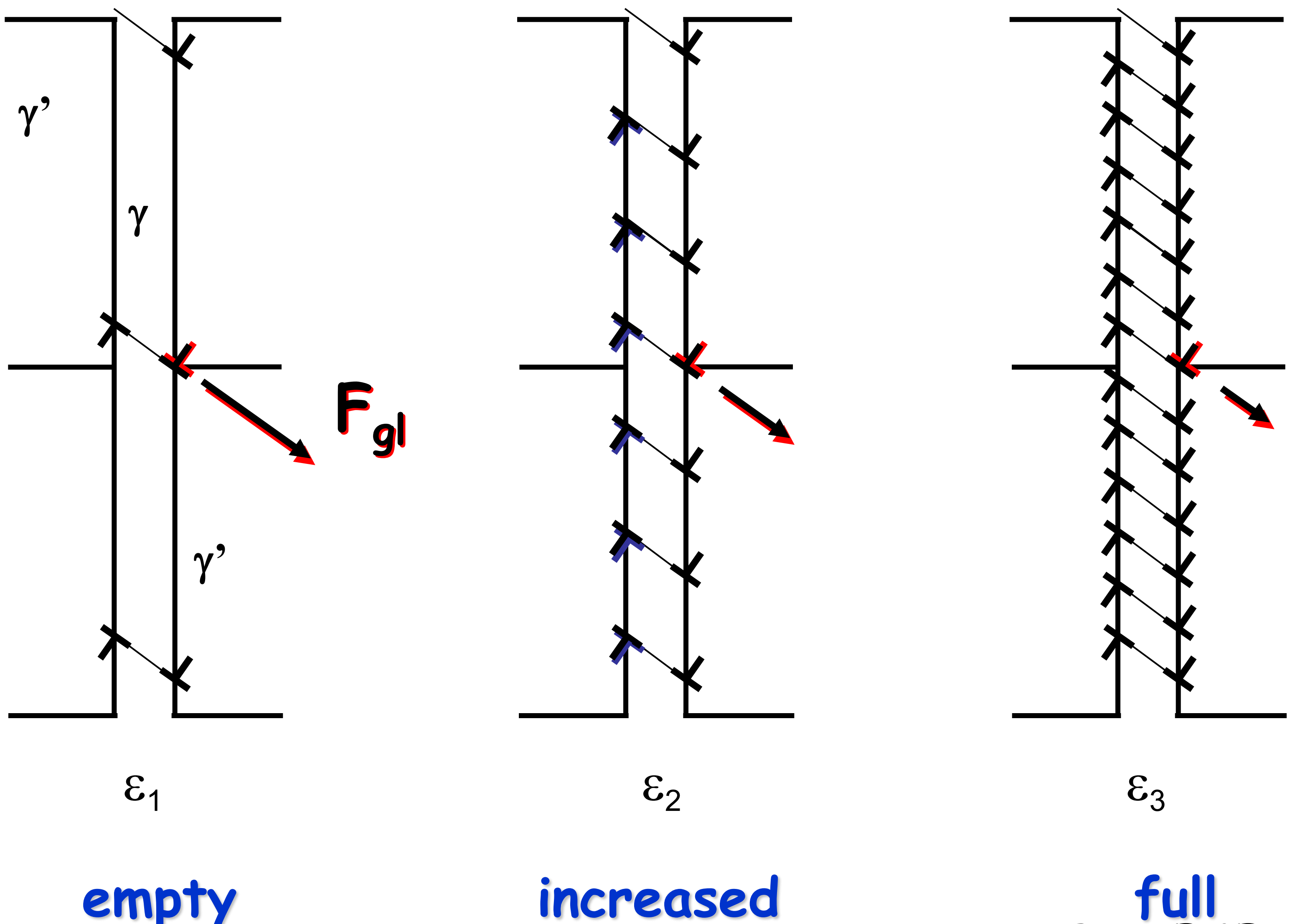
$$X = \frac{x}{D} \quad \text{and} \quad Y = \frac{y}{D}$$



Misfit
(ABAQUS)

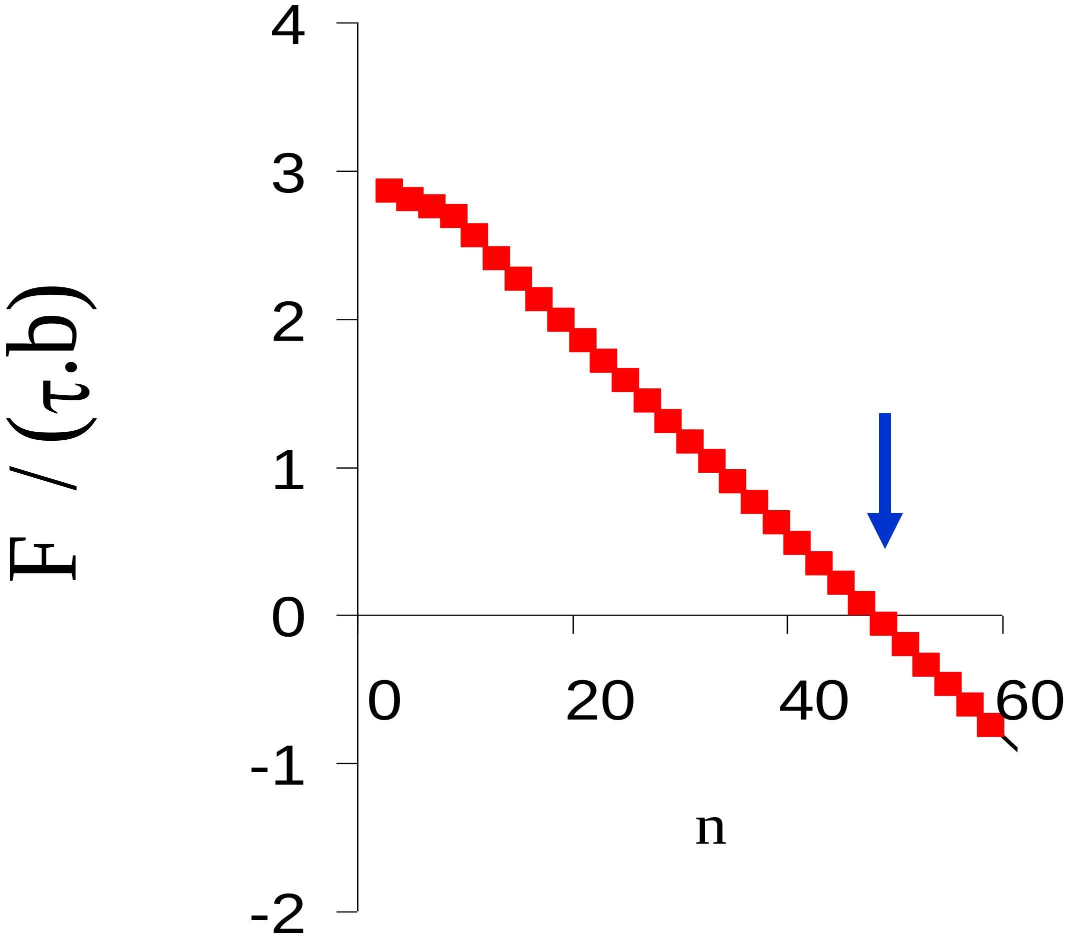
Dislocations
(Cottrell)

superposition



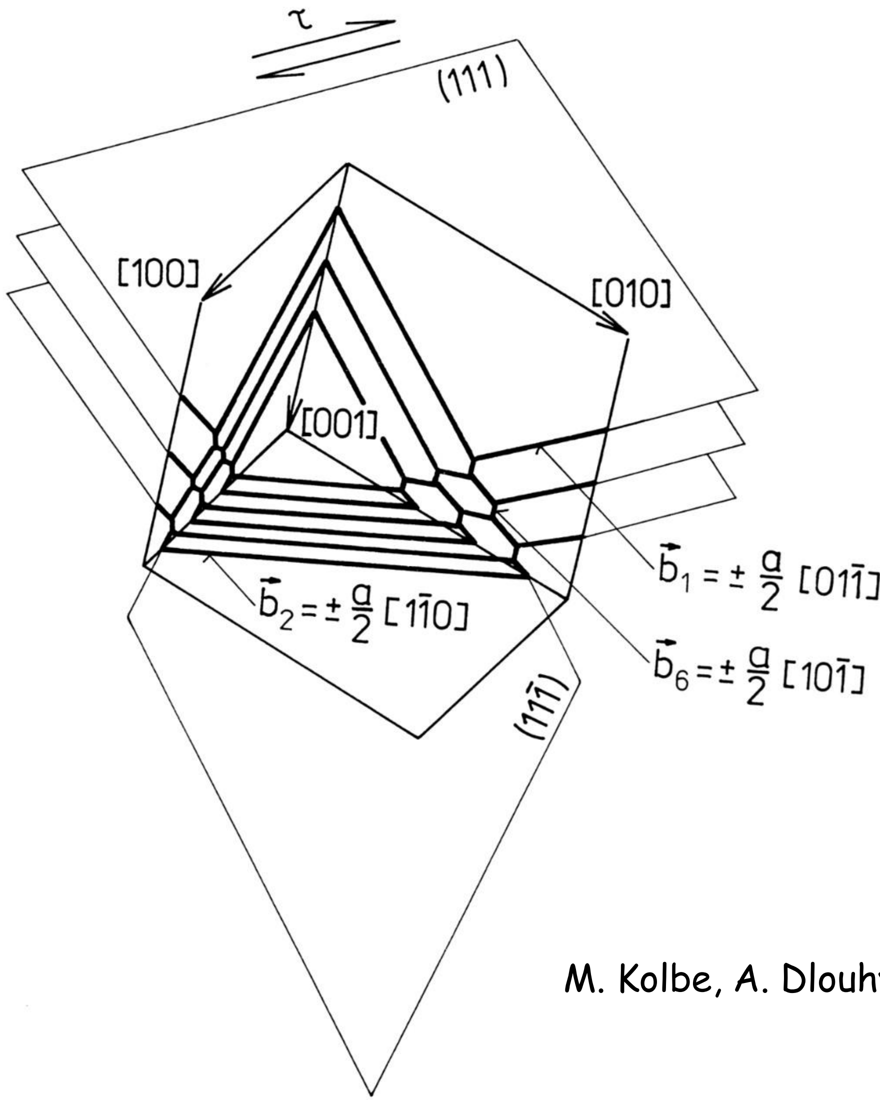
FAMSE-GEII-90

evolution of Peach Köhler force:

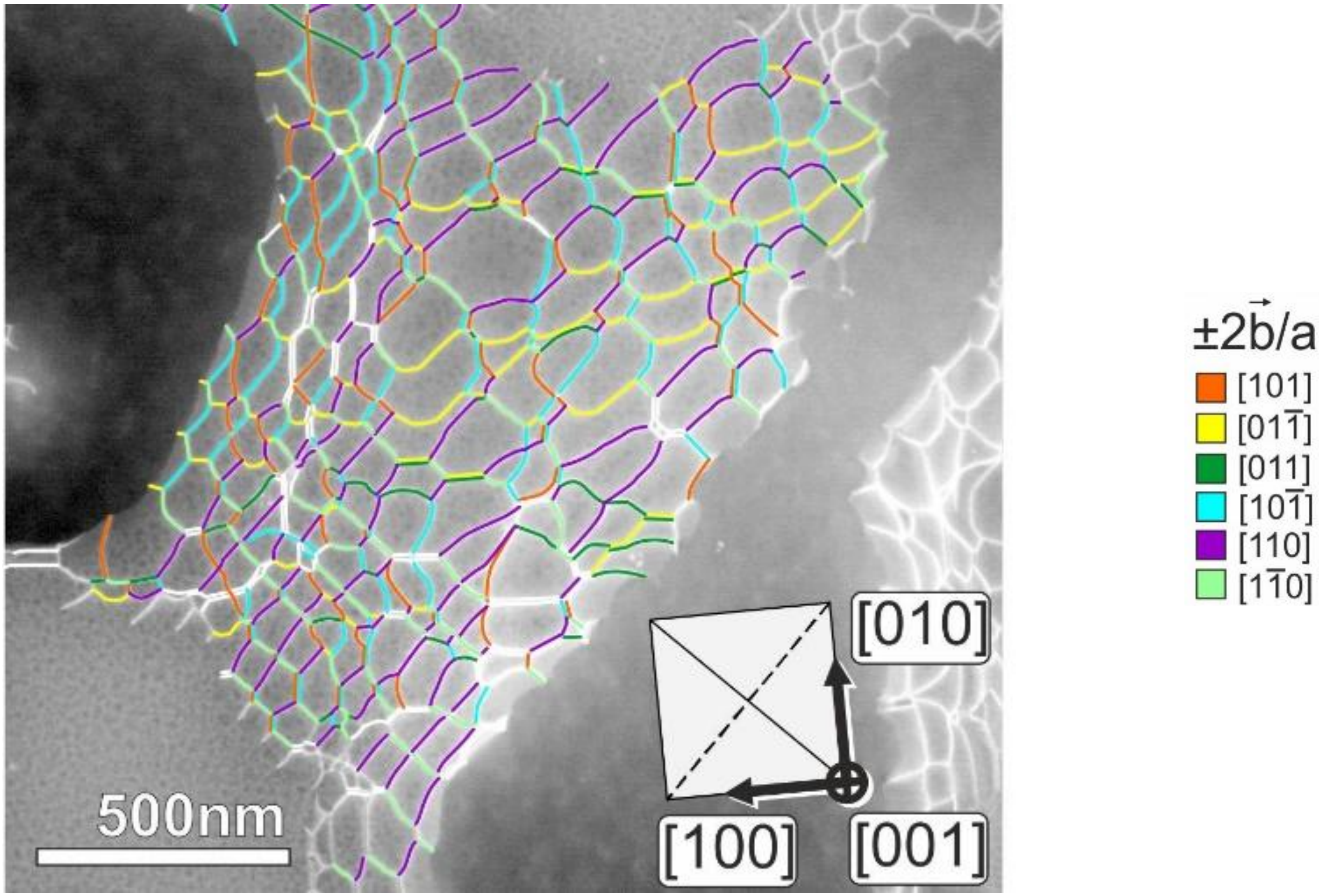


calculated dislocation spacing:

$$d = (\sqrt{2} \cdot D_{\gamma'}) / 48$$



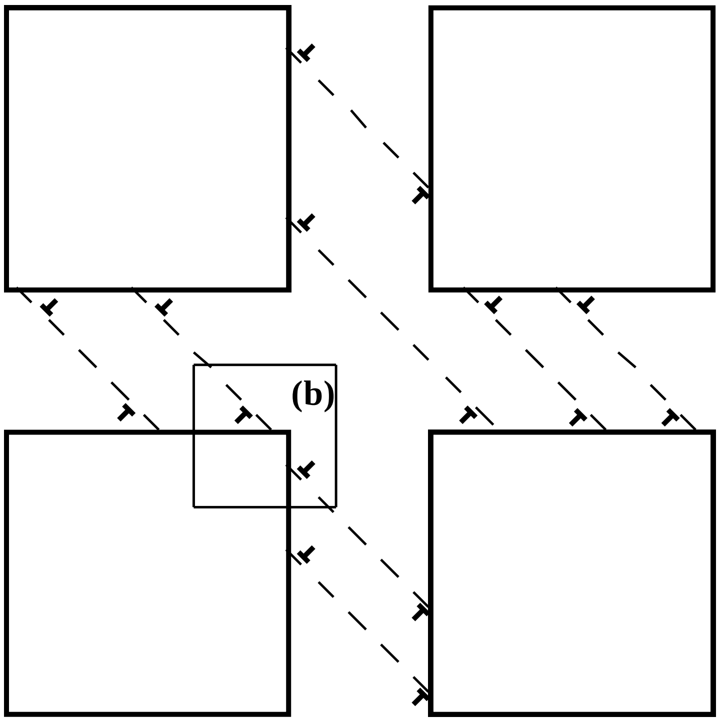
M. Kolbe, A. Dlouhy, G. Eggeler, Mat. Sci. Eng. A, 1998


500nm
[010]
[100]
[001]
±2b/a
[101]
[01$\bar{1}$]
[011]
[10$\bar{1}$]
[110]
[1$\bar{1}$0]

comparison experimental data - model:

	Feller-Kniepmeier and Link Met.Trans.A 1989 Vol. 20, p. 1233	Mayr, Eggler and Dlouhy Mat.Sci.Eng.A 1996 Vol. 207, p.51	Keller, Maier and Mughrabi Scripta Met. Mat. 1993 Vol. 28, p. 23	Probst-Hein, Dlouhy and Eggler Acta Mat. 1999 Vol. 47 p. 2497
type of work	**TEM**	**TEM**	**TEM**	**DDM**
alloy	SRR 99	CMSX6	SRR 99	systems with varying δ
loading condition	<100>-tension	<110>{111}-shear	<100>-tension	<110>{111}-shear
temperature in °C	980	1025	1050	1025*
stress (σ/τ) in MPa	170	85	200	85
TEM-Figures	8a	12	1 and 2	-
d_1 [nm]	**52**	**26**	**32**	**23** (δ = - 0.003)
d_2 [nm]	**55**	**42**	**60**	**57** (δ = - 0.0002)
d_3 [nm]	**82**	**76**	**83**	**113** (δ = 0.0005)

recovery at γ'-corners:

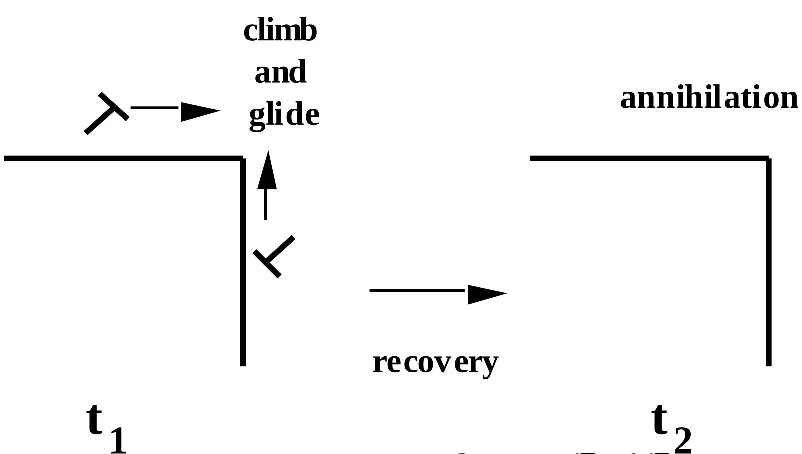


(a)

comparison with experiments:

recovery is important!

corner recovery ceases as rafting proceeds:



(b)

Section summary - dislocations

Dislocations are responsible for the high temperature deformation of single crystal superalloys.

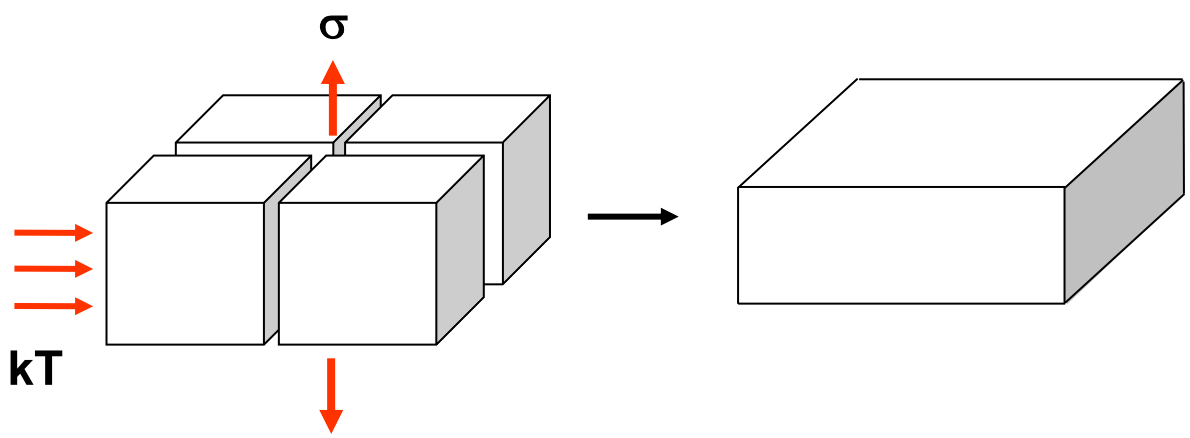
They squeeze into thin γ-channels. There is a leading screw segment which runs through the channels. In doing so, networks form at the γ/γ'-interfaces.

Each dislocation segment experiences Peach Köhler glide and climb forces which result from the local stress state.

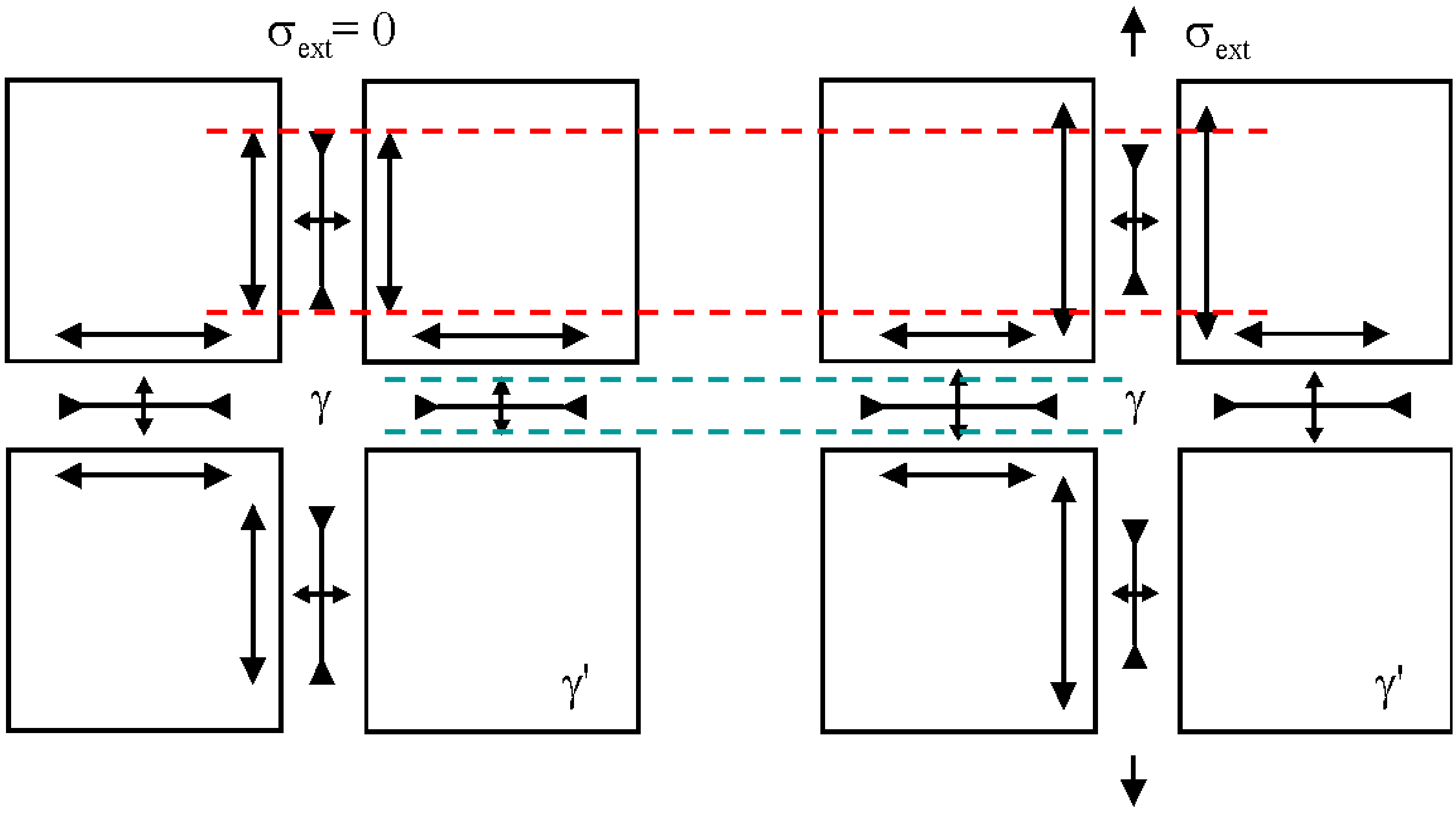
The local stress state results from a superposition of the externals stress (load in creep test, Schmid law), the local stress associated with misfit and the local stress associated with the neighbourhood of other dislocations.

Rafting

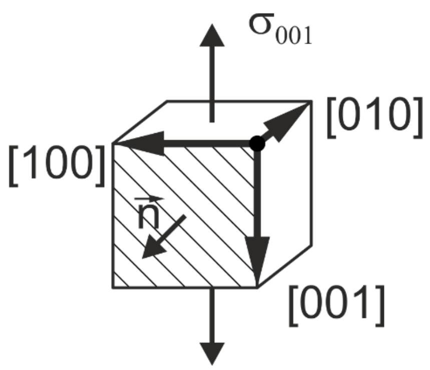
Rafting



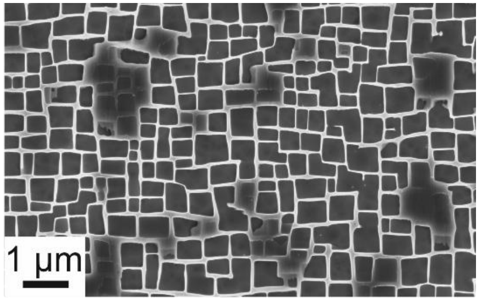
Directional coarsening of the γ'-phase. Cubes transforms into rafts by diffusion of alloy elements. This has to do with the Stress state in the γ/γ' microstructure.



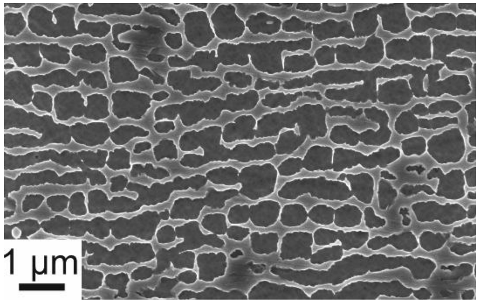
lattice misfit – coherency stresses



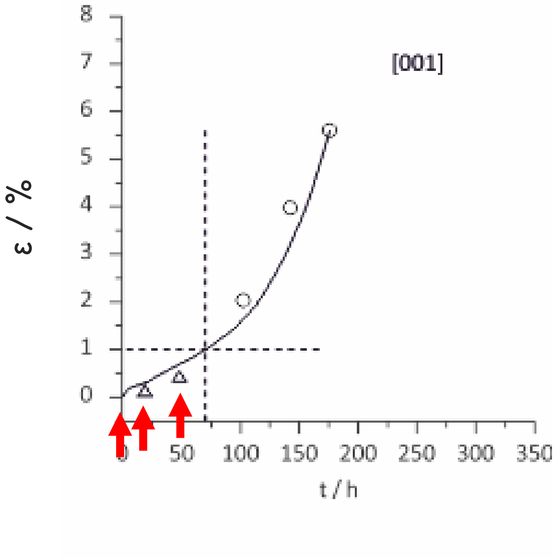
1293 K, 160 MPa

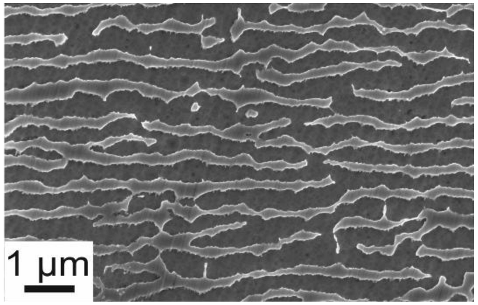


0 %

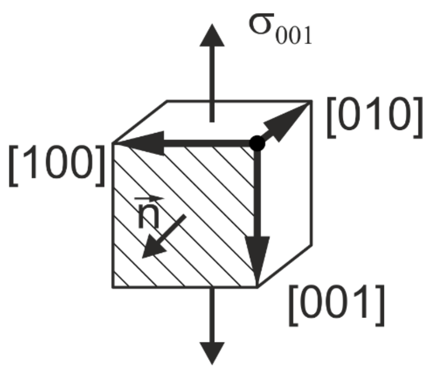


0,1 % (81 h)

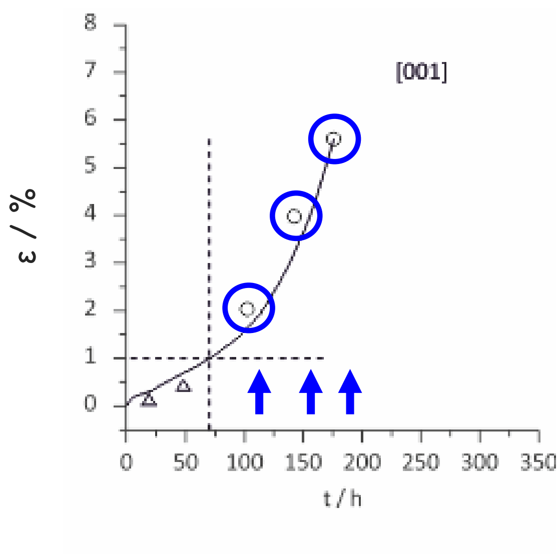


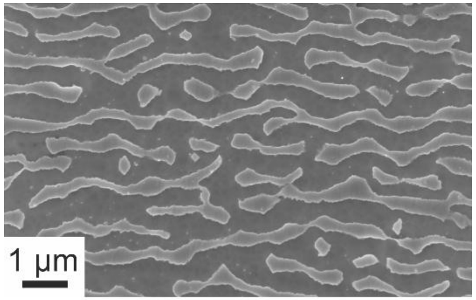


0,4 % (169 h)

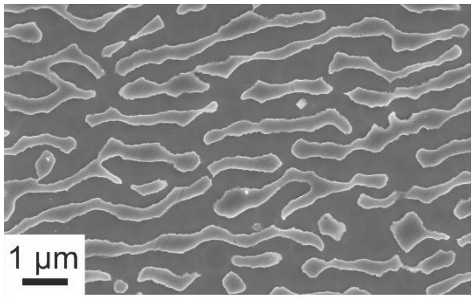


1293 K, 160 MPa

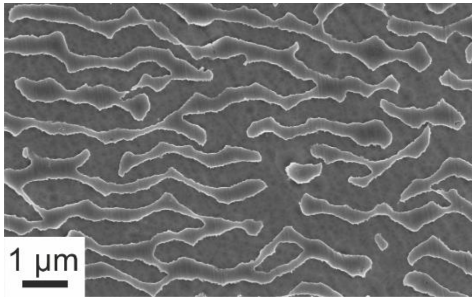




2 %



4 %



6 %

Section summary - rafting

Rafting is the directional coarsening of the γ'-phase.

Rafting has been most frequently observed in <100> tensile tests for SX with negative misfit. Rafts then form perpendicular to the direction of the applied stress.

In the 1000°C temperature range, rafting is fast (occurs in a few hours).

Outlook - creep materials science:

- Can we provide a model which accounts for dislocation plasticity and rafting?
- What happens during low temperature and high stress creep?
- How does alloy compostion affect creep (atomistic and microstructural view)?
- New experiments: high resolution TEM, 3D AP, nanomechanics, …
- New technologies: fluidized bed cooling, HIP treatments, additivie manufacturing, ...

Summary

- SX for first stage blades in advanced gas turbines
- SFB/TR 103
- Processing of superalloys
- Dendrites / interdendritic regions
- γ/γ'-microstructure
- Misfit of γ and γ'
- Elements of micromechanical modelling (simplified 2D discussion)
- Rafting

Questions for self control

1. What are single crystal Ni-based superalloys (SX) used for?
2. Which research fields must interact to progress SX technology?
3. Name five elements which are typically alloyed to Ni-base SX?
4. How does the Bridgman process work (drawing)?
5. Explain the formation of flat and irregular interfaces on the basis of temperature gradients across solid/liquid interfaces. What does the term constitutional undercooling mean?
6. What is the reason for the multiple step solutio heat treatment of SX?
7. Which large scale microstructural heterogeneity in SX is related to solidification?
8. What is a typical dendrite spacing?
9. Which small scale heterogeneity characterizes the microstructure of SX?
10. Why does the well known γ/γ'-microstructure consists of γ'-cubes with edges parallel to <100>-direction? Which role does the lattice misfit between the two phases and the elastic anistropy play in this context?

Questions for self control ctd.

11. How do dislocations enter γ-channels? Explain the terms „leading screw segment and 60°-dislocation!
12. Why and how do dislocation networks form around γ'-particles.
13. Explain how the microstructure evolves during creep. Which processes govern decreasing creep rates during primary creep and the minimum creep rate? What processes occur during tertiary creep?
14. What happens when an ordinary ½<110> dislocation enters the ordered γ'-phase?
15. What is rafting?
16. What is a Peach Koehler force? What are the contributions to the overall stress state which determines the Peach Köhler force which acts on a γ-channel dislocation?