TOYOHASHI UNIVERSITY OF TECHNOLOGY

SPring-8

Time-resolved 3D measurements of mechanical behaviours in aluminium alloys

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with the collaborations of D. Leclere[1], L. Qian[1], H. Oogo[1], Z.A.B. Shamsdin[1], Y. Suzuki[2], A. Takeuchi[2], K. Uesugi[2] and M. Kobayashi[1]

1. Toyohashi University of Tech.,Toyohashi, Japan
2. Japan Synchrotron Radiation Research Institute

- Current status of synchrotron microtomography
- Various advanced 3D/4D image analyses
  I. Microstructural tracking for strain mapping
  II. Grain Boundary Tracking (GBT)
  III. Diffraction-Amalgamated GBT (DAGT) technique for crystallographic analysis
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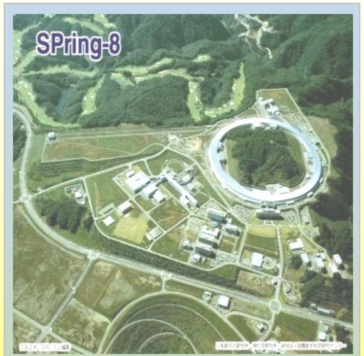
X-ray microtomography (CT): Imaging to appl.

Courtesy of Mr. Taki in Nihon Visual Science, Inc.

Current status of various X-ray CT apparatuses

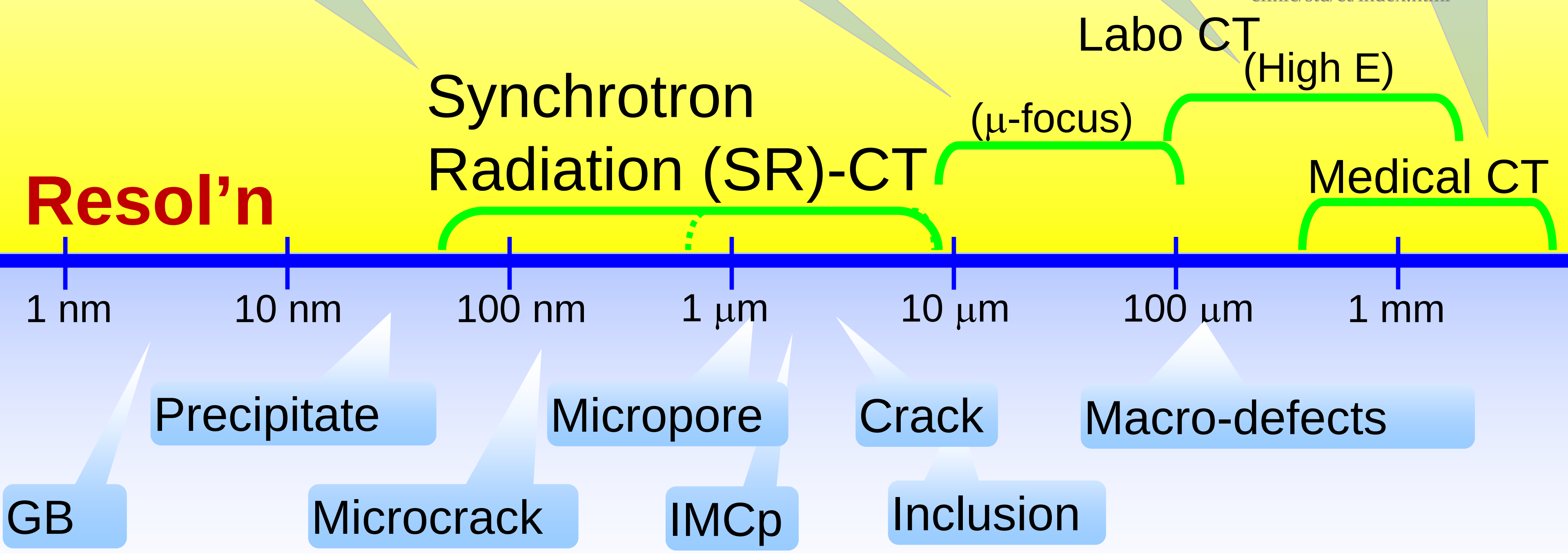


http://www.spring8.or.jp/ja/


http://www.shimadzu.co.jp/ndi/products/x_ryct/x_ryct04.html


http://www.bio-imaging. com/indsystems.asp

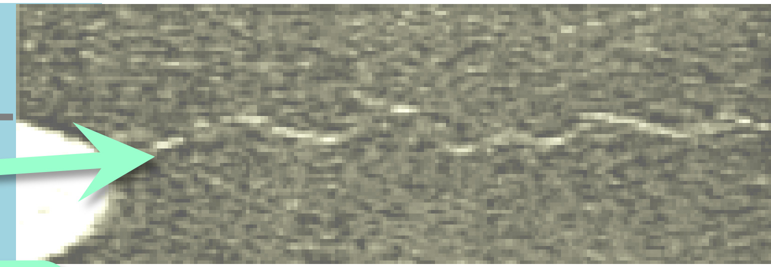

http://www.med.shimadzu.co.jp/clinic/std/ct/index.html



μ-structural features of materials

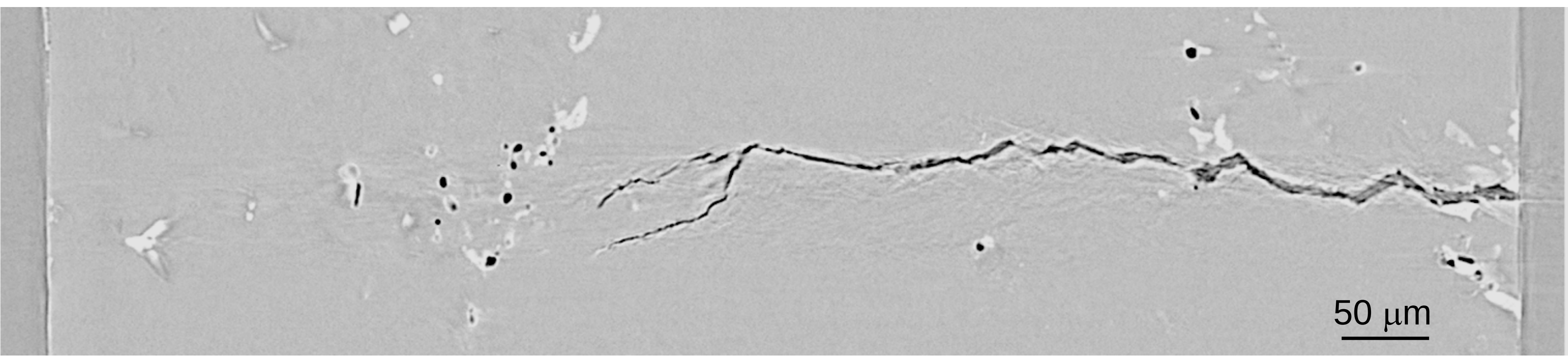
SR-CT application to a cracked metals

	(voxel size in µm³)
1999 (Stock@Cornell HESS)	6.4
2003 (Toda@ESRF)	0.7
2004 (Toda, Uesugi@SPring-8)	0.47
2006 (Toda, Takeuchi@SPring-8)	0.048



Fatigue crack (White)

Stock, et al., Phil. Trans. R. Soc. Lond. A, 357(1999), 2755



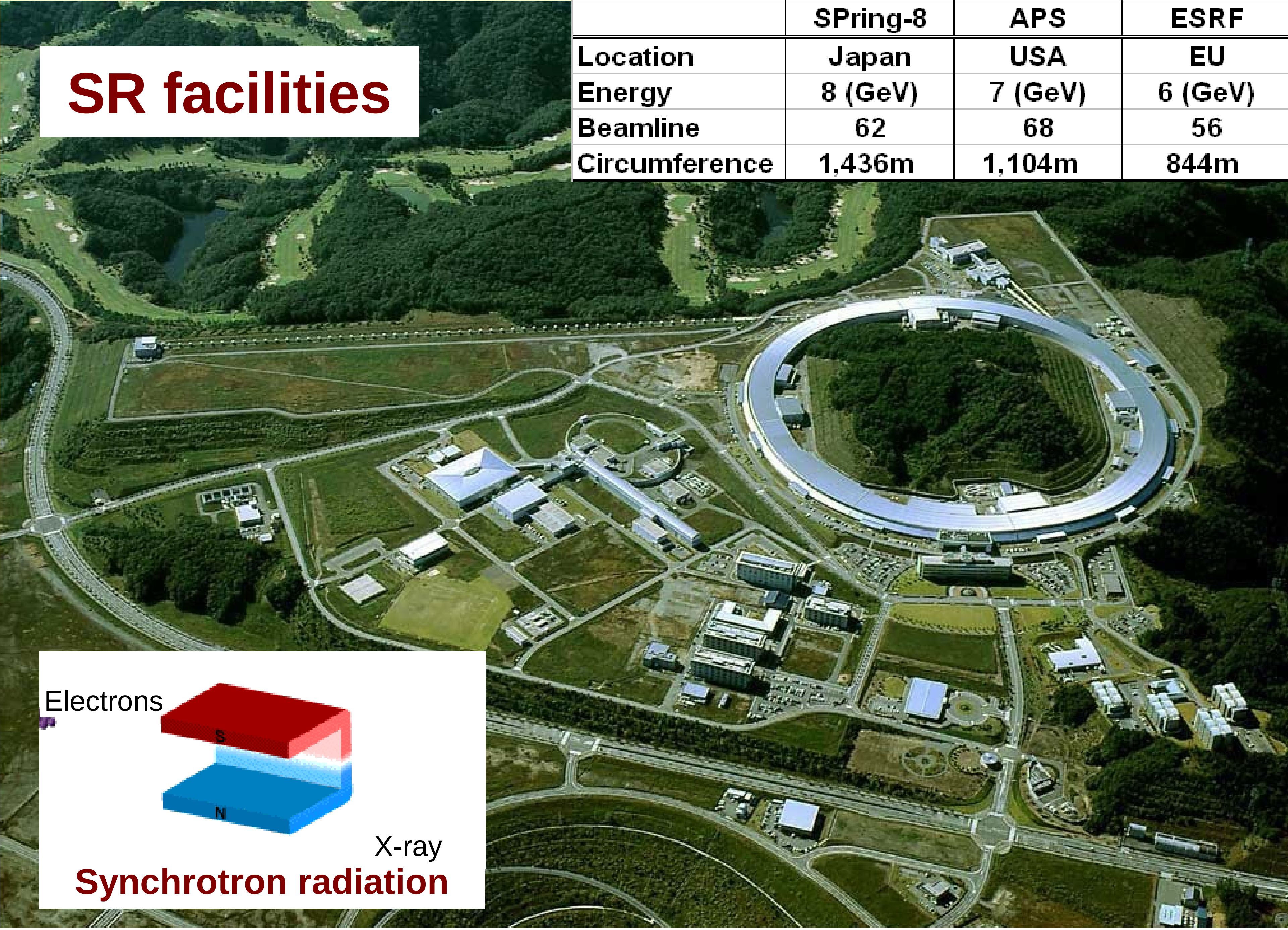
Fatigue crack (Black): Crack opening ~ a few micron

Toda, Sinclair, et al., Phil. Mag. A, 83(2003), 2429.

- Good visualisation of a crack and its opening behaviour
- µ-structural features visible owing to the high resolution

SR facilities

	SPring-8	APS	ESRF
Location	Japan	USA	EU
Energy	8 (GeV)	7 (GeV)	6 (GeV)
Beamline	62	68	56
Circumference	1,436m	1,104m	844m



Synchrotron radiation

http://www.spring8.or.jp/e/

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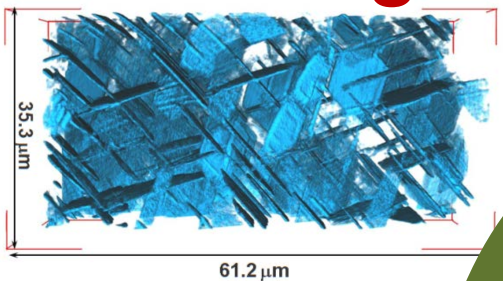
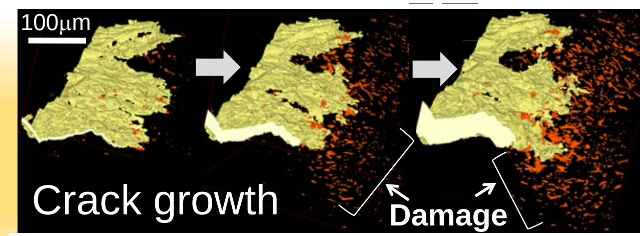
3D/4D image analysis techniques developed

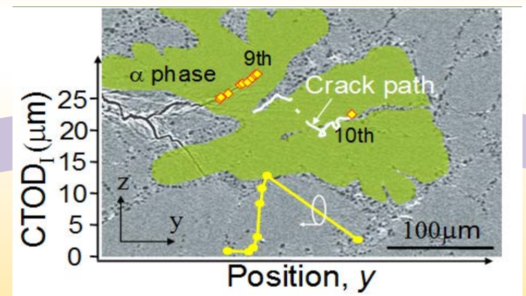
Imaging



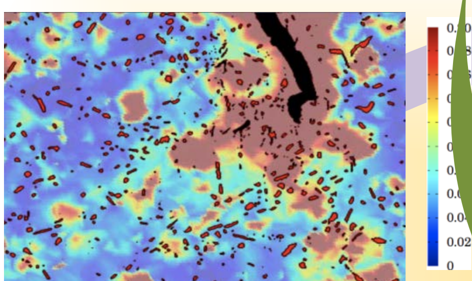
3D imaging



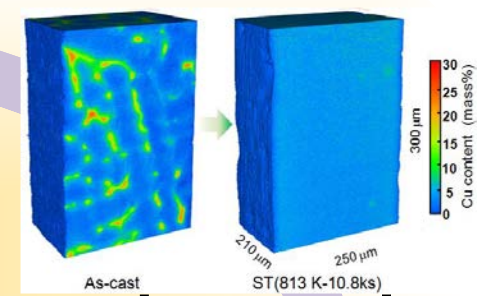
4D imaging



Crack driving force mapping



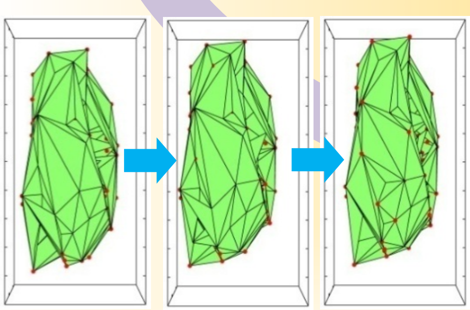
4D ε mapping



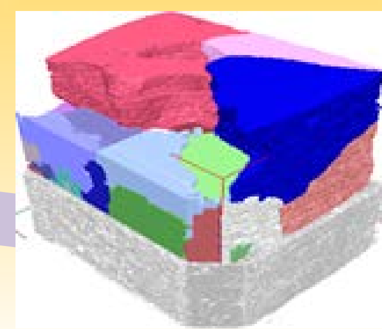
Elemental mapping

Quantification

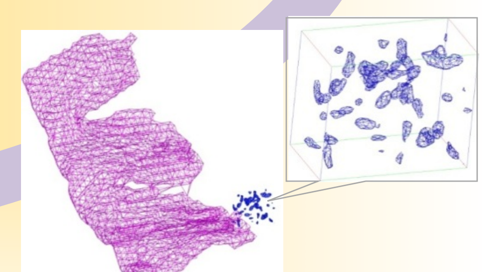
- Some of these hadn't been accessible experimentally



Grain tracking

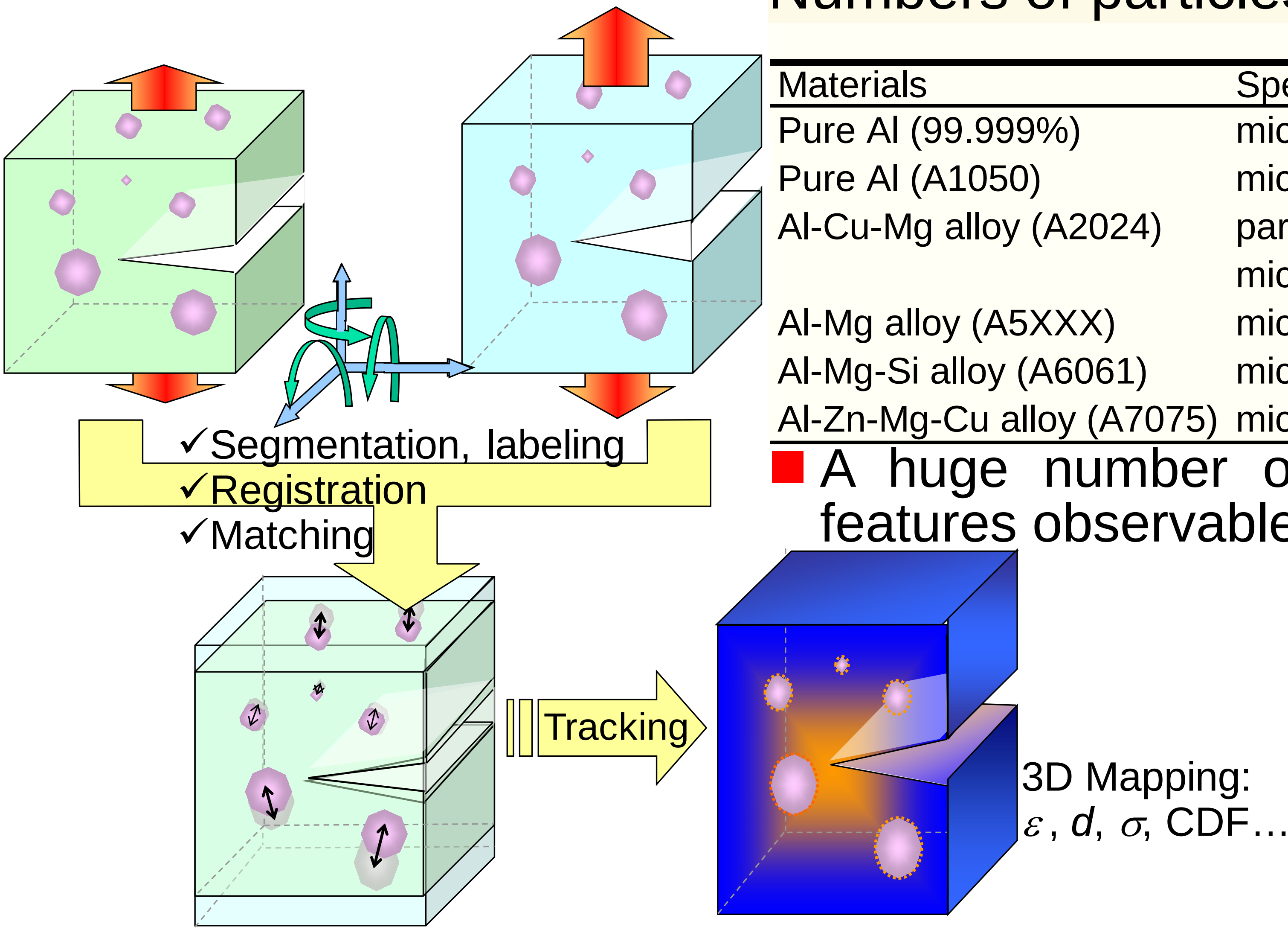


Crystallographic orientation



3D image-based simulation

Microstructural Tracking Technique



Numbers of particles/pores in Al

(1/mm^3)

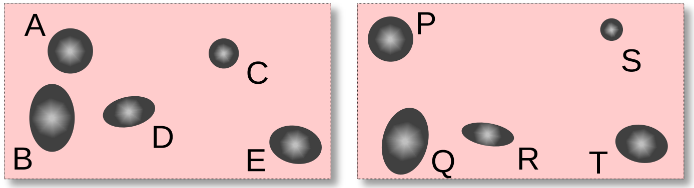
Materials	Species	Number
Pure Al (99.999%)	micro-pores	**3,036**
Pure Al (A1050)	micro-pores	**10,375**
Al-Cu-Mg alloy (A2024)	particles	**150,185**
	micro-pores	**63,111**
Al-Mg alloy (A5XXX)	micro-pores	**20,160**
Al-Mg-Si alloy (A6061)	micro-pores	**21,184**
Al-Zn-Mg-Cu alloy (A7075)	micro-pores	**28,852**

- A huge number of μ-structural features observable

- Physical displacement of each particle/pore tracked
- Unique experimental technique to obtain 3D/4D mapping of mechanical quantities

Kobayashi, Toda, et al., Acta Mater., 56(2008), 2167

Details of microstructural tracking



nth. image (n+1)th. image

Matching Parameter, M_p

$M_p \geq M_p^{th1}$ — No → Rejected markers

Yes ↓

$\Delta M_p \geq M_p^{th2}$ — Yes → Matched markers

No ↓

Pended marker

↓

SPM

Matched → Matched markers; No → Rejected markers

Matched markers

Rejected markers

M_p^{th1}, M_p^{th2}: Threshold value for tracking

Matching parameter, M_p

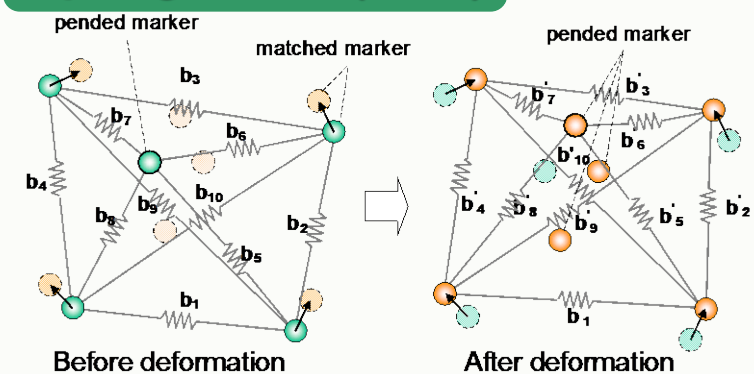
$$M_p = \alpha \times M_L + \beta \times M_V + \gamma \times M_S$$

M_L: Parameter for location, M_V: Parameter for volume

M_S: Parameter for surface area

α, β, γ: Parameter's weight ($\alpha + \beta + \gamma = 1$)

Spring model (SPM)



Total energy of imaginary spring, E_{sp}

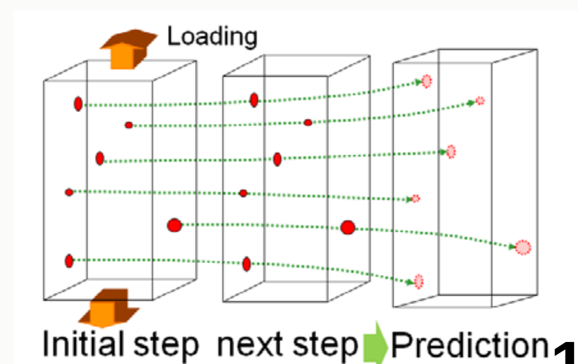
$$E_{sp} = \frac{1}{N_{sp}} \sum_{i=1}^{N_{sp}} \frac{|\mathbf{b}'_i - \mathbf{b}_i|}{|\mathbf{b}_i|}$$

N_{sp}: number of springs

b and b': spring vectors

Trajectory prediction

- Data extrapolation by polynomial approximation
- Physical theories utilized if it is appropriate



Kobayashi, Toda, et al., Acta Mater., 56(2008), 2167

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Another key tech. for GBT: GB visualisation

Immersing TP in liquidus metal

↓

Diffusion into GB.

*Diffusion length: $\bar{x} = (2Dt)^{1/2}$

(T = 323K, t = 500s)

↓

Grain interior, $\bar{x} = 2.51\times10^{-5}$ μm

Grain boundary, $\bar{x}$ = 79.5 μm

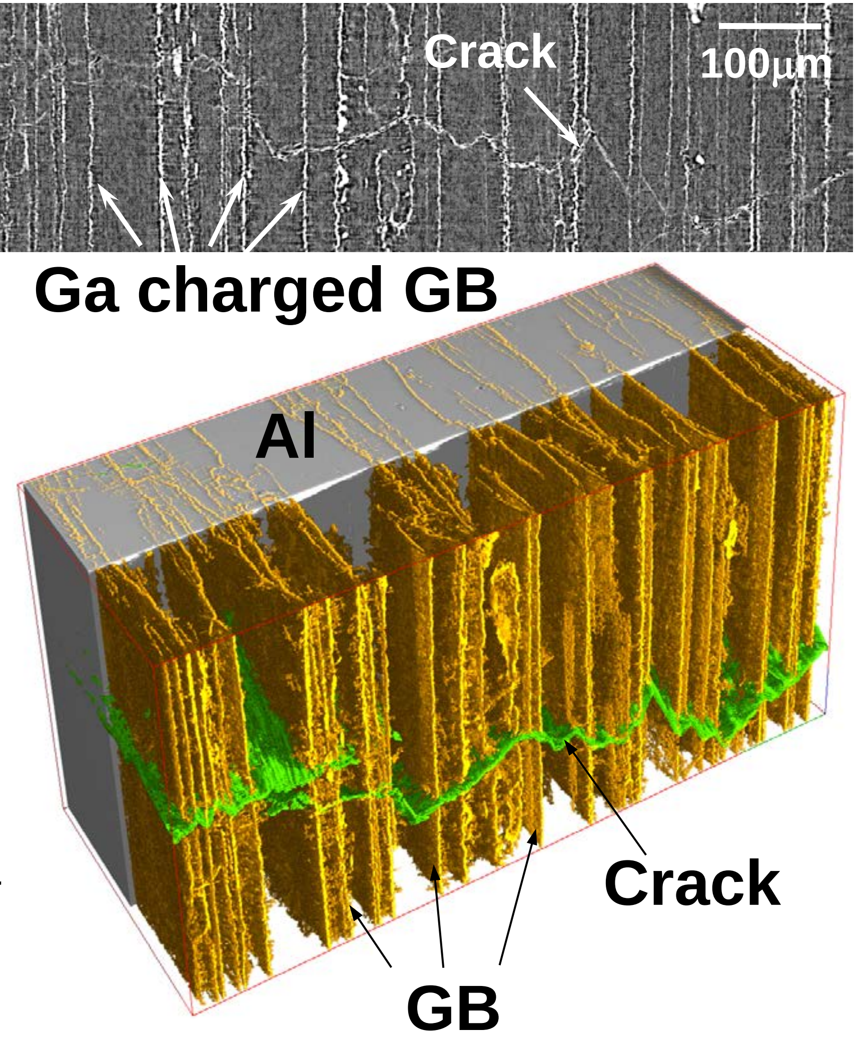
Observation (absorption contrast)

*Transmitted intensity:

$$I = I_0 \exp\{-(m_M t_M + m_{Ga} t_{Ga})\}$$

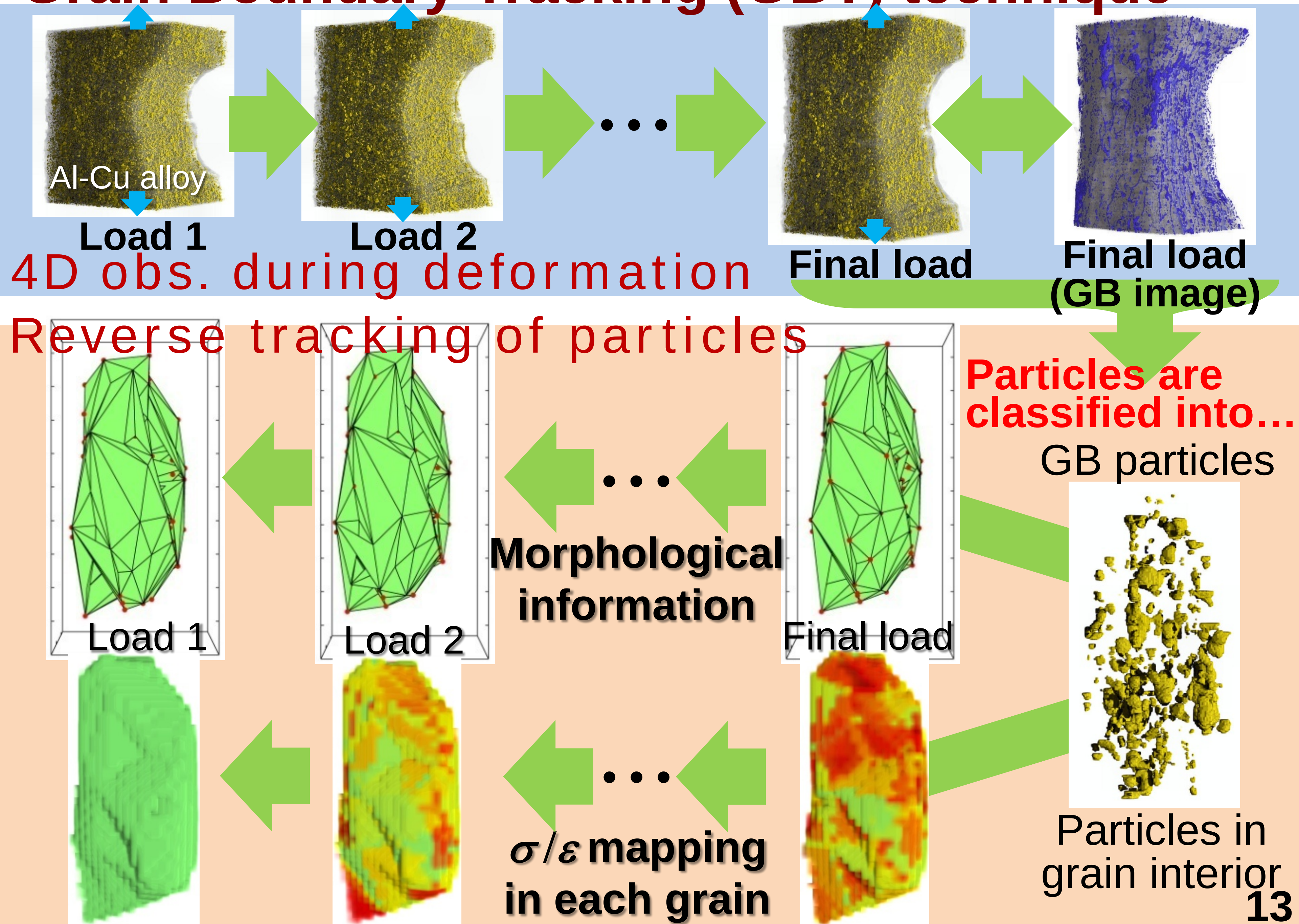
Pairs of metals that cause LME

Material	Embrittling liquids
Al	Hg, Ga, In, Sn, Pb, Cd, Zn, Na
Cu	Hg, In, Ga, Bi, Zn, Li, Sn, Pb
Ti	Hg, Cd, Ag
Mg	Na, K, Rb, Cs, Zn
Fe	Hg, In, Li, Sn, Pb, Cd, Zn, Cu



Khor, et al. J. of Physics: Condensed Matter, vol.16, 2004, S3511-S3515.

Grain Boundary Tracking (GBT) technique



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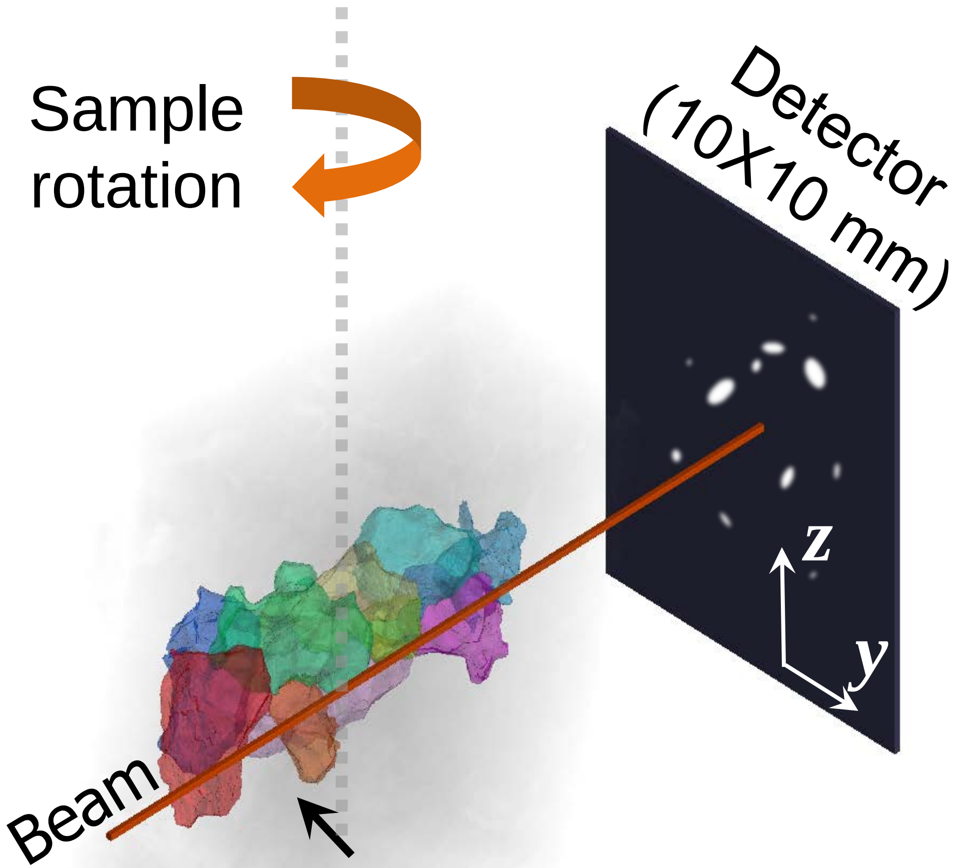
XRD using an X-ray pencil beam for DAGT



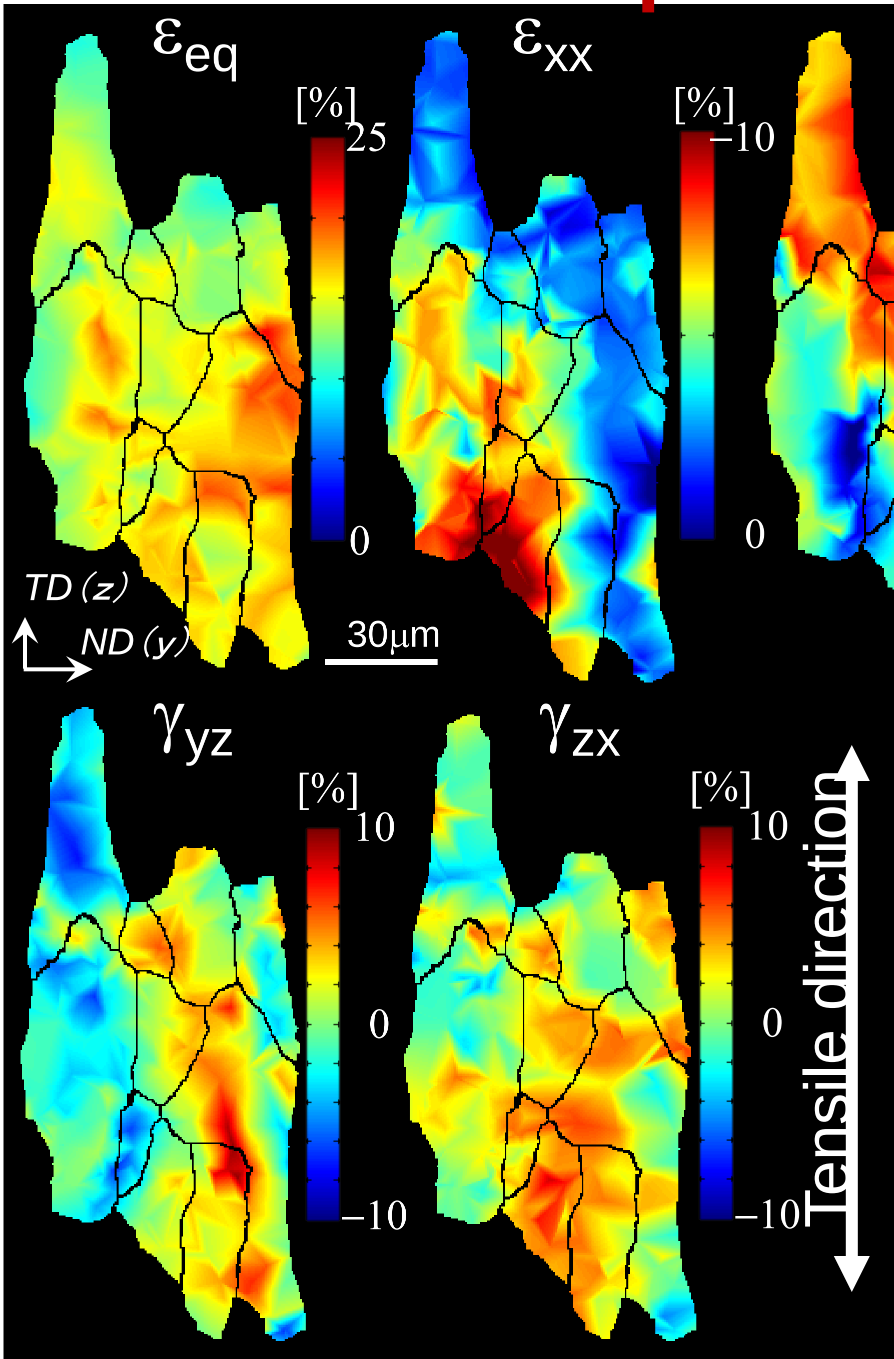
A grain of interest (Red)

Schematic demonstrating the experimental setup and how the beam interacts with an individual grain.

The experiment was carried out at SPring-8 synchrotron facility (Hyogo, Japan) with the following parameter

Beam line: BL20XU
Energy: 35 keV
Camera: 2000x2000 CMOS
Sintillator: Gd_2O_2SiTb
Beam Size: 10 μm x 10 μm
Sample-detector: 13 mm
***z*-step:** 16 μm x 20
***y*-step:** 10 μm x 60
Rotation: 0° -180°
Exposure: 0.1s / 1°

Local strain map on a virtual cross-section



- Strain map at macroscopic strain of 10.6 %
- Large strain variation in every single grain, that is extensively developed across grain boundaries

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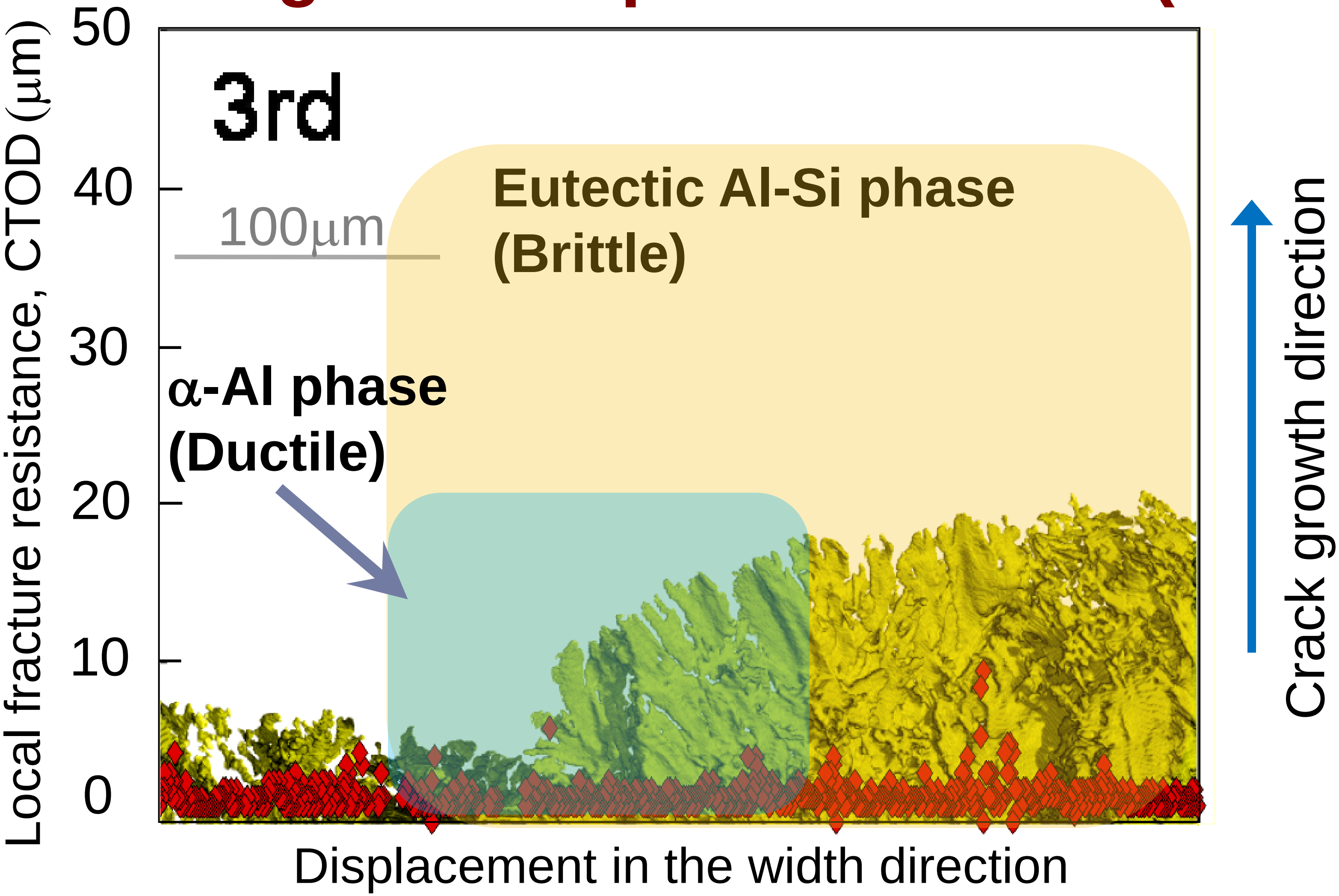
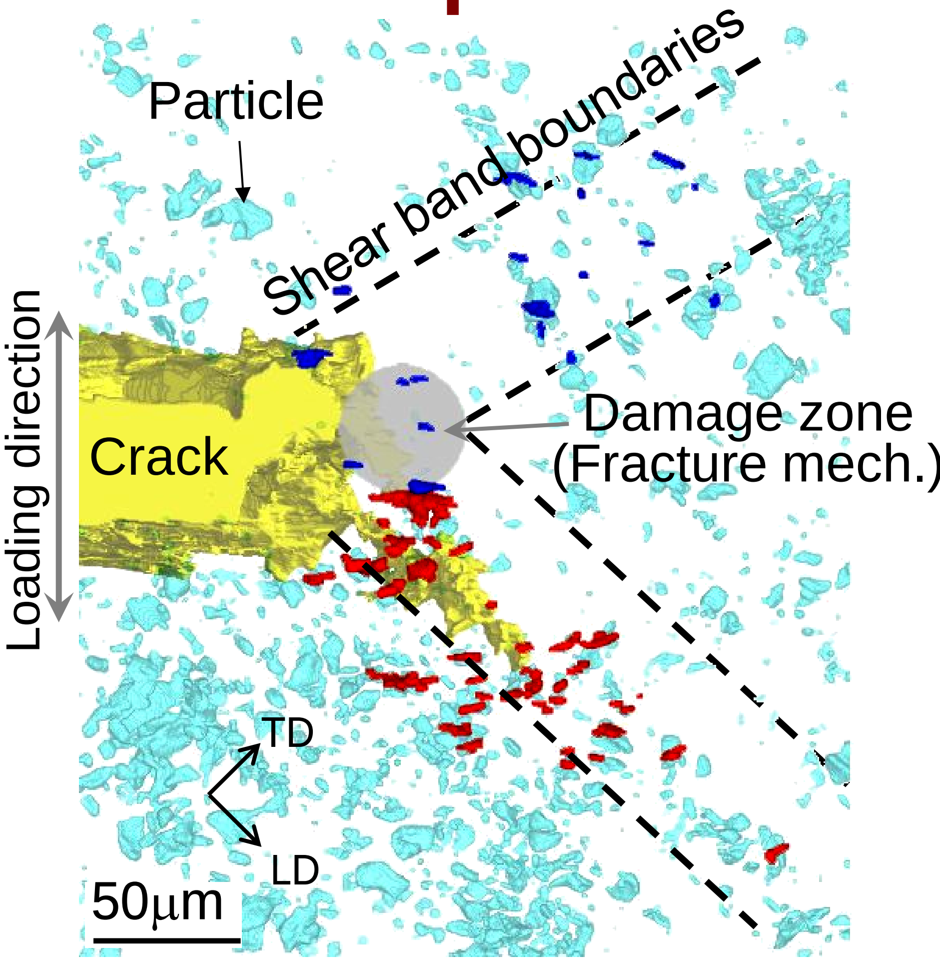
Evaluation of local fracture resistance: Crack growth through a dual-phase material (Al-7Si)

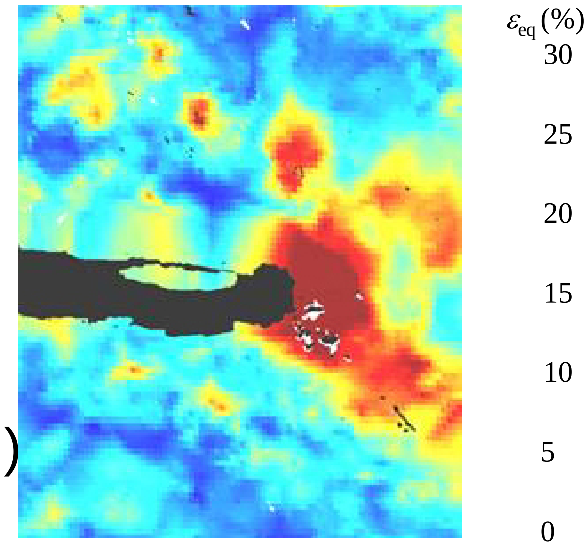


- Premature crack propagation in a brittle eutectic Al-Si phase
- No substantial crack growth in a ductile α-Al phase

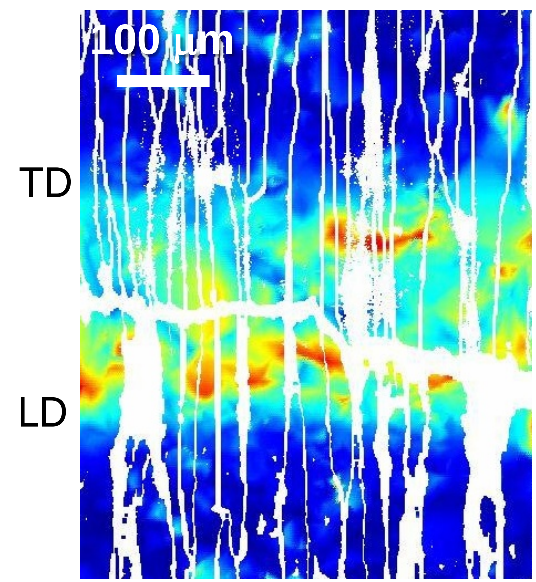
H. Toda, et al., Acta Mater., 56(2008), 6027

Extensive particle damage distribution





Lateral view



Front view
(GB visualized)

- Damage observed extensively ahead of the crack tip far beyond the large strain region assumed in the fracture mechanics. It exhibits shear-band-like localization.
- Effects of underlying texture obvious

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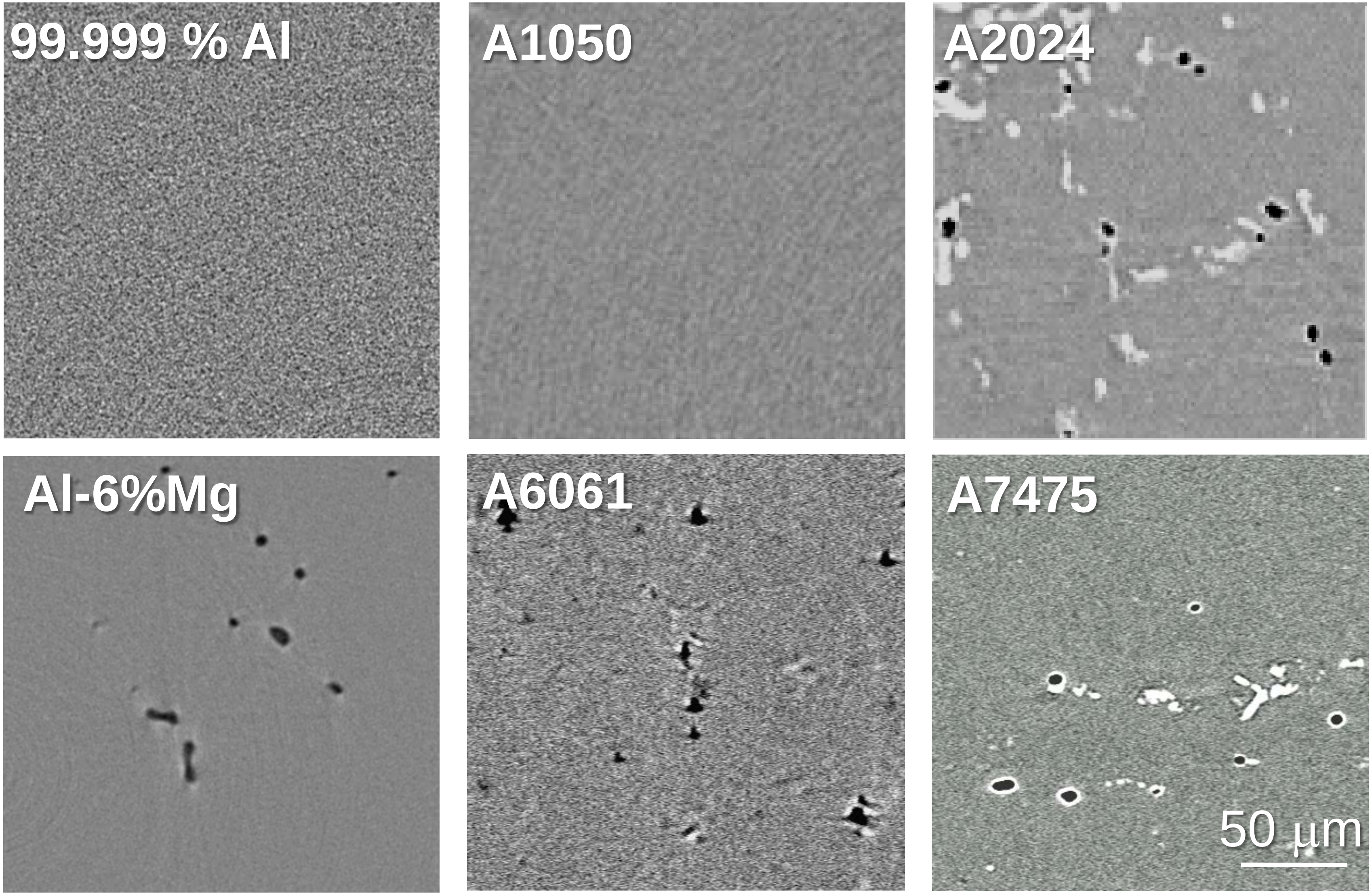
Long-unrecognized pores in various alloys



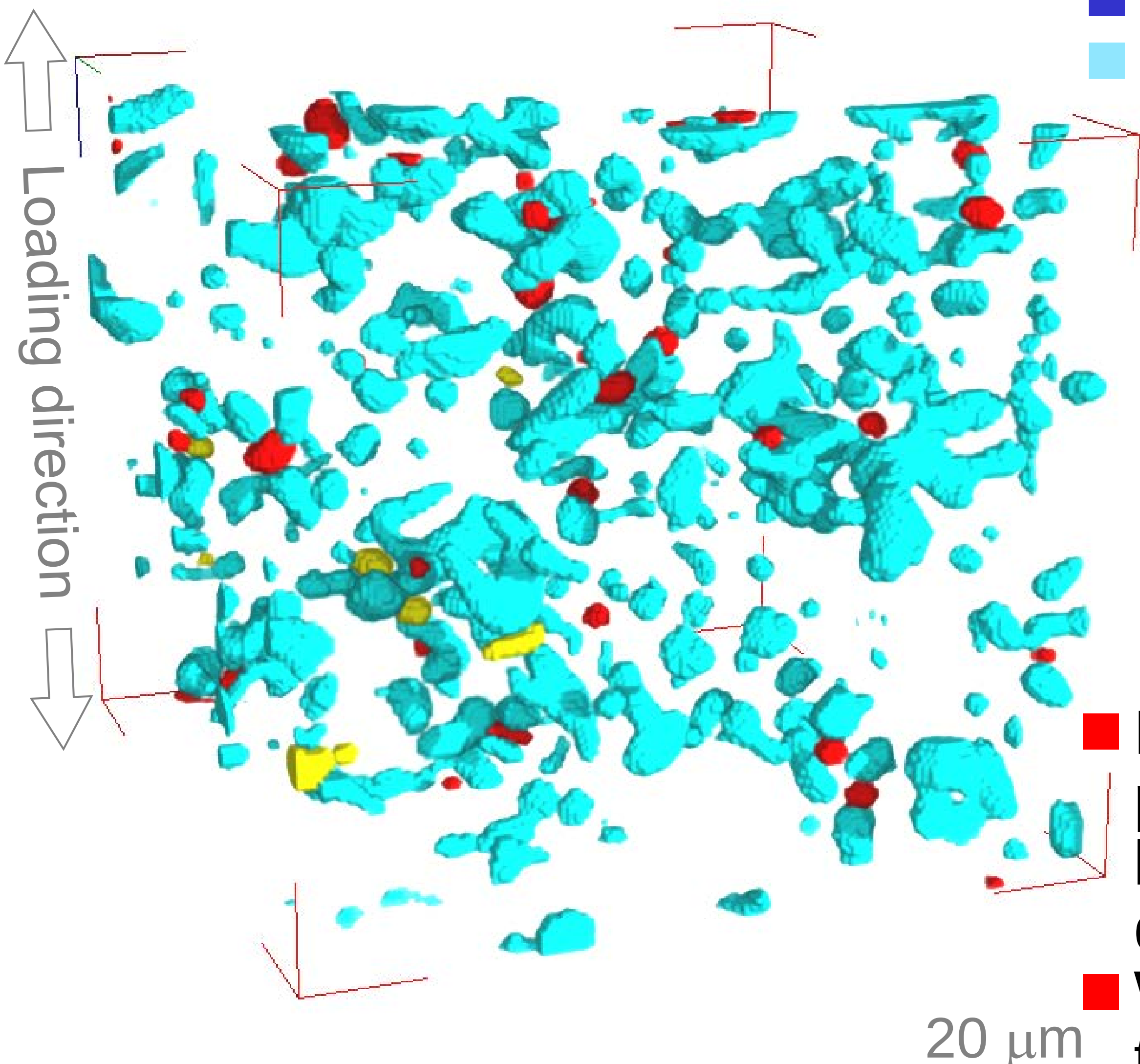
- High-density micro pores observed even in wrought aluminium alloys by employing the high-resolution X-ray tomography

Toda, et al., Acta Mater., 57(2009), 2277

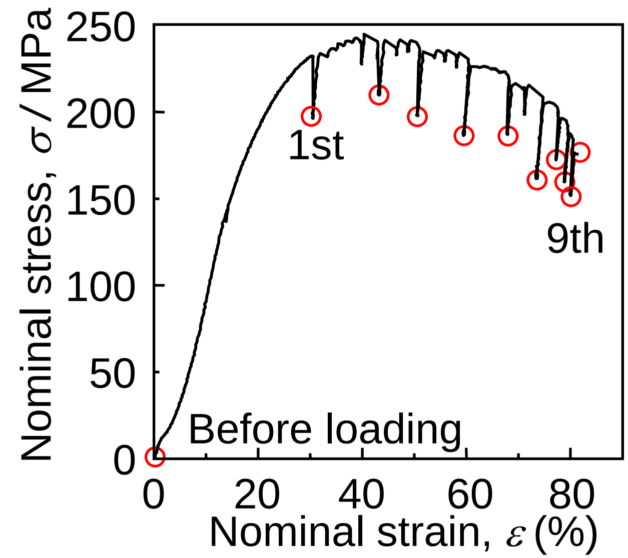
True origin of ductile fracture

Toda, et al., Philos. Mag., (2012), under review

- Pores caused the final fracture
- The other pores
- Void from particle damage
- Particles



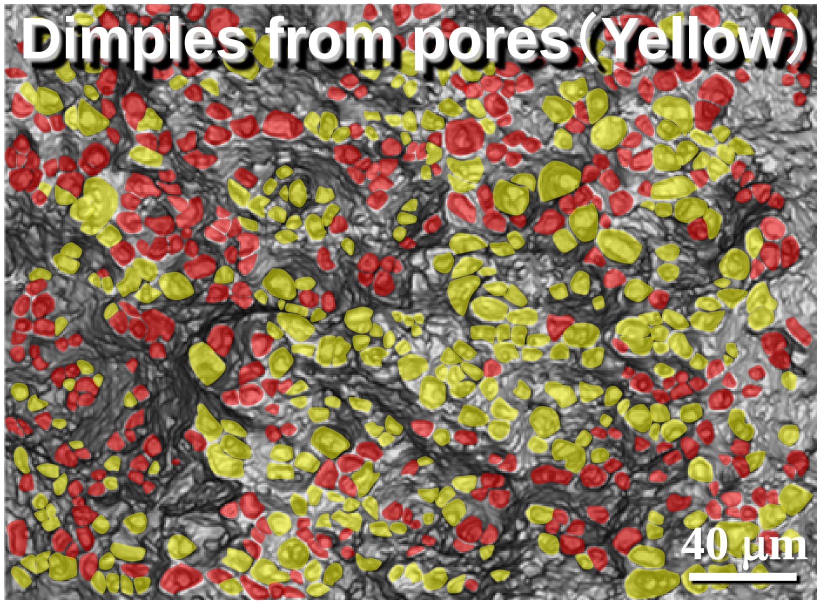
3D fracture behavior of a 2024 Al



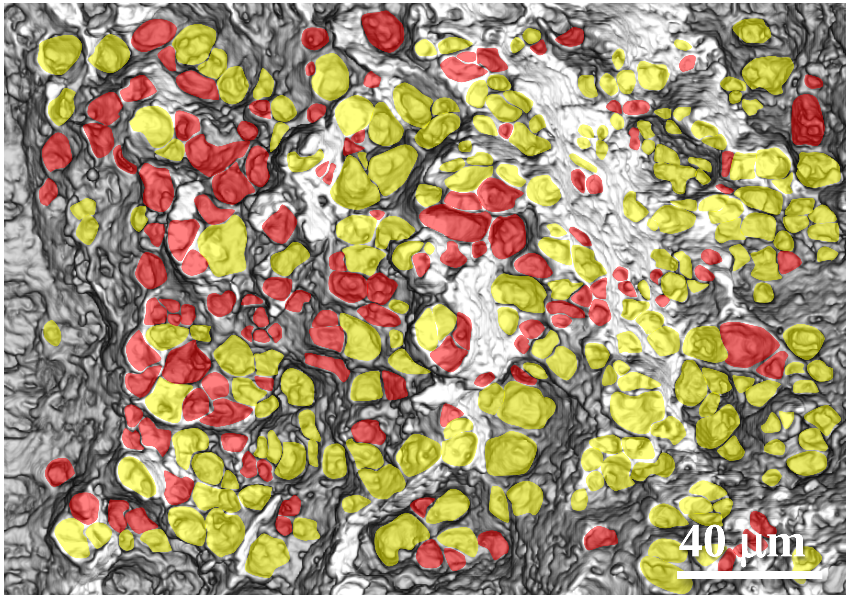
Stress-strain curve

- Pre-existing pores exhibit premature growth under loading, thereby inducing ductile fracture
- Well-known particle fracture mechanism operates only incidentally

Dimple patters originated from H pores



Fracture surface (Unnotched)



Fracture surface (Pre-cracked)

Dimple patterns originated from micro pores in the three TPs

Properties		Unnotched	Notched	Pre crack
Avg. diameter (µm)	Micro pore	3.7	4.6	4.2
	Particle damage	3.3	3.6	3.8
Areal fraction (%)	Micro pore	54.6 <	62.3 <	67.1
	Particle damage	45.4	37.7	32.9

- Dimples originated from H pores occupy more than 50 %
- Fractional area increases with the increase in stress triaxiality

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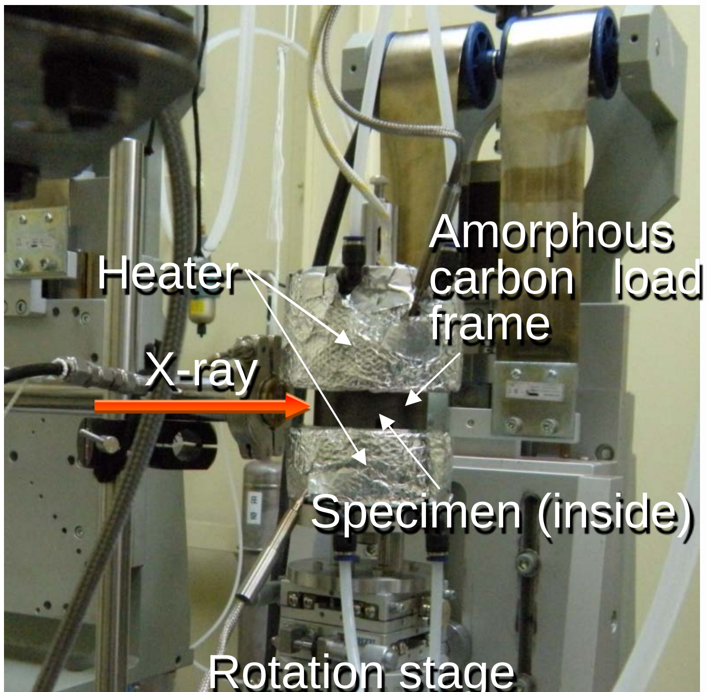
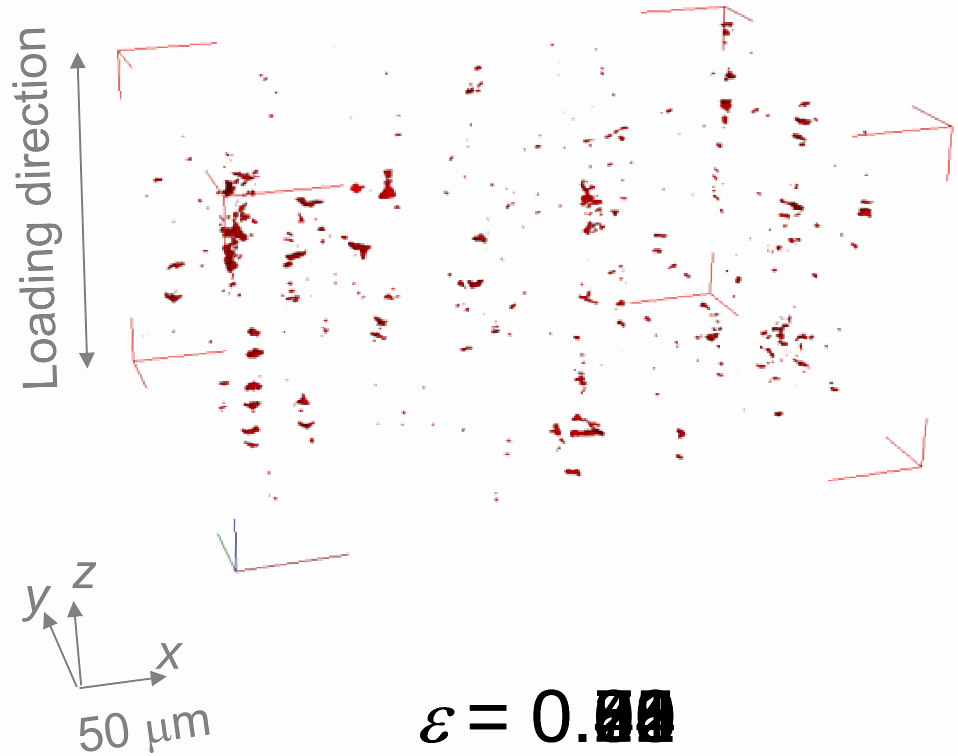
Effects of H pores on cavitation at high T

- In-situ tension of Al-4.5Mg at 773 K at $\dot{\varepsilon}$ of $10^{-2}s^{-1}$
- Trans-/inter-granular transition regime

Toda, et al., Acta Mater., under review, (2012)



High temperature test rig

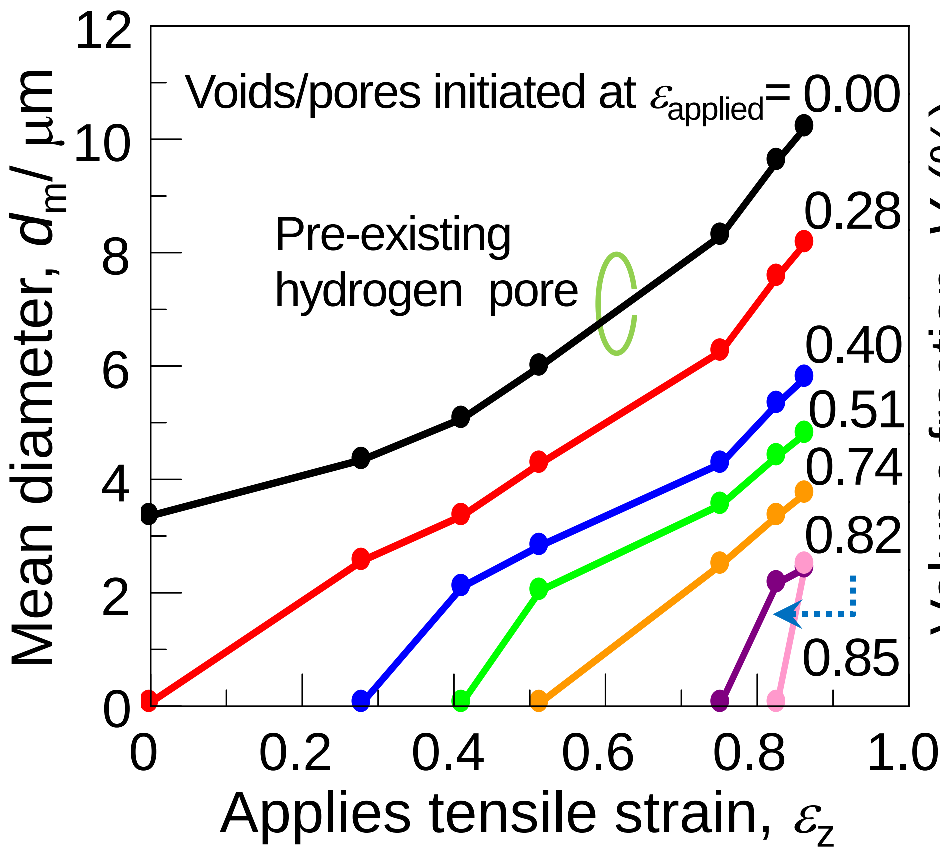


Growth of pores/cavities under tention

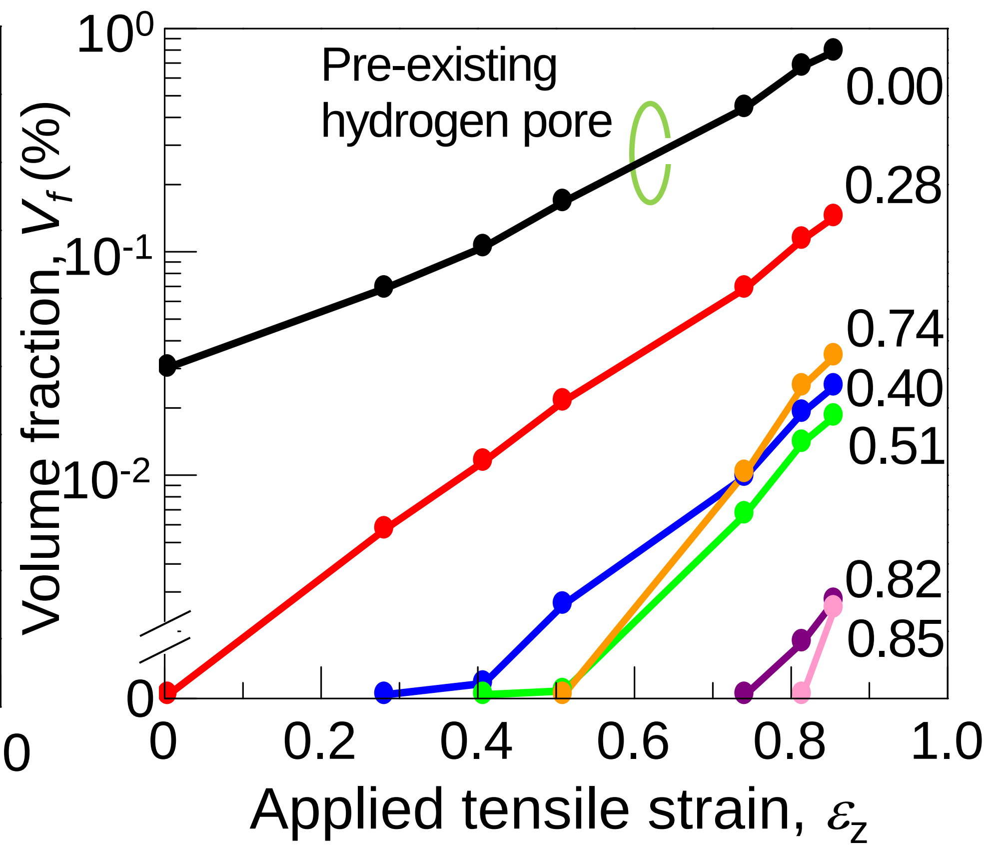
■Origin of the common extensive cavitation during high temperature deformation investigated as well

Growth of H pore / creep cavity

Toda, et al., Acta Mater., under review, (2012)



Change in the diameter of creep cavity initiated at each applied ε



Change in the V_f of creep cavity initiated at each applied ε

- Growth behaviors are similar between pores and creep cavities
- Pre-existing pores and creep cavities initiated early tend to grow bigger: Pre-existing pores finally account for 64 % in V_f

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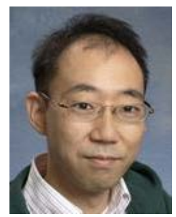
5 yr project "A new concept breakthrough in materials development: Reverse 4D materials engineering"

【Research Group】

Toda / Kobayashi Lab
(Toyohashi Tech)

Maters Sci and Engng



Recruiting !

Comp Solid Mech

Kuwazuru Lab
(Fukui Univ)

Information Tech

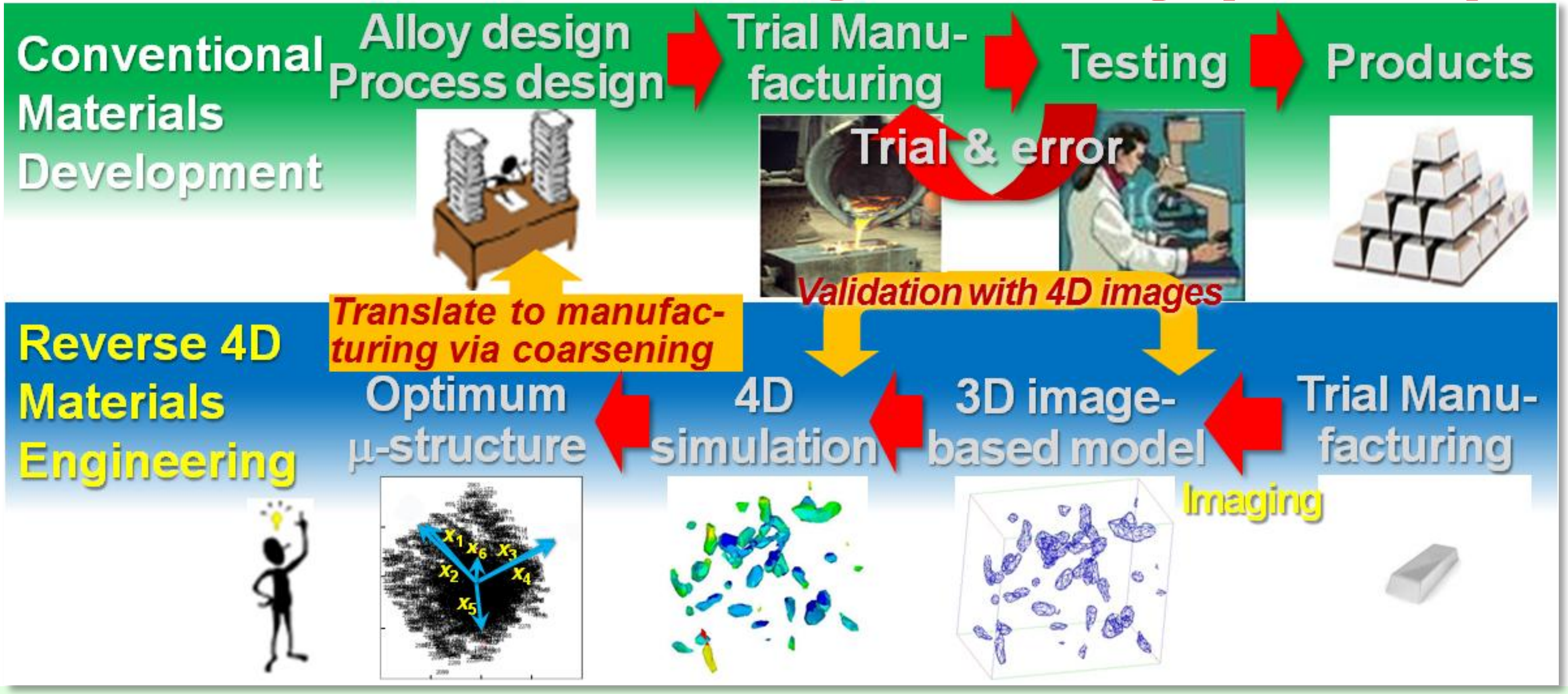
Batres Lab
(Toyohashi Tech)

【Term of Project】 FY2012-2016

【Budget Allocation】 2.1 Million dollars

【Grant】 Grant-in-Aid for Scientific Research (S) from the Japan Society for the Promotion of Science (JSPS)

Reverse 4D Materials Engineering (*R4ME*)



- The conventional materials development process inevitably needs trial and error. Averaged properties evaluated after sampling limited μ-structural features.
- R4ME enables rapid development of high-performance materials in which μ-structures are virtually optimized by means of IBS
- To render R4ME a practical technique, the representation of a given complex 3D μ-structure is 'coarsened' to make it suitable to conventional materials design techniques

Coarsening

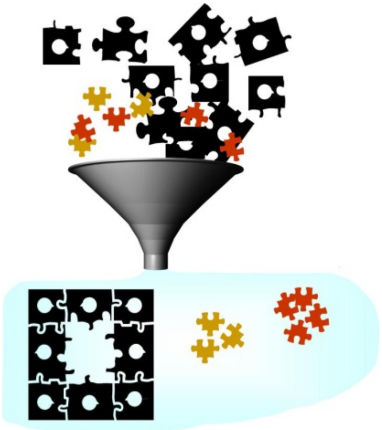
Basic concept

Features to be coarsened:
Particles, GB, crystallographic grains, alloying elements…

Reduction of model dimension

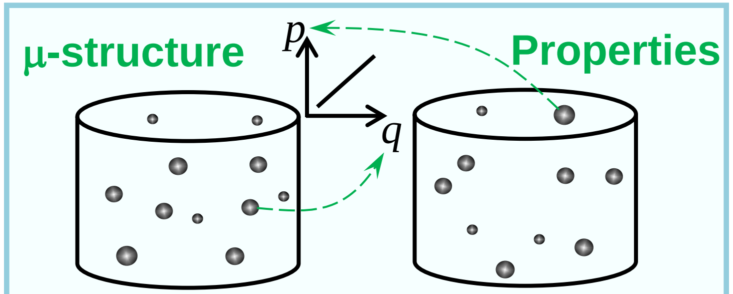
<u>CONSTITUENTS</u>:
Morphology, distribution, crystallog-raphic info., concentration…

<u>STEPPER</u>:
Velocity, changing rate, probabilistic parameter…



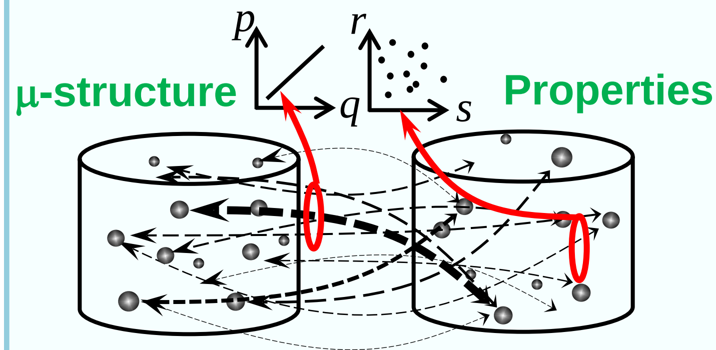
Procedures

Data mining: Statistical anal. (Multiple regression anal., response surface), NN model, RBF network…



Cvntl. understanding

*Evaluation with arbitrarily chosen limited correlations



Coarsening

*Comprehensive, panoramic view of all mater. behaviors

- Coarsening process thoroughly filters out a staggering amount of μ-structural information in a given complex 3D μ-structure
- Can also be answer to "Information explosion" in 4D imaging

Summary

- Recent ***3D/4D imaging*** provides direct observation of complex and dynamic phenomena. Physical displacements of μ-structural features have been used to obtain ***local ε, crack driving forces and crystallographic information in 3D/4D***. Especially DAGT (Diffraction-Amalgamated Grain-boundary Tracking) technique provides a description of the crystallographic orientations in polycrystalline materials from an XRD analysis in addition to the morphological and mechanical information obtained from the 3D imaging.
- ‘***Reverse 4D Materials Engineering***’ (R4ME), has been proposed through the utilization of the advanced imaging and quantification techniques. It optimizes μ-structures by means of an accurate image-based simulation in which multi-scale 3D structures of existing materials are accurately reproduced.

Acknowledgement: Grant-in-Aids for Science Research (S) (FY 2012～2016) for R4ME and (A) (FY 2008～2012) for DAGT from the Japan Society for the Promotion of Science (JSPS)