A revolution in the field of metallography aided by the Laser Ultrasonic technology

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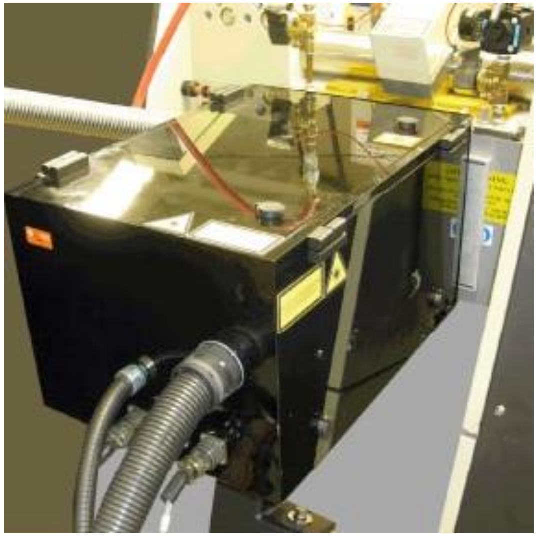
Introduction and topics

- ✓ Laser Ultrasonics for Metallurgy (LUMet)
- ✓ Austenite grain size in steel
- ✓ Grain growth in INCONEL 718
- ✓ Phase transformation and static recrystallization
- ✓ Dynamic recrystallisation in austenite
- ✓ Simulation of ultrasound propagation in anisotropic polycrystalline media

A commercial sensor for metallurgist

- ✓ Laser Ultrasonics for Metallurgy (LUMet)
- ✓ Attachment to a Gleeble thermo-mechanical simulator
- ✓ Dedicated sensor for measurements during processing of metals



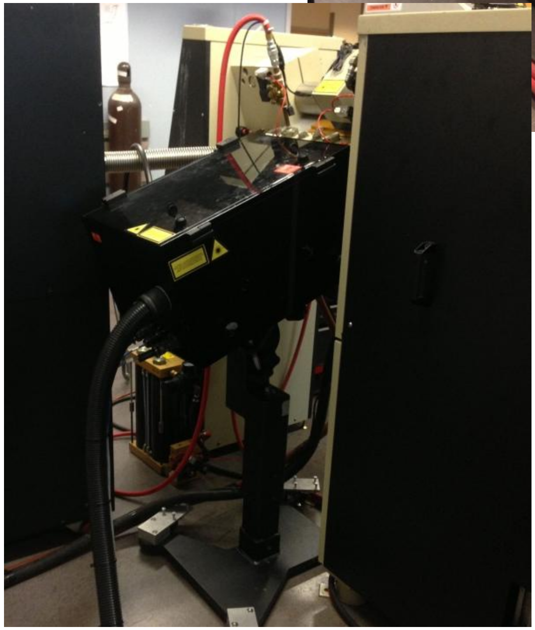
NRC·CNRC
Industrial
Materials
Institute

TECNAR

DSI
Dynamic Systems Inc.

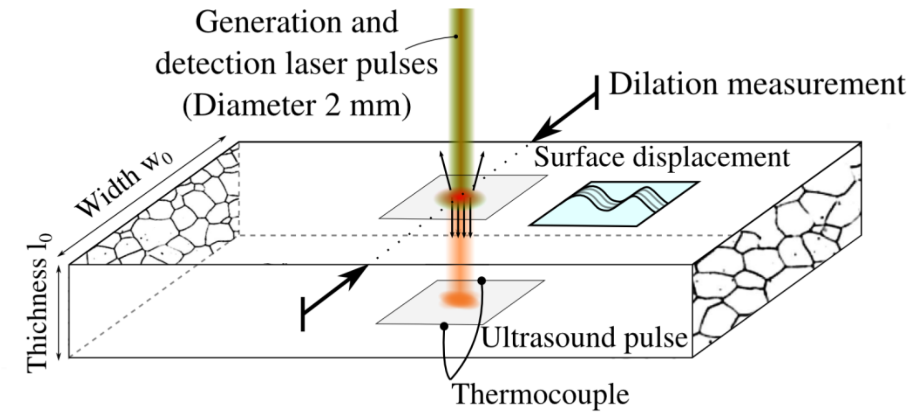
LUMet : Technical specifications

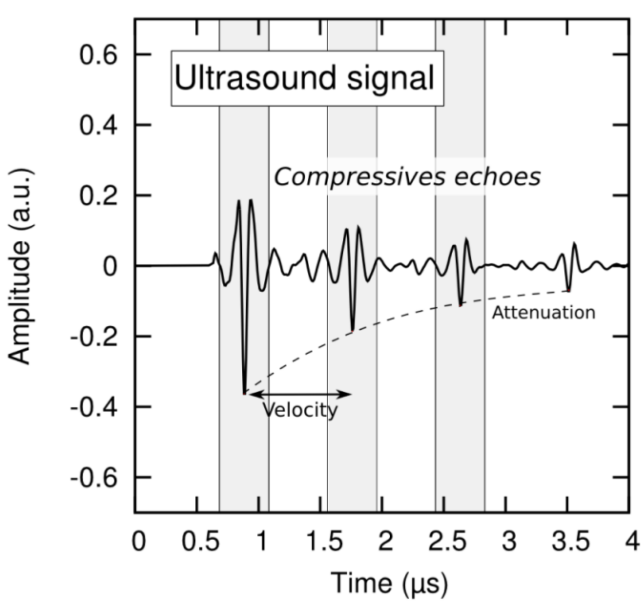
- ✓ Generation pulse laser: Frequency double Q-switch Nd:YAG (532nm, 72mJ, 9 ns)
- ✓ Detection pulse laser: Frequency stabilized Nd:YAG (1064nm, 90 µs)
- ✓ Photorefractive interferometer
- ✓ Bandwidth: 4 to 20 MHz
- ✓ Up to 50 pulses per second



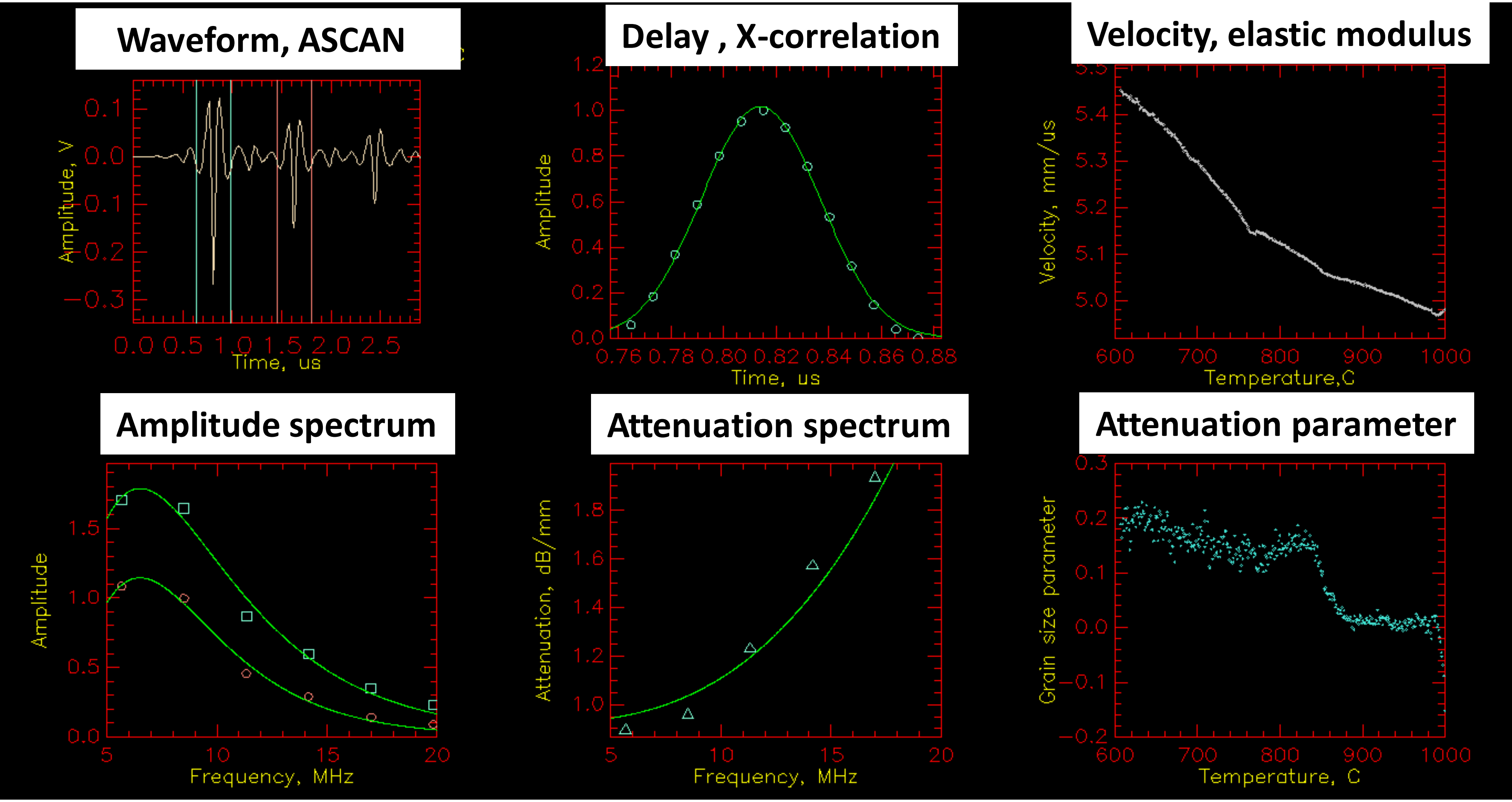
LUMet : Methodology & applications

- ✓ Generation and detection of ultrasonic wave by lasers
- ✓ Velocity is related to elasticity → Texture
- ✓ Attenuation mainly due to grains scattering → Grain size
- ✓ Recrystallization, phase transformation, grain growth



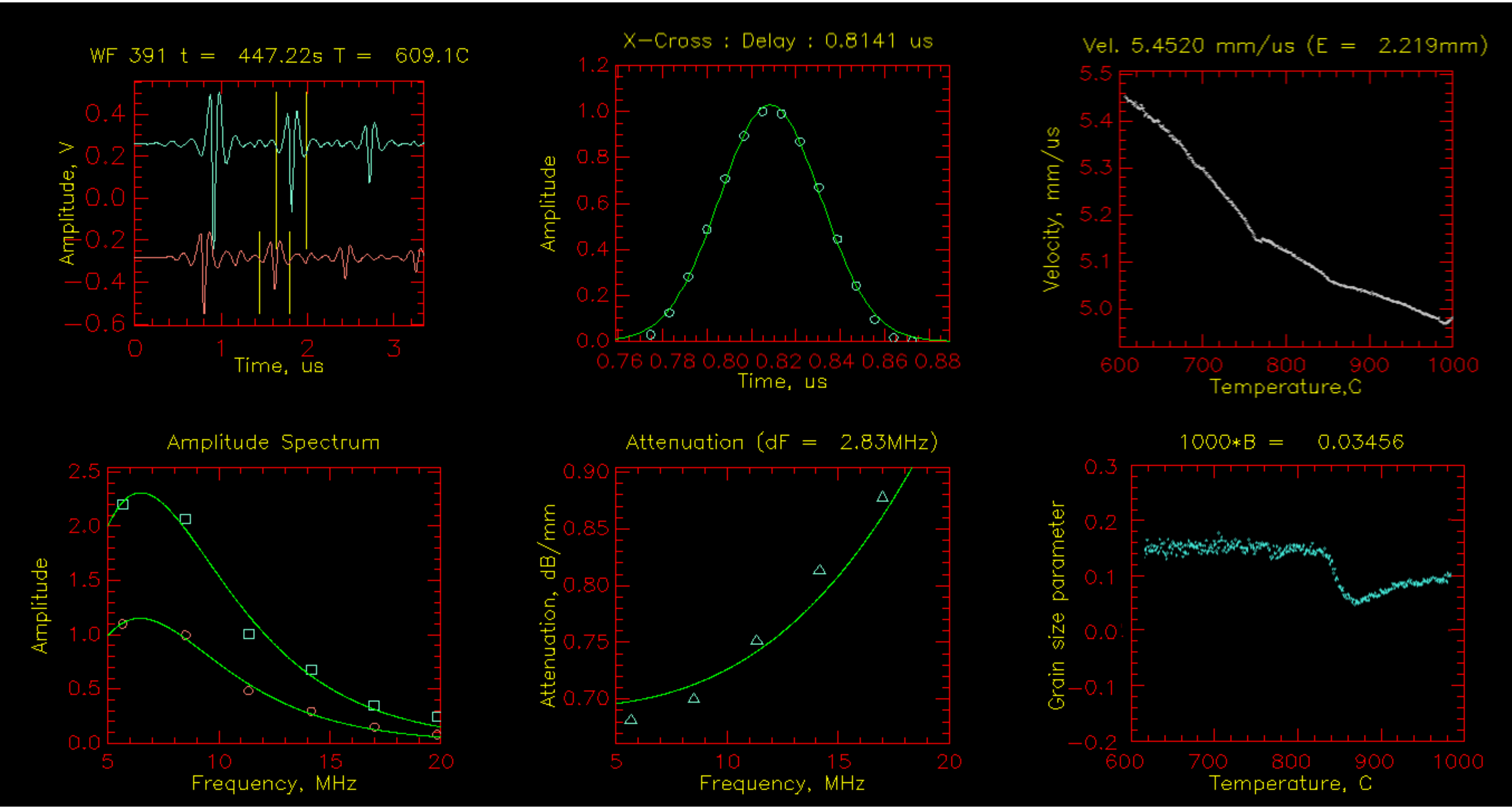


Comparing echoes from the same pulse



Ultrasound properties for a given microstructure

Comparing echoes from the two different pulses

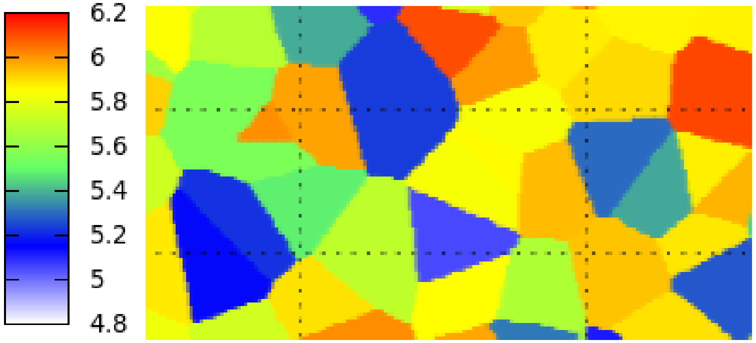


Ultrasound properties of a microstructure relative to another one

Why attenuation relates to grain size ?

- ✓ Relatively anisotropic material
- ✓ Large mismatch in the elastic properties from grain to grain
- ✓ Attenuation : Diffraction + scattering + internal friction
- ✓ Experiments and theories agree on the existence of scattering regimes

Velocity map



$$\alpha(f) = C(T)D^{n-1}f^n$$

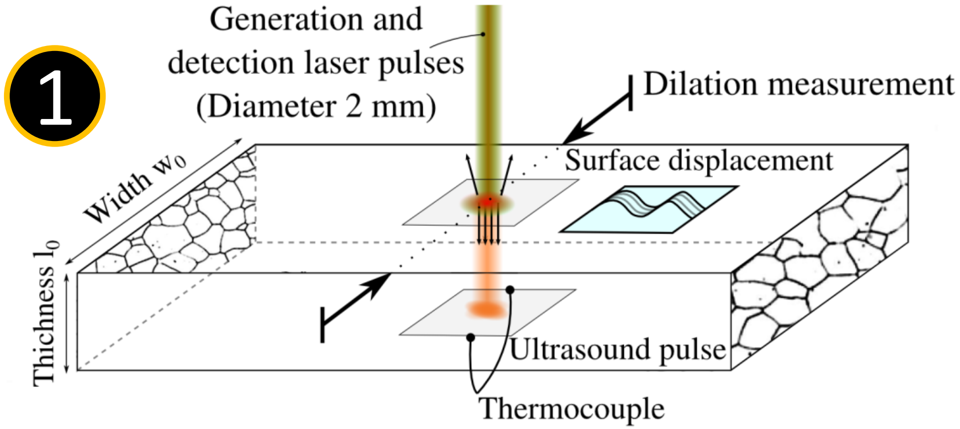
$$\lambda >> D \text{ , } n = 4$$
$$\lambda \approx D \text{ , } n = 2$$

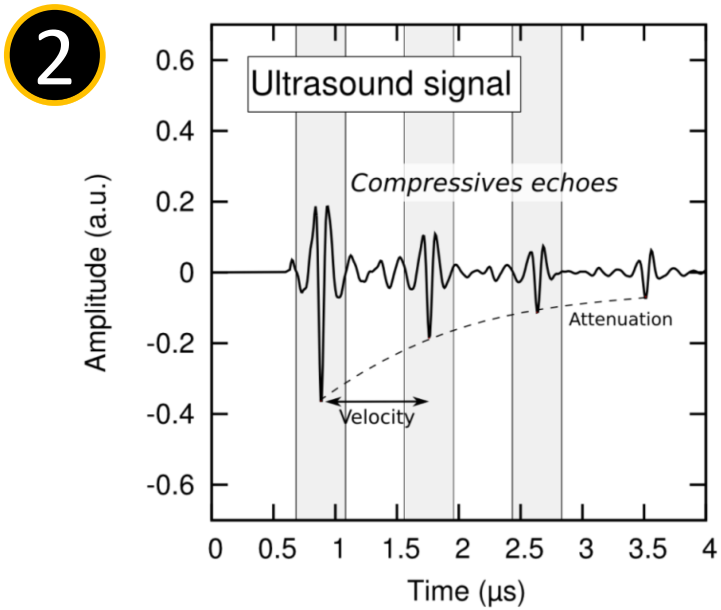
<u>LUMet in steel</u>

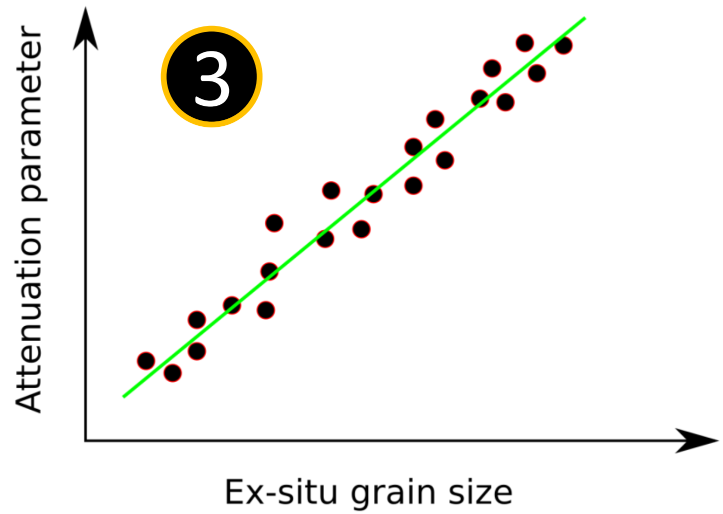
300 < λ < 1000 (µm)

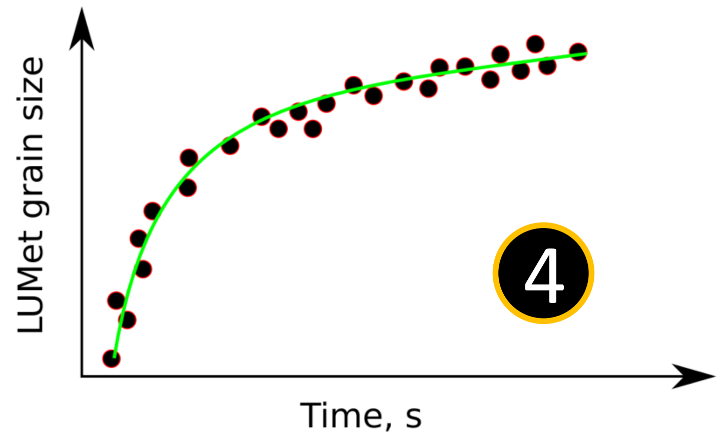
5 < D < 500 (µm)

Measure grain size with LUMet



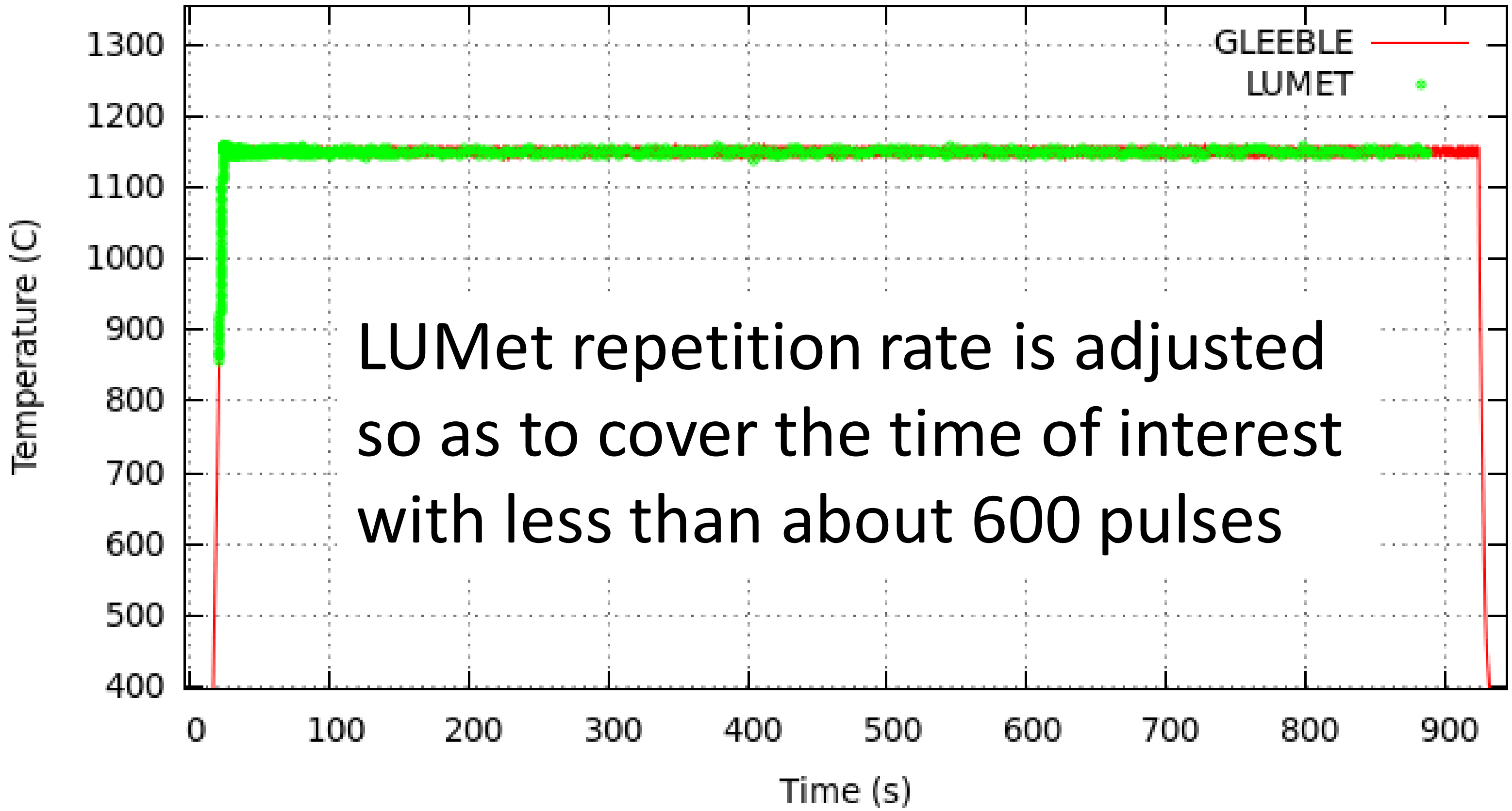






In-situ austenite grain size evaluation

✓ X80 linepipe steel, 100°C/s, 1150°C, 15 min



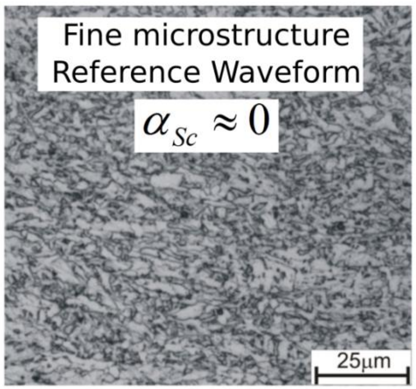
LUMet repetition rate is adjusted so as to cover the time of interest with less than about 600 pulses

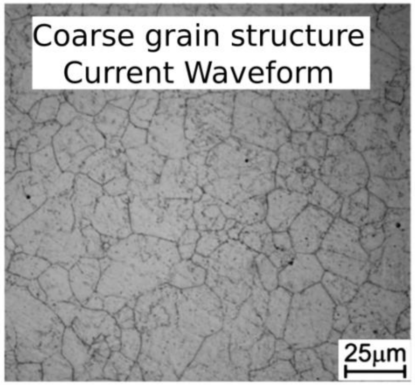
Methodology

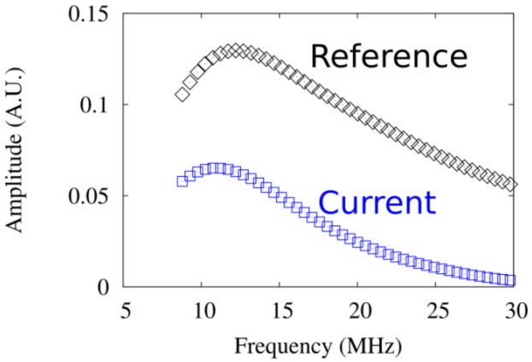
✓ Attenuation spectrum from single echo technique

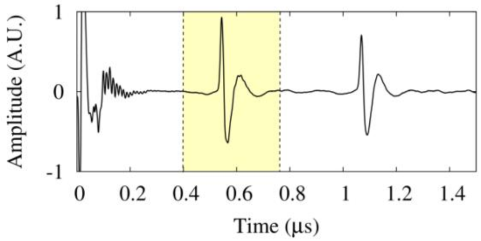
$$\alpha = \alpha_D + \alpha_{Sc}$$

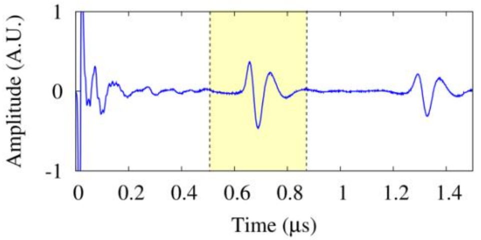
$$\alpha_{Sc} = \frac{20}{2e}\log\left(\frac{A_{Current}}{A_{Reference}}\right)$$

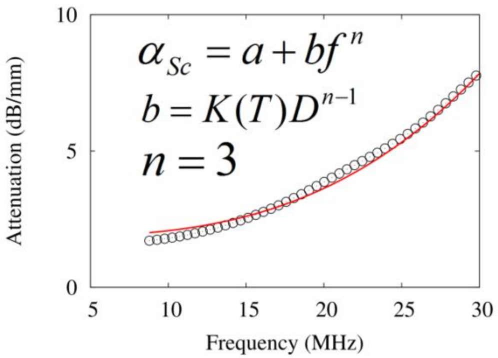








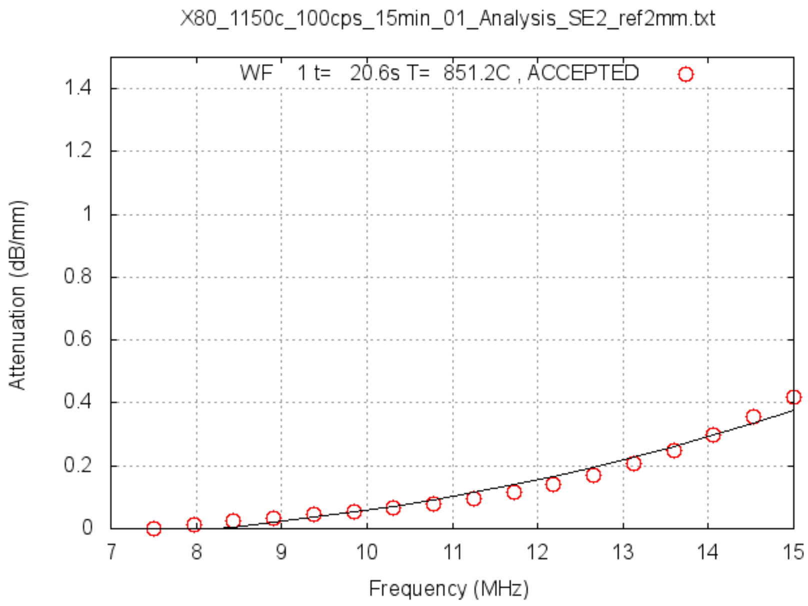


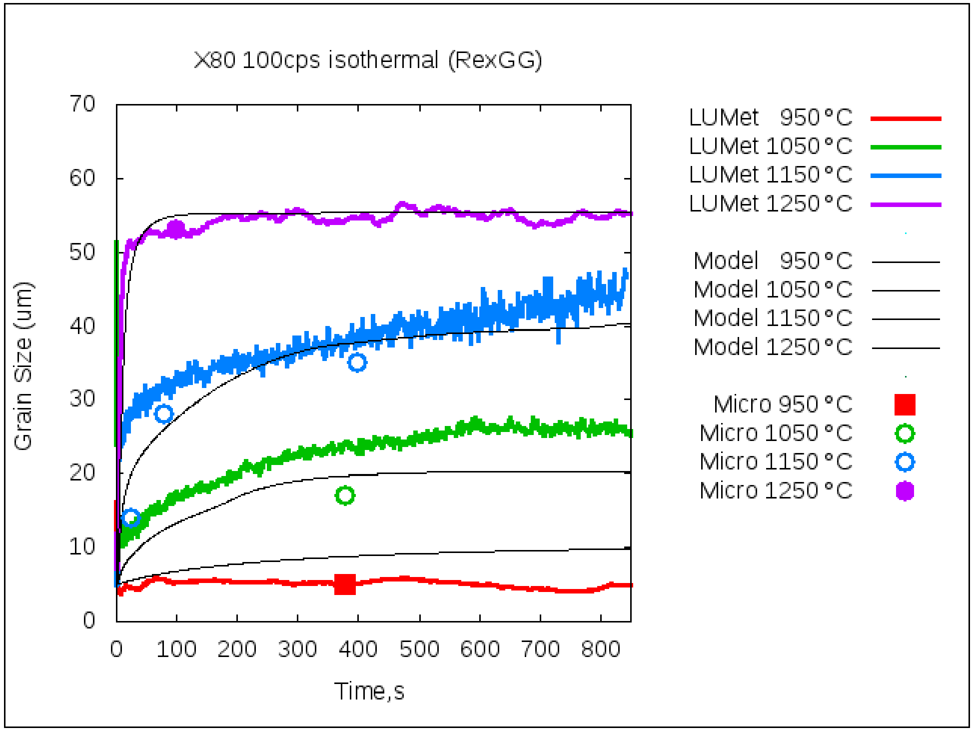


✓ Calibration available for austenite in low alloy steel : S.E. Kruger et al., Iron Steel Technol, (2005), 2(10),25

Grain size parameter

- Regression on attenuation spectrum
- Evaluation of grain size from available calibration data

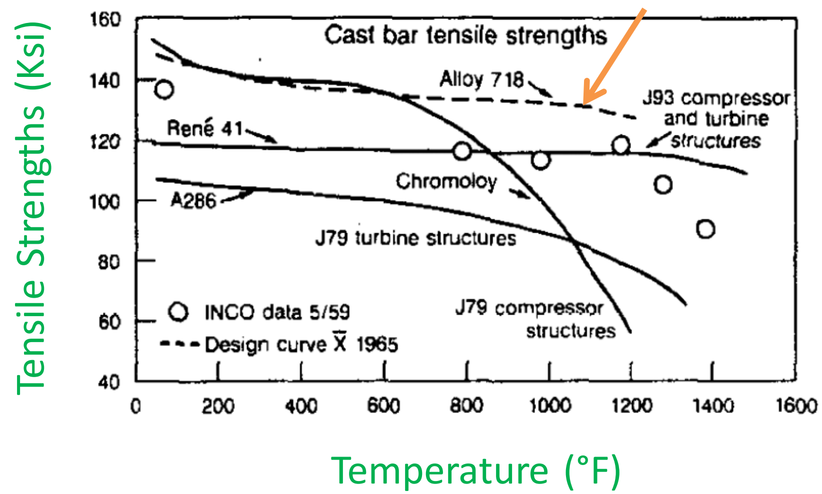




Evolution of microstructure in INCONEL 718 superalloy

- ✓ Inconel 718 used in aviation industry
- ✓ High strength, stable at elevated temperature
- ✓ Dynamic recrystallization may occurs during forging
- ✓ **First step, monitoring of grain growth**
- ✓ … Static recrystallization, …dynamic measurements

±1 Gpa at 650°C



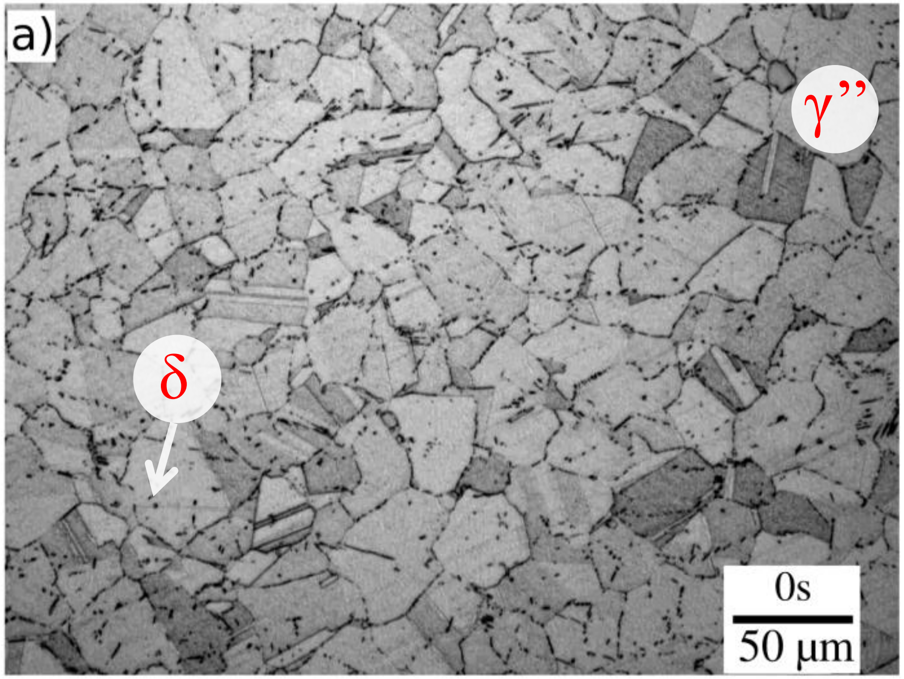
J. F. Barker
The initial years of alloy 718. TMS Superalloys 718, 625, 706 and various derivatives (1989)

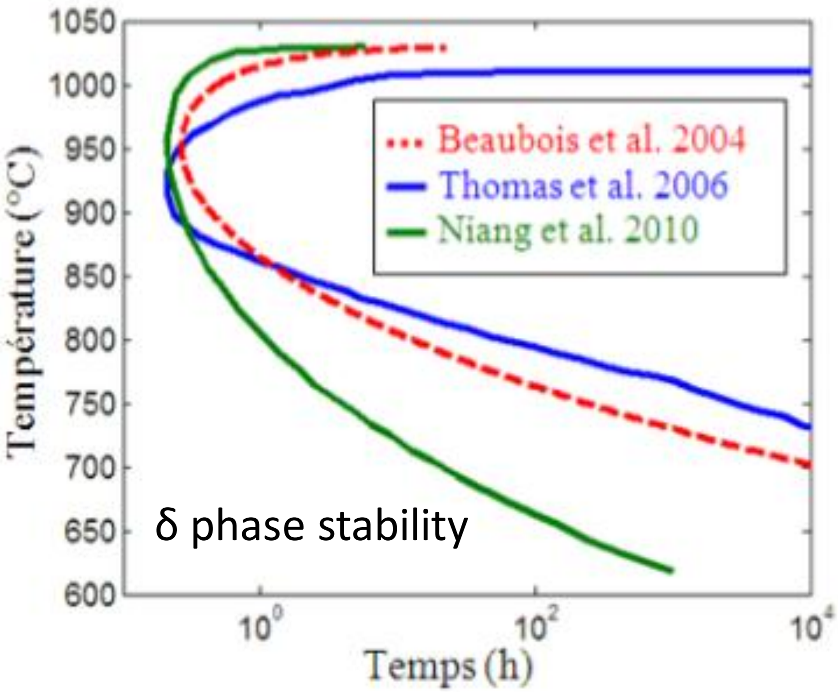
Material INCONEL 718

- ✓ Composition (Key elements wt.fraction)

  0.52Ni, 0.19Cr, 0.19Fe, Mo, Nb, Ta, Ti, Al, Co

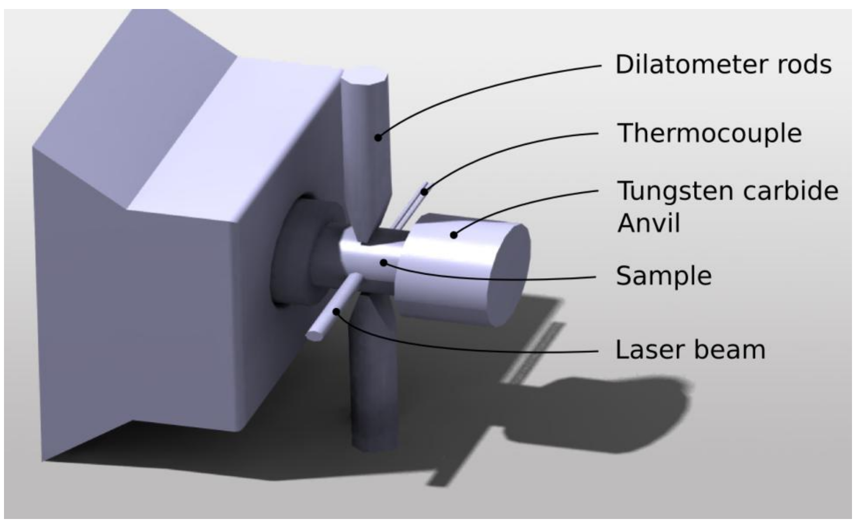
- ✓ Grain size 24 µm, globular delta phase precipitates

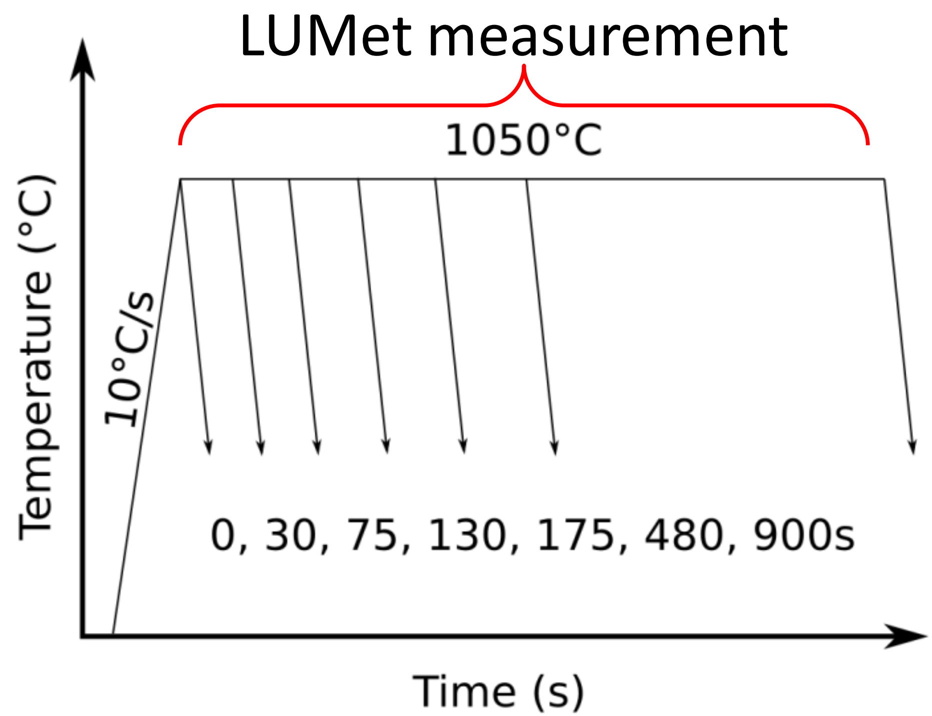




Experiments

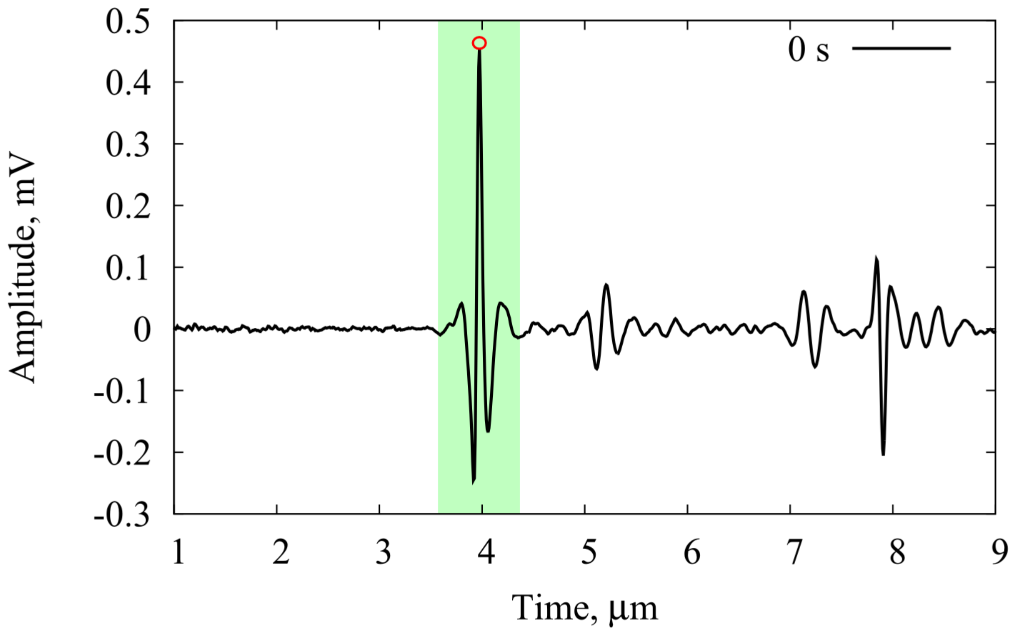
- Isothermal holding at 1050°C for various time
- Attenuation measurement under isothermal conditions
- Validation with metallography

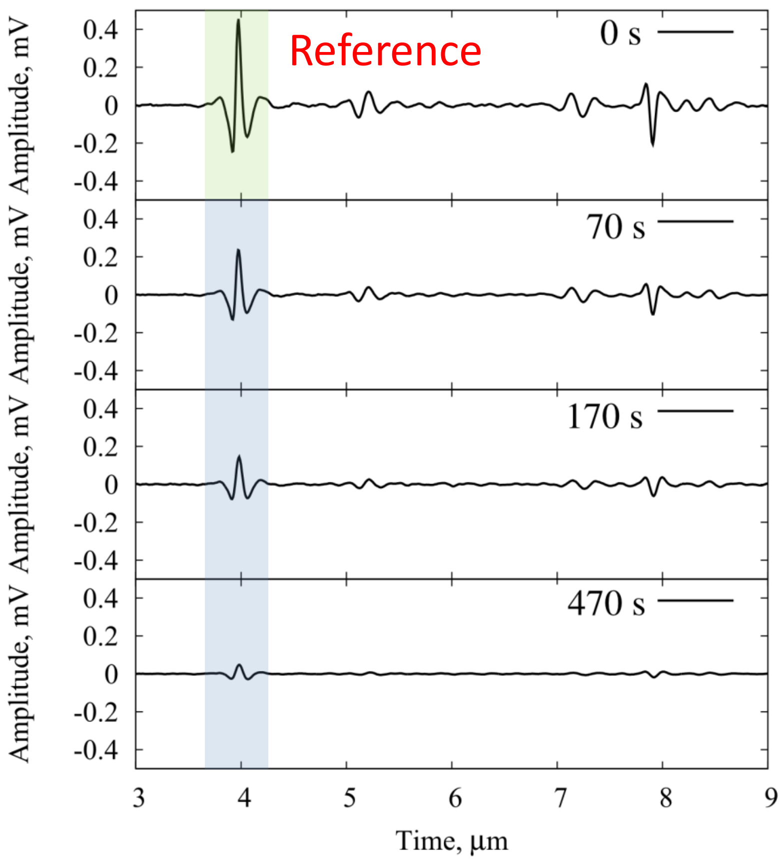




Ultrasound signal

- ✓ For each waveform, analysis of the frequency content of the first echo relative to the echo measured in the initial state





Modified single echo technique

- ✓ Ideally, reference waveform is measured in fine grain material, negligible scattering by grain

$$\alpha(f) = -\frac{20}{2D} log10\left(\frac{g(f)A_{sc}(f)}{g(f)}\right)$$

$\leftarrow$ t ≠ t_{ref}

$\leftarrow$ t = t_{ref}

$$\alpha\,(f) = a + CD^{n-1}f^{n}$$

$$\alpha\,(f) = a + bf^{3}$$

$$b \propto CD^{2}$$

- ✓ Absolute grain size measurement

Modified single echo technique

- ✓ Here, reference has scattering contribution

$$\alpha(f) = -\frac{20}{2D} log10\left(\frac{g(f)A_{sc1}(f)}{g(f)A_{sc0}(f)}\right)$$

← t ≠ t_{ref}

← t = t_{ref}

$$\alpha(f) = \alpha_1(f) - \alpha_0(f)$$

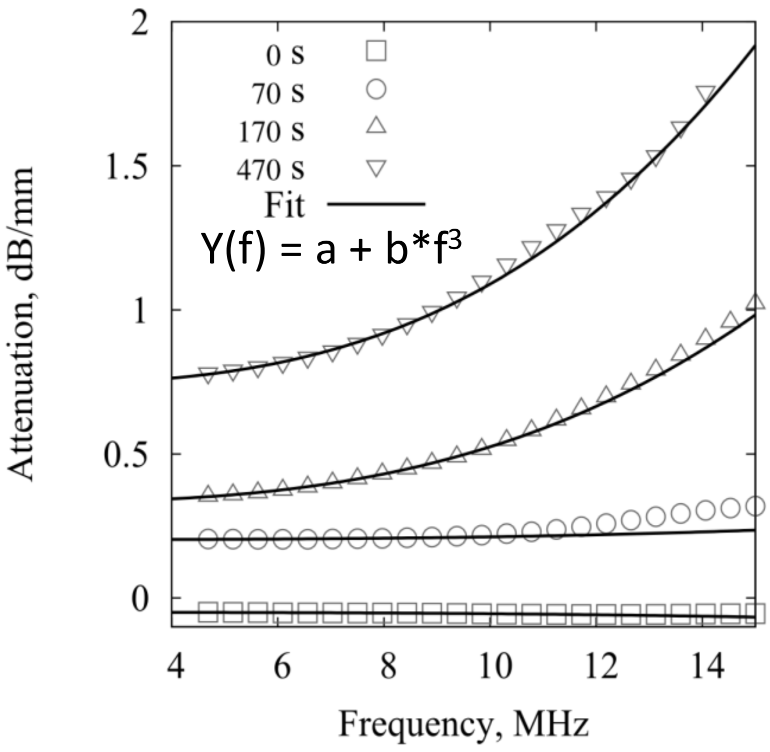
$$\alpha\ (f) = a \quad + C(D_1^{n-1} - D_0^{n-1})f^n$$

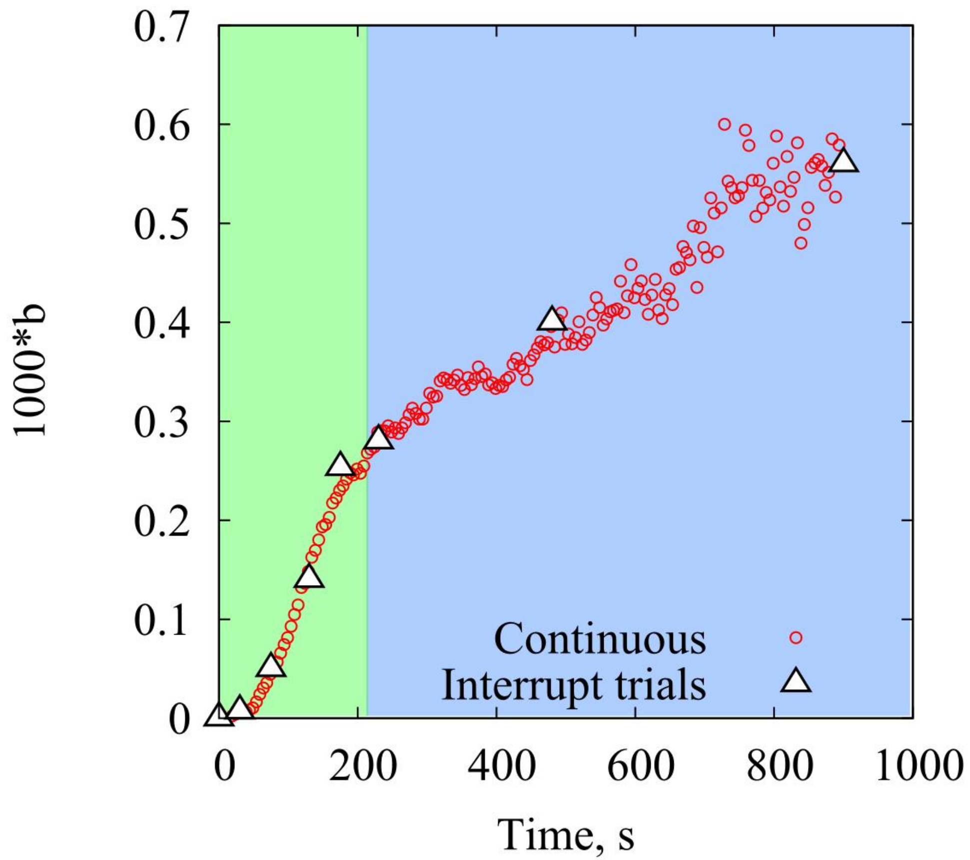
$$b \propto C\left({D_1}^2 - {D_0}^2\right)$$

- ✓ Relative change in grain size

Attenuation, grain size parameter

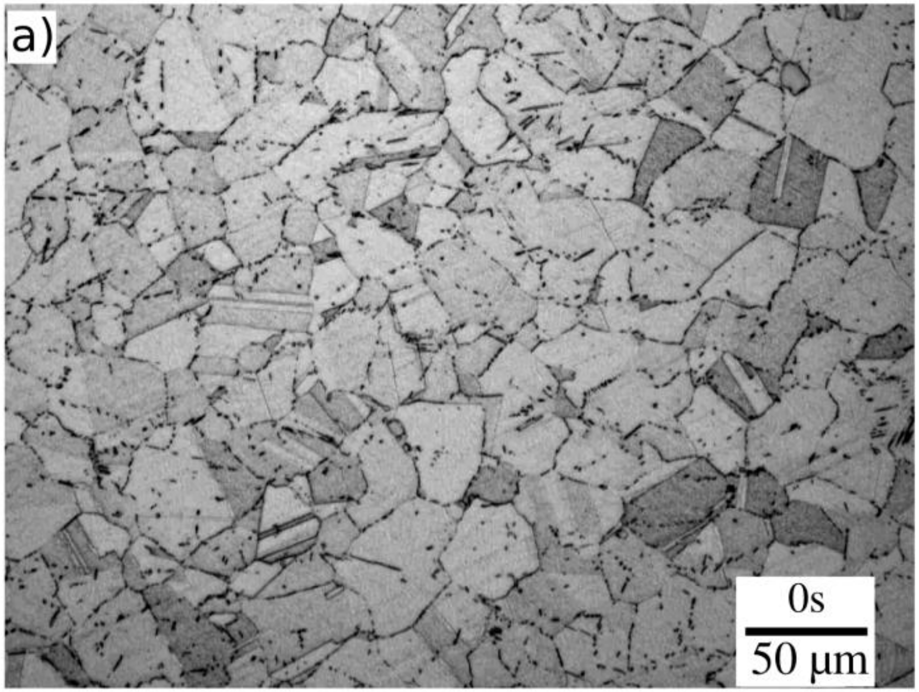
- ✓ Systematic evaluation of the grain size parameter b from the measured attenuation spectrum

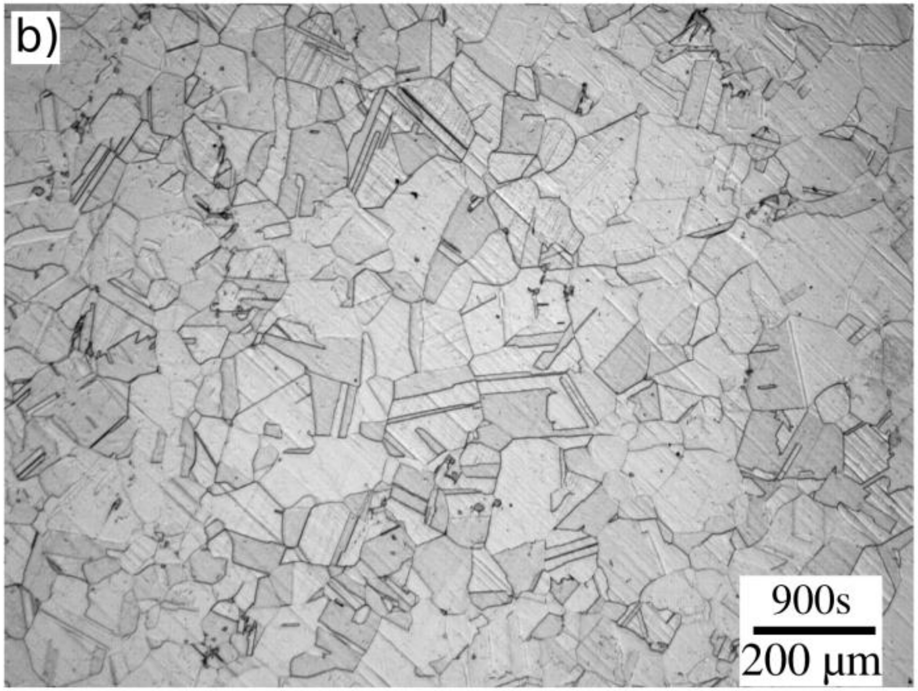




Initial and final stage

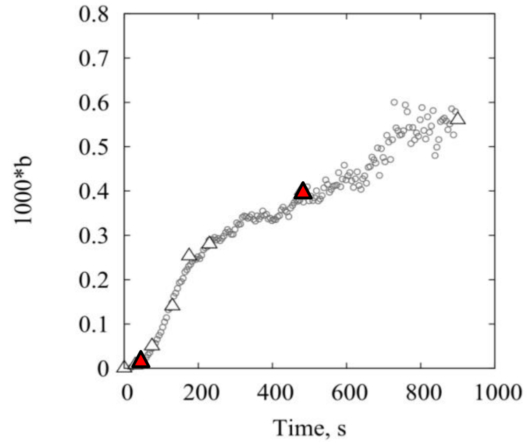
- ✓ Average grain size increases by a factor of 4 during the 15 mn annealing
- ✓ 900 s: Delta phase is almost fully dissolved
- ✓ 900 s: Formation of annealing twins

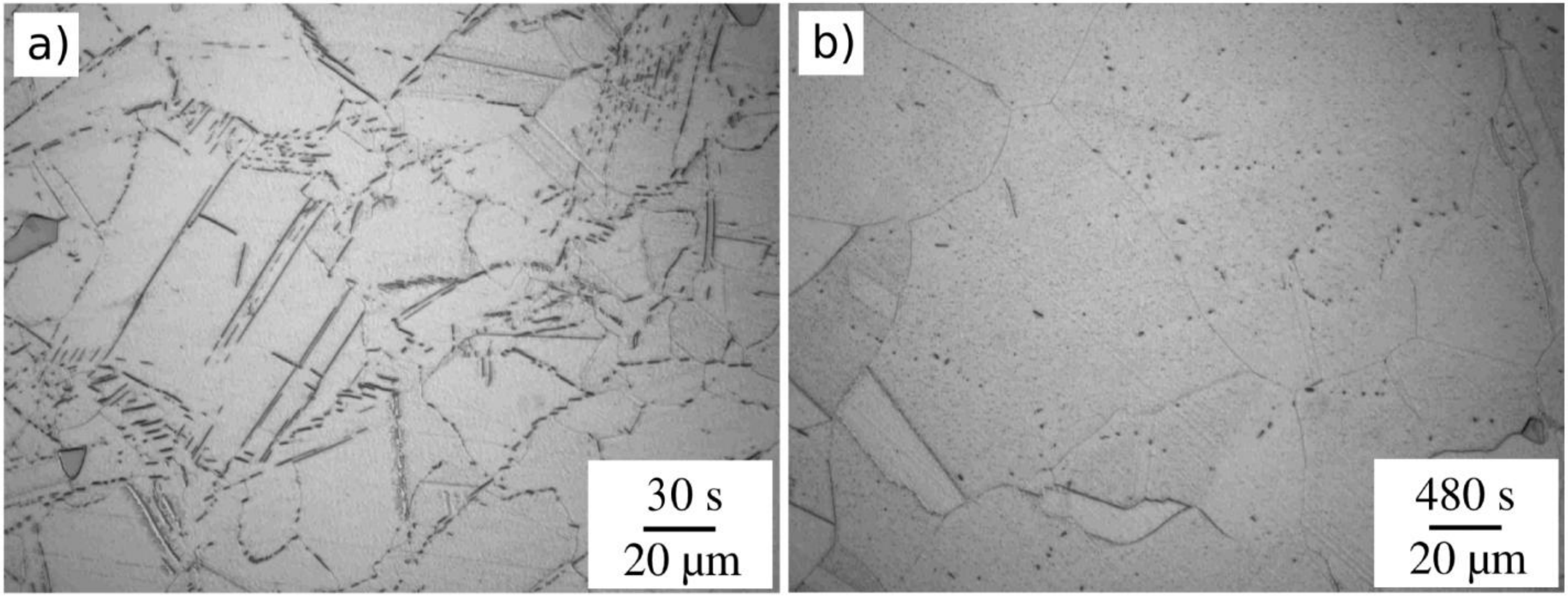




Evolution of Delta phase

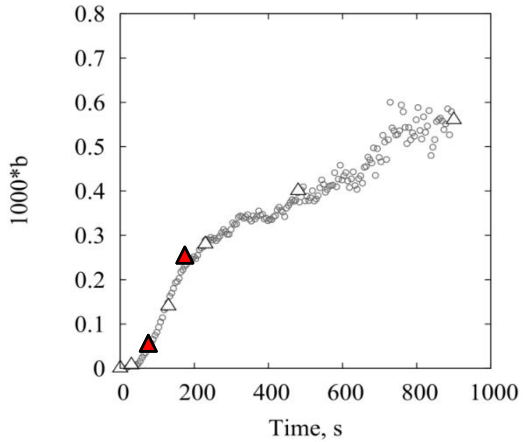
- ✓ 30s : Coarsening and dissolution of delta phase
- ✓ 480s: Small fraction of delta phase remains, most GB are unpinned

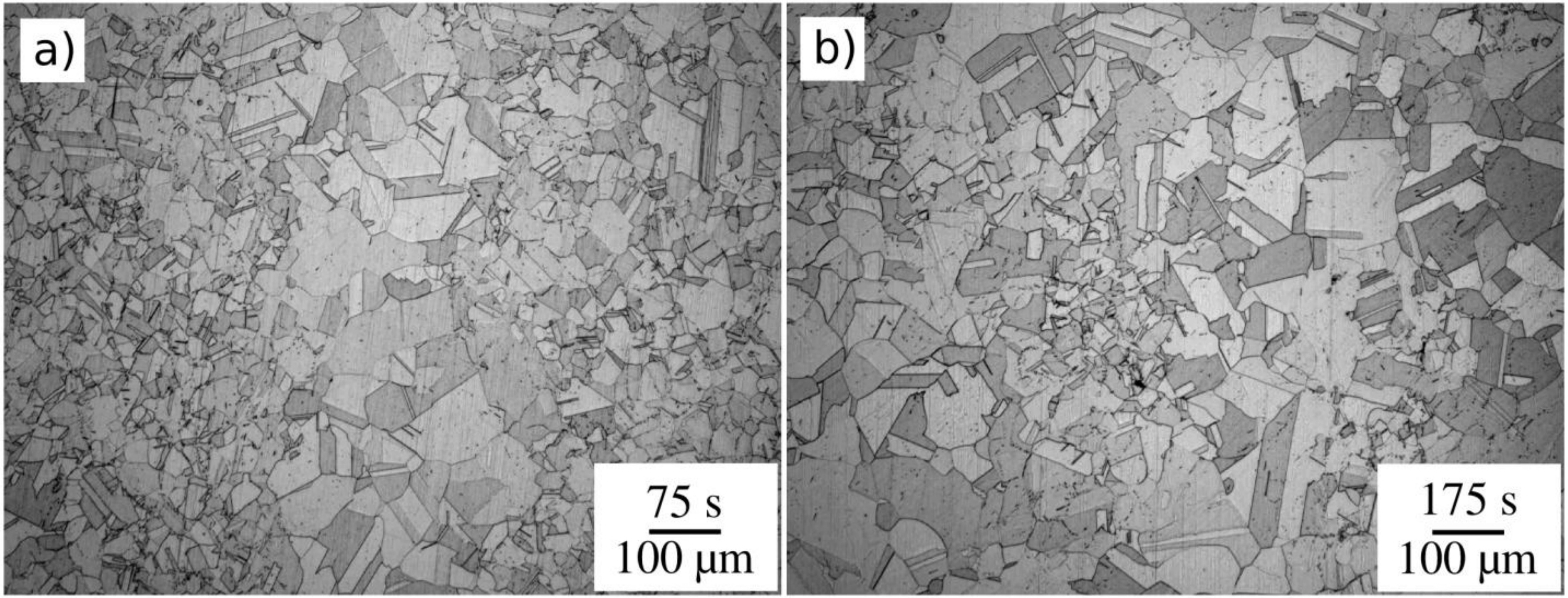




Heterogeneous grain structure

- ✓ 75s : Faster grain growth in certain area of the sample
- ✓ 480s: Few zones with small grains remains





Mean grain size, distribution

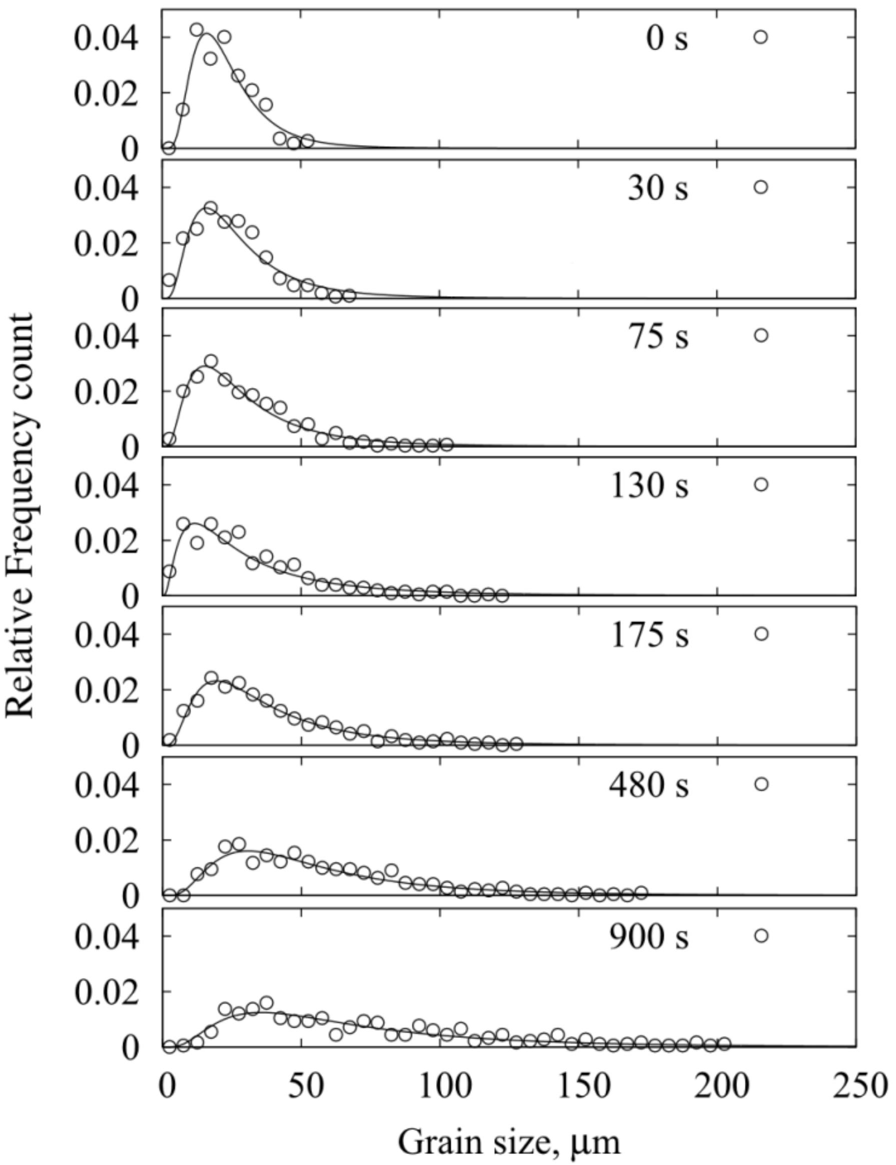
- ✓ Quantitative analysis of optically measured grain area

$$EQAD = \sqrt{\pi \bar{A}/4}$$

- ✓ Log normal distribution, M, S

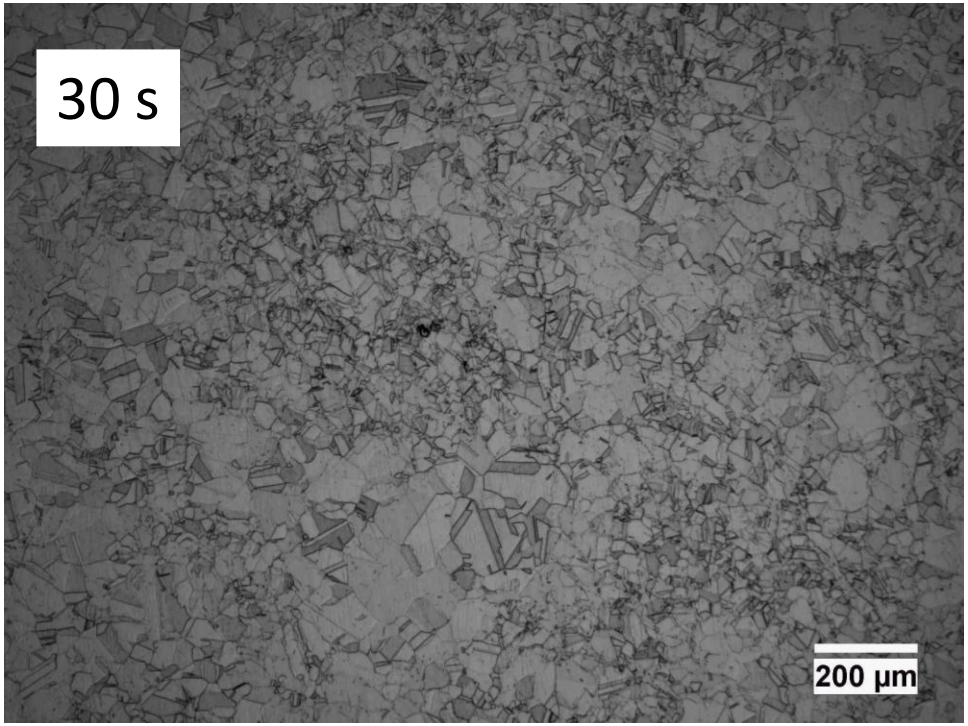
$$\mu = \exp(M + \frac{S^2}{2})$$

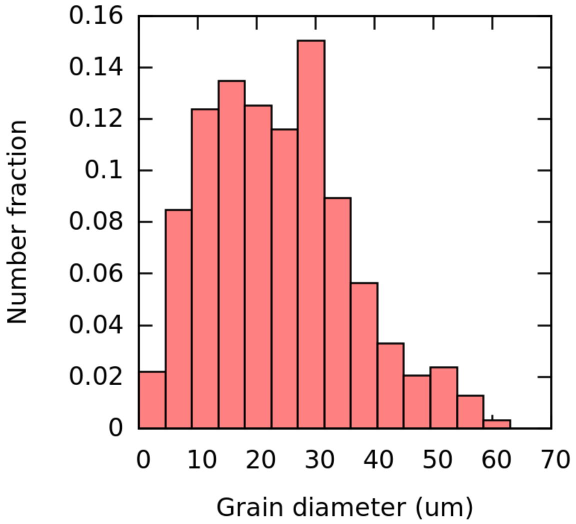
Time	EQAD	μ(M)	σ(S)
0	**24**	**24** (3.05)	**13** (0.52)
30	**27**	**29** (3.17)	**20** (0.63)
75	**33**	**32** (3.20)	**25** (0.70)
130	**37**	**38** (3.24)	**42** (0.89)
175	**44**	**40** (3.45)	**32** (0.70)
230	**46**	**43** (3.50)	**32** (0.68)
480	**62**	**59** (3.86)	**42** (0.65)
900	**82**	**74** (4.07)	**60** (0.70)

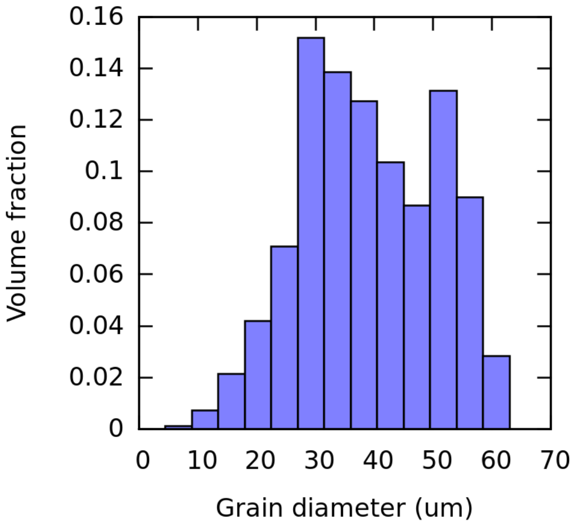


Volume fraction

- ✓ Large volume occupied by larger grains at the early time of the holding
- ✓ Not clearly a bi-modal grain distribution

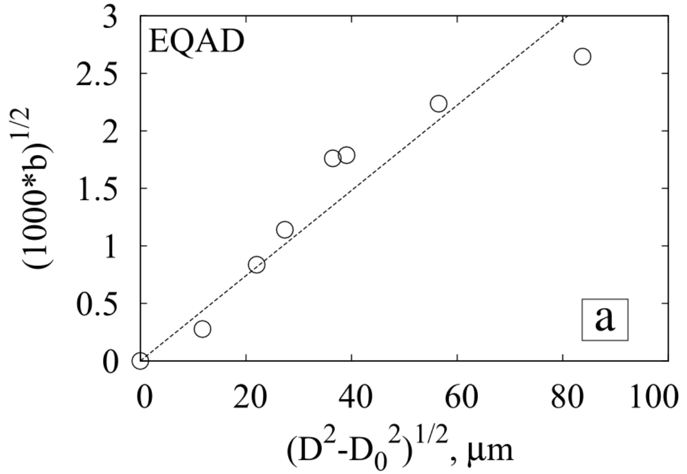


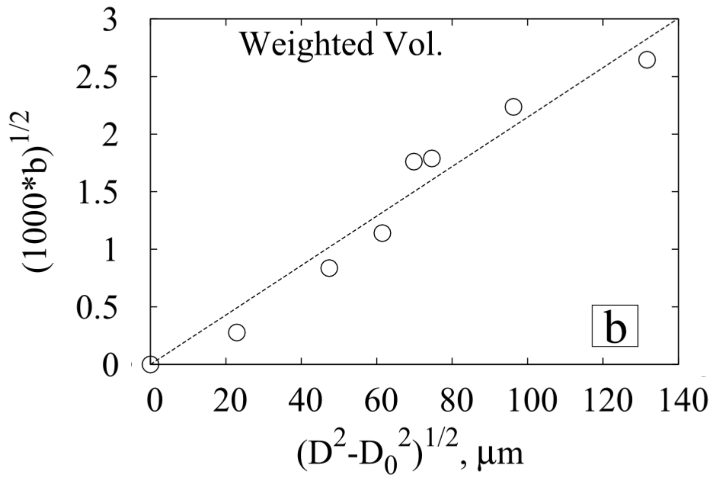




Attempt to build calibration

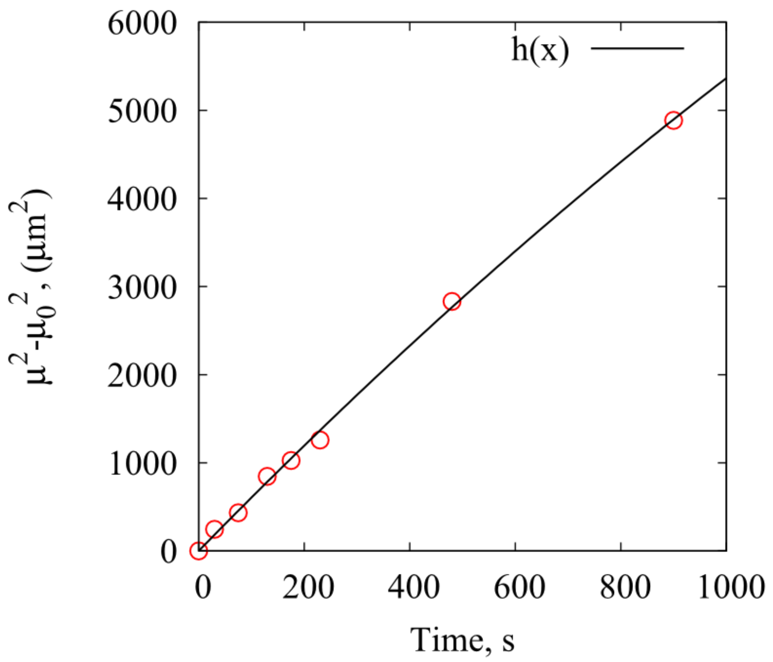
- ✓ Simple relation between mean grain size and grain size parameter ?
- ✓ Mean grain size only is not correctly describing the evolution of the size distribution.
- ✓ LUMet seems sensitive to variation in the distribution.
- ✓ Larger grains contribute more to the LU signal

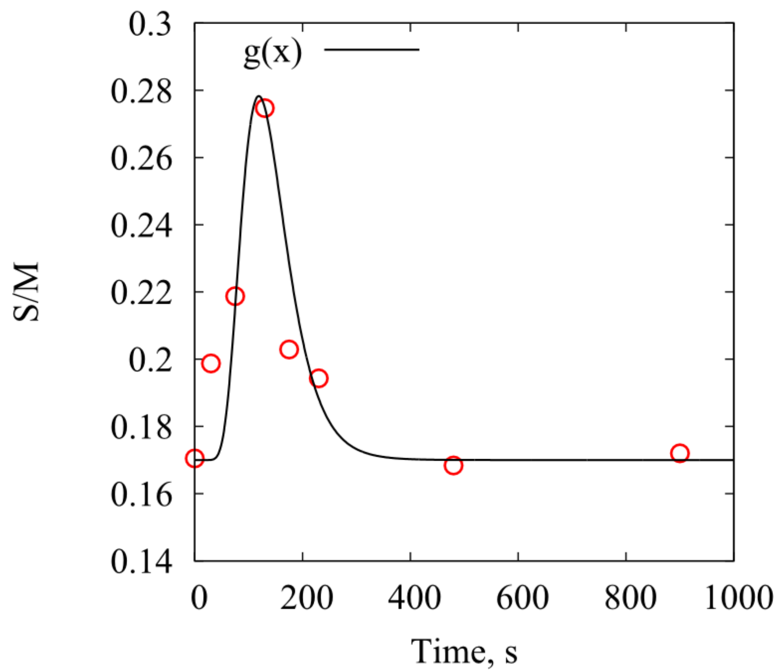




Attenuation and size distribution

- ✓ Evaluate the expected variation of the attenuation according to a measured size distribution
- ✓ Empirical approach based on scattering theory
- ✓ Construction of time dependant distribution F(S,M,t)



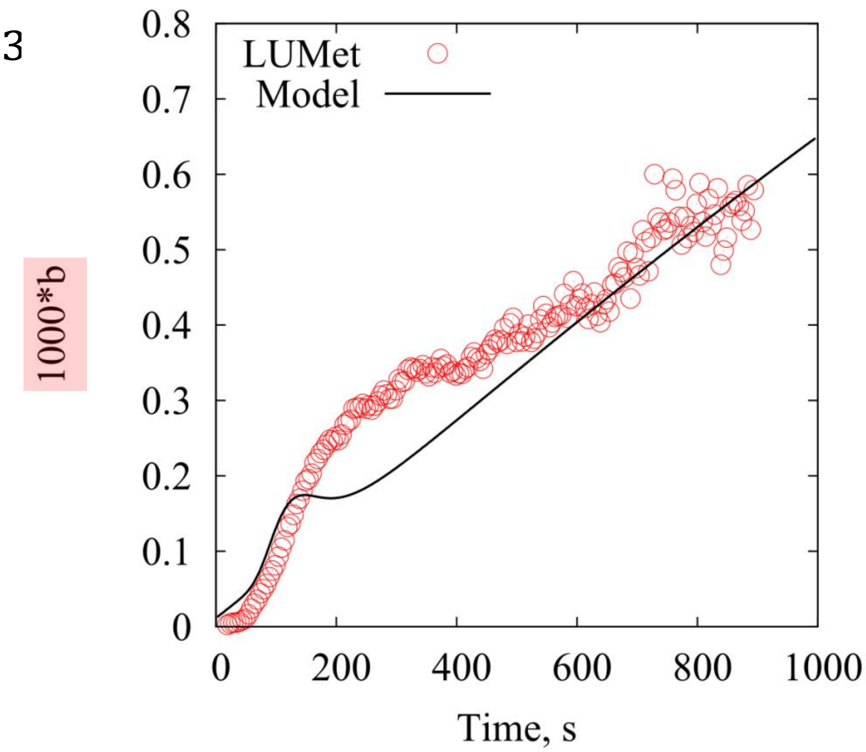


Prediction of the attenuation parameter

- ✓ Attenuation spectrum weighted by the distribution of grain size in the materials

$$\alpha\big(f, F(D)\big) = Cst \cdot \sum_{i}^{n} F(D_i)D_i{}^{2} f^{3}$$

- ✓ Evidence of two regimes
- ✓ Transition is still not very well described
- ✓ To simplistic approach, may be aid by Finite Element simulations



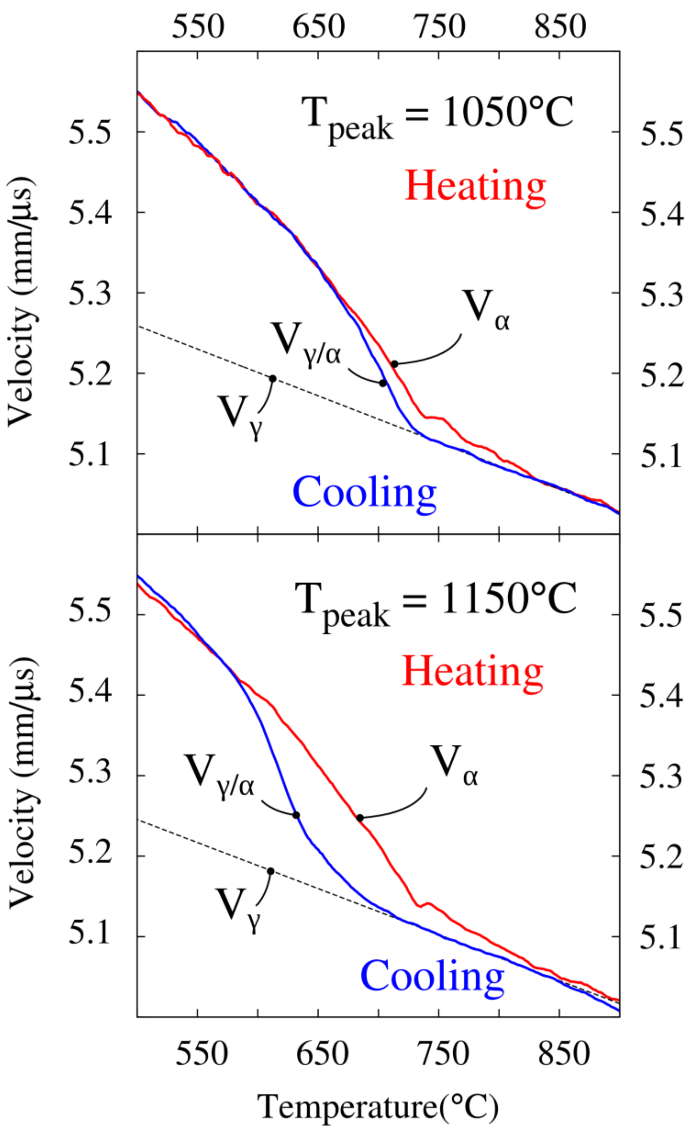
Monitoring of phase transformation

- Velocity difference between ferrite and austenite

$$v \cong \sqrt{\frac{\lambda + 2\mu}{\rho}}$$

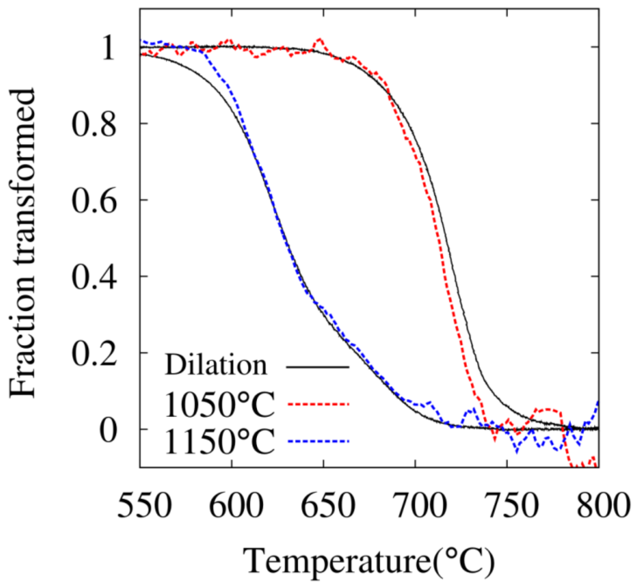
- Application of the lever-rule method on ultrasonic velocity

$$f_{\alpha} = \frac{v_{\gamma} - v_{\gamma/\alpha}}{v_{\gamma} - v_{\alpha}}$$

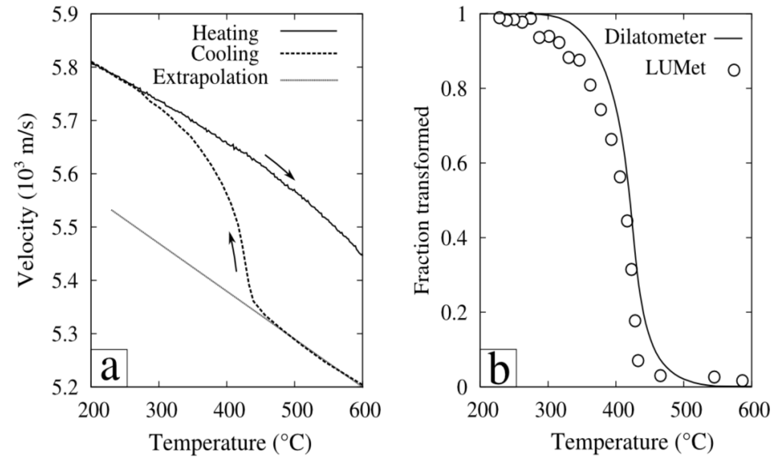


Fraction transformed

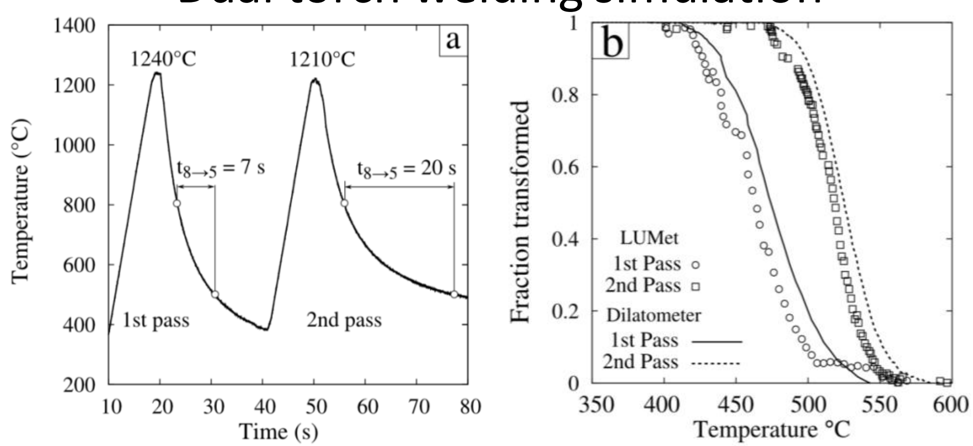
Continuous cooling rate 3,10 C/s



High cooling rate 150C/s



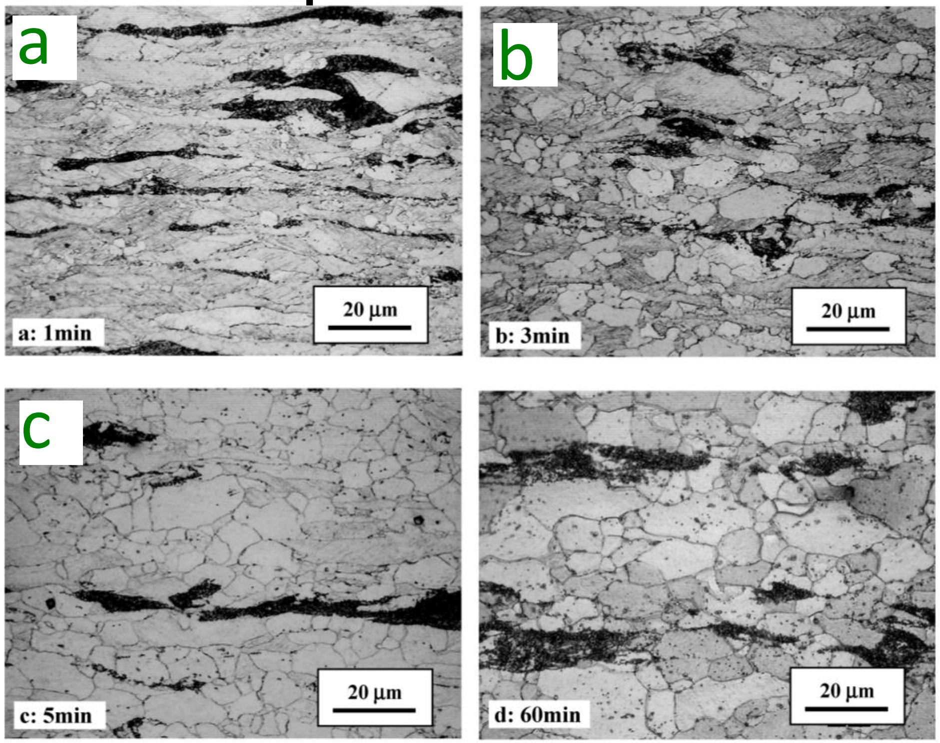
Dual torch welding simulation



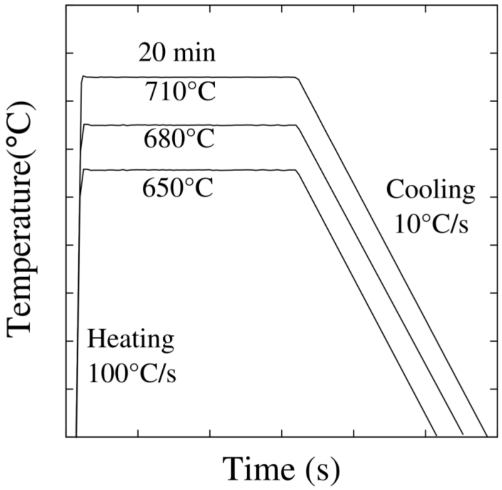
Monitoring of static recrystalization

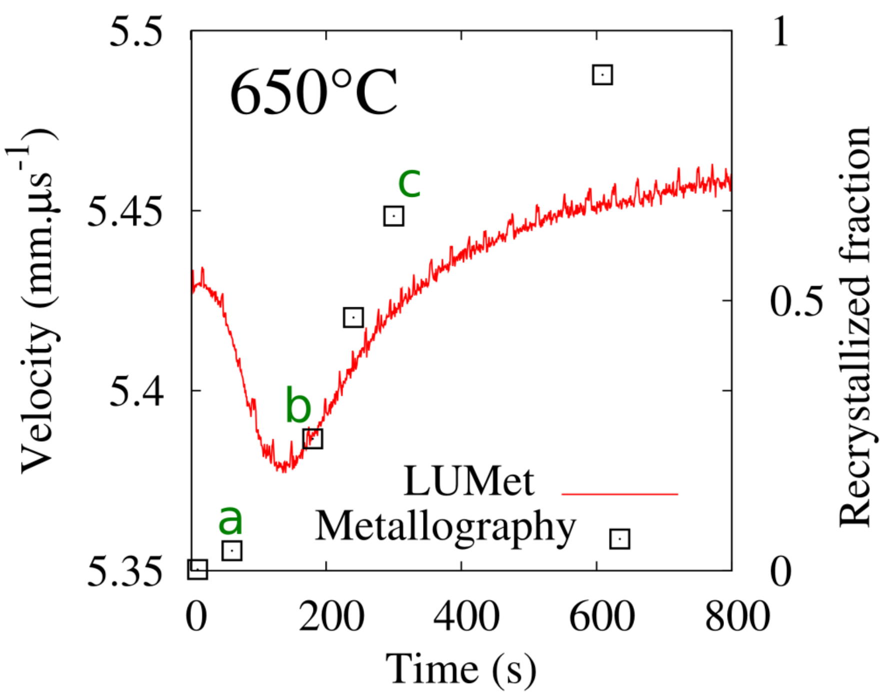
✓ Dual Phase Steel 55% Cold Rolled

Interrupt treatment at 650ºC

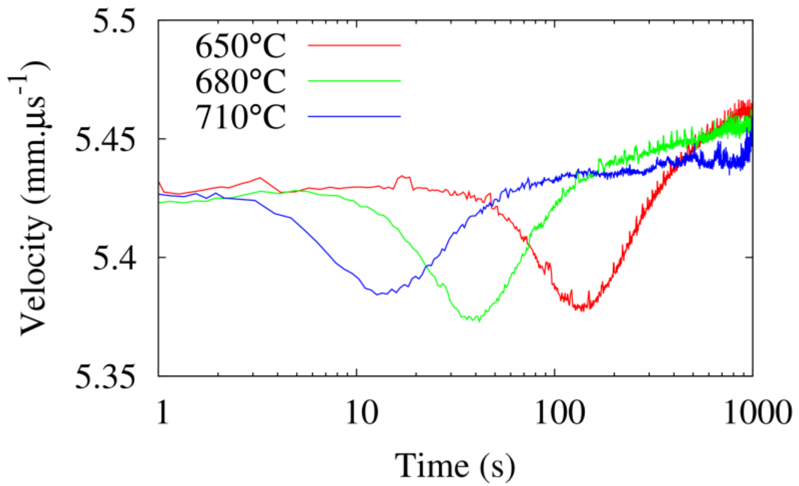


Huang J, Poole W J, Militzer M, Metall. Mater Trans.(2004) 35A, pp3363





Modified lever rule method



Segmentation of the velocity

↓

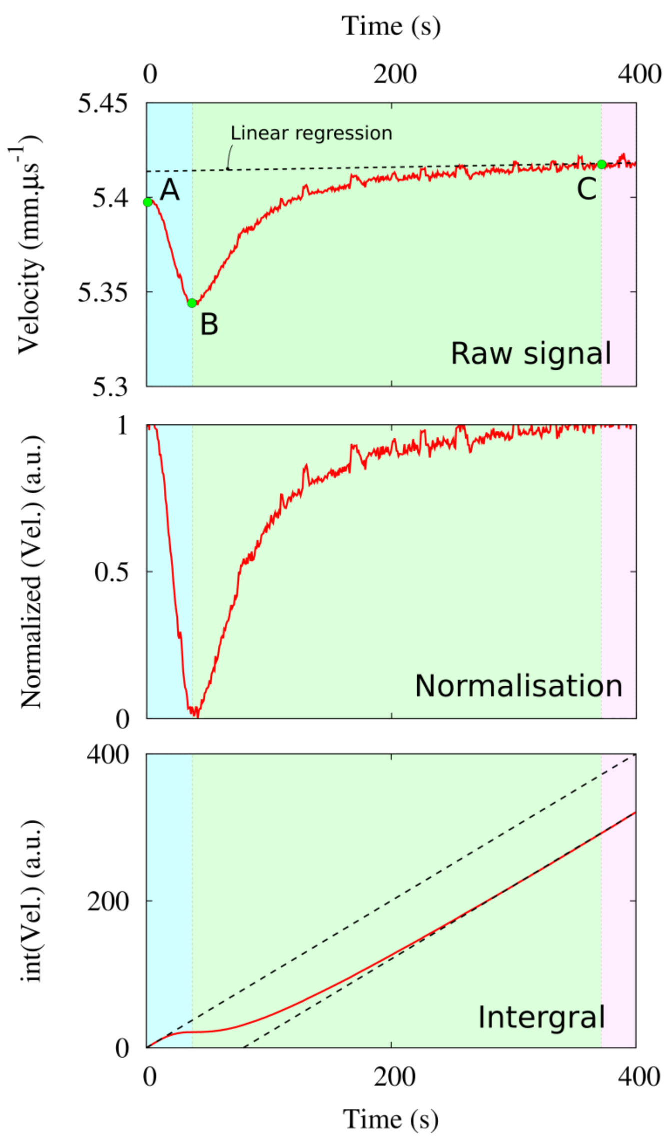
Normalisation of individual segment

↓

Integration of the normalized signal

↓

Application of the lever rule



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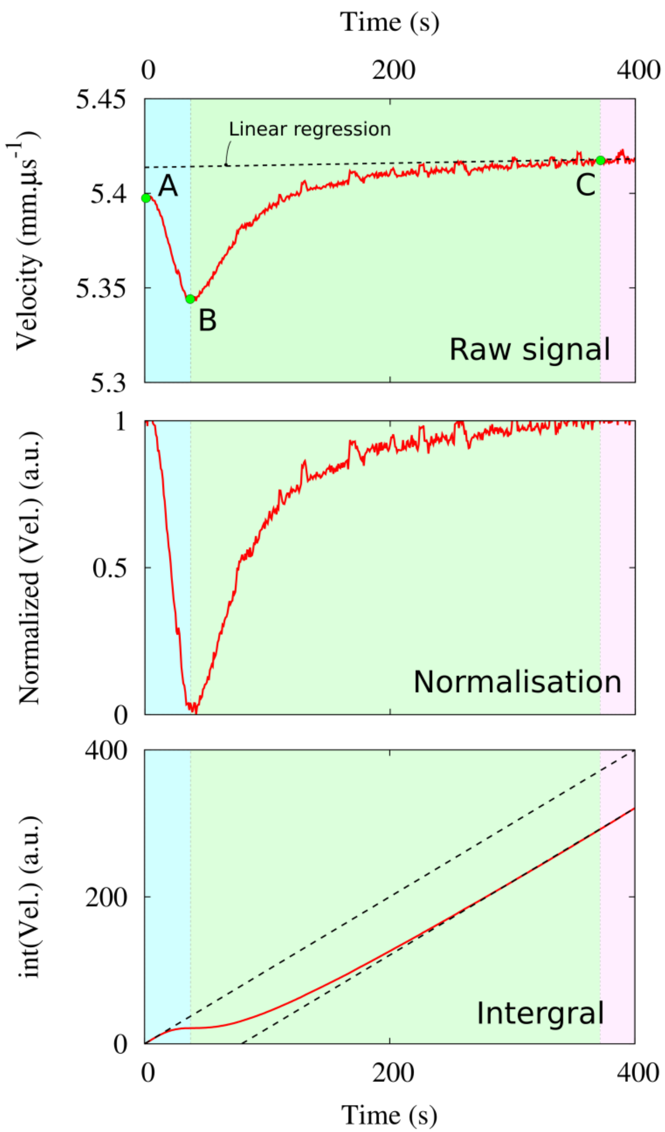
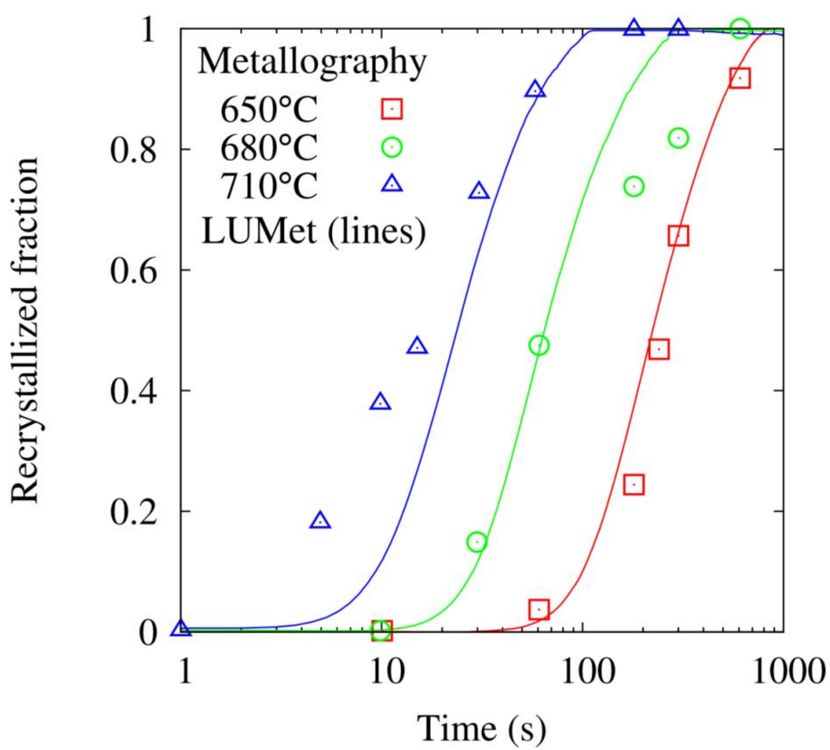
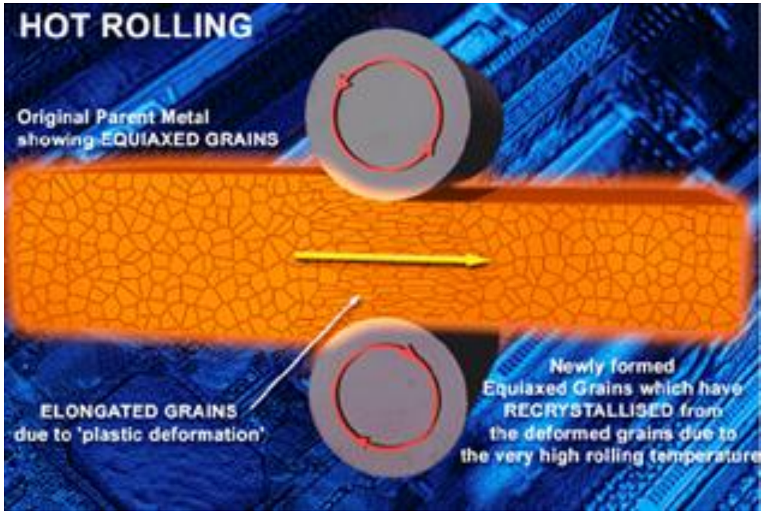


- ✓ Evolution of the recrystallized fraction validated with metallographic observations



Dynamic recrystallization in Mo-TRIP steel

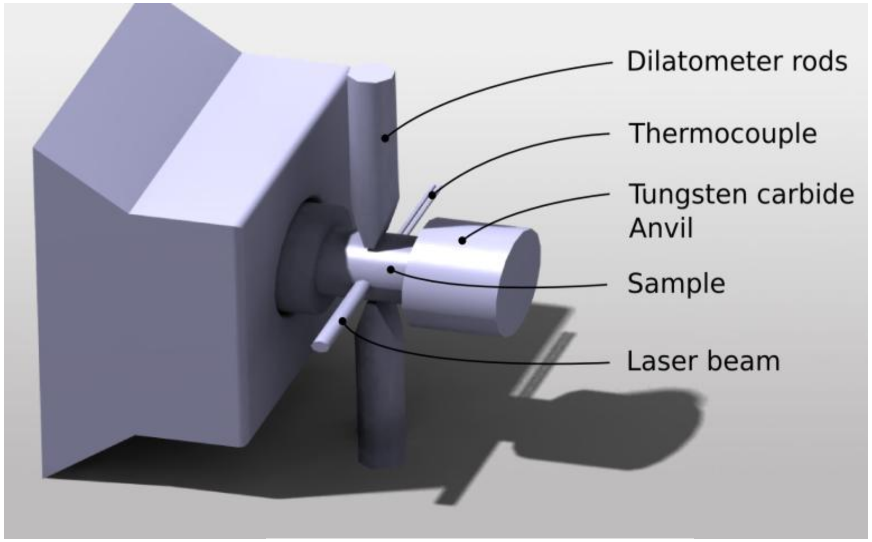
- ✓ Monitoring in-situ the microstructure evolution in steel after and during hot-deformation
- ✓ Work-hardening, recovery, recrystallization
- ✓ Static or dynamic processes
- ✓ Challenging processes to characterise in austenite with existing techniques
- ✓ What type of information can LUMet provide ?

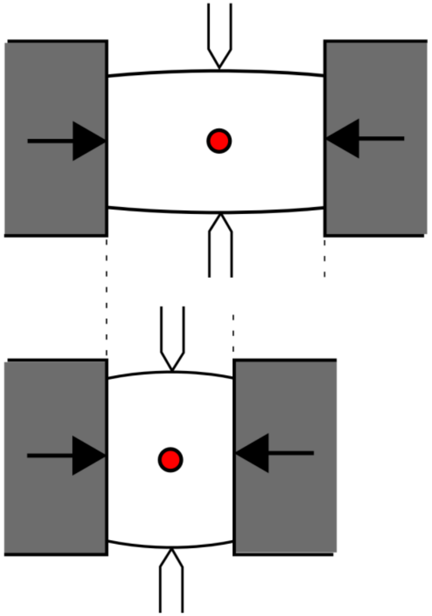


Caption:
Charles Sturt University, Sydney, Australia, Material Engineering

Material and setup

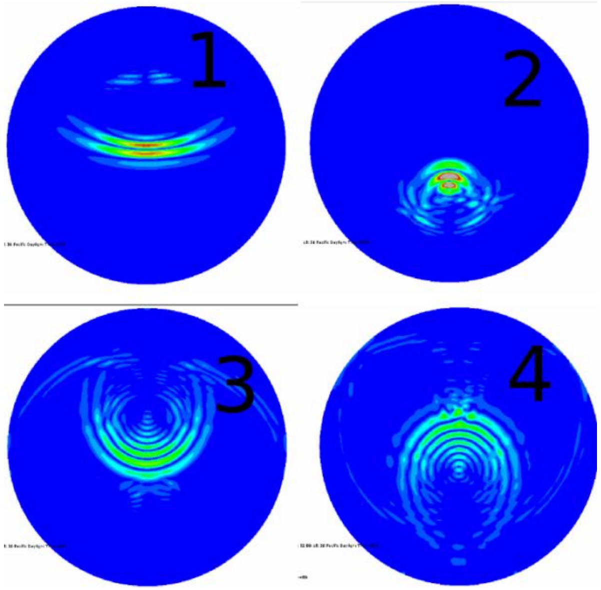
- ✓ Mo-TRIP, forged bar
- ✓ Composition (wt %) C0.19, Mn1.5, Si1.6,Mo0.2
- ✓ Cylindrical sample 10*15
- ✓ Uniaxial compression tests
- ✓ Translating laser table to follow the center of the sample

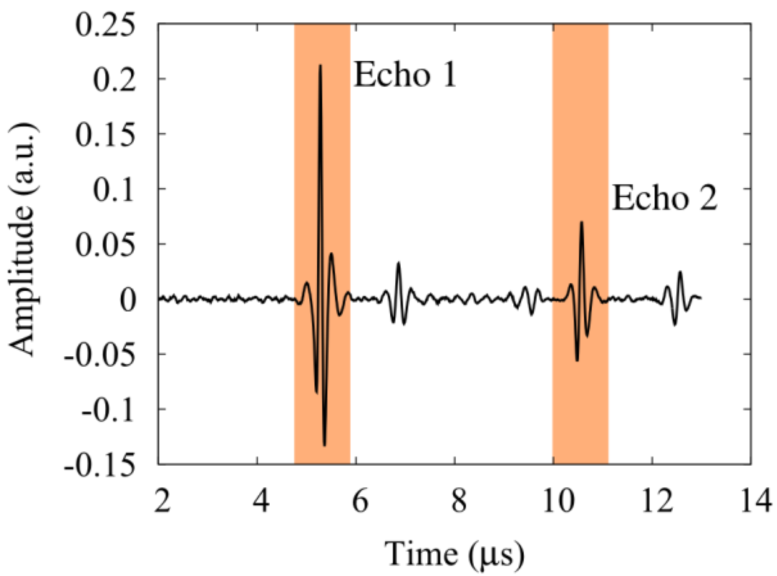




Complex waveform

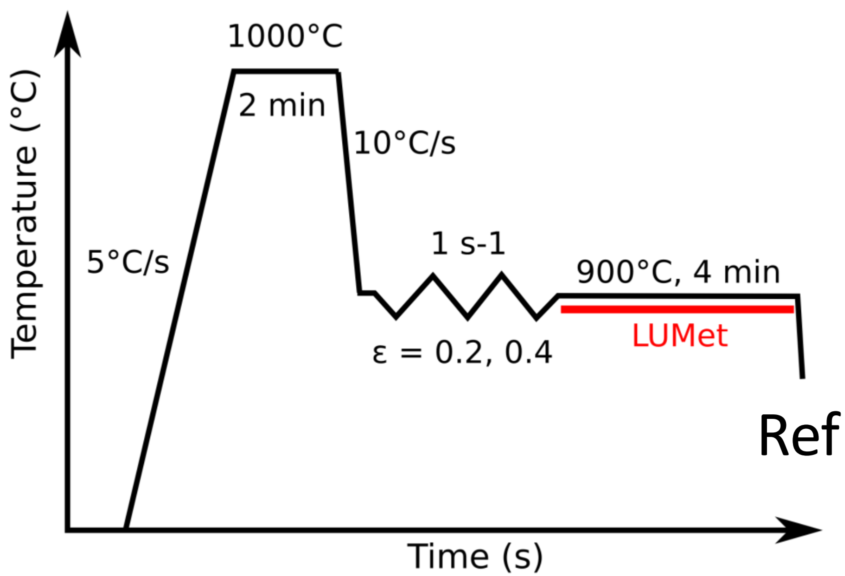
- ✓ Multiple acoustic waves are being generated and start to propagate in various directions
- ✓ High intensity pressure pulse travels across the diameter
- ✓ Surface wave of lower amplitude propagates around the circumference
- ✓ Lower amplitude pressure waves reflect and mode-convert into shear waves

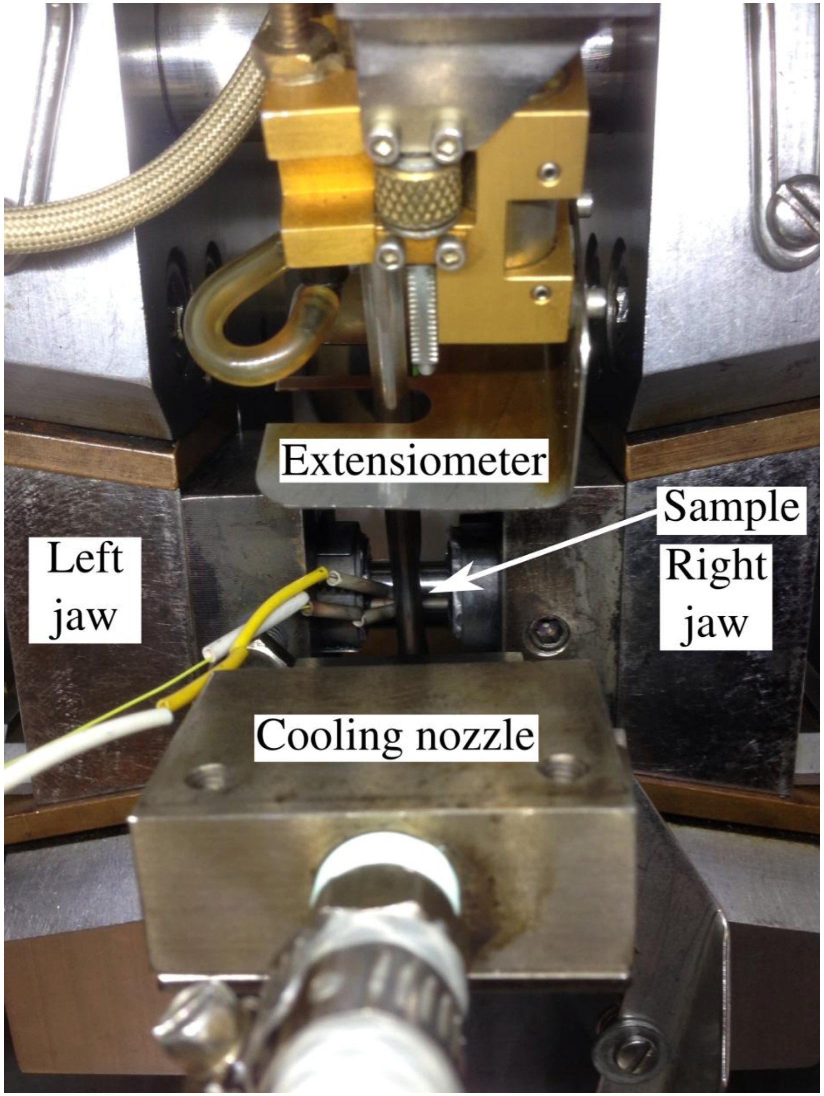




Static recrystallization in austenite

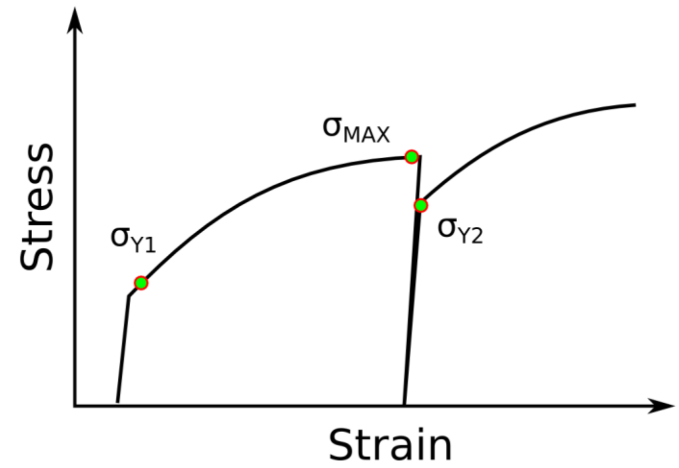
- ✓ Grain size measurement after hot-deformation
- ✓ Strain = 0.2 and 0.4
- ✓ Single echo technique + Available austenite calibration



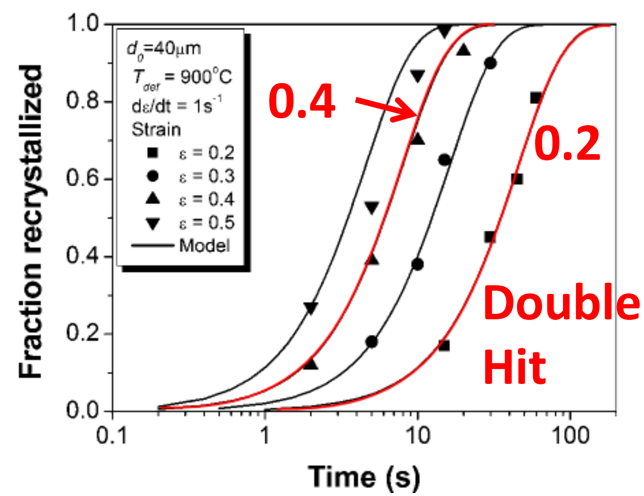


Recrystallization kinetics

- ✓ Initial austenite grain size prior to deformation: 40 µm (*Liu.D et al. Met. Mater. Trans 38A, 2007, pp 897*)
- ✓ Recrystallization kinetics measured from interrupt compression test (double hit tests).

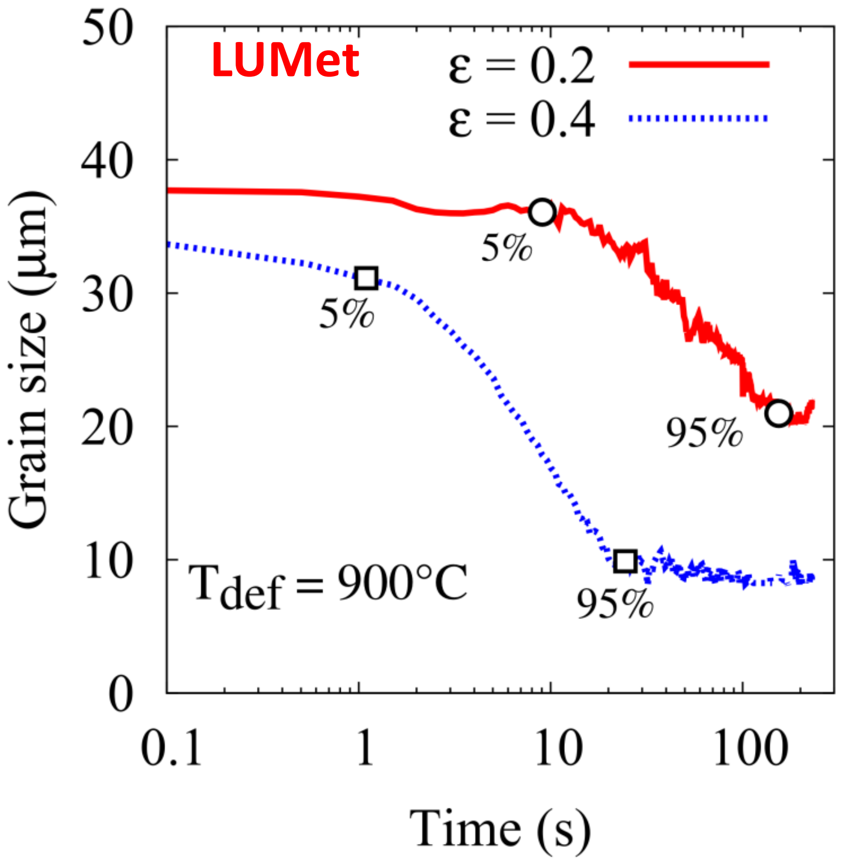


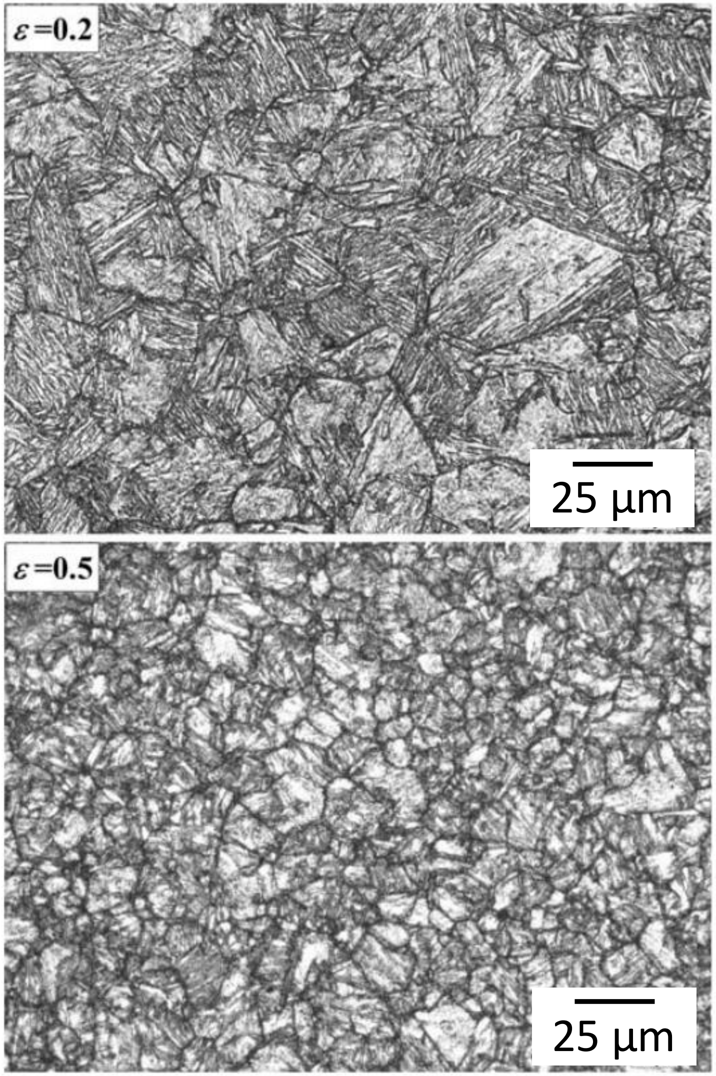
$$F_s = \frac{\sigma_{\max} - \sigma_{Y2}}{\sigma_{\max} - \sigma_{Y1}}$$



Austenite grain size evolution

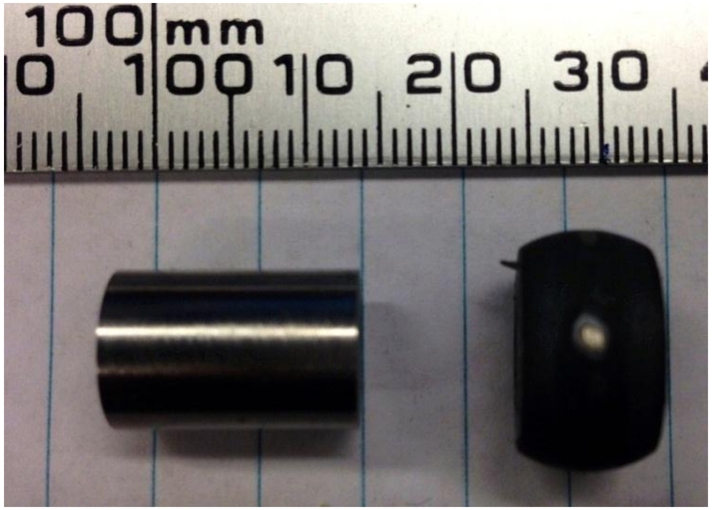
- ✓ Larger grain refinement at higher deformation strain

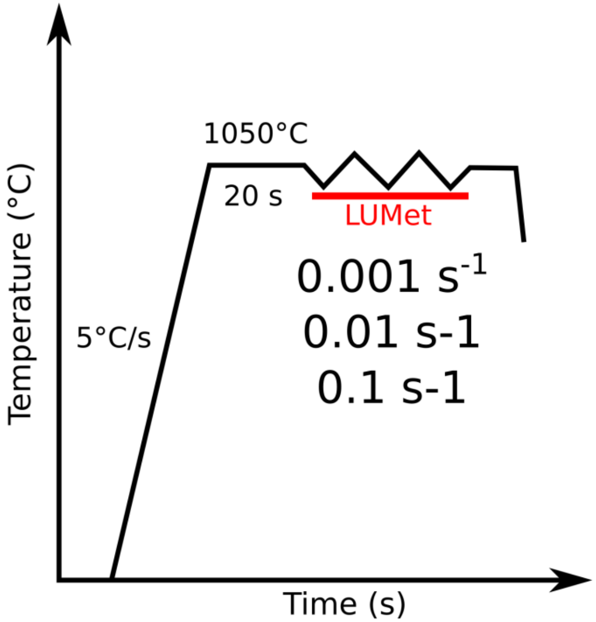


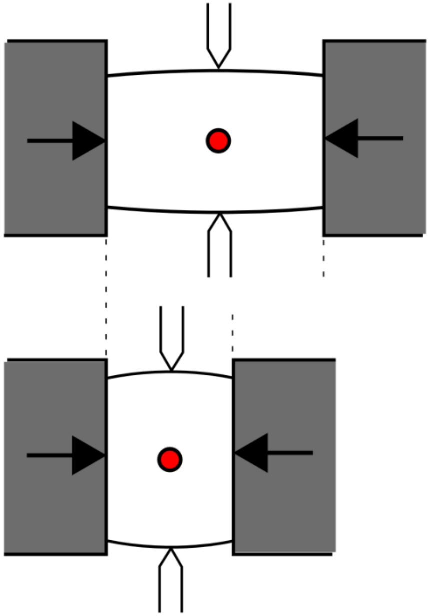


Hot-compression experiment

- ✓ Attenuation measurement during hot-compressions
- ✓ 3 strain rates



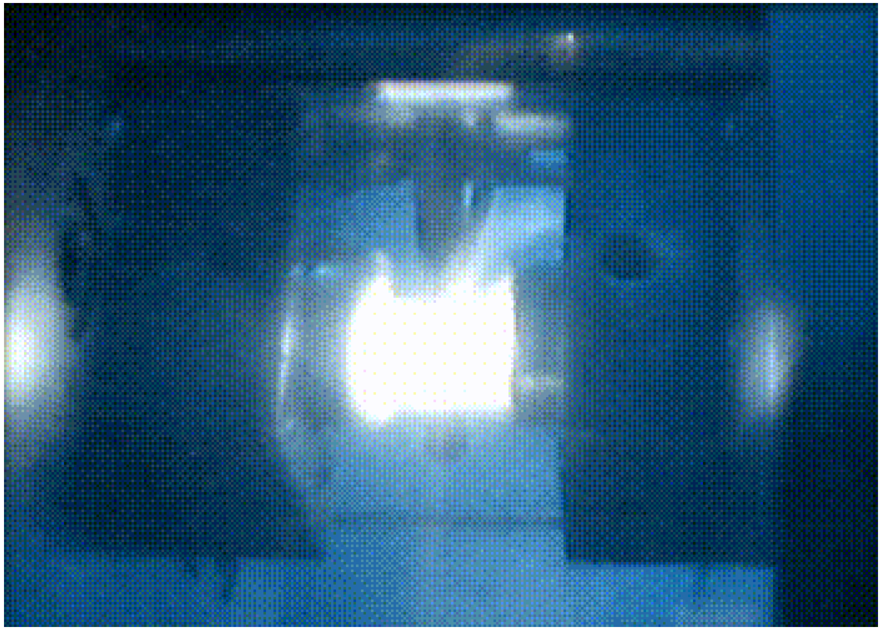


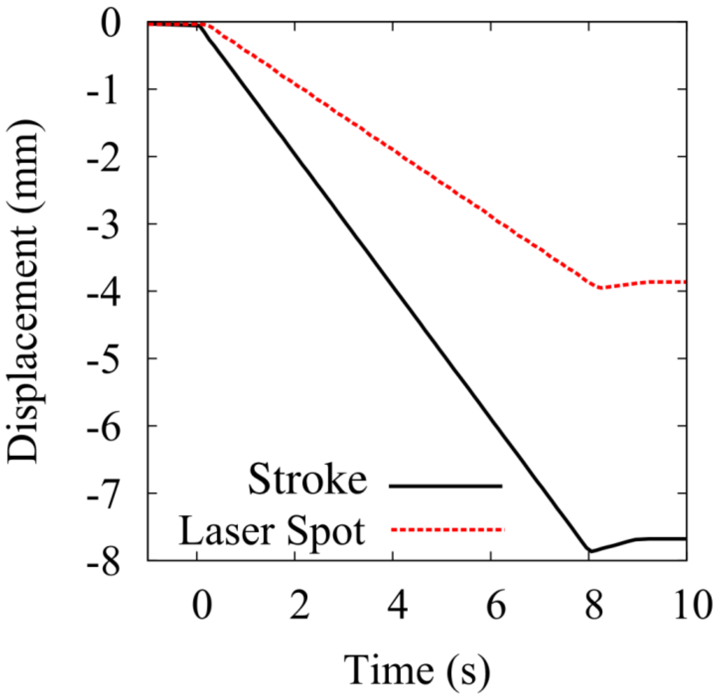


In-situ monitoring during deformation

✓ Successfully follow the center of the sample up to a rate of 1mm/s^{-1}

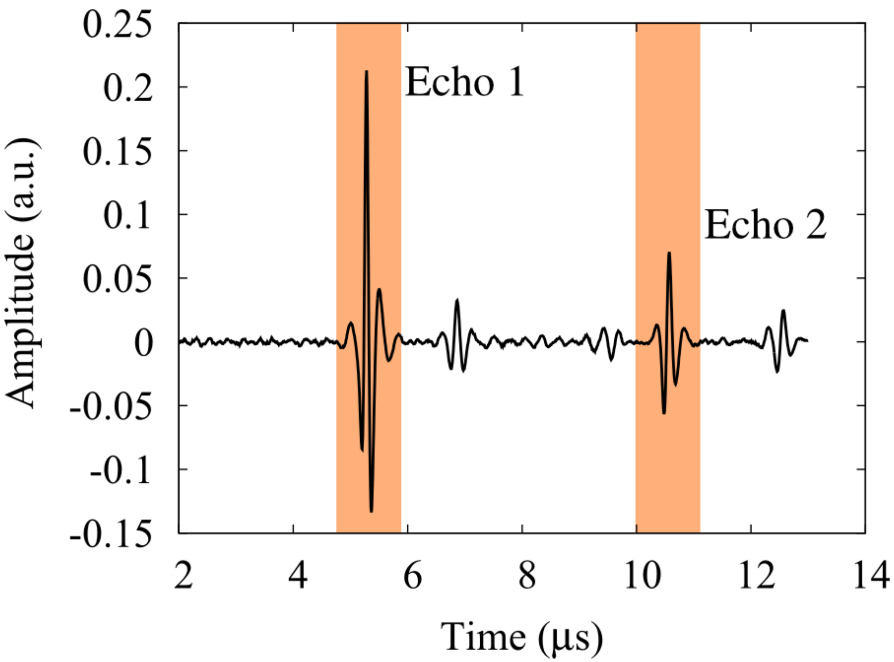
Video of in-situ LUMet inspection





Analysis methodology

- ✓ Sample is continuously changing in shape
- ✓ No reference microstructure can be obtained
- ✓ Attenuation calculated by the ratio of amplitude spectrum of first and second echoes
- ✓ Variation of attenuation at a single frequency

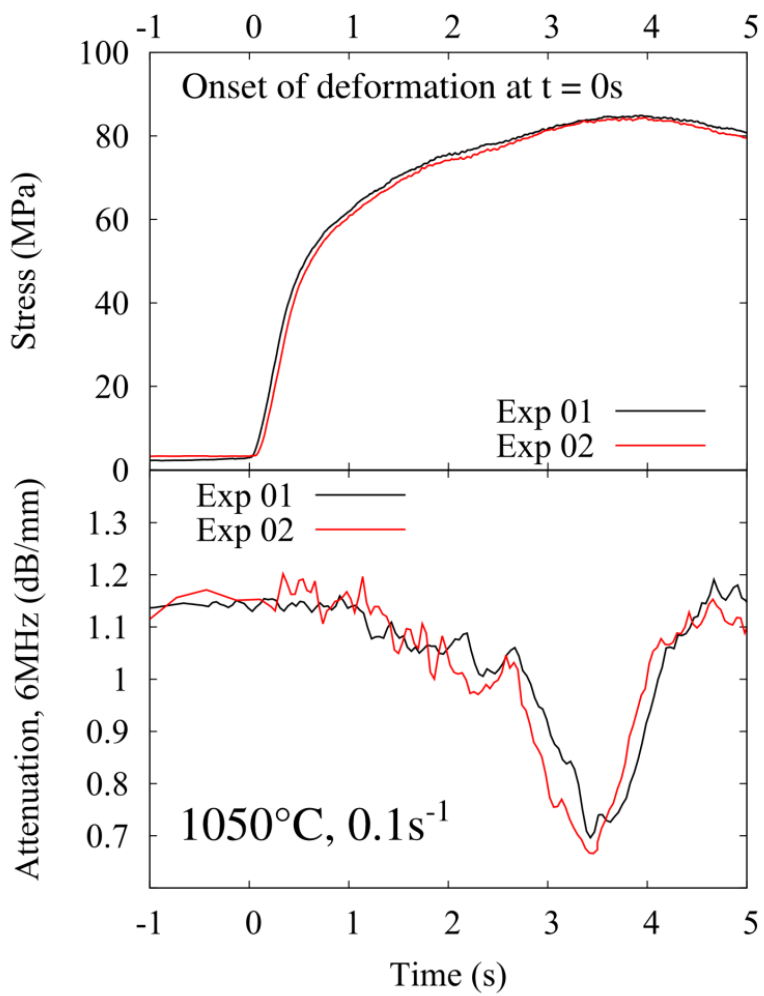


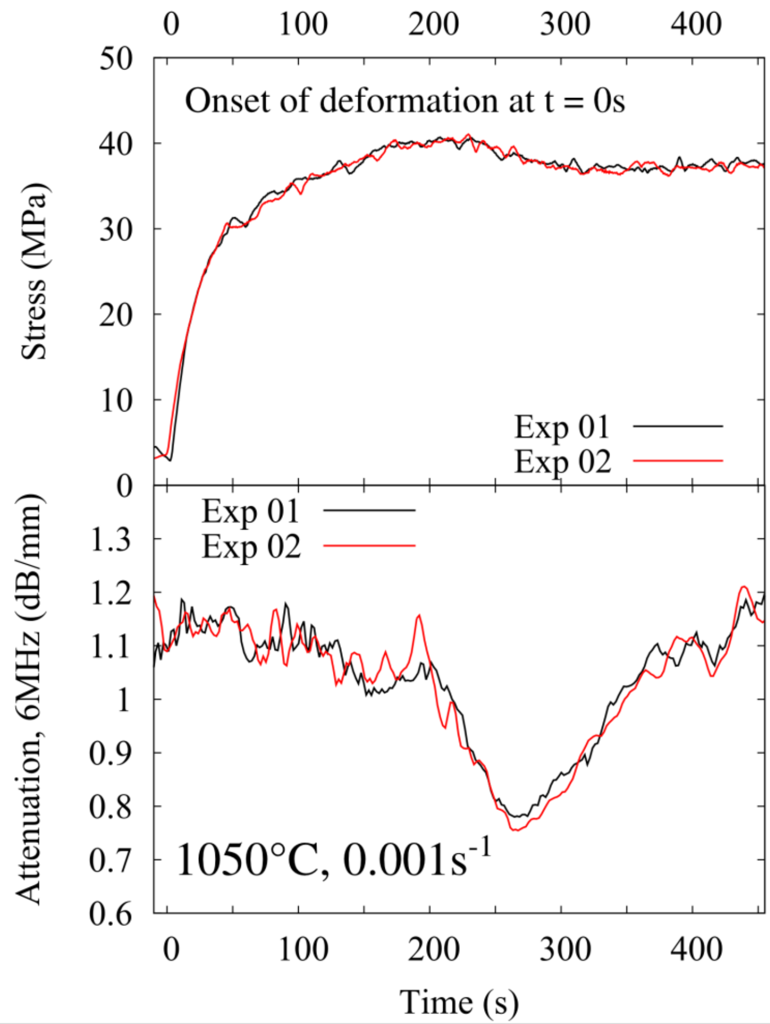
$$\alpha(f) = -\frac{20}{2D(1+\varepsilon)} log10\left(\frac{1}{g}\frac{A_2(f)}{A_1(f)}\right)$$

$$v = \frac{2(D+\xi)}{\tau}$$

Attenuation change during deformation

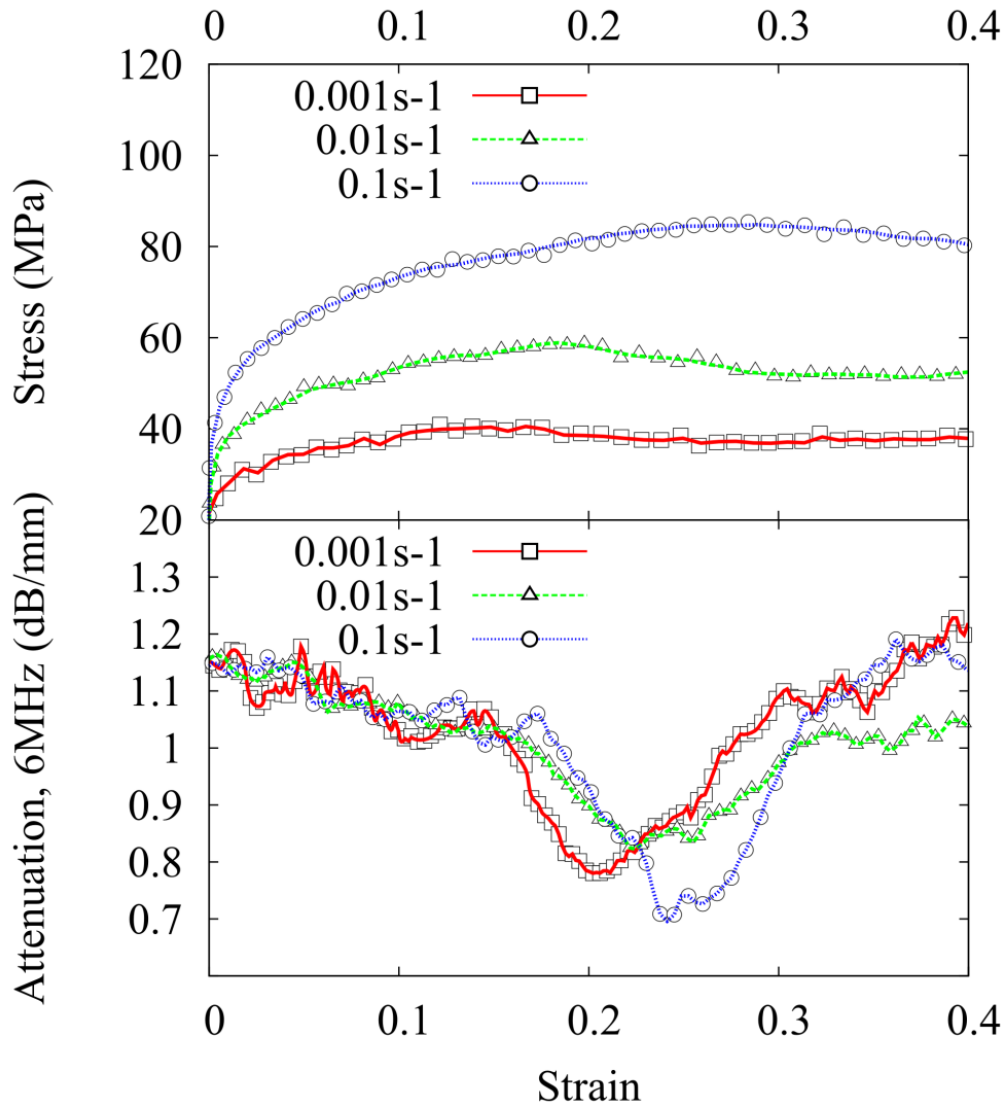
- ✓ Measurements are reproduced to test repeatability





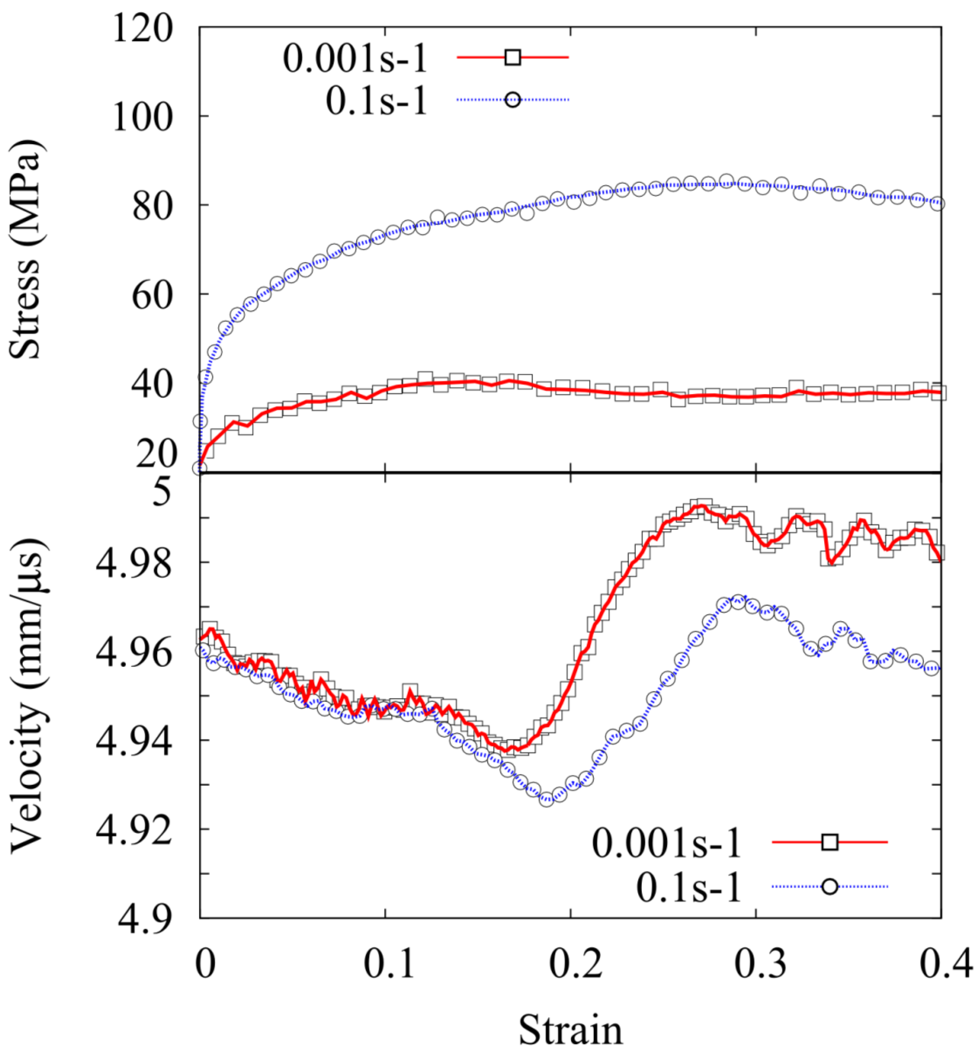
Effect of strain rate on attenuation

- ✓ Higher strain rate delays the onset of dynamic recrystallization.
- ✓ 1/ Attenuation decreases at a constant rate
  2/ later, marked decrease
  3/ increases again at decreasing rate
- ✓ Minimum of attenuation is shifted toward higher strain



Effect of strain rate on velocity

- ✓ When recrystallization occurs, this should be associated to a change in the bulk texture in austenite
- ✓ Several stages are evidenced on the velocity curve during the deformation
- ✓ Transition depends on strain rate



Simulation of ultrasound propagation in anisotropic polycrystalline media

- ✓ Three main requisites
  - ✓ Materials properties (stiffness, density, grain size, morphology and crystallographic orientation)
  - ✓ Finite Element engine (Sample geometry, meshing, wave generation, propagation and detection of pulse)
  - ✓ Waveform analysing tool (Extract ultrasound pulse properties, velocities, attenuation spectrum)

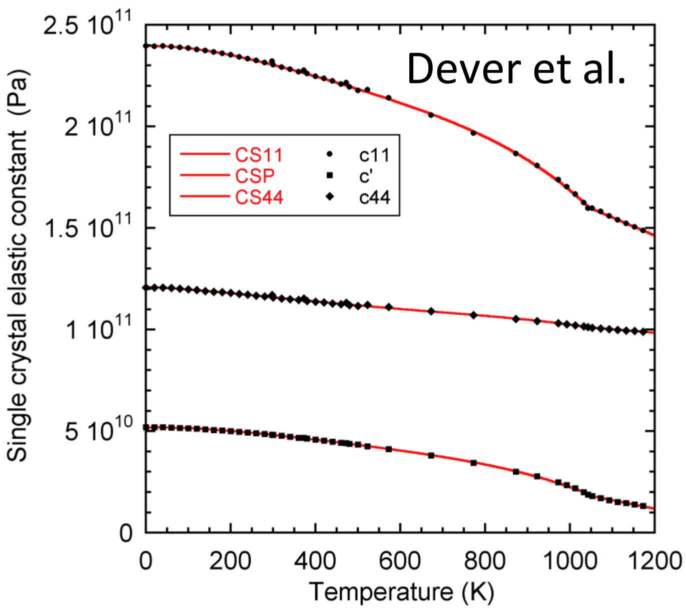
Materials properties

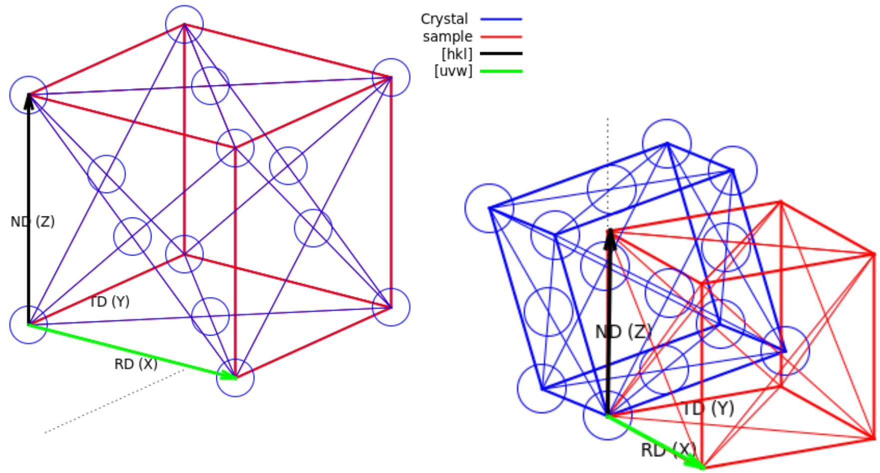
- ✓ BCC-iron (well documented, relatively highly anisotropic)

$$\boldsymbol{\sigma} = \boldsymbol{c} : \boldsymbol{\epsilon}$$

- ✓ Rotation of stiffness tensor (Euler's angles)

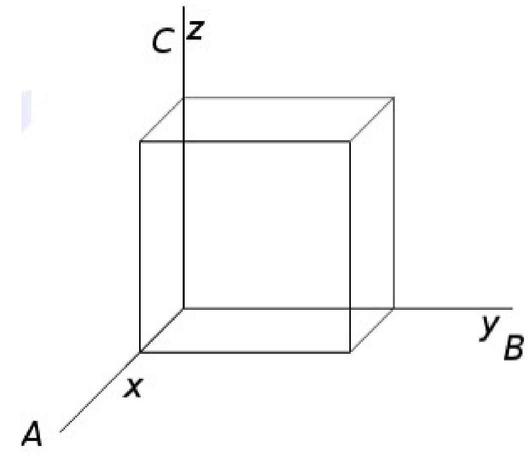
$$\boldsymbol{c}' = \boldsymbol{M}_{ZYZ}\, \boldsymbol{c}\ \ \bar{\boldsymbol{M}}_{ZYZ}$$





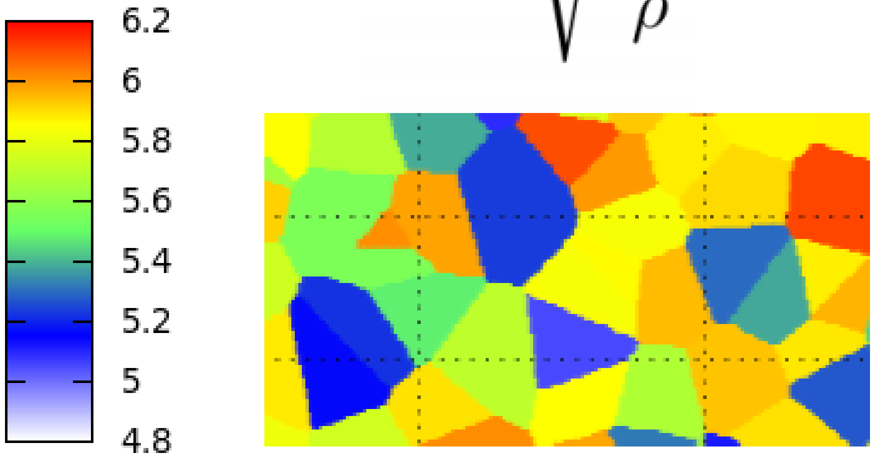
Crystal orientation, propagation direction

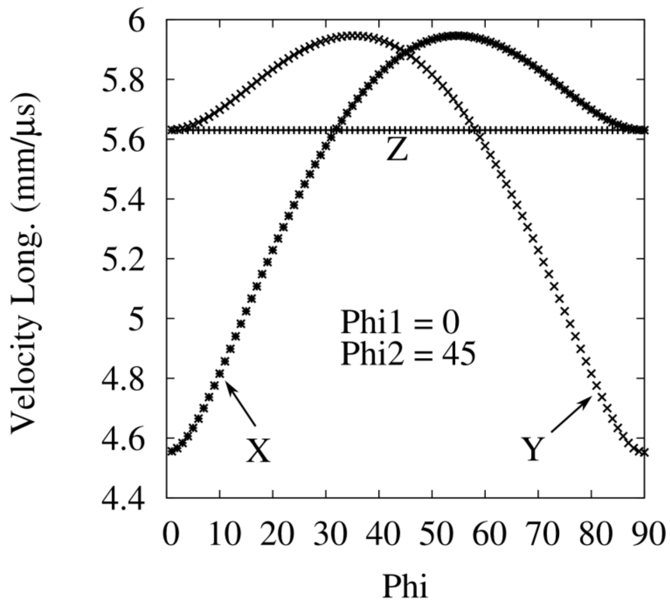
✓ Wave propagating in a particular direction of the sample



$$T_{ik}(\vec{n}) = C_{ijkl}\vec{n}_j\vec{n}_l$$

$$V_p = \sqrt{\frac{\lambda_1}{\rho}}$$





FEM calculation using ABAQUS

Dynamic explicit analysis

Dynamic equilibrium

$$\ddot{\boldsymbol{u}}^{(i)} = \boldsymbol{M}^{-1} \cdot \left(\boldsymbol{F}^{(i)} - \boldsymbol{I}^{(i)}\right)$$

$\boldsymbol{u}$: position of a node

i : increment number

$\boldsymbol{F}^{(i)}$: external applied forces

$\boldsymbol{I}^{(i)}$: internal element forces

Explicit integration

$$\dot{\boldsymbol{u}}^{\left(i+\frac{1}{2}\right)} = \dot{\boldsymbol{u}}^{\left(i-\frac{1}{2}\right)} + \frac{\Delta t^{(i+1)} + \Delta t^{(i)}}{2} . \ddot{\boldsymbol{u}}^{(i)}$$

$$\boldsymbol{u}^{(i+1)} = \boldsymbol{u}^{(i)} + \Delta t . \dot{\boldsymbol{u}}^{\left(i+\frac{1}{2}\right)}$$

Constitutive laws
+ boundary conditions
and loads

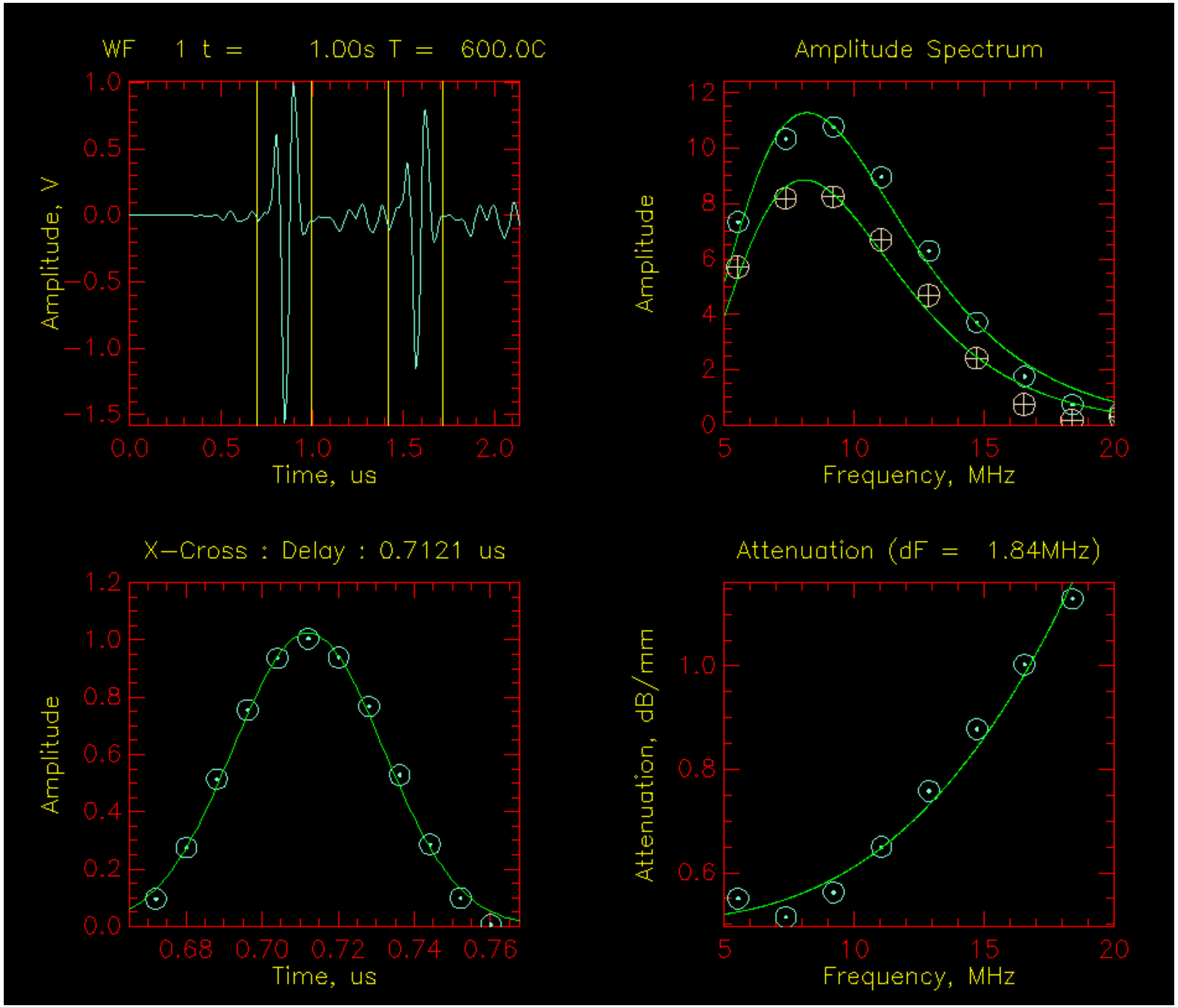
<u>Stability limit:</u>

$$\Delta t = min\left(\frac{L_e}{c_d}\right)$$

Waveform analysis

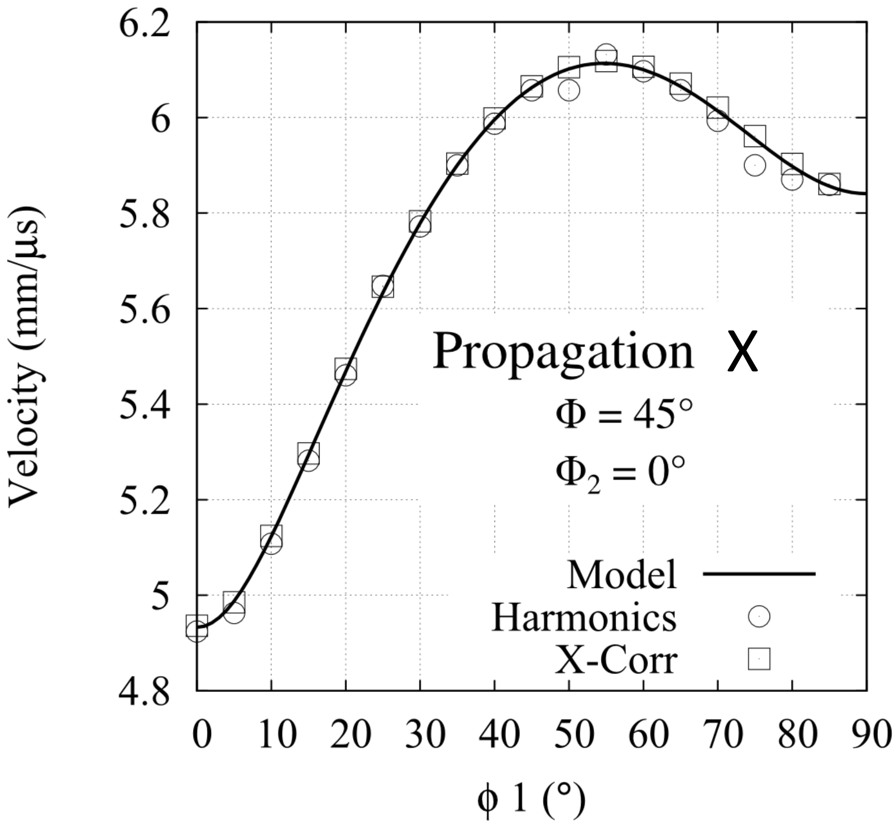
$$A = (1 - 2\pi^2 f^2 t^2) e^{-\pi^2 f^2 t^2}$$

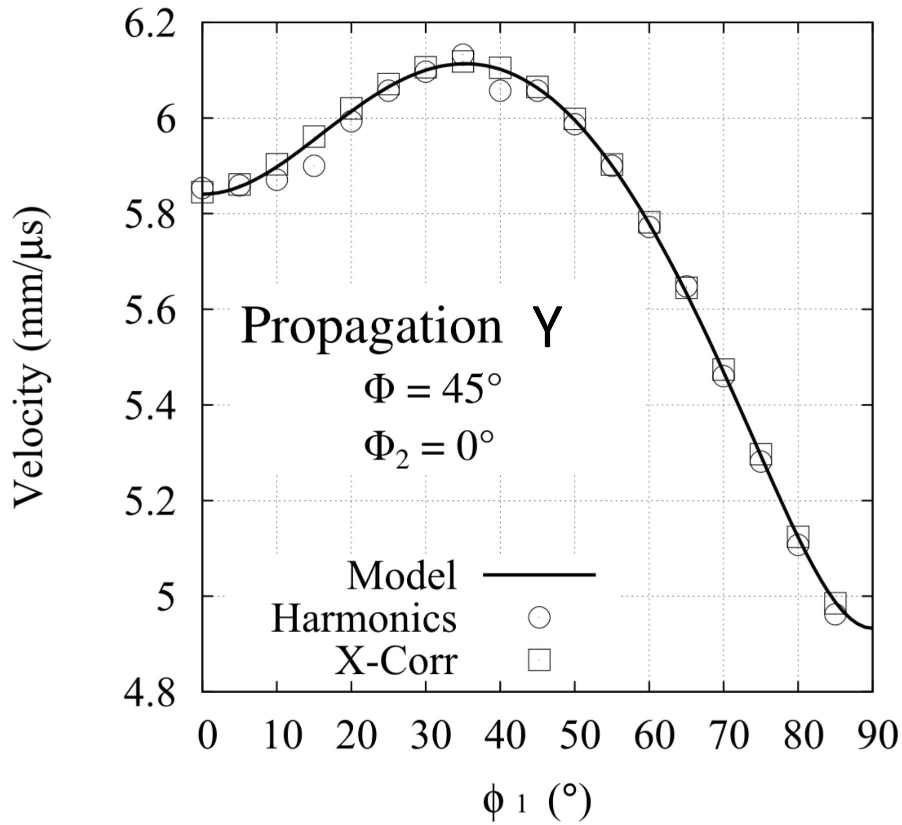
- ✓ Echo analysis
- ✓ Windowing, Cross-correlation, spectrum evaluation
- ✓ Velocity, attenuation



Ultrasound velocity in single crystal

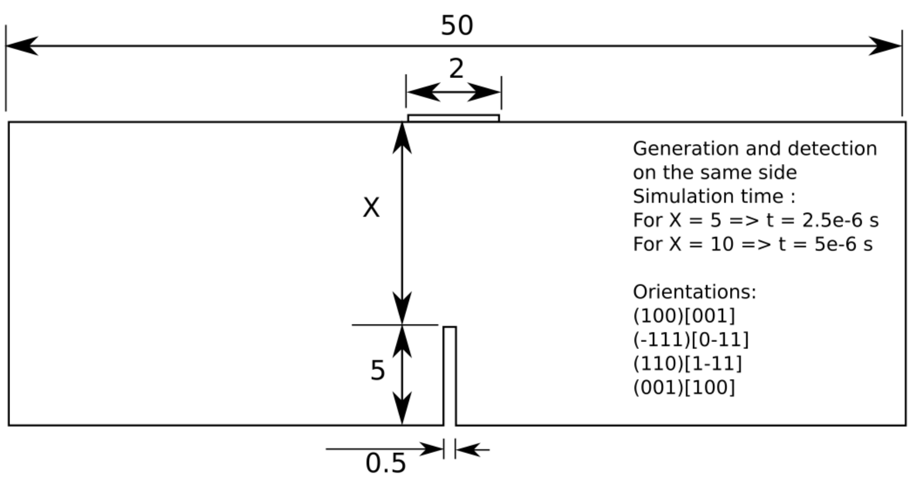
✓ Wave propagating along the X, Y in single crystal with specific crystallographic orientation

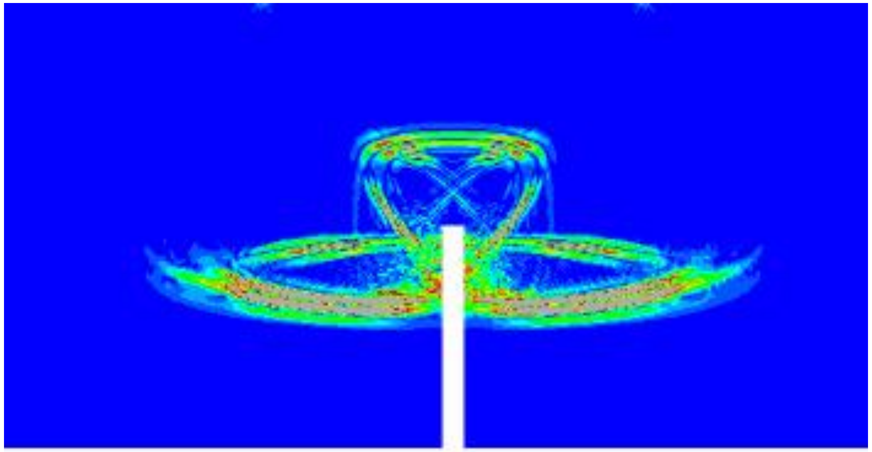




Ultrasound dispersion in single crystal

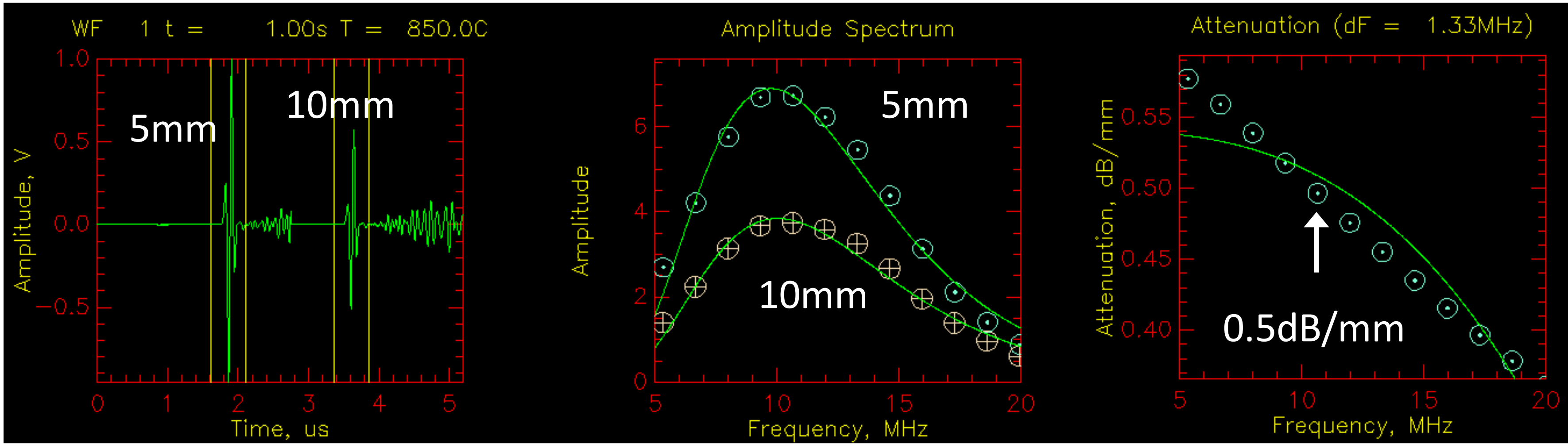
- ✓ Wave propagating in single crystal with specific crystallographic orientation
- ✓ Evaluation of the dispersion of the ultrasound pulse with respect to the crystallographic orientation



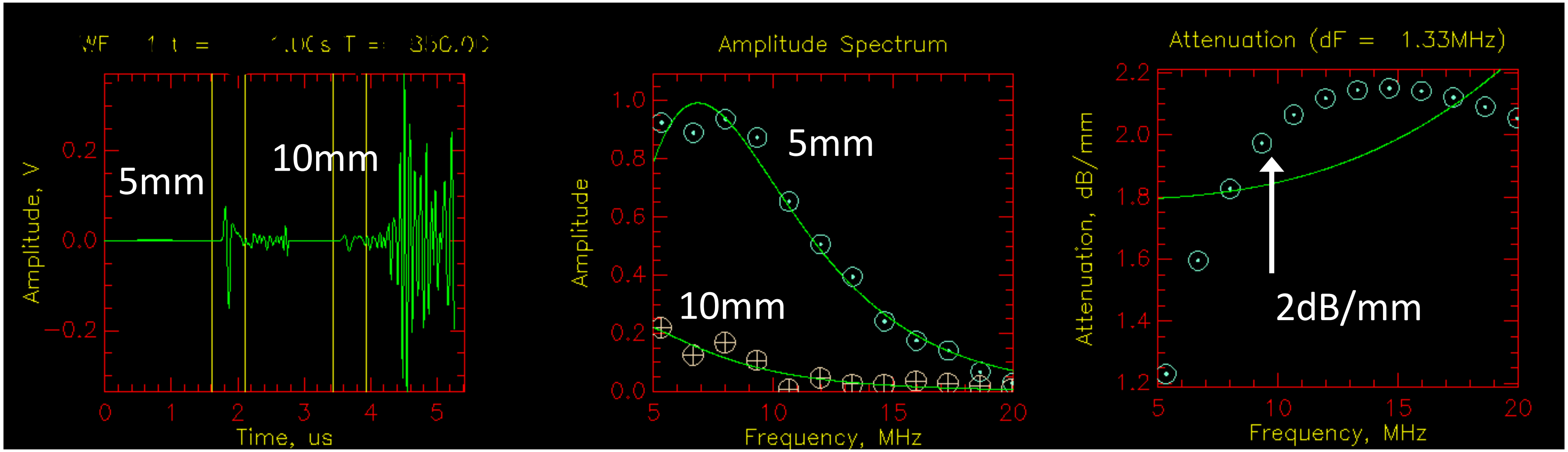


Ultrasound dispersion in single crystal

✓ Orientation (1 0 0) [0 0 1] : Weak dispersion

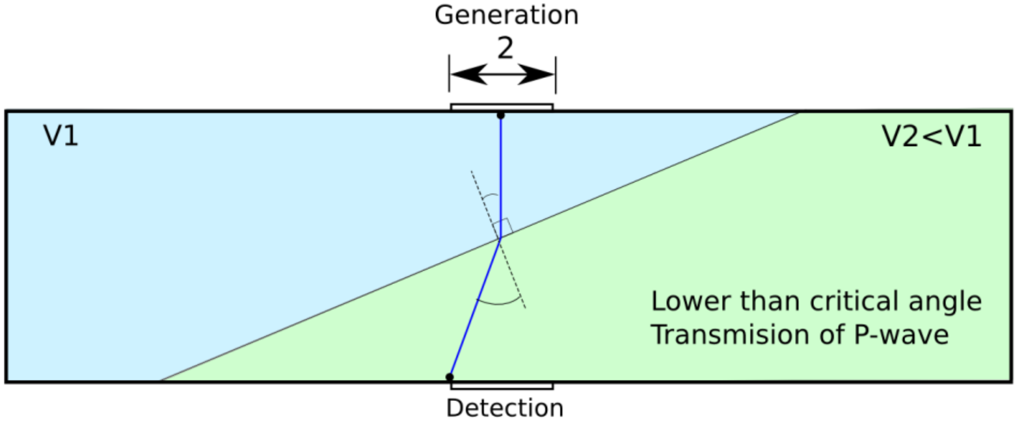


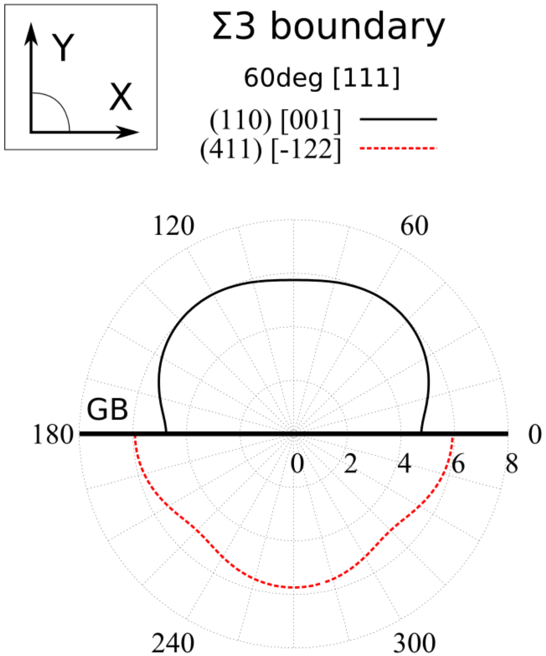
✓ Orientation (1 1 0) [1 -1 1] : Strong dispersion

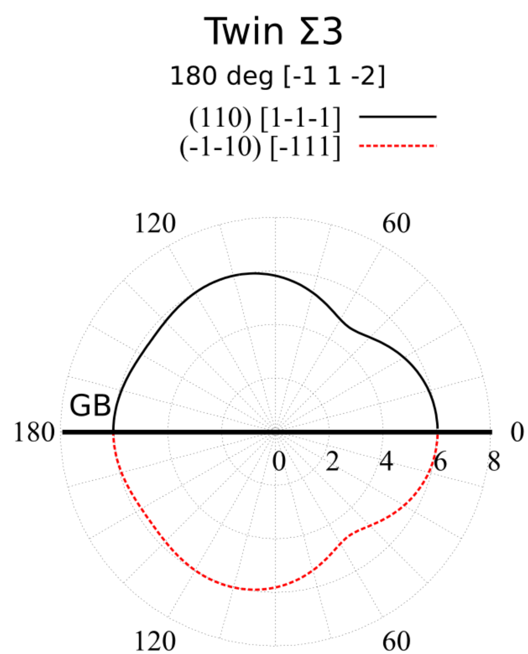


Bi-crystal: misorientation, incidence angle

- ✓ Study of incidence angle with grain boundary
- ✓ Effect of misorientation between two grains
- ✓ Anisotropic elastic mismatch between two grains

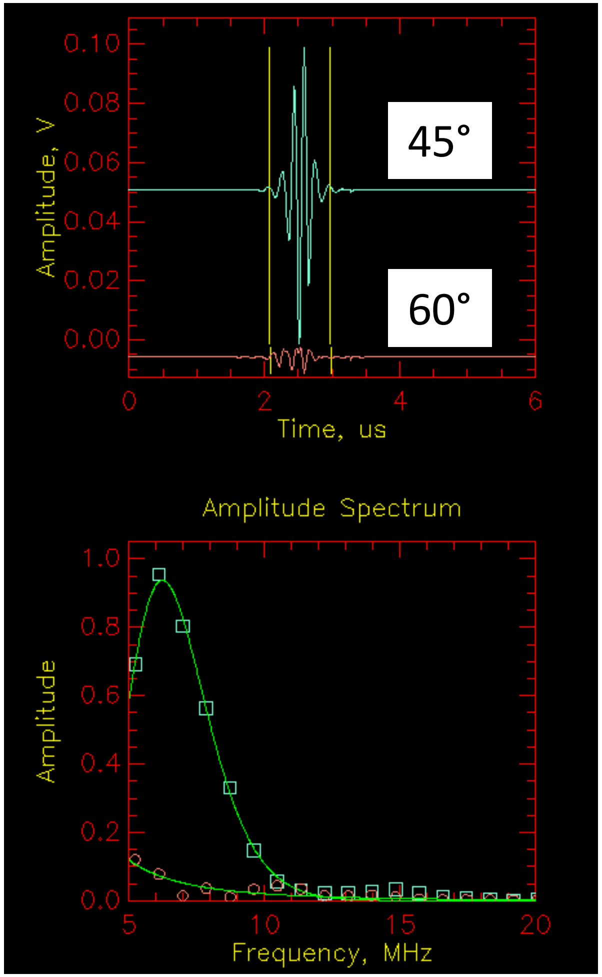


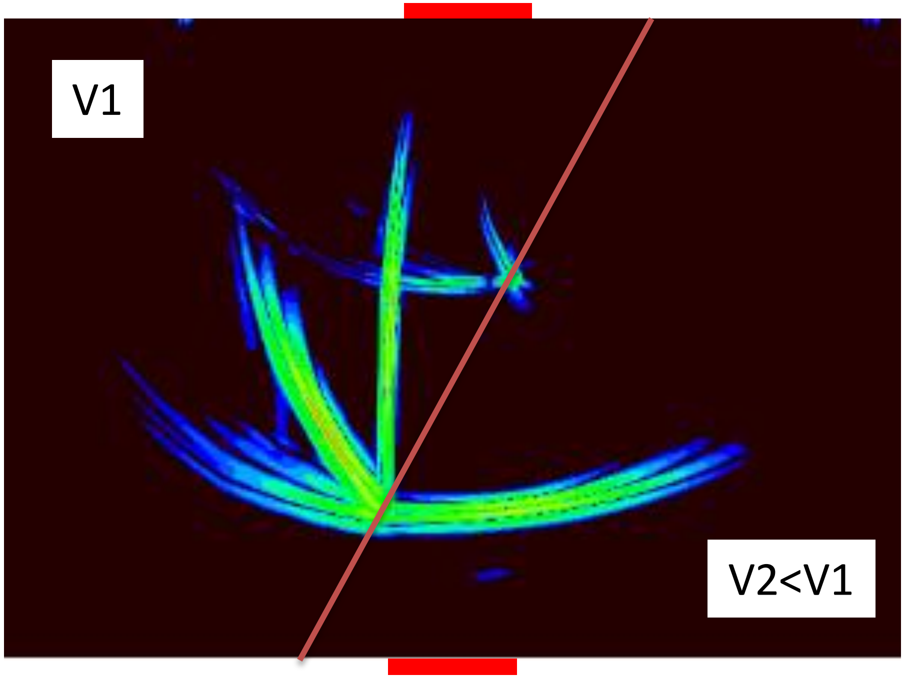




Bi-crystal: isotropic case

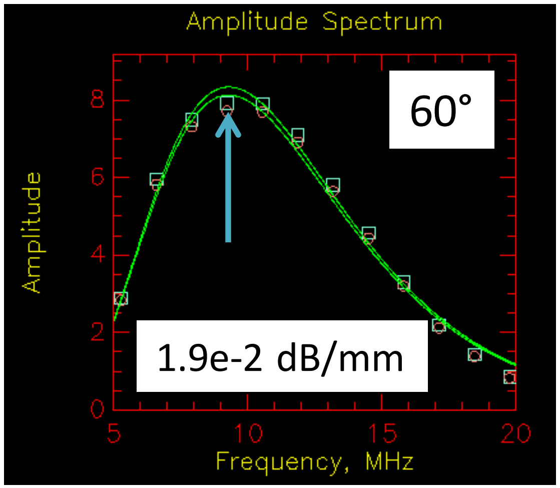
- ✓ Compare energy collected at 45° with higher incidence angle

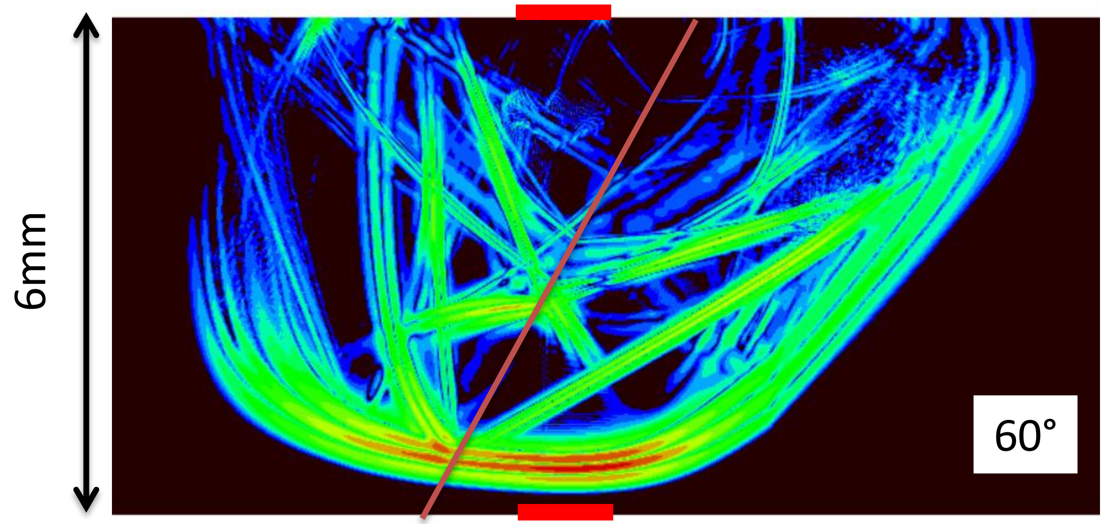


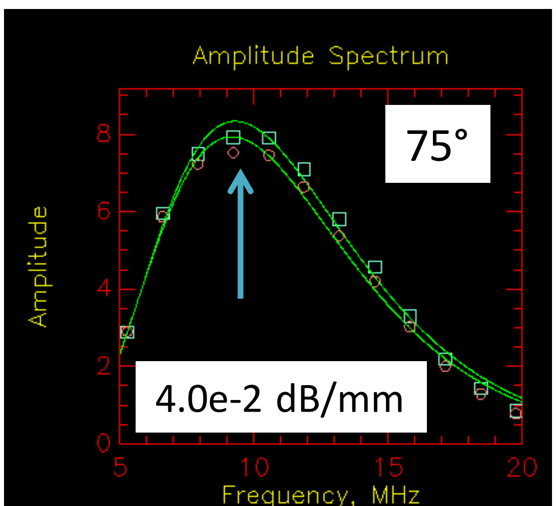


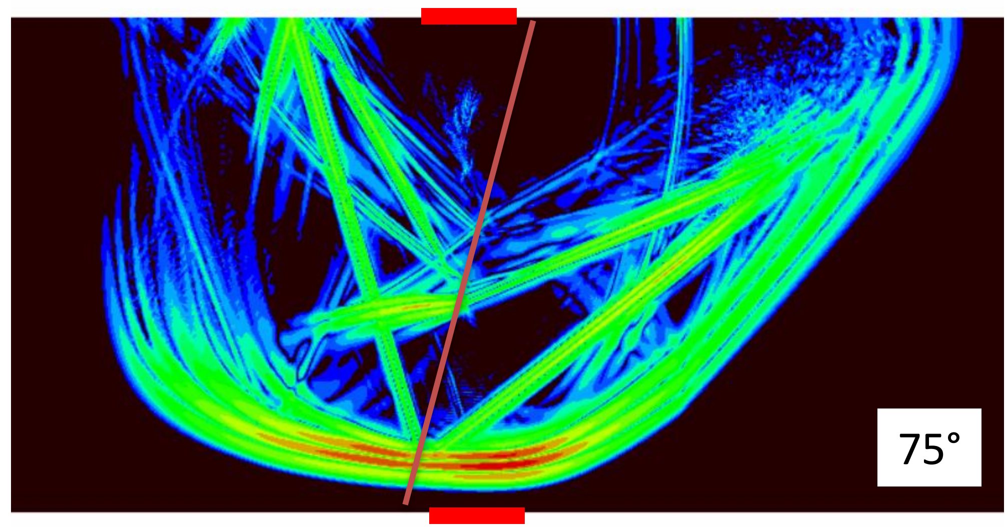
Bi-crystal: anisotropic case

- ✓ Twin boundary, Reference : Incidence angle 45°



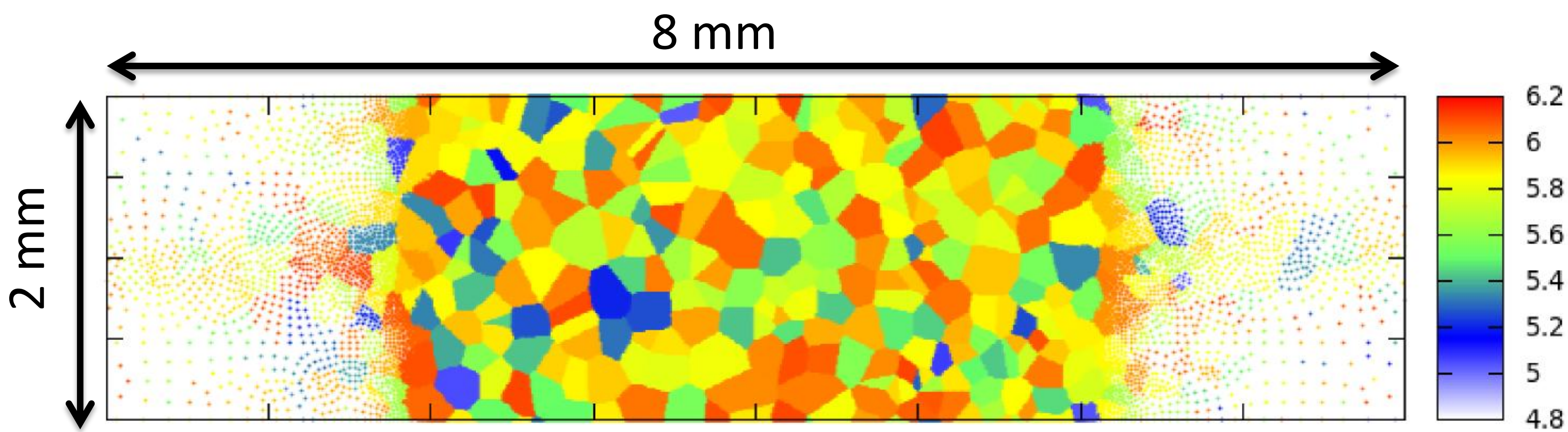






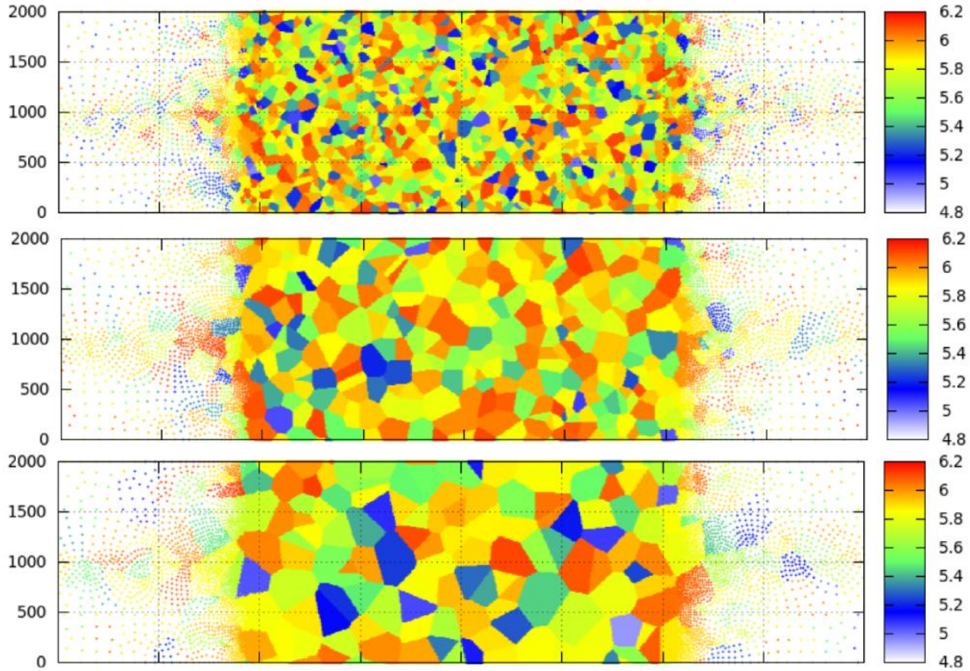
Polycrystalline material

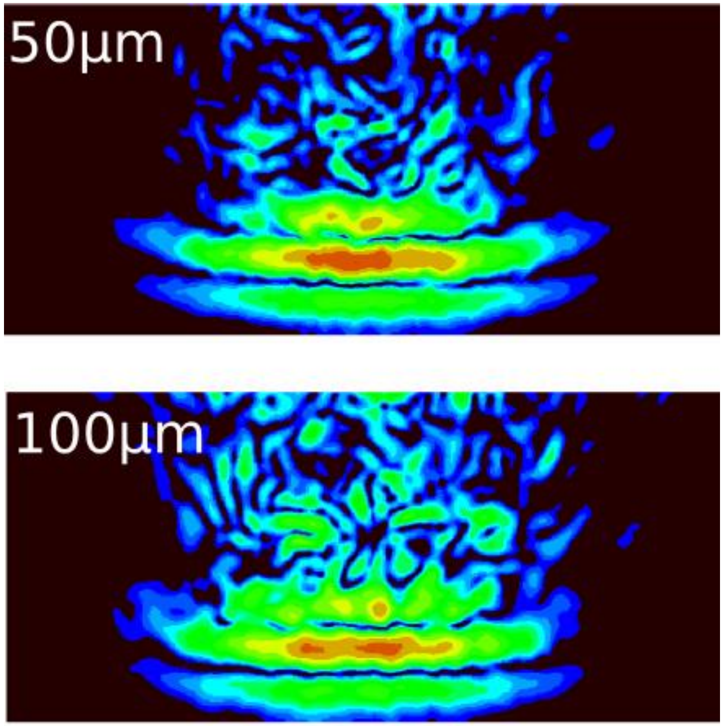
- ✓ Voronoi tessellation
- ✓ Attribution of random orientation (Rodrigues space, axis angle)
- ✓ Attribute rotated stiffness tensors to elements of the mesh for each grain.



Polycrystalline material

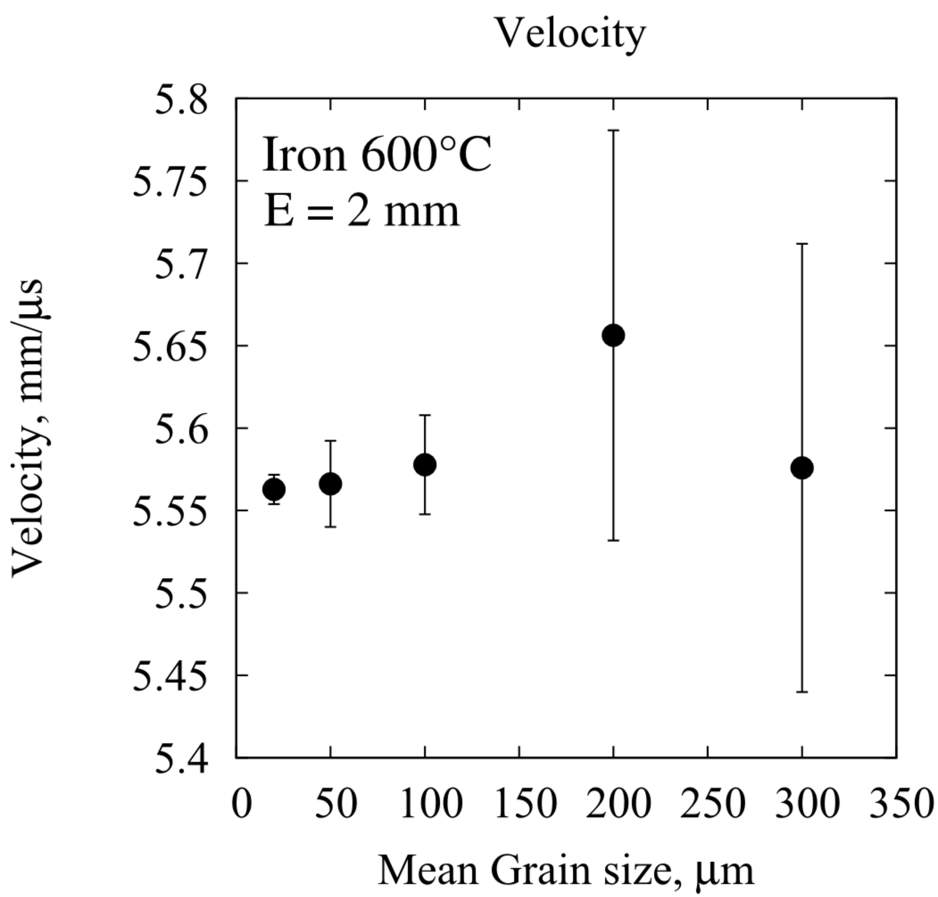
✓ Generation of grain size from 20 to 300 µm

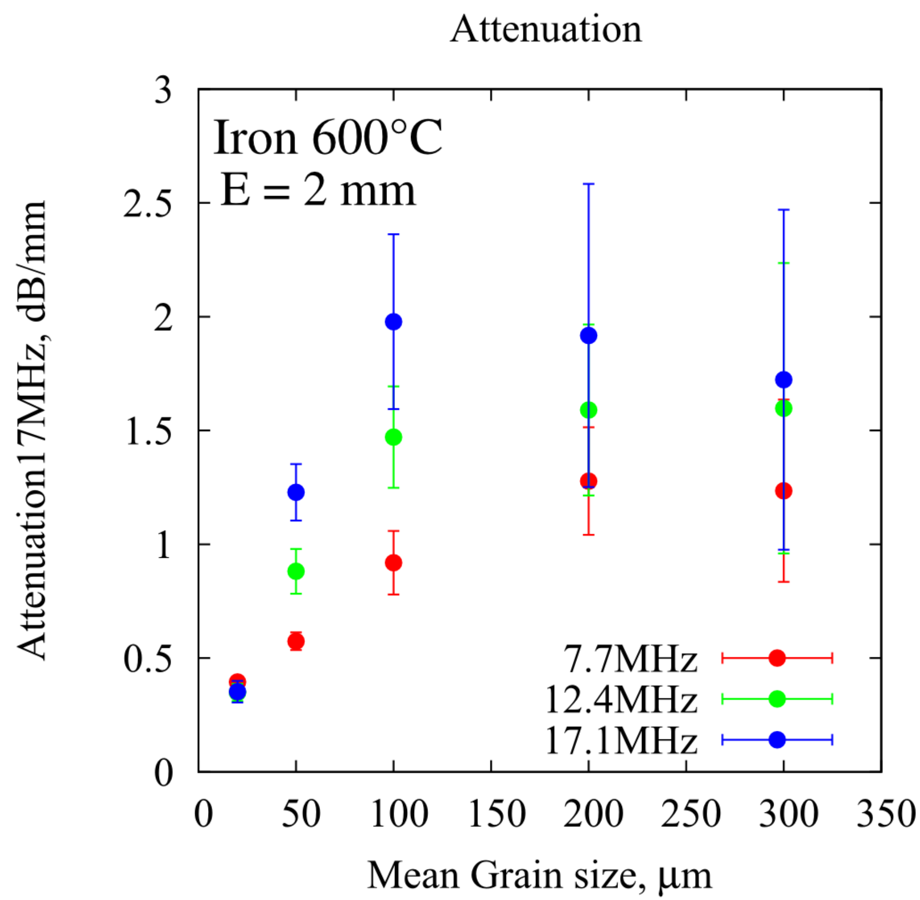




✓ Analysis of ultrasound properties after propagation in the generated structure

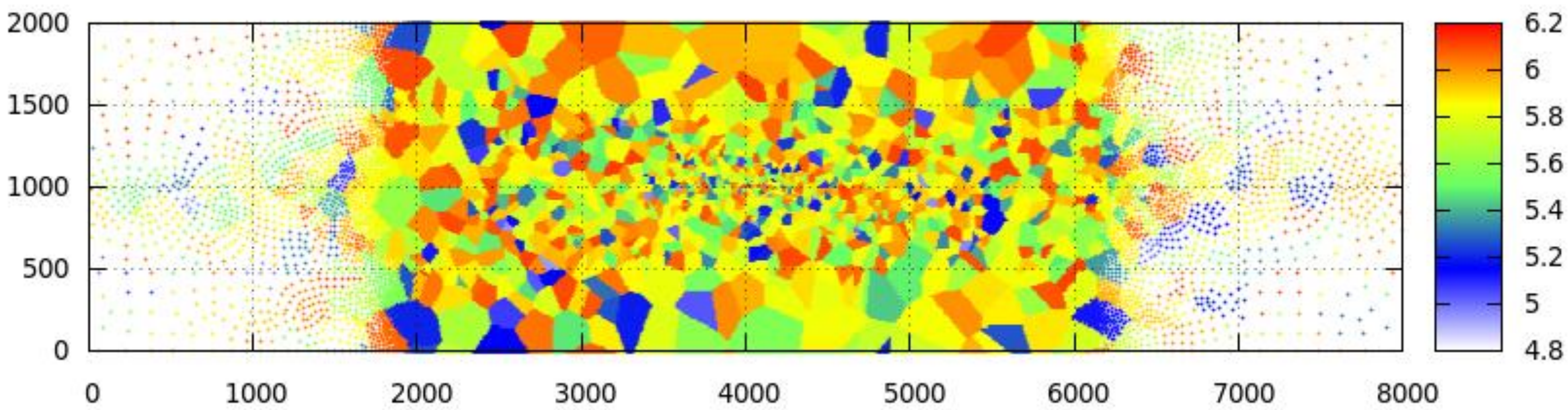
Grain size/tickness on ultrasound properties





Grain clusters with various size

- ✓ Play with the pseudo random distribution of center for the Voronoi tessellation
- ✓ Effect of size distribution, cluster of grains, …



Conclusions & Outlook

- ✓ **LUMet – disruptive sensor technique for Research and Development, process modelling and process control: Innovative microstructure design for better steels**
- ✓ **First time monitoring of dynamic processes**