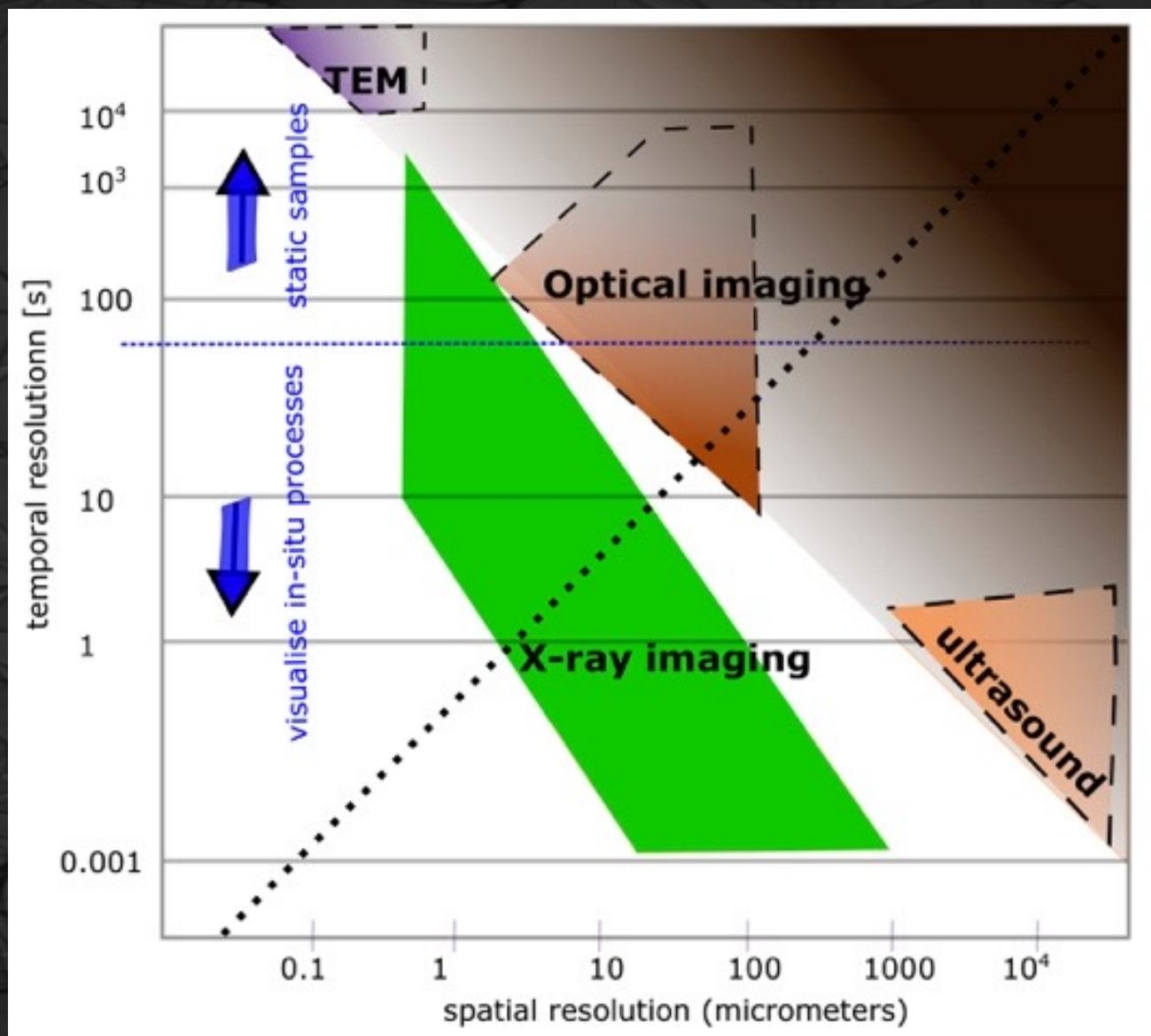


# Examples of advanced X-ray characterization of (mainly) alloys?

**Rajmund Mokso**  
**DTU Physics, Denmark & MAX IV Laboratory in Lund, Sweden**

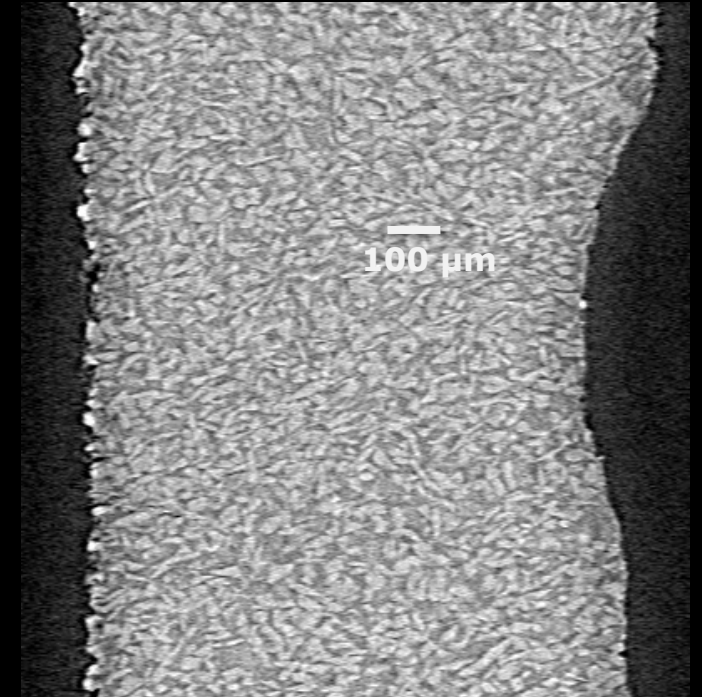
# Imaging methods with 3D capabilities



# Fast tomography: in-situ visualization of fracture

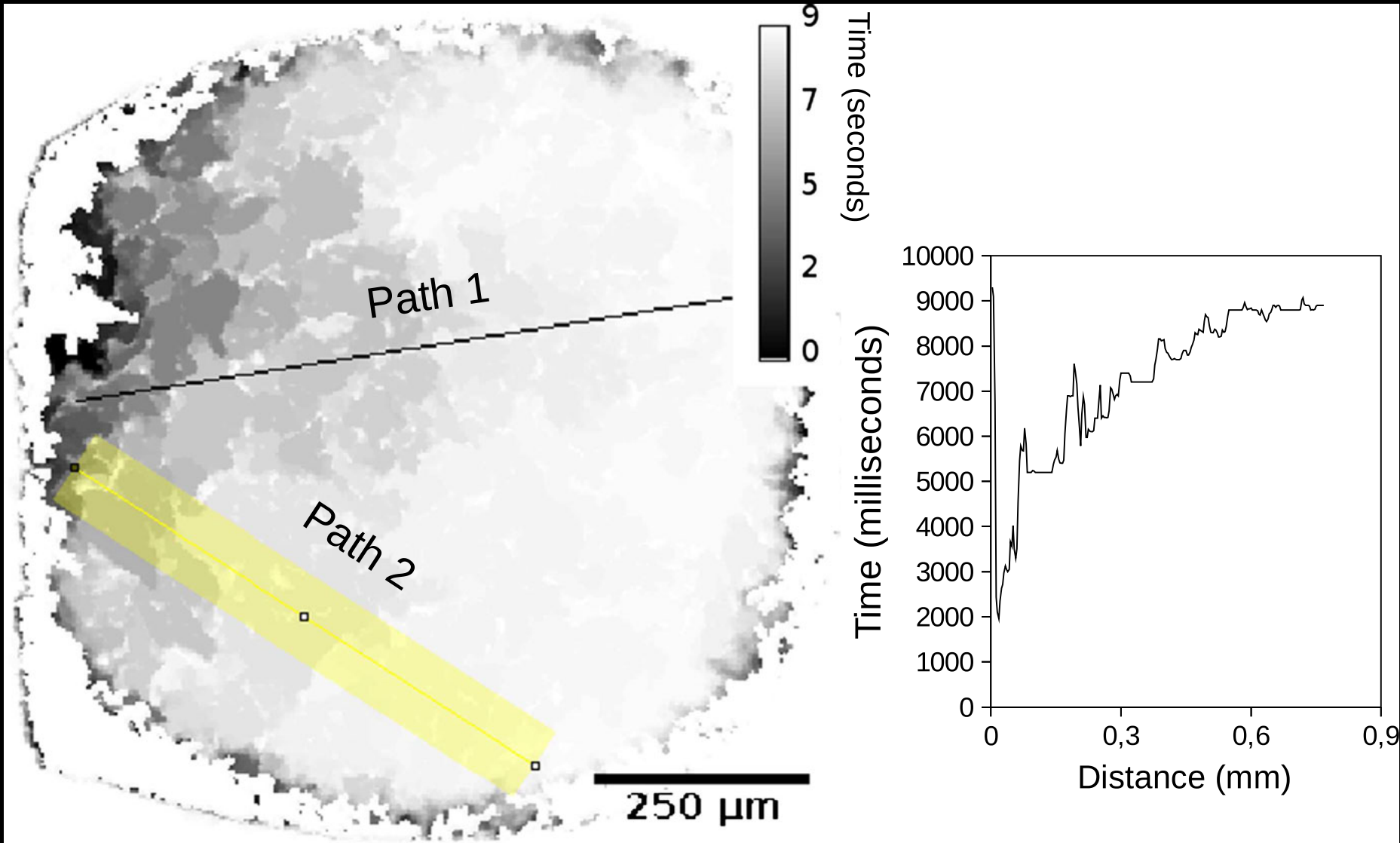


Al - Alumina sample (Eric Maire, Joel Lachambre, INSA Lyon, France)



Vertical tomographic slice,  
time separation  $\sim 50$  ms,  
voxel =  $3\mu\text{m}$

# Fatigue cracking in Alumina sample



Maire et al., Int J Fracture 2016

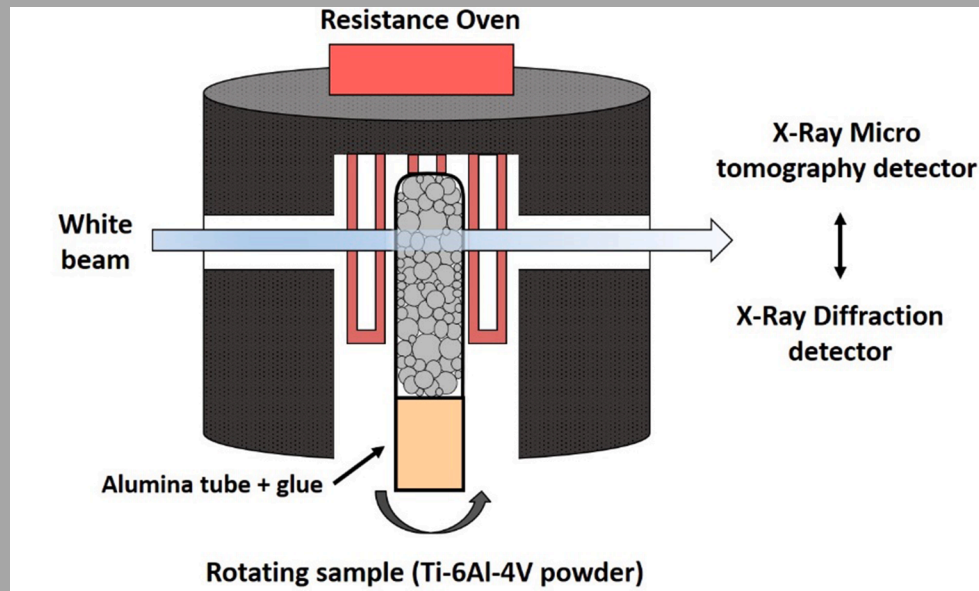
Full length article

*In situ* synchrotron study of sintering of gas-atomized Ti-6Al-4 V powder using concomitant micro-tomography and X-ray diffraction: Effect of particle size and interstitials on densification and phase transformation kinetics

M. Pontoreau<sup>a</sup>, M. Coffigniez<sup>a</sup>, V. Trillaud<sup>a</sup>, C. Le Bourlot<sup>a</sup>, J. Lachambre<sup>a</sup>, L. Gremillard<sup>a</sup>, M. Perez<sup>a</sup>, E. Maire<sup>a</sup>, J. Adrien<sup>a</sup>, P. Steyer<sup>a</sup>, T. Douillard<sup>a</sup>, A. King<sup>b</sup>, X. Boulnat<sup>a,\*</sup>

<sup>a</sup> INSA Lyon, University of Lyon, MATEIS, UMR CNRS 5510, F69621 Villeurbanne, France

<sup>b</sup> Synchrotron Soleil, Psiché Beamline, F-91190 Gif-sur-Yvette, France



Ti-6Al-4 V gas-atomized powders

# Ti-6Al-4 Sintering

*In-situ tomographic microscopy*

*Voxel size = 650 nm*

*X-ray energy (pink) ~ 30-80 keV*

*Acquisition time ~ 6 s*

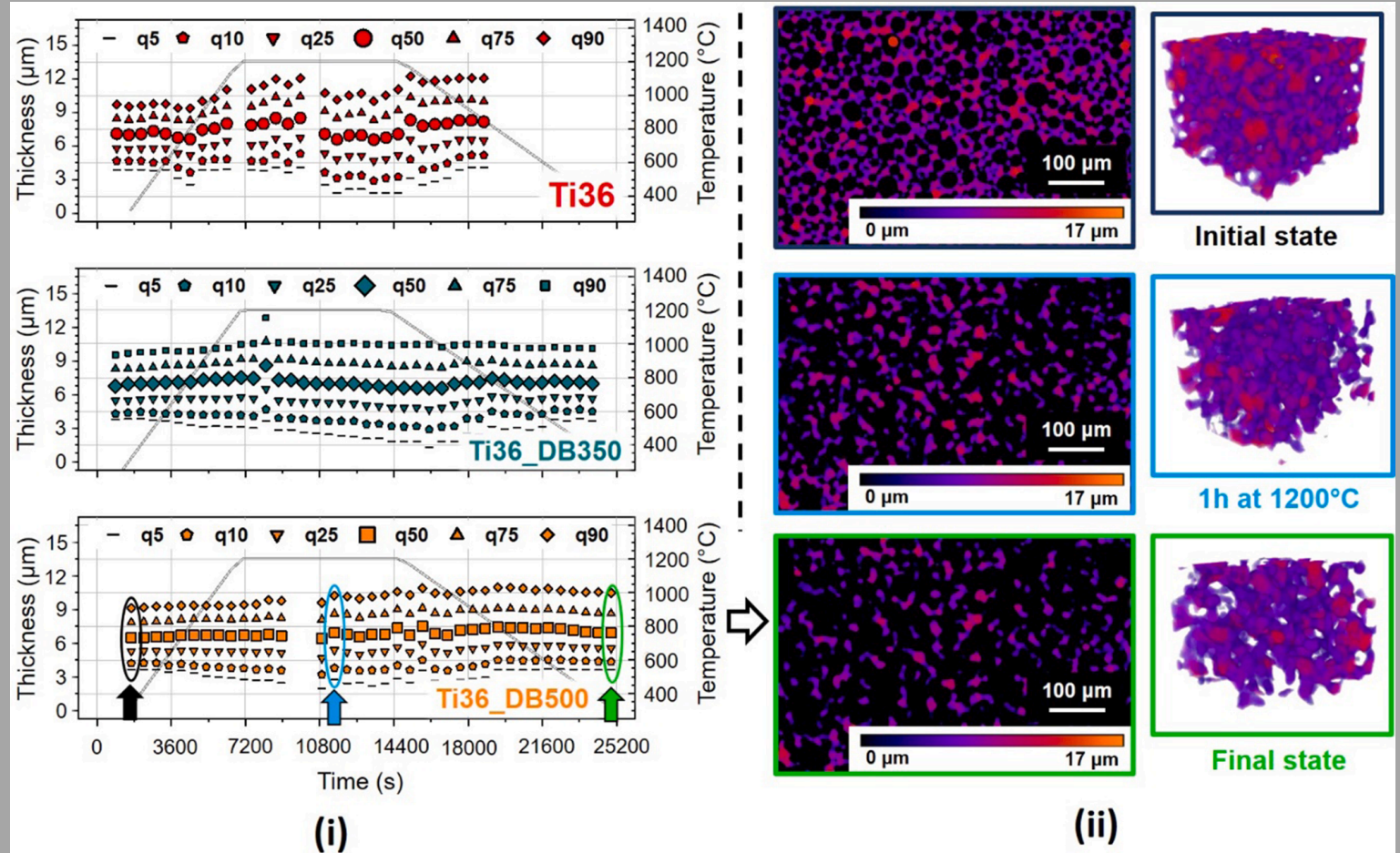
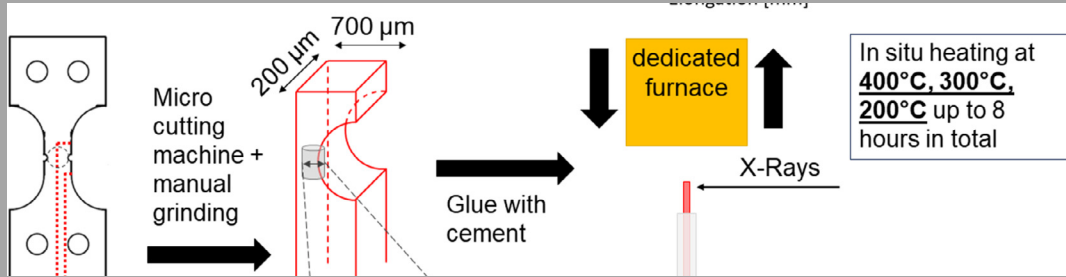


Fig. 4. Interconnected pore evolution for all samples with (i) the evolution of the thickness of their branches and (ii) 2D and 3D ( $135 \times 108 \times 133 \mu\text{m}^3$ ) representation of local thickness plugin used on sample Ti36\_DB500 (one slice and less than 1 vol% of total 3D volume).

During sintering, the thickness of the interconnected pore is globally stable despite the shrinkage of the interconnected pore network => suggests that the decrease in volume of the interconnected pores network only happens by the closure of the branches composing the interconnected pore rather than an isotropic shrinkage.



A new precipitation based healing strategy for metals demonstrated on the commercial Al 6063 alloy.



Full length article

### A new healing strategy for metals: Programmed damage and repair

Mariia Arsenko<sup>a</sup>, Florent Hannard<sup>a</sup>, Lipeng Ding<sup>a,b,c</sup>, Lv Zhao<sup>a,d</sup>, Eric Maire<sup>e</sup>, Julie Villanova<sup>f</sup>, Hosni Idrissi<sup>a,b</sup>, Aude Simar<sup>a,\*</sup>

<sup>a</sup> Institute of Mechanics, Materials and Civil Engineering, UCLouvain, Louvain-la-Neuve 1348, Belgium

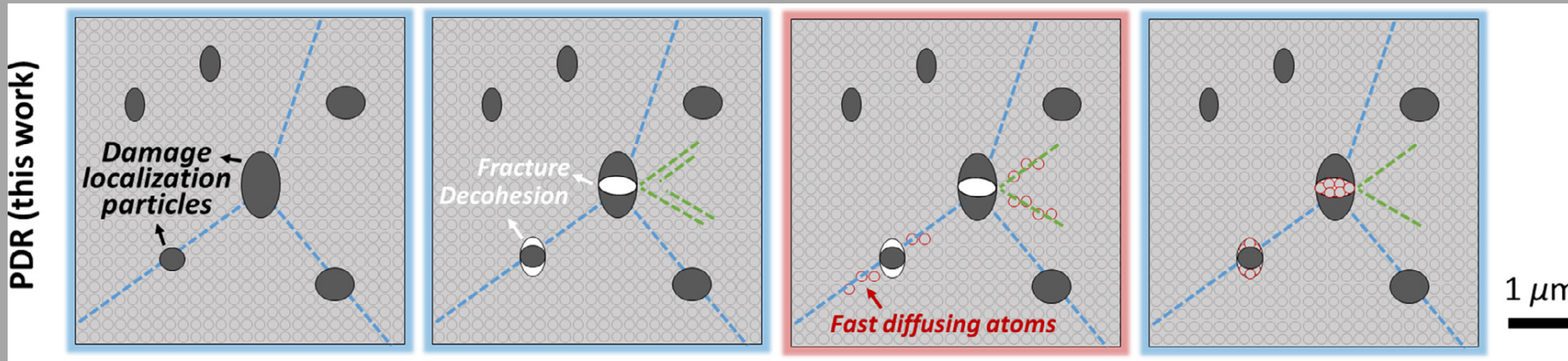
<sup>b</sup> Electron Microscopy for Materials Science, University of Antwerp, Antwerp 2000, Belgium

<sup>c</sup> Key laboratory for Light-Weight Materials, Nanjing Tech University, Nanjing 211816, China

<sup>d</sup> Department of Mechanics, School of Aerospace Engineering, Huazhong University of Science and Technology, Wuhan 430074, China

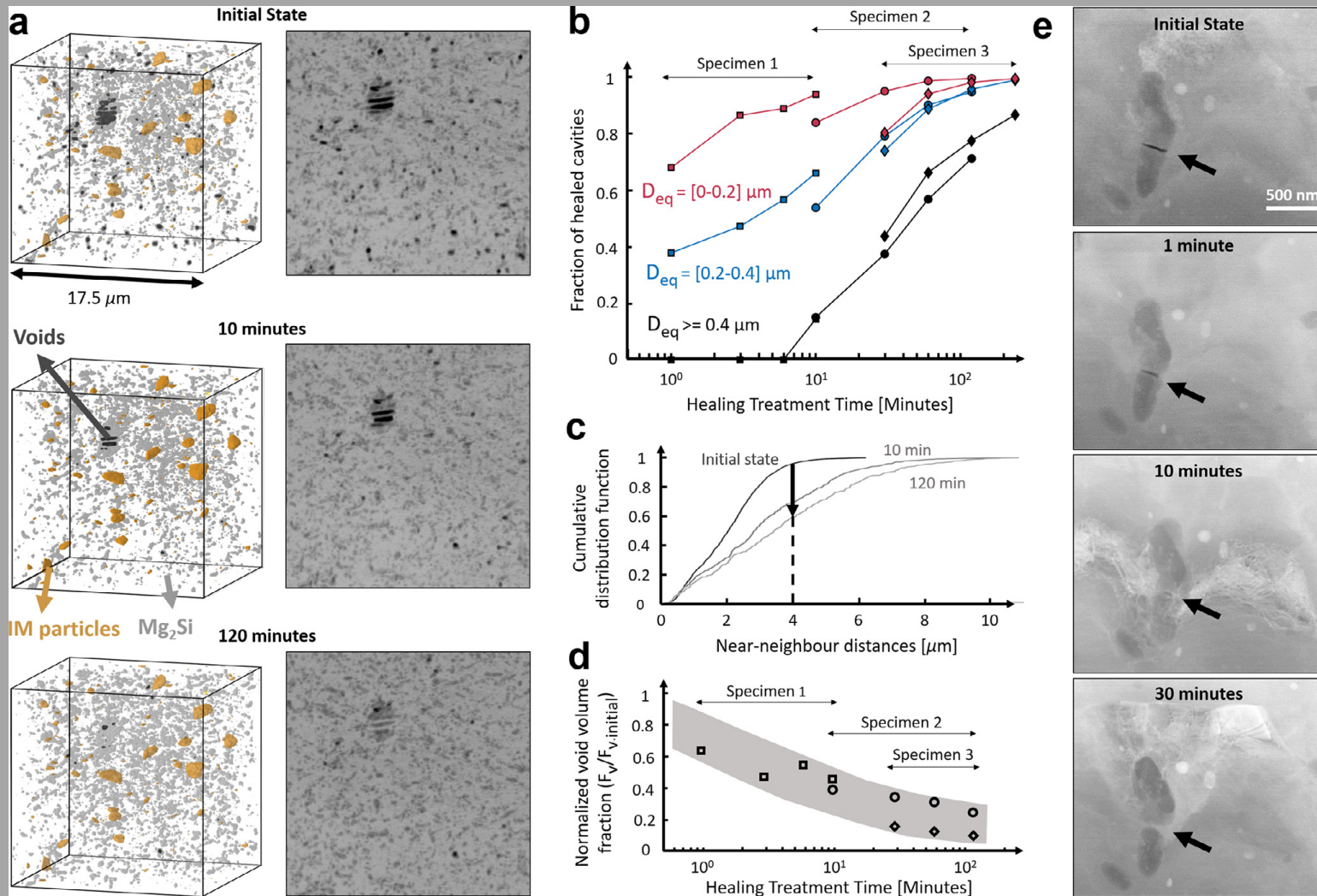
<sup>e</sup> Mateis, INSA Lyon, Université de Lyon, Villeurbanne F-69621, France

<sup>f</sup> ESRF - The European Synchrotron, Grenoble 38043, France



Programmed Damage and Repair

# Healing in metals



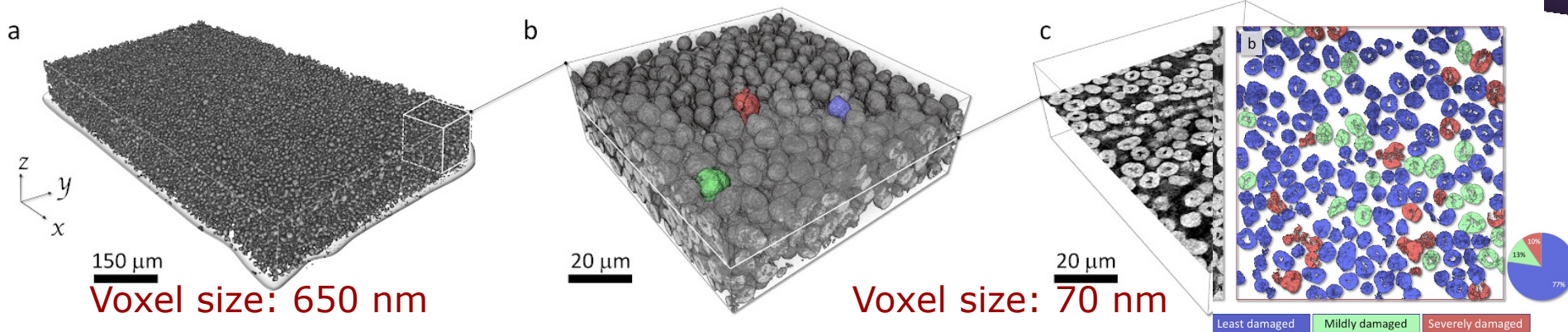
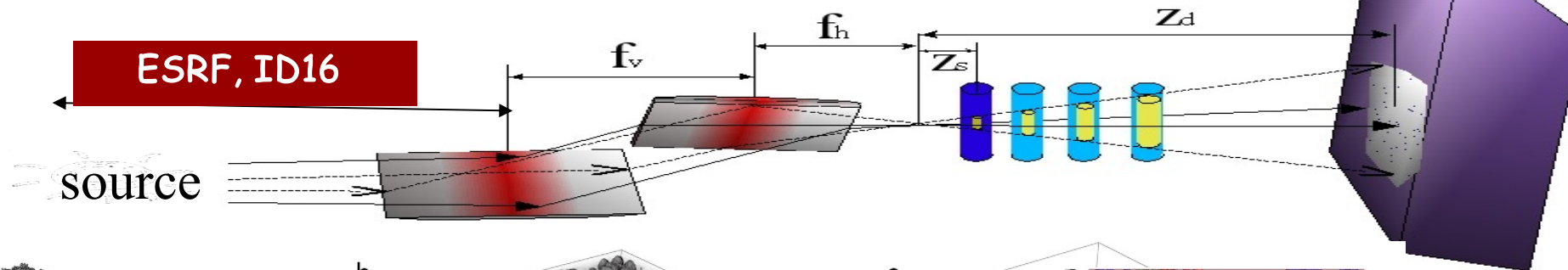
*In-situ holotomography with*  
*Voxel size = 35 nm*  
*X-ray energy = 17.5 keV*  
*Acquisition time ~ 20 min*

*Fully exploited coherence properties of the new machines.*

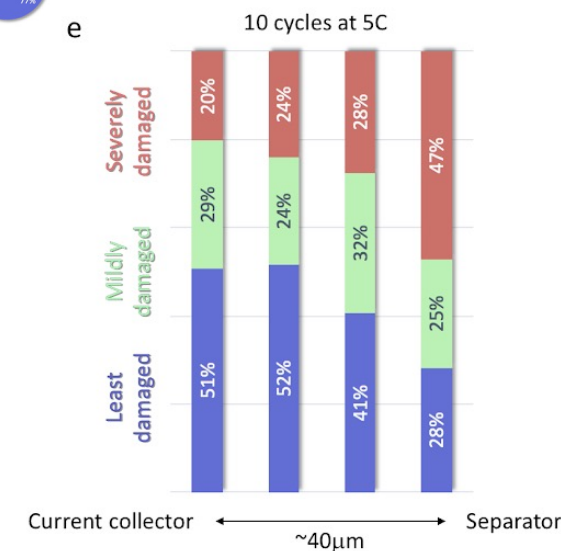
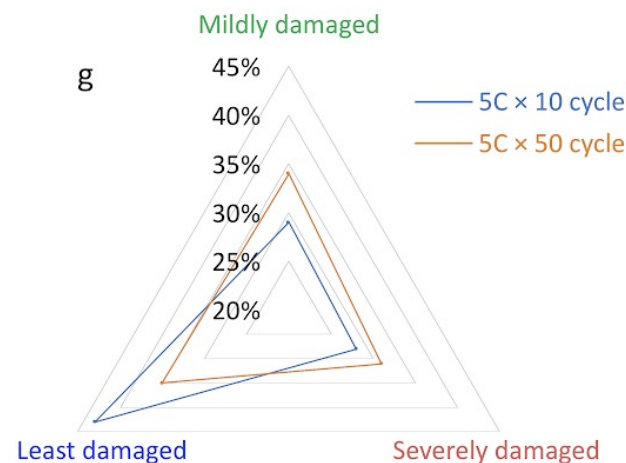
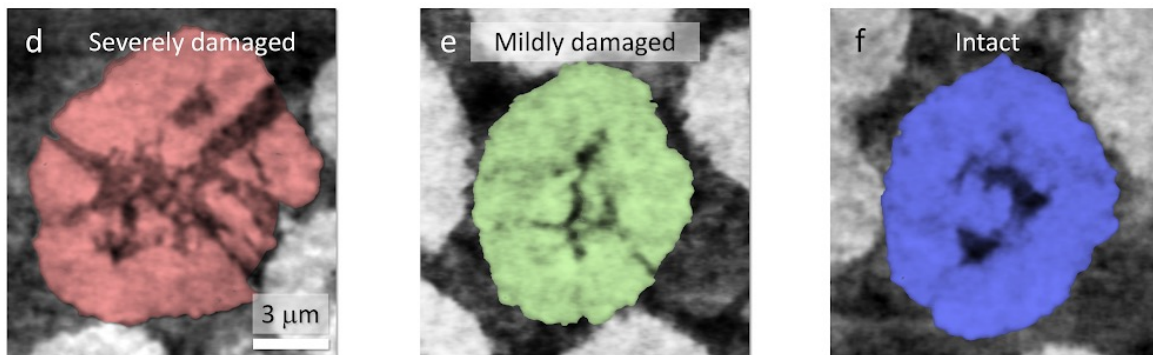
**Fig. 6.** Healing evolution with time at 400°C. (a) 3D volumes and corresponding Minimum Intensity Projections (MinIP) in the initial state, after 10 min and after 2 h at 400 °C; (voids are in black, intermetallic particles in yellow and  $\text{Mg}_2\text{Si}$  particles in grey). (b) Evolution of the number of healed cavities with healing time for different size classes: Specimens 1 and 2 correspond to 0.6 mm of global elongation; Specimen 3 corresponds to 0.7 mm of global elongation (see Supplementary material 2 for details on each specimen). (c) Cumulative distribution function (CDF) of the near-neighbour distances between voids as a function of the heating time. (d) Evolution of the void volume fraction ( $F_v$ ), normalised by the value in the initial state ( $F_{v,\text{init}}$ ). (e) HAADF-STEM images showing healing evolution with time. The black arrows indicate the position of the crack.



# Nanoimaging of particles in LiNi type cathode material



Ni rich cathode material:  $\text{LiNi}_{1-x-y}\text{Mn}_x\text{Co}_y\text{O}_2$



Y. Yang, Y. Liu, et al., Adv. Energy Mater. (2019)

# Critical components of success

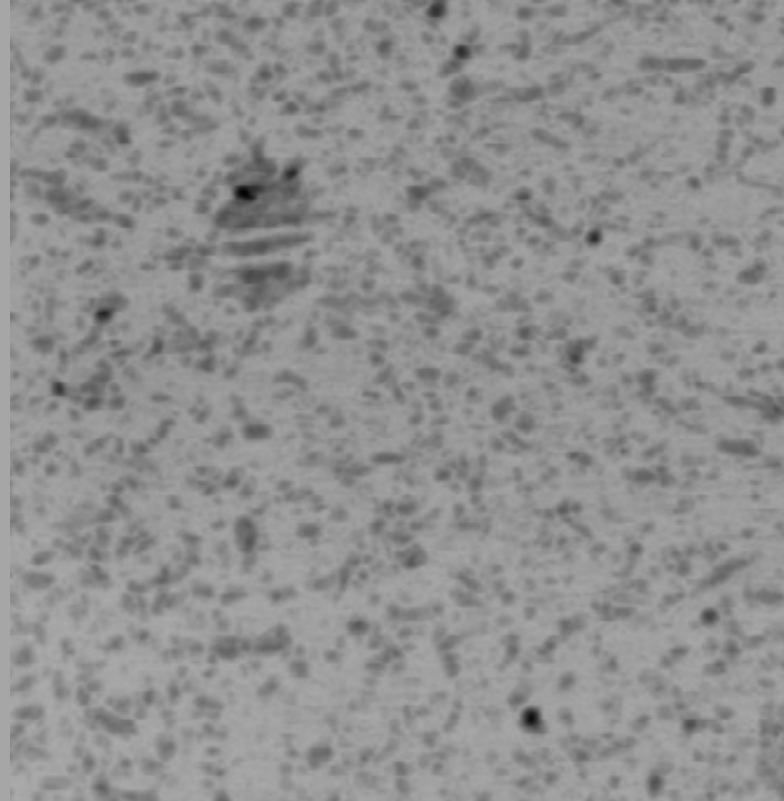
weak contrast between metallic phases



*Optimized phase contrast acquisition and reconstruction*



**Full exploitation of coherence of the beam**



need of reliable and non-manual quantification of fine and complex structures



*Artefacts-free reconstructions (avoid rings, gradients, structured noise – local tomo)*



**Clean X-ray wavefront**  
*As perfect (and as few) beamline optics as possible*

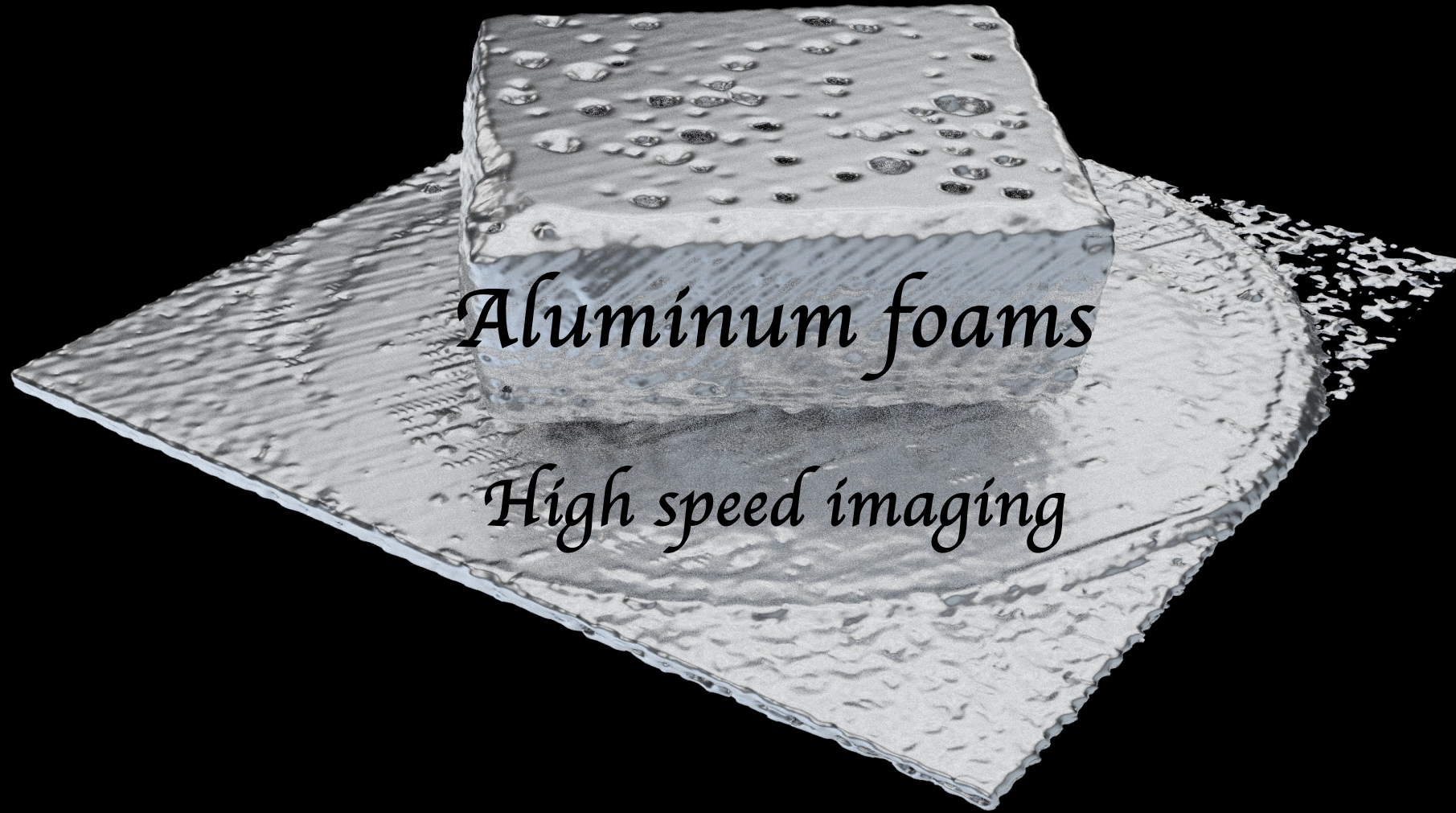
Representative volumes



*Maximized aspect ration between FOV and resolution*



*Large beam / detector*



*Aluminum foams*

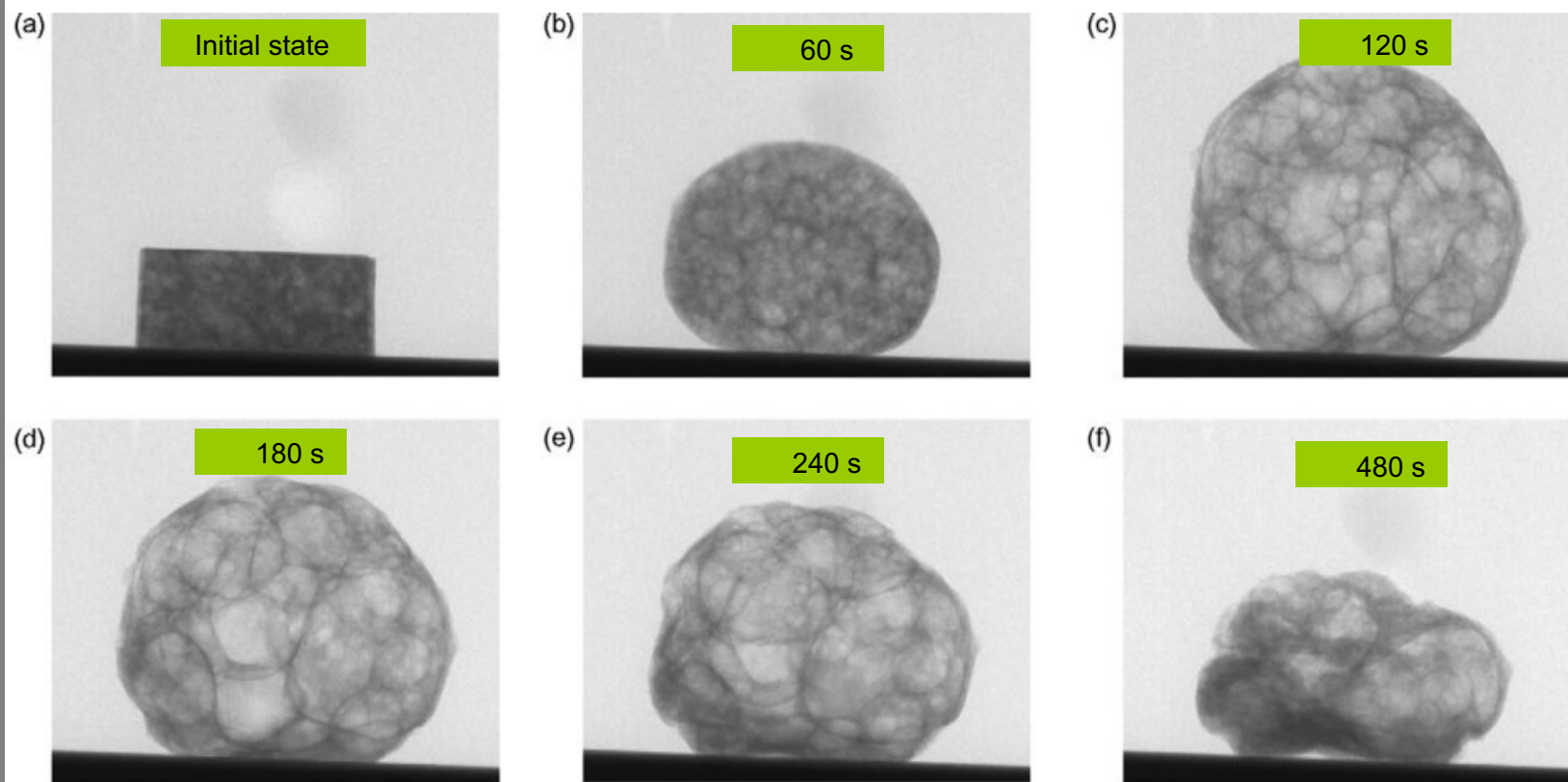
*High speed imaging*

# Why Alu?

1 IA											13 III A	14 IV A	15 V A	16 VI A	17 VII A	18 VIII A	
1 H Hydrogen 1.00794																2 He Helium 4.002602	
3 Li Lithium 6.941	4 Be Beryllium 9.012182											5 B Boron 10.811	6 C Carbon 12.0107	7 N Nitrogen 14.0067	8 O Oxygen 15.9994	9 F Fluorine 18.9984032	10 Ne Neon 20.1797
11 Na Sodium 22.98976928	12 Mg Magnesium 24.305	3 III B	4 IV B	5 V B	6 VI B	7 VII B	8 VIII B	9 VIII B	10 VIII B	11 IB	12 IIB	13 Al Aluminium 26.9815386	14 Si Silicon 28.0855	15 P Phosphorus 30.973762	16 S Sulfur 32.065	17 Cl Chlorine 35.453	18 Ar Argon 39.948
19 K Potassium 39.0983	20 Ca Calcium 40.078	21 Sc Scandium 44.9559	22 Ti Titanium 47.867	23 V Vanadium 50.9415	24 Cr Chromium 51.9961	25 Mn Manganese 54.938045	26 Fe Iron 55.845	27 Co Cobalt 58.933195	28 Ni Nickel 58.6934	29 Cu Copper 63.546	30 Zn Zinc 65.38	31 Ga Gallium 69.723	32 Ge Germanium 72.64	33 As Arsenic 74.9216	34 Se Selenium 78.96	35 Br Bromine 79.904	36 Kr Krypton 83.798
37 Rb Rubidium 85.4678	38 Sr Strontium 87.62	39 Y Yttrium 88.90585	40 Zr Zirconium 91.224	41 Nb Niobium 92.9063	42 Mo Molybdenum 95.96	43 Tc Technetium [98]	44 Ru Ruthenium 101.07	45 Rh Rhodium 102.9055	46 Pd Palladium 106.42	47 Ag Silver 107.8682	48 Cd Cadmium 112.411	49 In Indium 114.818	50 Sn Tin 118.71	51 Sb Antimony 121.76	52 Te Tellurium 127.6	53 I Iodine 126.90447	54 Xe Xenon 131.293
55 Cs Caesium 132.9054519	56 Ba Barium 137.327	57-71 Lanthanoids	72 Hf Hafnium 178.49	73 Ta Tantalum 180.94788	74 W Tungsten 183.84	75 Re Rhenium 186.207	76 Os Osmium 190.23	77 Ir Iridium 192.217	78 Pt Platinum 195.084	79 Au Gold 196.966569	80 Hg Mercury 200.59	81 Tl Thallium 204.3833	82 Pb Lead 207.2	83 Bi Bismuth 208.9804	84 Po Polonium [209]	85 At Astatine [210]	86 Rn Radon [222]
87 Fr Francium [223]	88 Ra Radium [226]	89-103 Actinoids	104 Rf Rutherfordium [267]	105 Db Dubnium [268]	106 Sg Seaborgium [271]	107 Bh Bohrium [272]	108 Hs Hassium [270]	109 Mt Meitnerium [276]	110 Ds Darmstadtium [281]	111 Rg Roentgenium [280]	112 Cn Copernicium [285]	113 Nh Nihonium [286]	114 Fl Flerovium [289]	115 Mc Moscovium [288]	116 Lv Livermorium [293]	117 Ts Tennessine [294]	118 Og Oganesson [294]

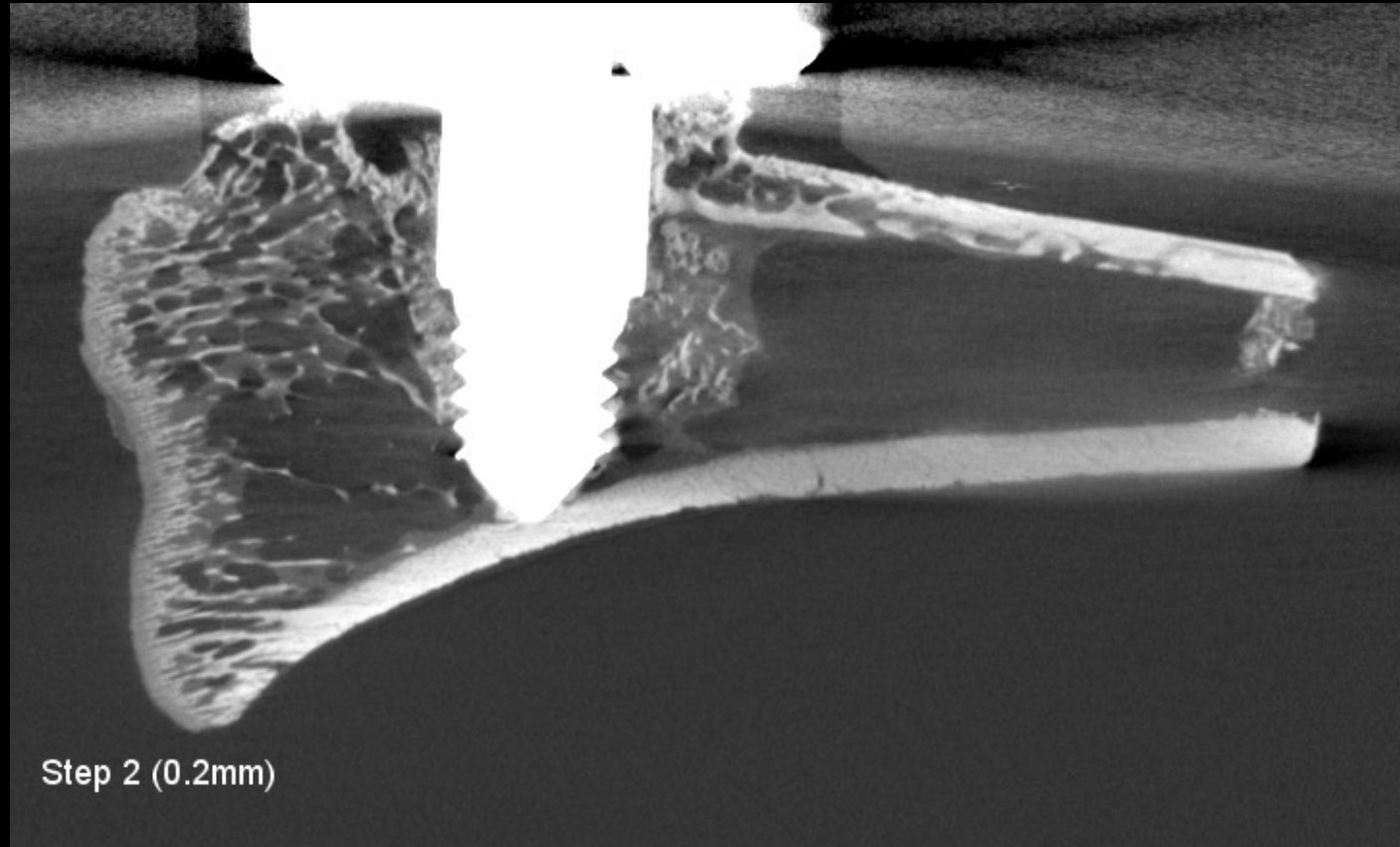
*Simplest to image  
relevant sized  
samples at a usual  
imaging beamline at  
3rd gen  
synchrotrons*

57 La Lanthanum 138.90547	58 Ce Cerium 140.116	59 Pr Praseodymium 140.90765	60 Nd Neodymium 144.242	61 Pm Promethium [145]	62 Sm Samarium 150.36	63 Eu Europium 151.964	64 Gd Gadolinium 157.25	65 Tb Terbium 158.9253	66 Dy Dysprosium 162.5	67 Ho Holmium 164.93032	68 Er Erbium 167.259	69 Tm Thulium 168.93421	70 Yb Ytterbium 173.054	71 Lu Lutetium 174.9668
89 Ac Actinium [227]	90 Th Thorium 232.03806	91 Pa Protactinium 231.03588	92 U Uranium 238.02891	93 Np Neptunium [237]	94 Pu Plutonium [244]	95 Am Americium [243]	96 Cm Curium [247]	97 Bk Berkelium [247]	98 Cf Californium [251]	99 Es Einsteinium [252]	100 Fm Fermium [257]	101 Md Mendelevium [258]	102 No Nobelium [262]	103 Lr Lawrencium [262]



Babcsan, Moreno, Banhart, *Colloids & Surfaces A* 2007

Precursor made from oxidized melt heated slightly above the melting point.



***Interfaces between high and low Z materials are difficult***



***The important contribution of phase contrast at high energies***

4D imaging lab,  
Zeiss, Solid mechanics, LU

Le Cann et al., Front. Bioeng. Biotechnol 2019

# Discussion

- I. Which type of materials are you interested in? (Al, Mg...)***
- II. Any interest in samples with more than 1 mm steel?***
- III. Are large batches of samples relevant? (automation of acquisition)***
- IV. For in-situ studies, do you have your own sample environment? Compatible with X-ray tomo?***

