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Laser ultrasonic measurements of grain size during processing of metals and alloys.

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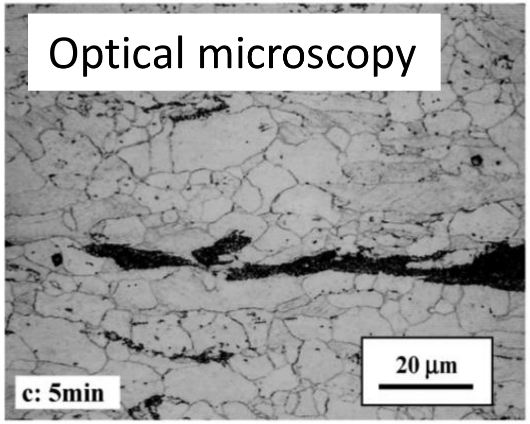
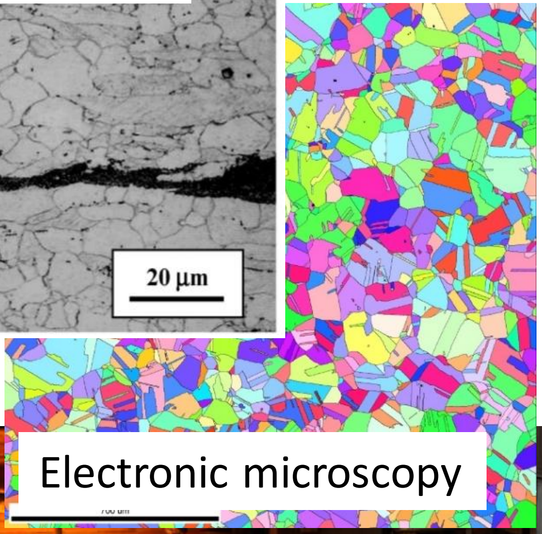
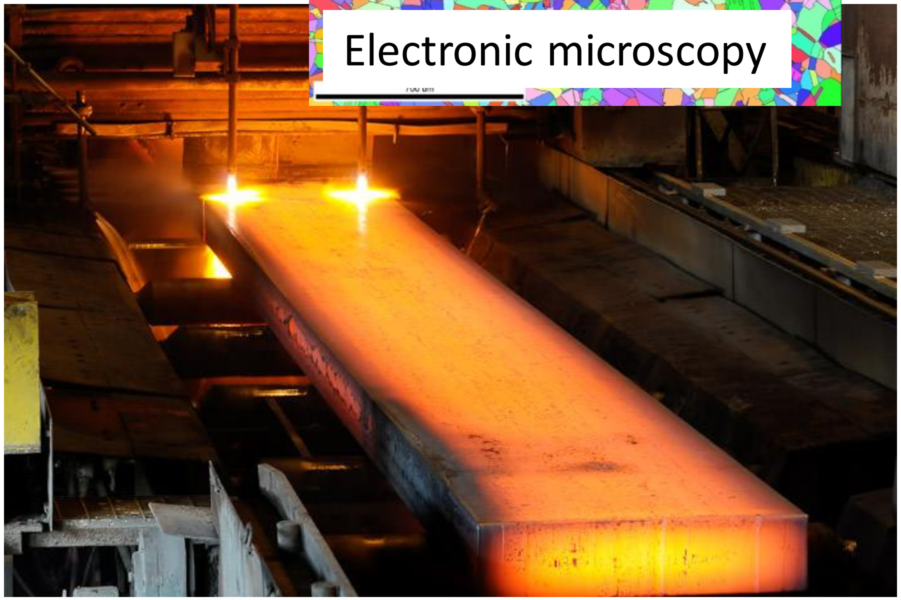
Acknowledgments: Warren Poole, Chad Sinclair, Mahsa Keyvani,

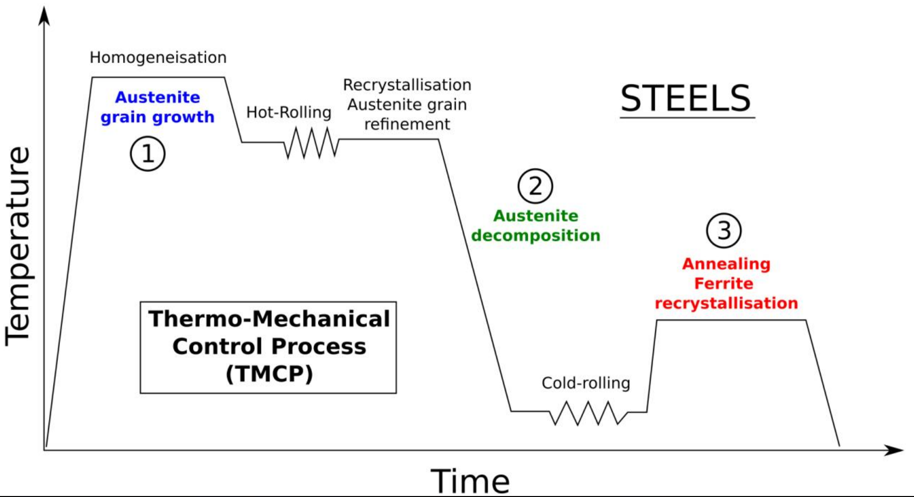
Real time microstructure control

- Complementary tool to control metallurgical processes
- Estimate optimum process parameters for novel metal and alloys

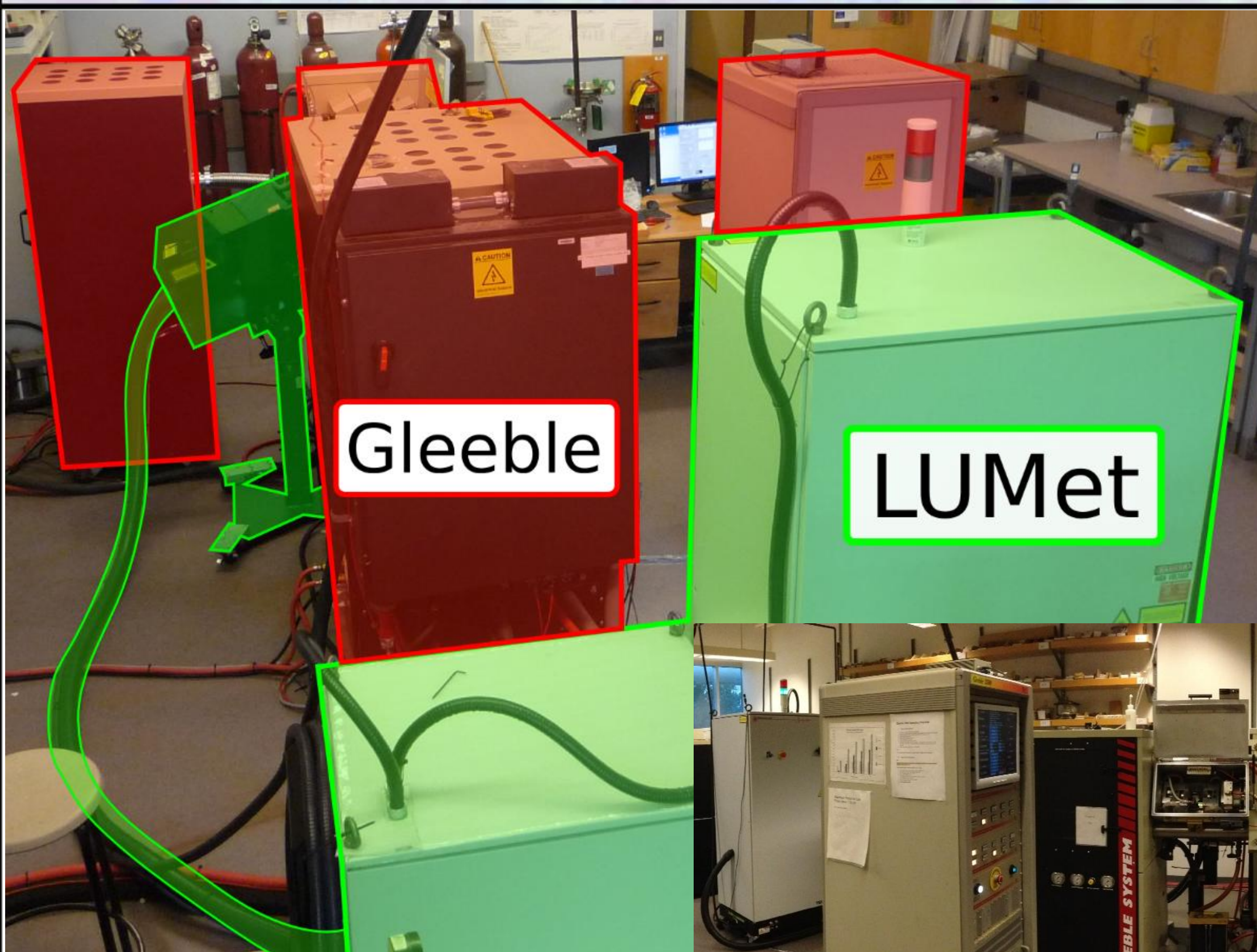








Thermo-mechanical processing lab



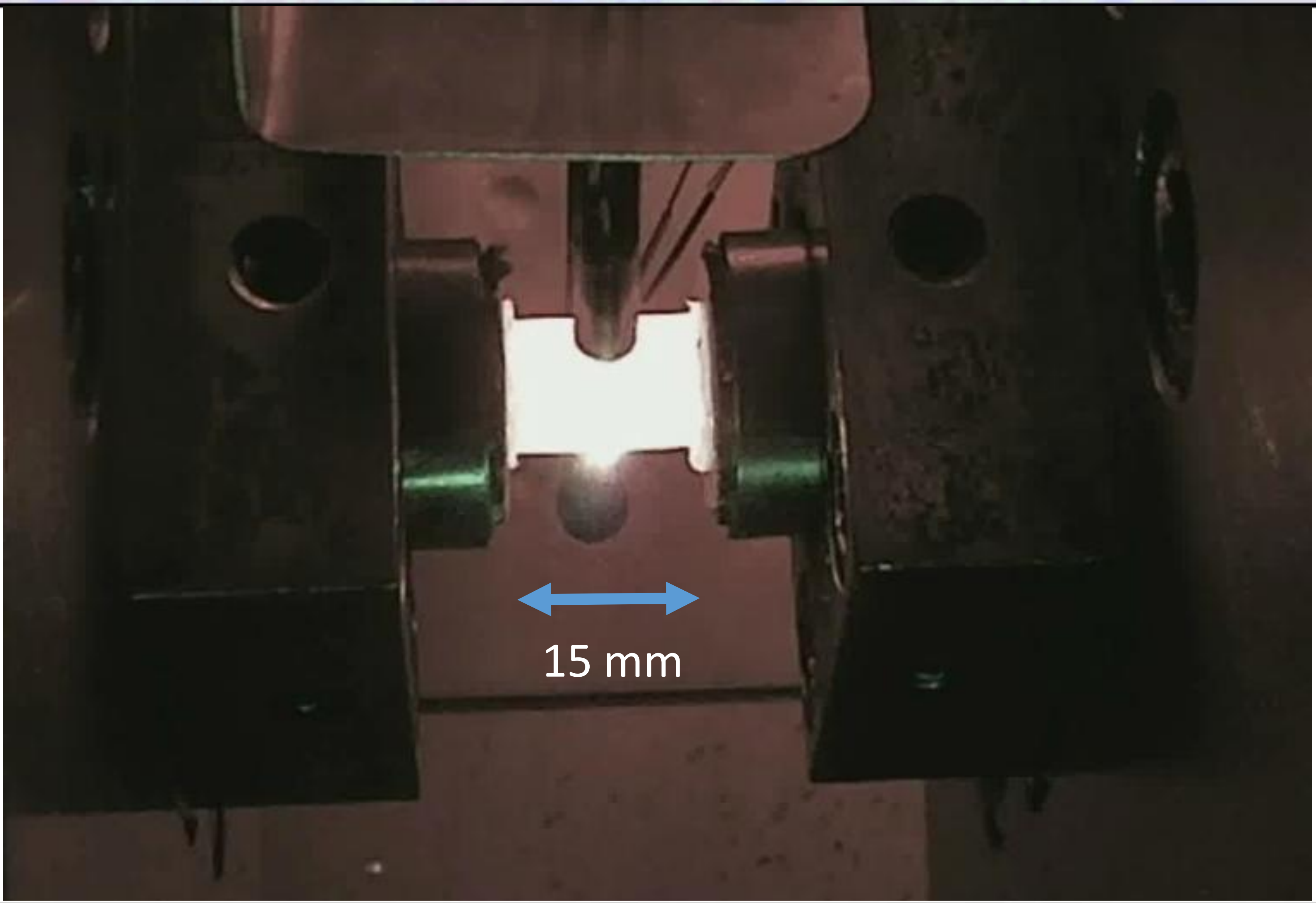
GLEEBLE

TECNAR
SENSORS FOR ADVANCED PROCESS CONTROL

NRC·CNRC

CTOME

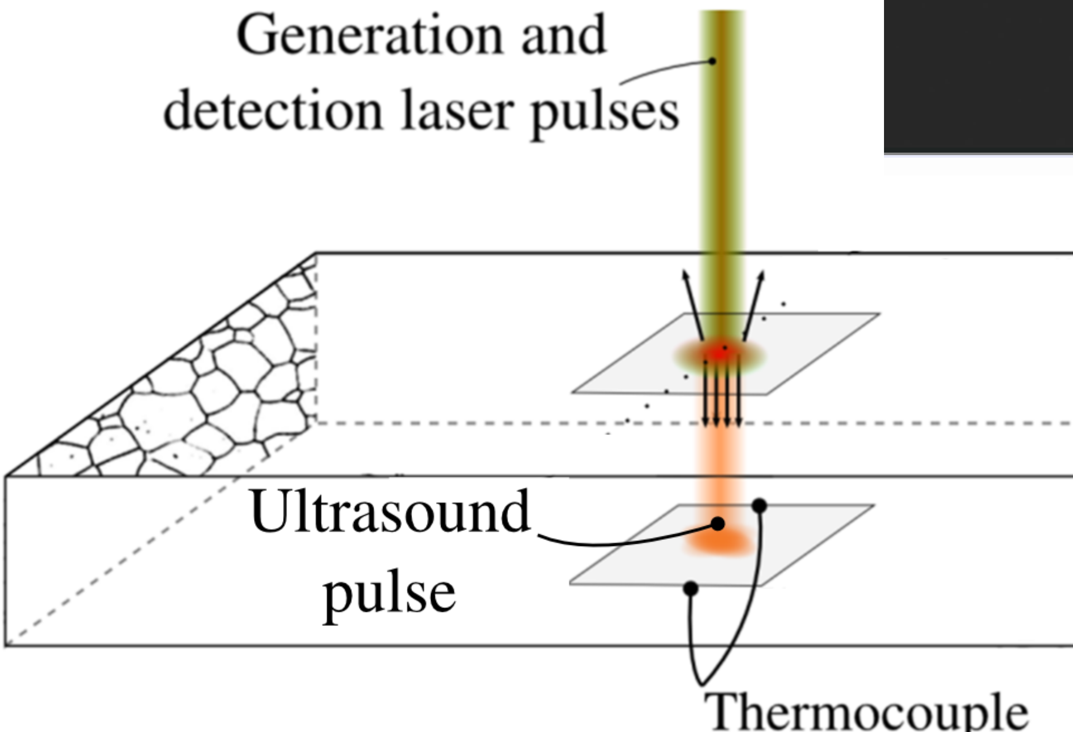
Real time sensing at high temperature

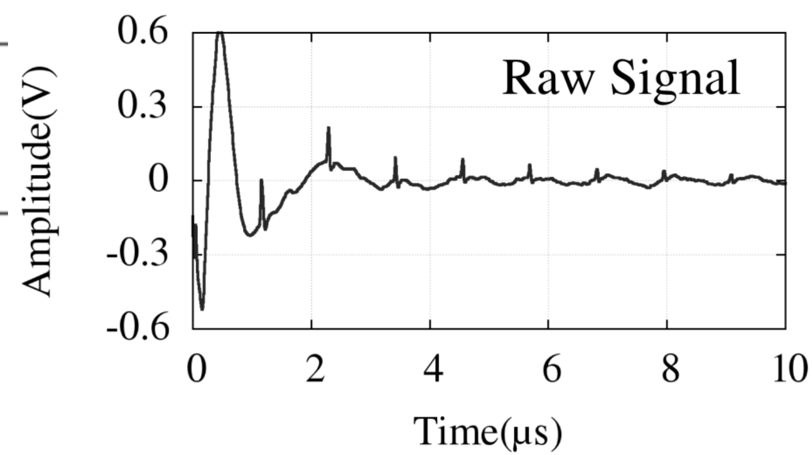


Principle of the technique

Broadband ultrasound pulse (2 to 30 MHz)

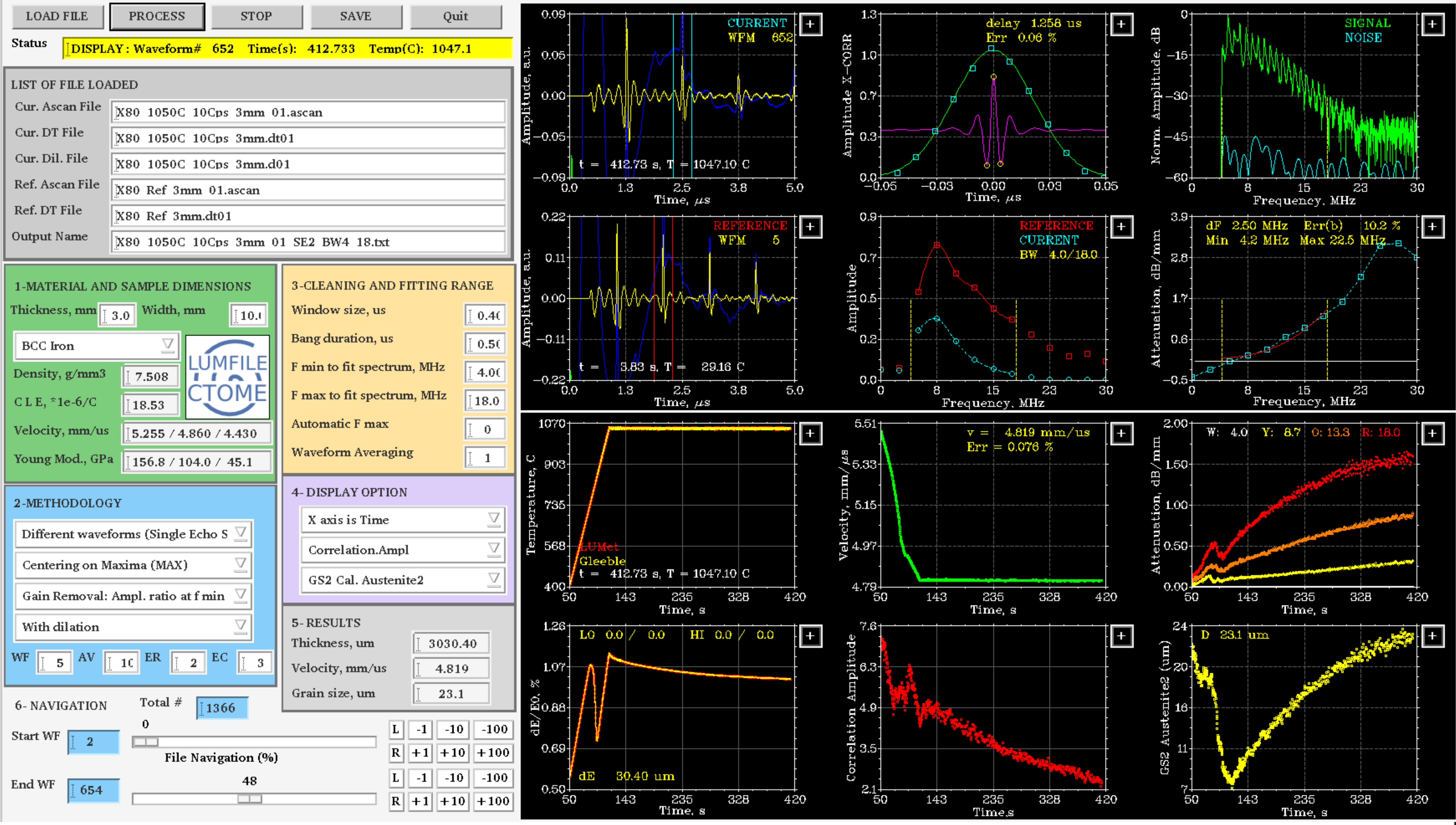
FEM simulation





Up to 50 waveforms measured per second

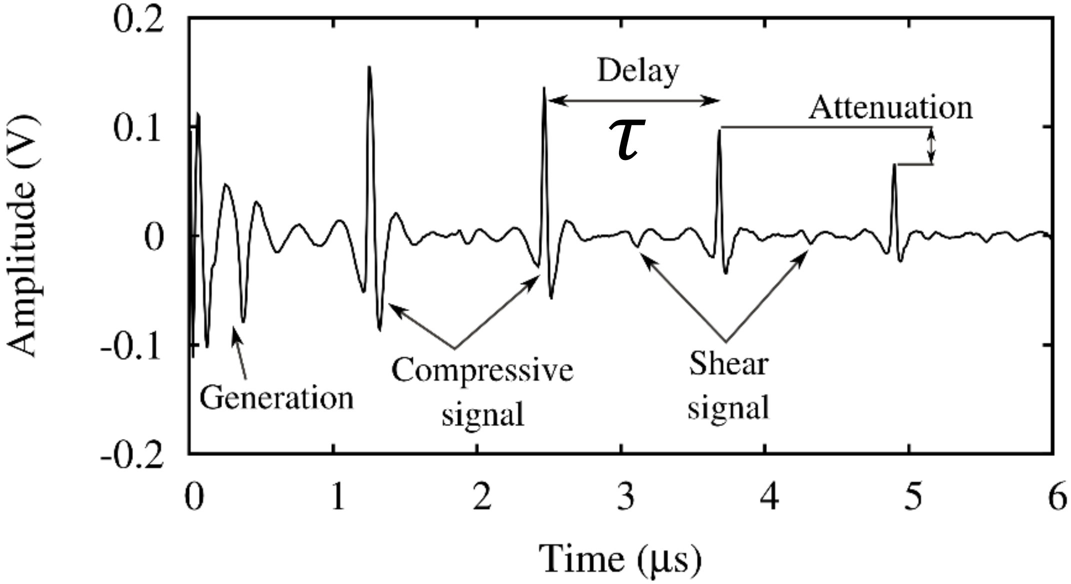
Analysis software

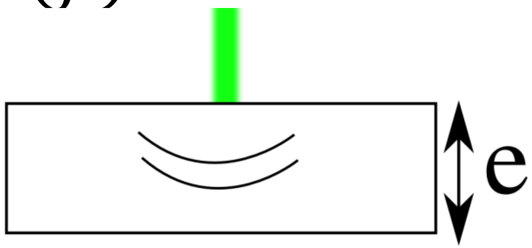


Measured ultrasonic parameters

- Properties of ultrasound compressional waves
- Time of arrival of echoes -> Velocity V
- Amplitude of echoes -> Attenuation $\alpha(f)$

Filtered signal



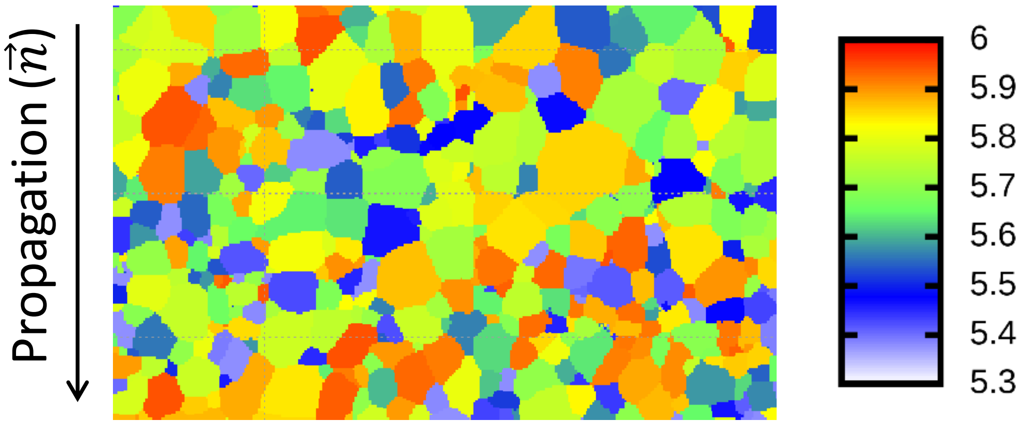


$$V = \frac{2(e+\epsilon)}{\tau}$$

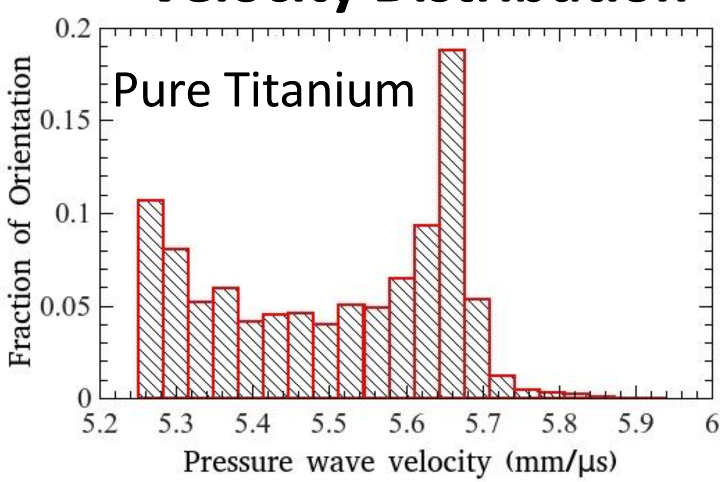
$$\alpha(f) = \frac{20}{2e}\log\left(\frac{A_{echo(i)}}{A_{echo(j)}}\right)$$

Velocity of ultrasonic wave

EBSD to **Velocity map** (mm/µs)



Velocity Distribution



Rotated Elastic Tensor

$$C_{ijkl} = \int c'_{ijkl} f(odf)$$

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

$$V = \sqrt{\sum_{Phases} \frac{K(odf)}{\rho}}$$

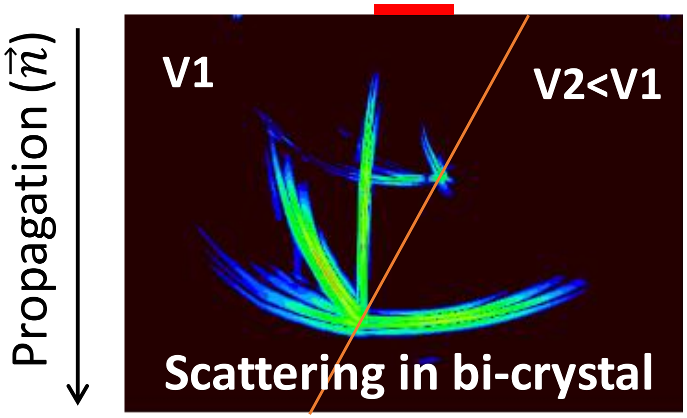
What can be investigated ?

Phase transformation

Second phase/Precipitation

Recrystallization

Attenuation and scattering by grain



Wave scattering depends on grain boundary disorientation and incidence angle and grain volume/size

In polycrystalline metals, scattering depends on ultrasonic wavelength

Rayleigh Region	$\alpha(D,\lambda) = C_r D^3 \lambda^{-4}, \lambda \gg D$
Stochastic Region	$\alpha(D,\lambda) = C_s D \lambda^{-2}, \lambda \cong D$
Diffusion Region	$\alpha(D,\lambda) = C_d / D, \lambda \ll D$

How to estimate the grain size ?

1) Reference sample D_0
2) **ONE ECHO METHOD**

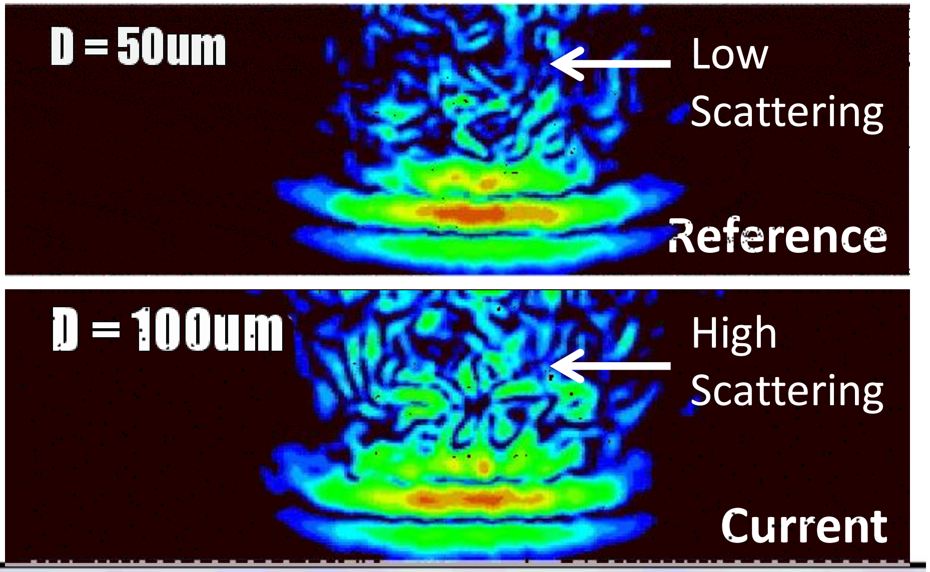
$$\alpha(f) = a + b f^n$$

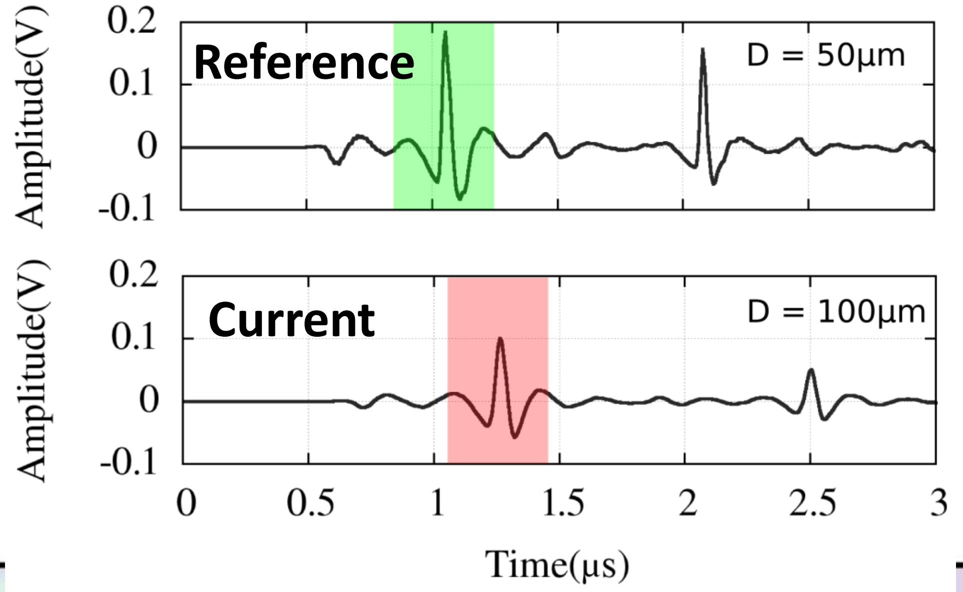
Frequency dependant grain size parameter

Isolate only grain scattering

$$b = C(T)\left[D_i^{n-1}(t) - D_0^{n-1}(t_0)\right] f^n$$

Measurement precision < 10 %



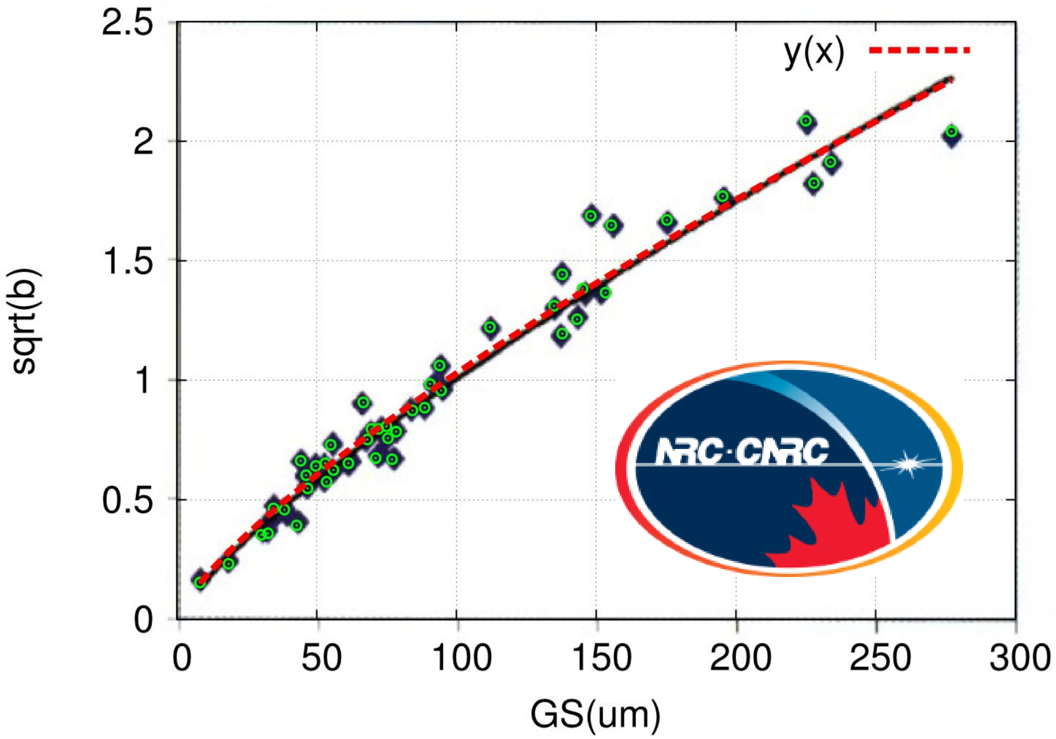


Application to austenite in steel

1) Reference fine grain sample at room temperature

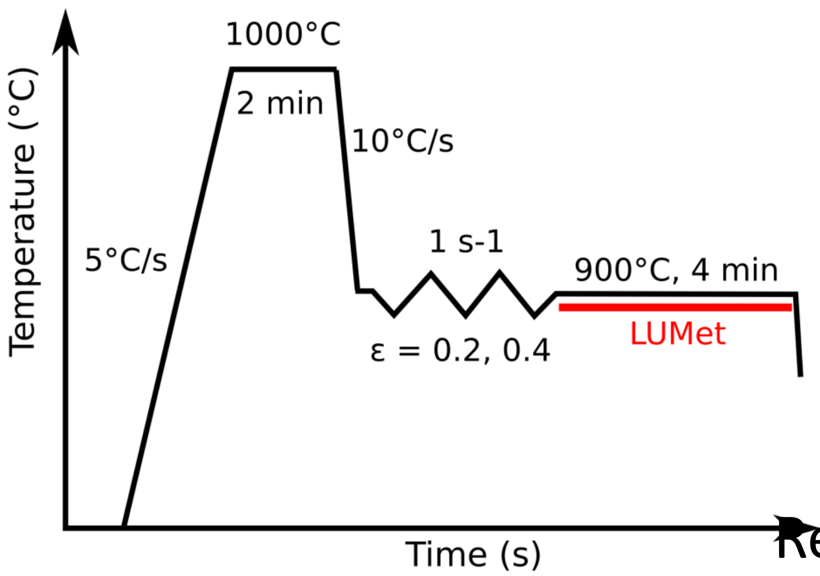
$$\alpha(f) = a + bf^3$$

2) Calibration developed at Timken (S.E. Kruger et al., Iron Steel Technol, (2005), 2(10),25



Application to hot rolling processes

- ✓Grain size measurement after hot-deformation in Mo-TRIP steel
- ✓Strain = 0.2 and 0.4



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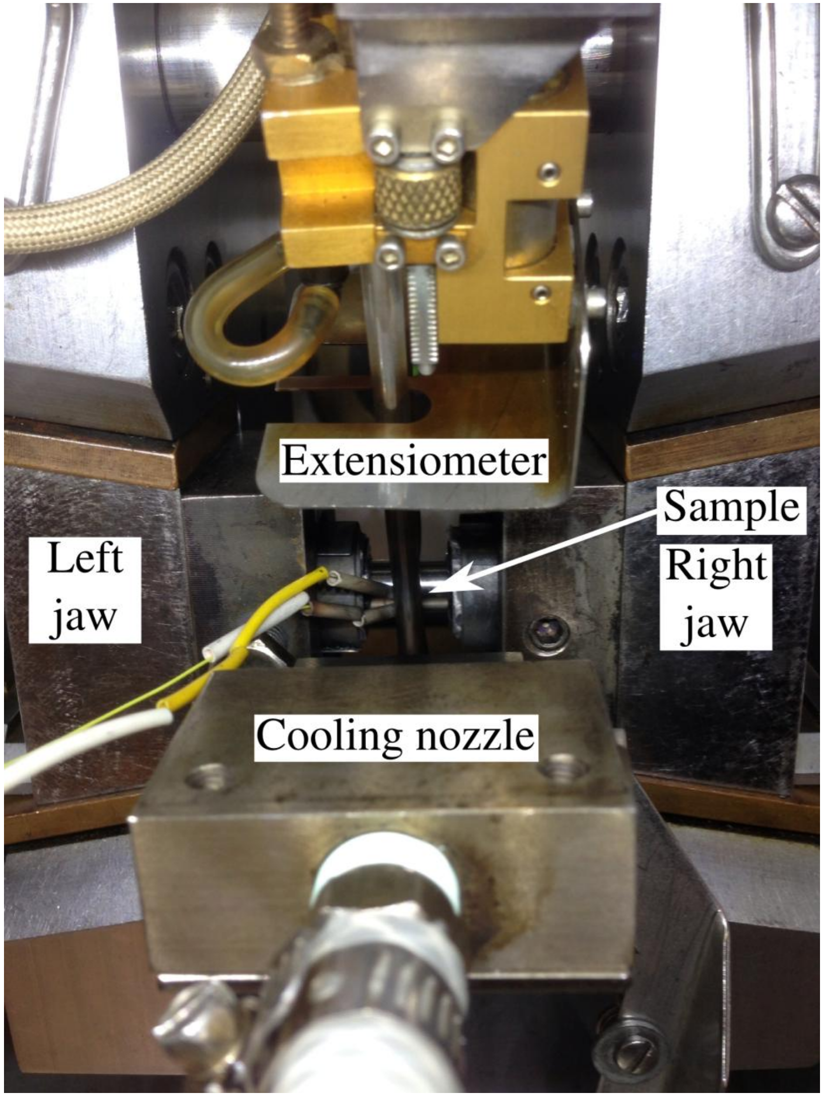
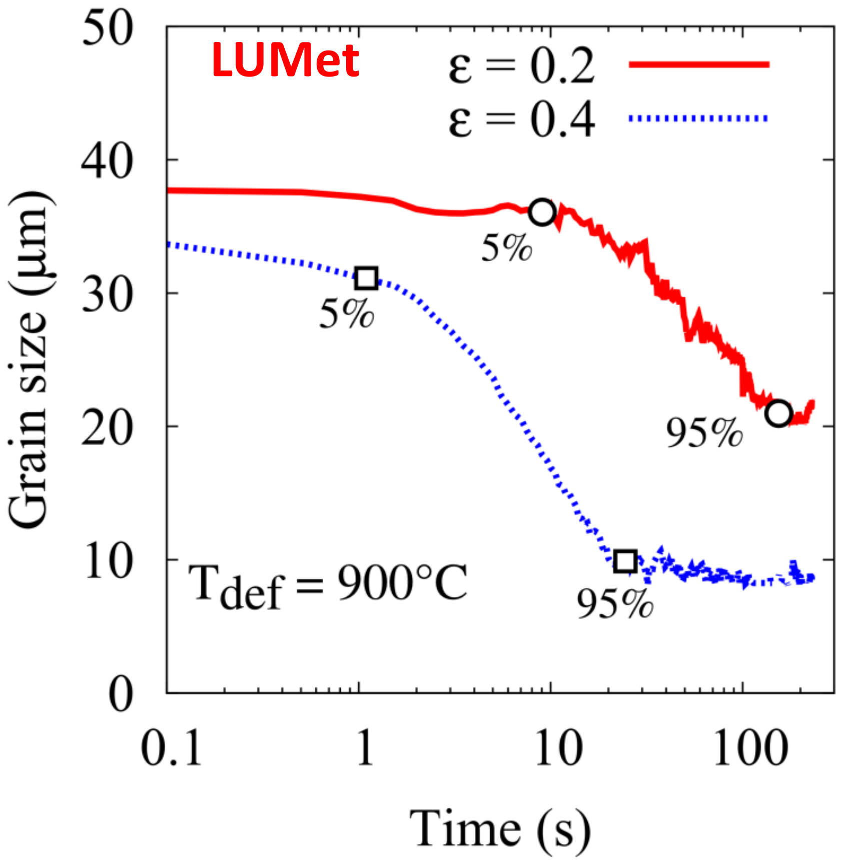
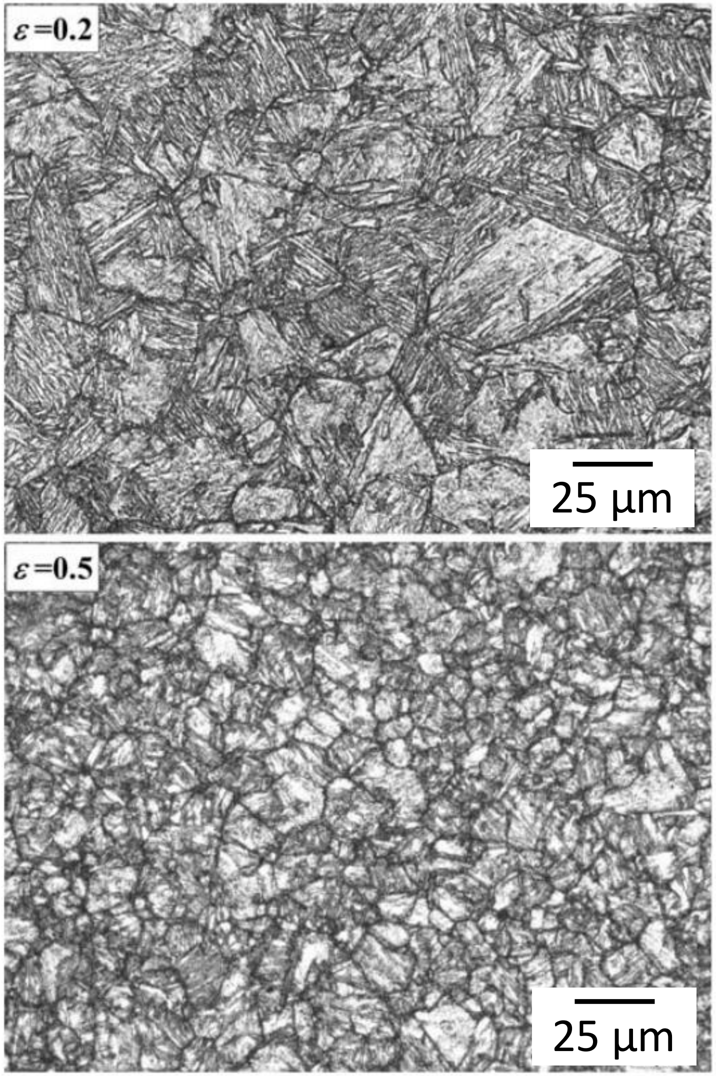


Austenite grain refinement

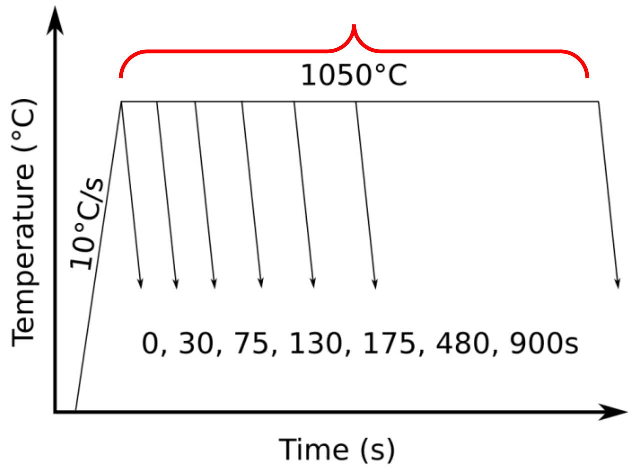
✓ Larger grain refinement at higher deformation strain

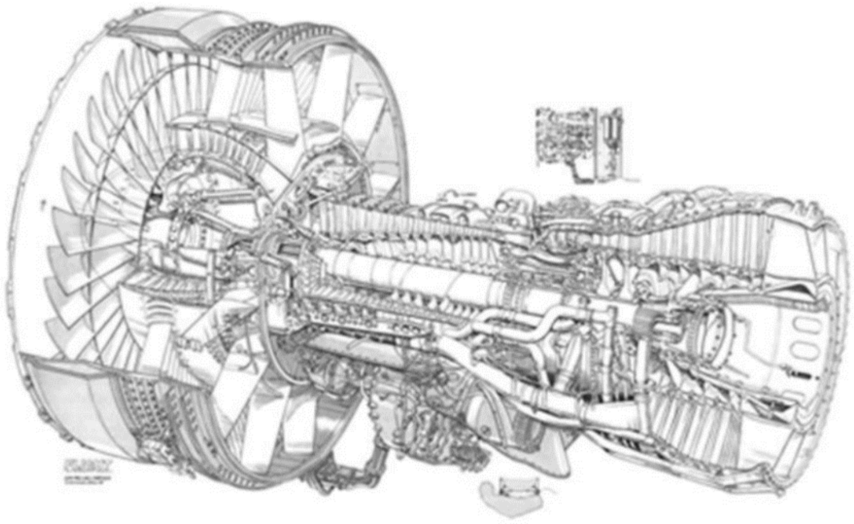


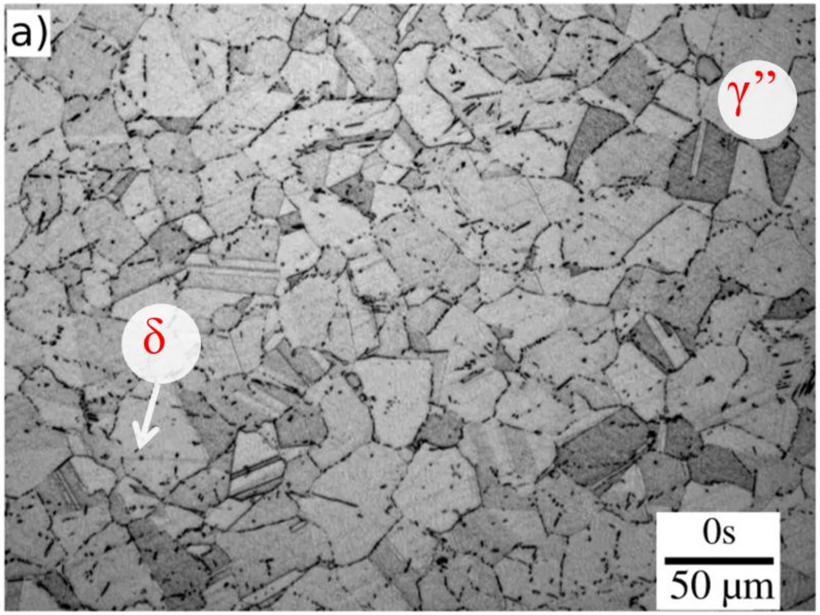


Nickel based super alloys

- Control the grain growth + dissolution of second phase particles prior to forging
- Starting structure has 20 µm polygonal grain
- + 2 to 3 % of delta phase precipitates

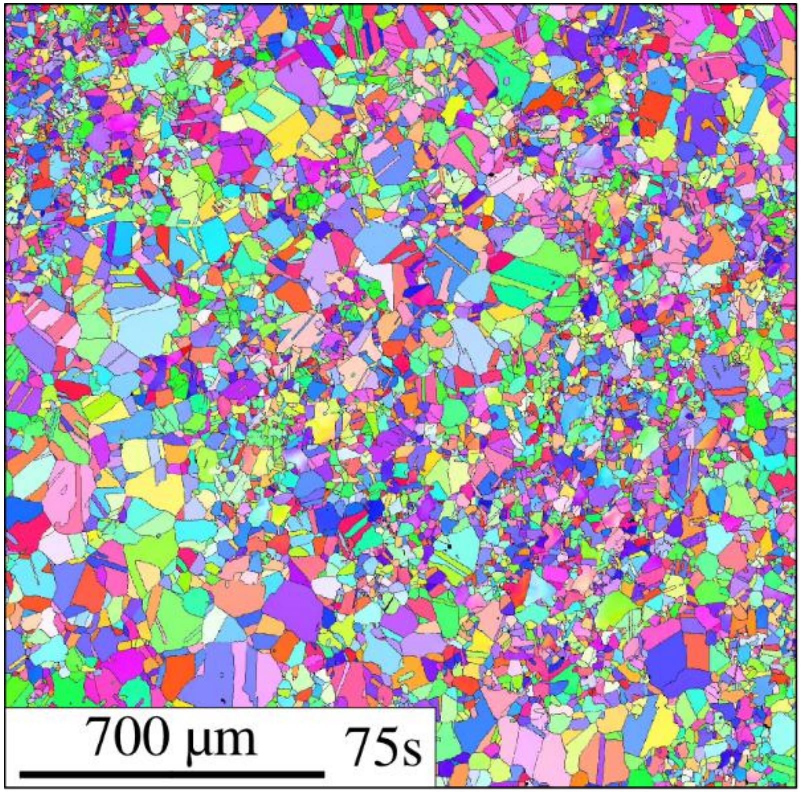


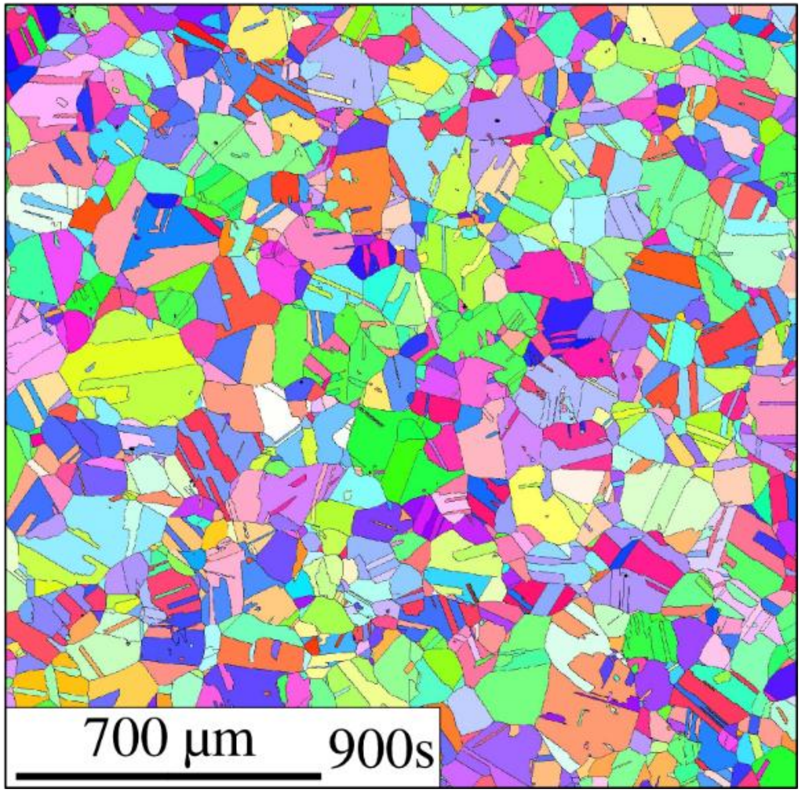




Stage of heterogeneous grain growth

- Local Nb microsegregations affect the stability of the second phase leading to heterogeneous grain grow (Fraction of large and small grains)

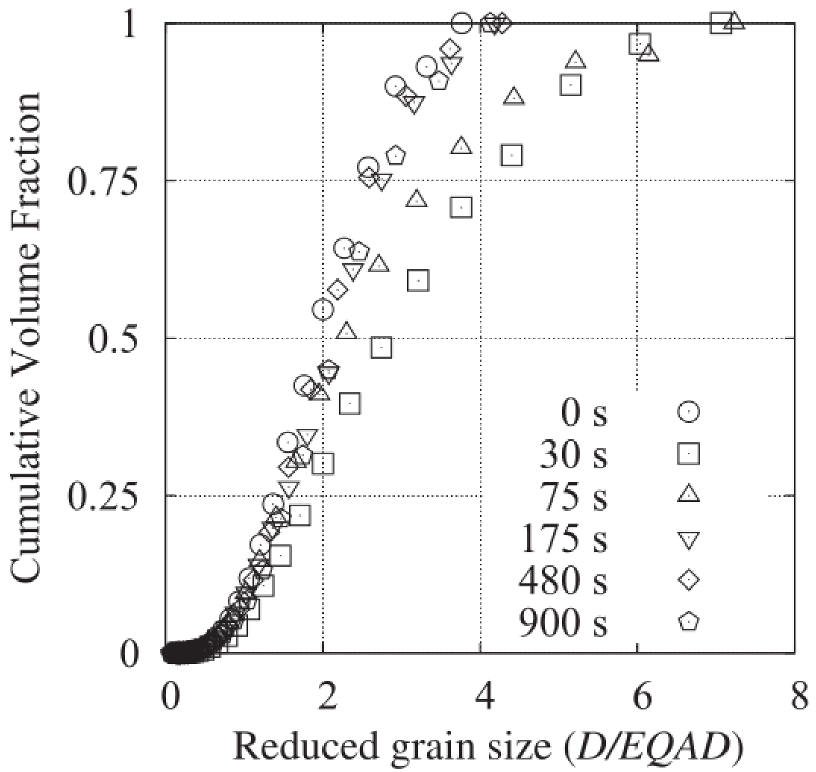




Metallographic analysis

- Evaluation of the mean grain size EQAD = $\sqrt{4\bar{A}/\pi}$
- Maximum 1% largest grain diameter

Time (s)	*EQAD*(μm)	D_{MAX}(μm)	$\frac{D_{MAX}}{EQAD}$
0	15	56	3.7
30	18	120	6.7
75	19	139	7.3
175	33	139	4.2
480	36	155	4.3
900	42	172	4.1

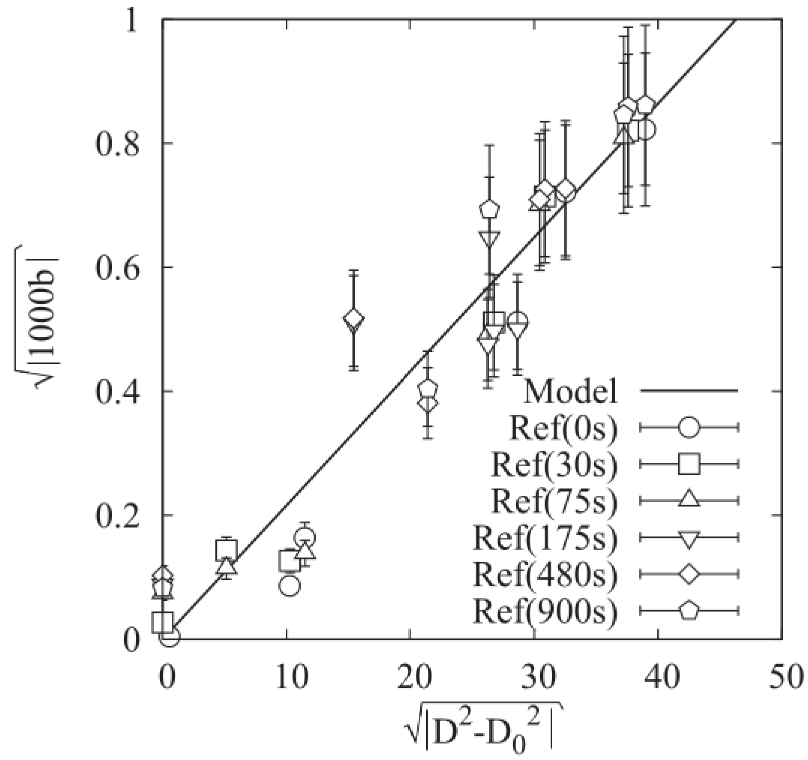


Correlation at 1050°C

- Evolution of the scattering parameter b with the relative change in mean grain size.
- Direct measurement of the coefficient C*

$$\sqrt{|1000 \cdot b\ (t_i, D_i)|} = C^* \sqrt{\left|D_i^2(t_i) - D_0^2(t_0)\right|}$$

Linear regression coefficient
C* = 0.022



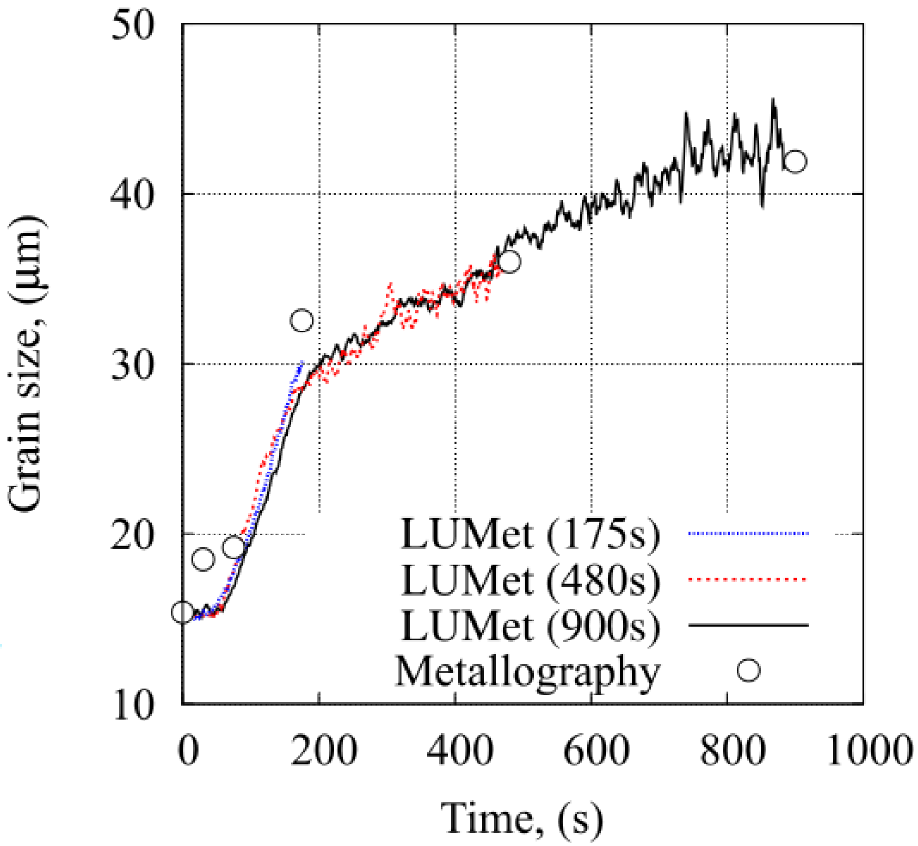
Grain grow tests

- Insight into the grain growth behavior.
- Different grain growth stages

- 1) Zener $\frac{dD}{dt} = K\left(\frac{1}{D} - P_0\right)$

- 2) Rapid grain growth

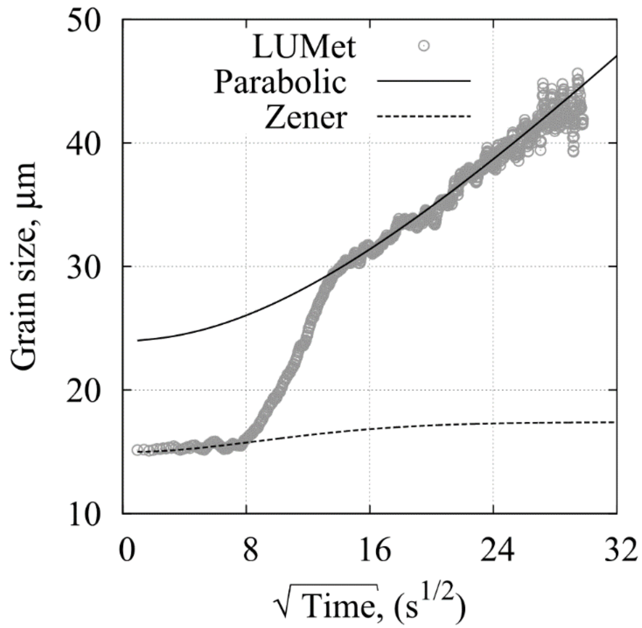
- 3) Parabolic $D^2 - D_{init}^2 = Kt$

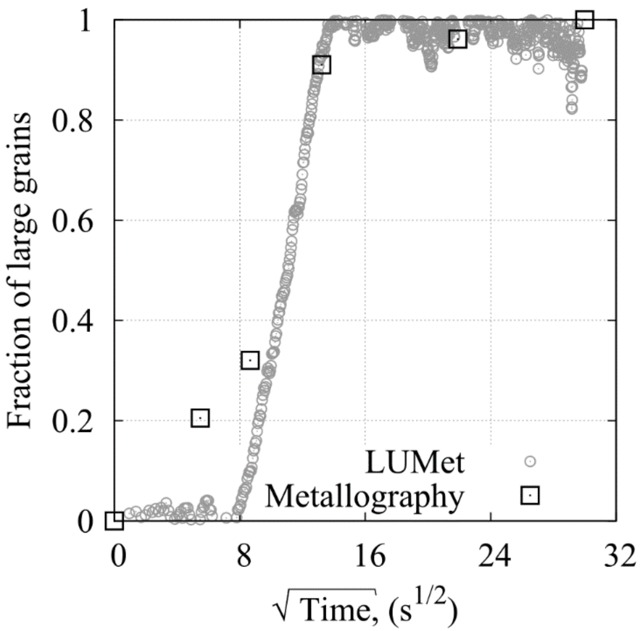


Criteria for abnormal grain growth

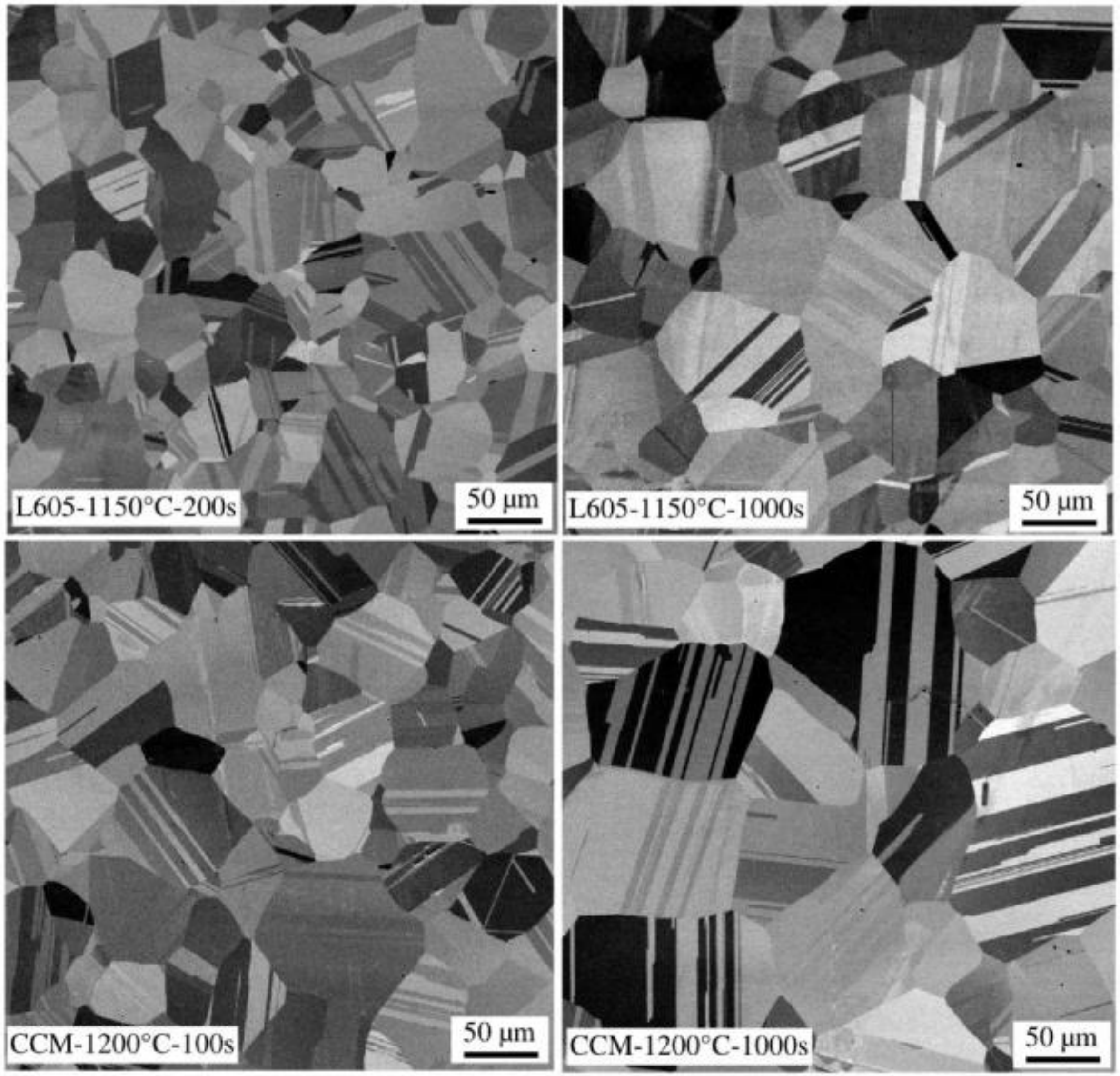
- Normalization procedure
- Time at the onset of abnormal grain growth

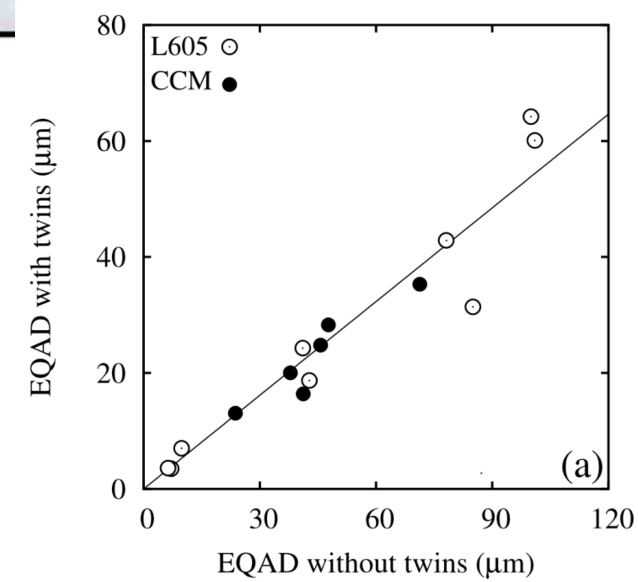
D_{init} (μm)	K ($\mu m^2.s^{-1}$)	P_0 (μm^{-1})
24	1.6	0.0574

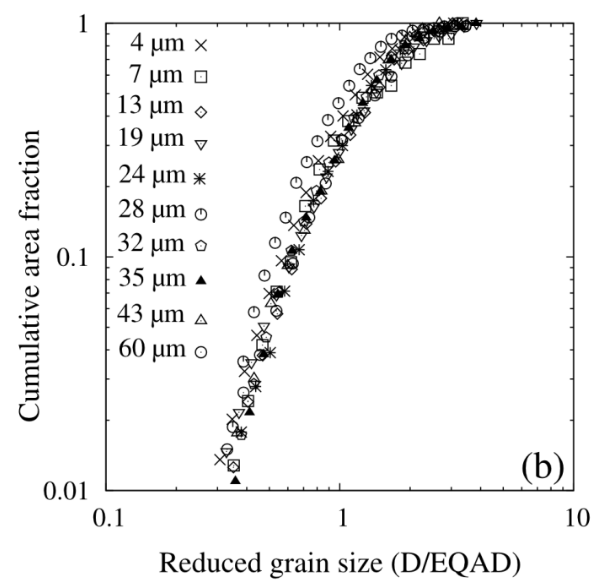




Cobalt super alloys



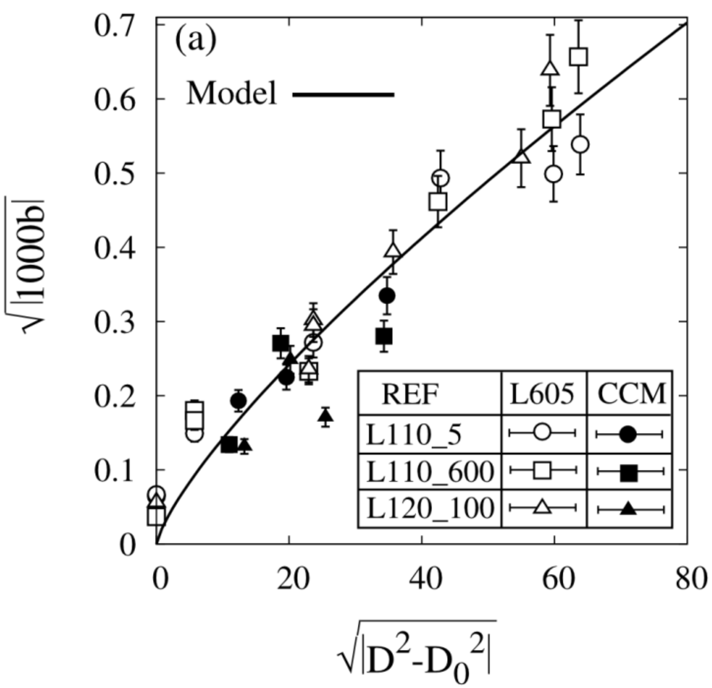




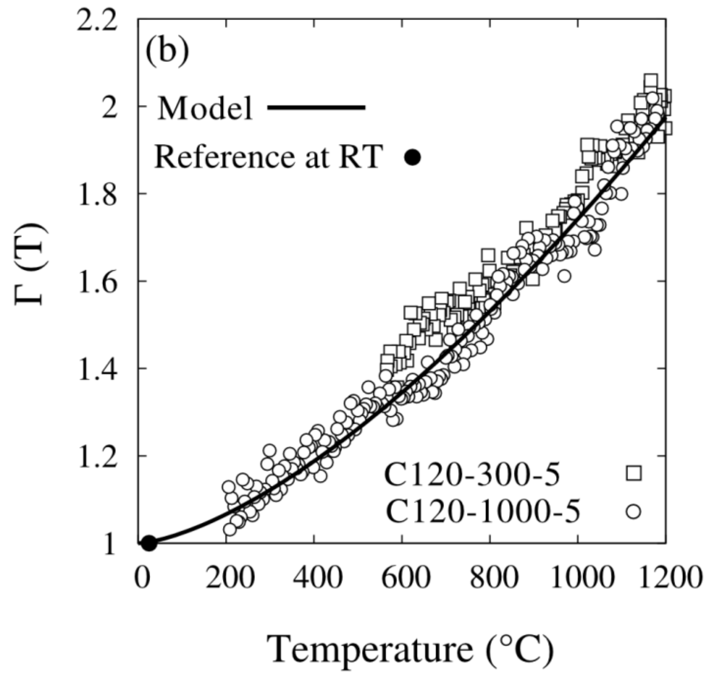
Empirical correlation

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

$$\sqrt{|1000b|} = \Gamma(T)\delta\left(\sqrt{\left|D_i{}^2 - D_0^2\right|}\right)^{1-\varepsilon}$$



Grain size dependence

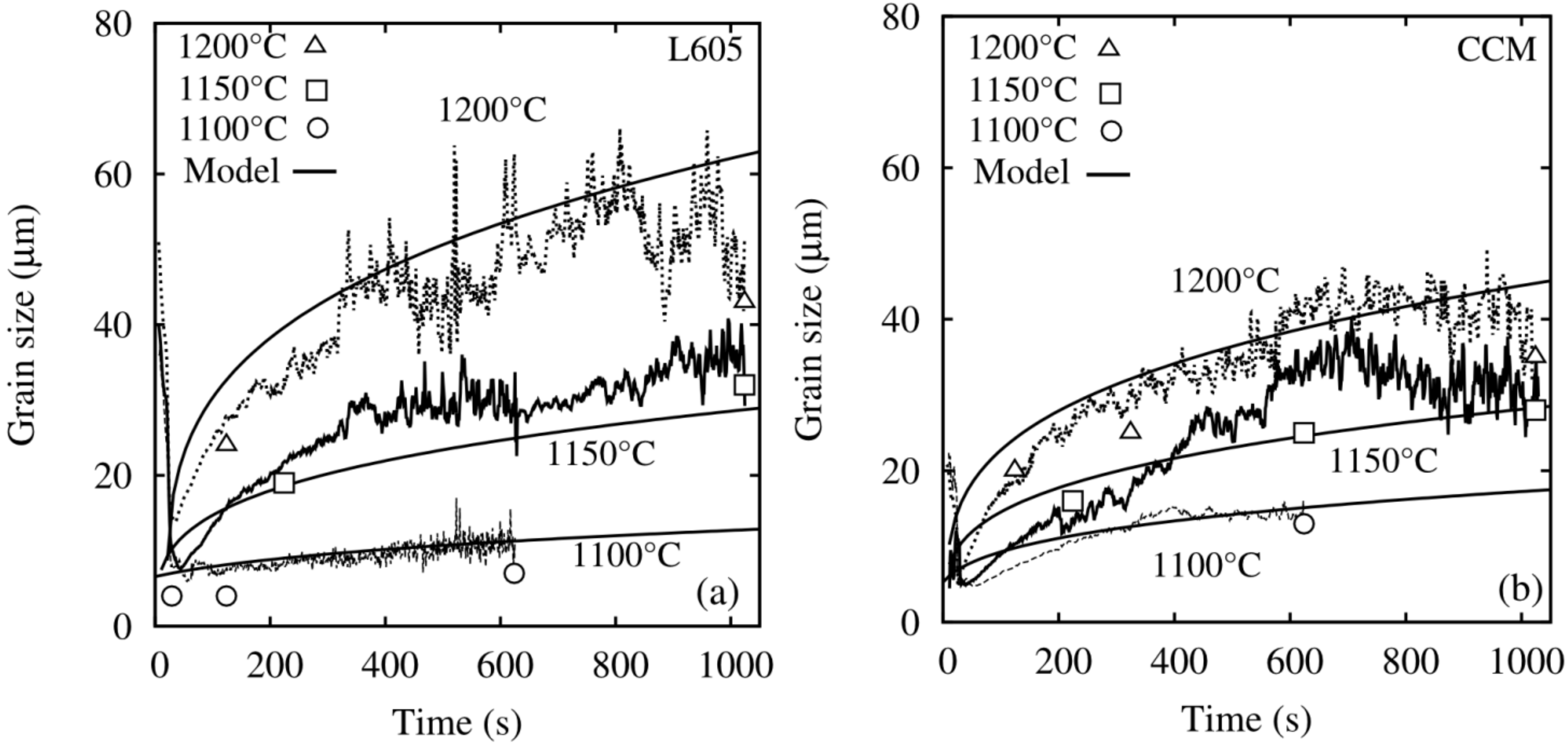


Temperature dependence

Grain growth measurements

Grain growth model $D^m - D_{init}^m = \Phi(T)t$

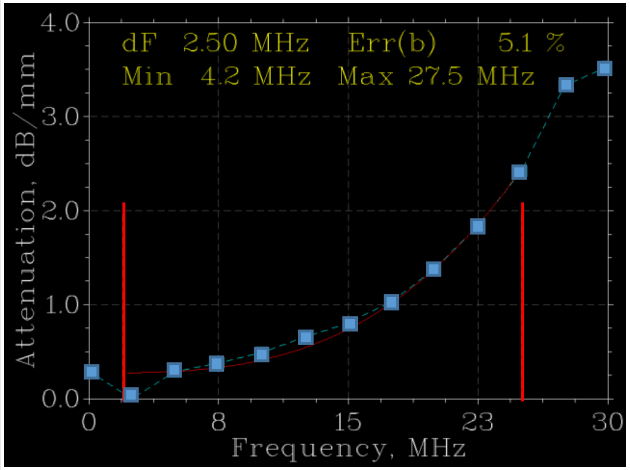
Effective mobility $\Phi(T) = \lambda_1 \exp(-\frac{\lambda_2}{kT})$



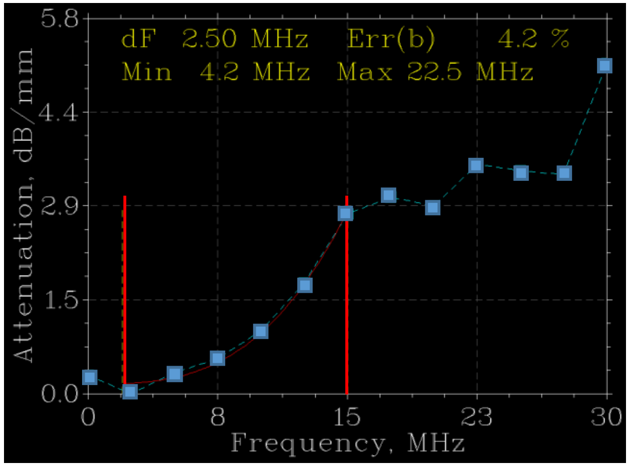
Closer look at the attenuation spectrum

- These empirical approaches require adjusting the effective bandwidth
- Because they do not account for multiple regimes of scattering

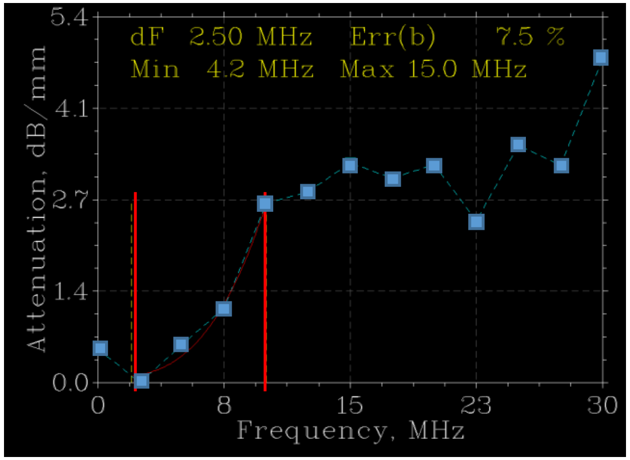
$$\alpha(f) = a + bf^3$$



D = 10 µm



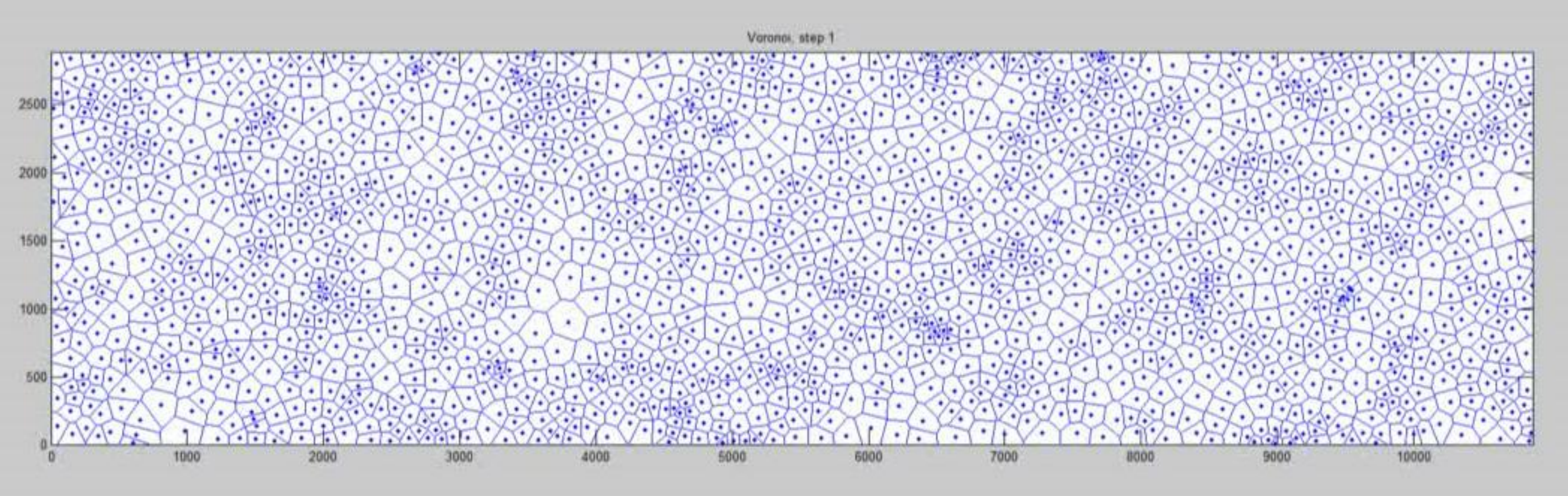
D = 60 µm



D = 130 µm

Computer generated grain structure

- Finite element simulation of wave propagation on polycrystalline materials
- Centroidal voronoi tessellation: all cells have 6 faces but the final structure is not ordered



Material properties

- Single crystal stiffness tensor
- FCC iron at 1423 K (Zarestky et al., 1987 , Phys.Rev. B 35(9), pp.4500)
- Single crystal elastic constant:

c_{11} = 154 GPa

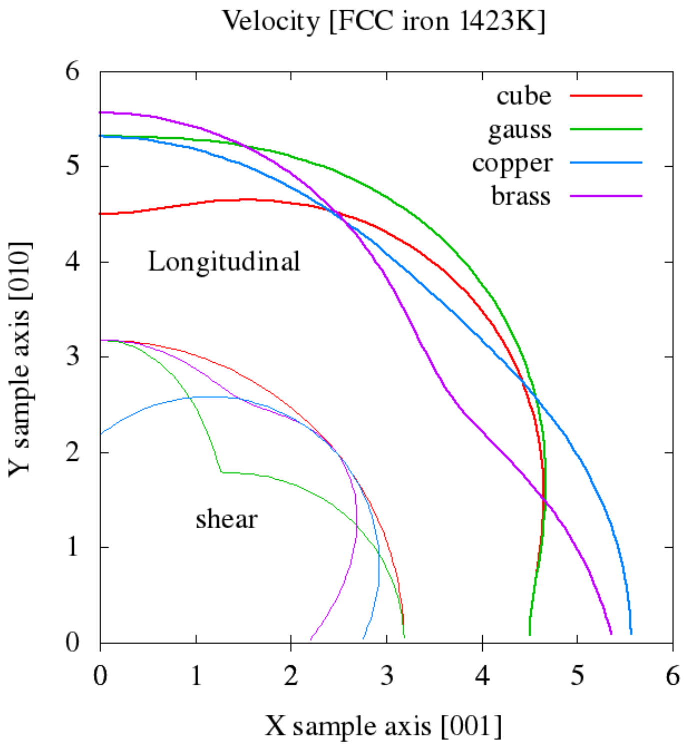
c_{12} = 122 GPa

c_{44} = 77 Gpa

Zener Anisotropy factor

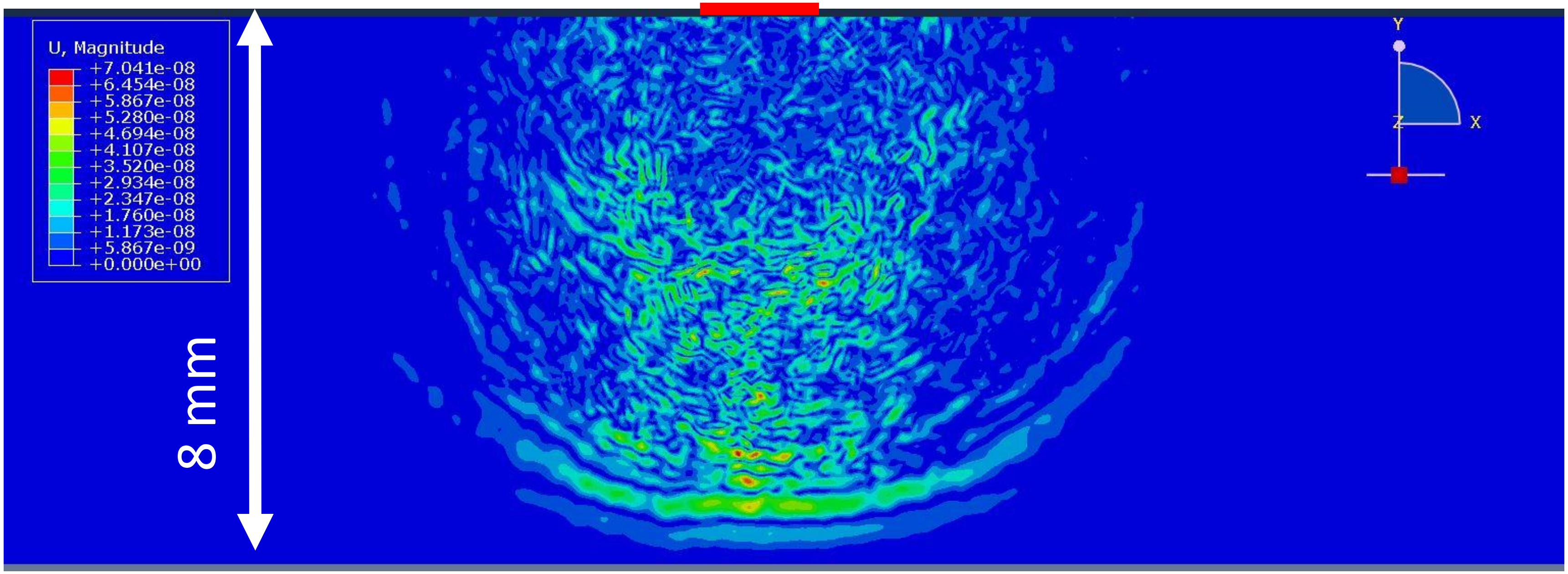
c_{44}/c' = 4.8

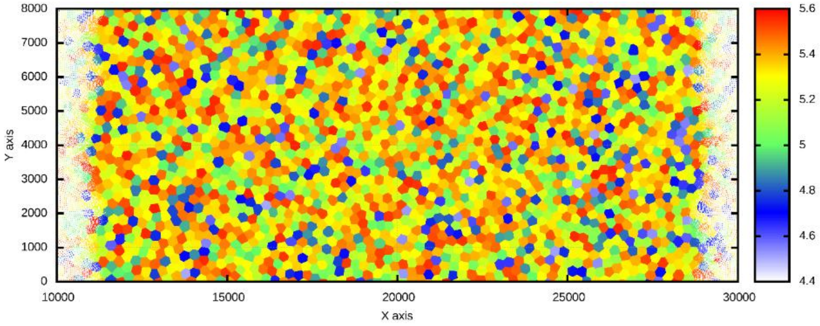
- Crystallographic orientation

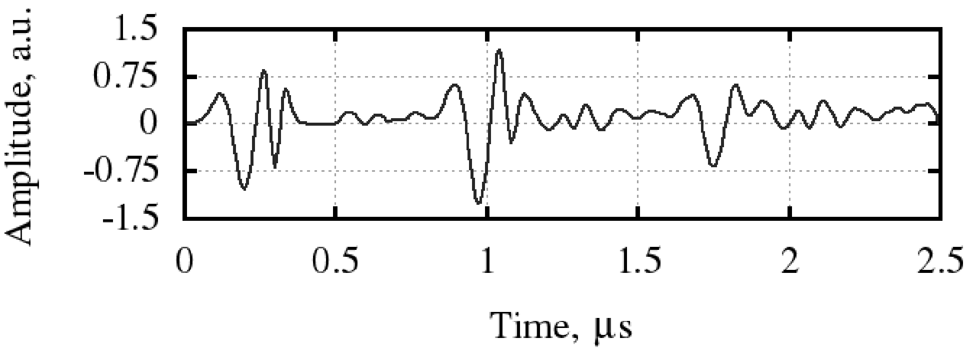


FEM simulation of ultrasound propagation

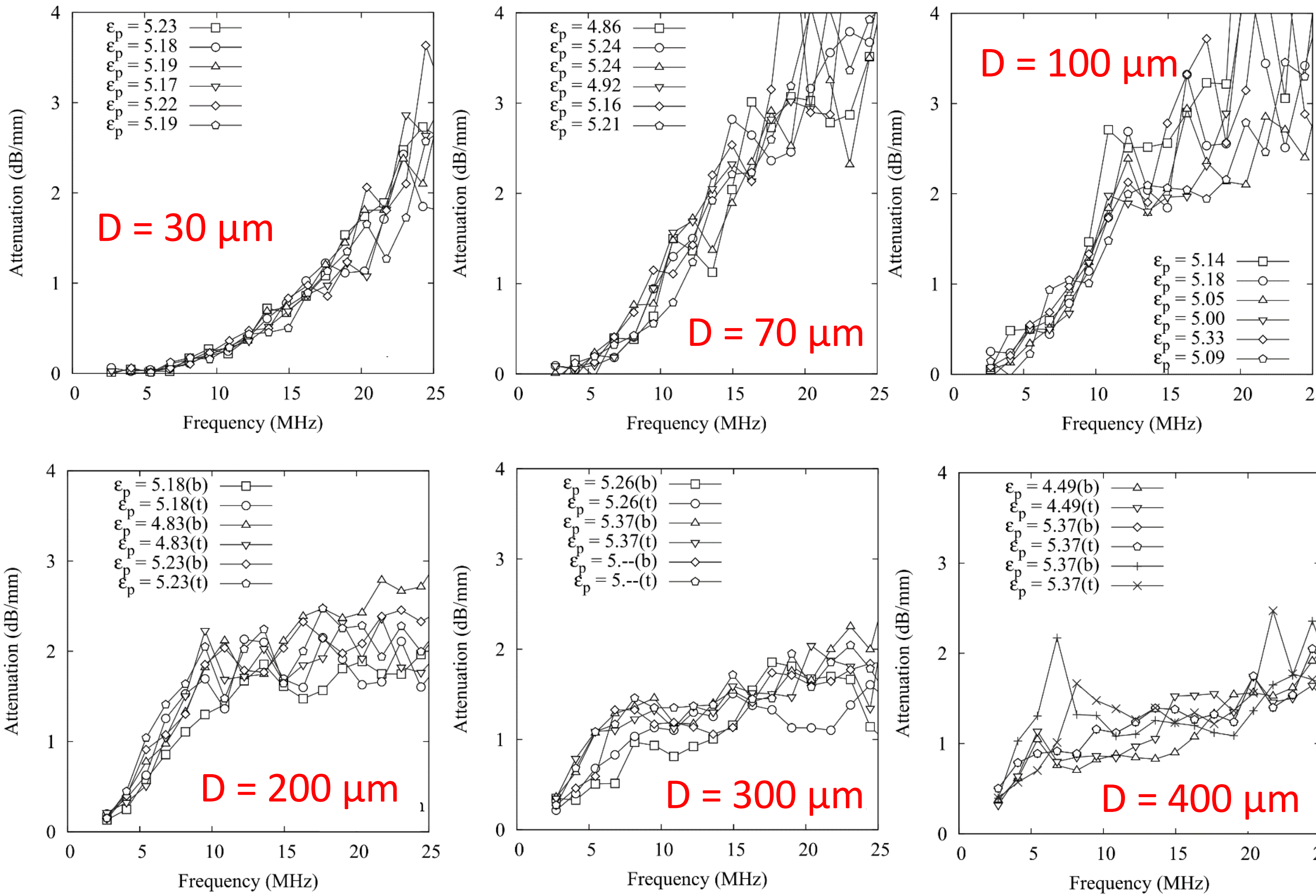
- Displacement field for austenite (D = 300µm)





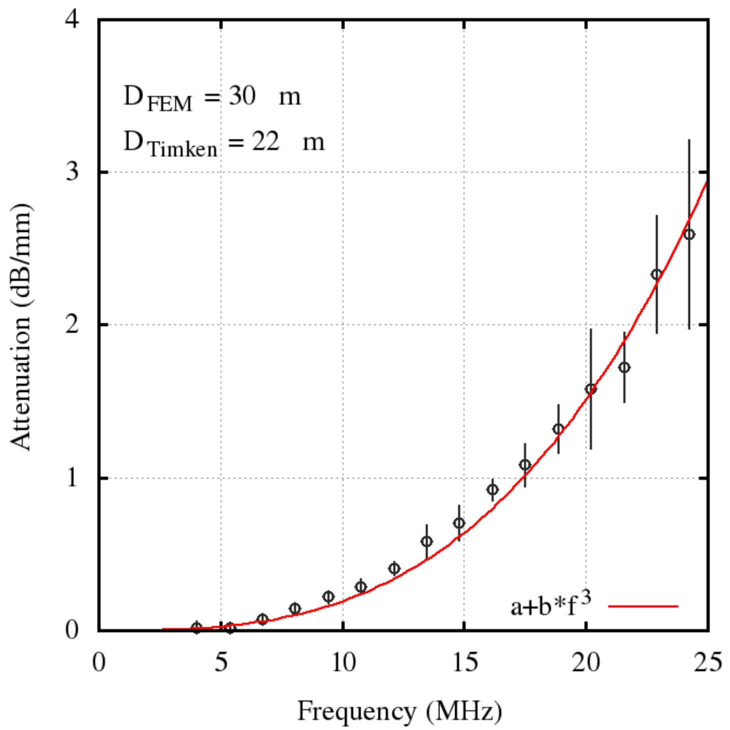


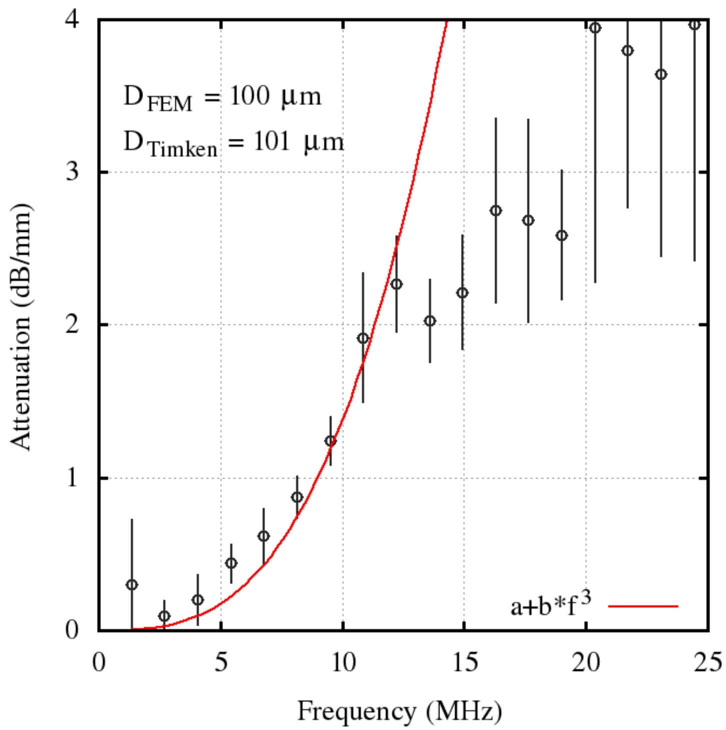
Results: Attenuation spectrum



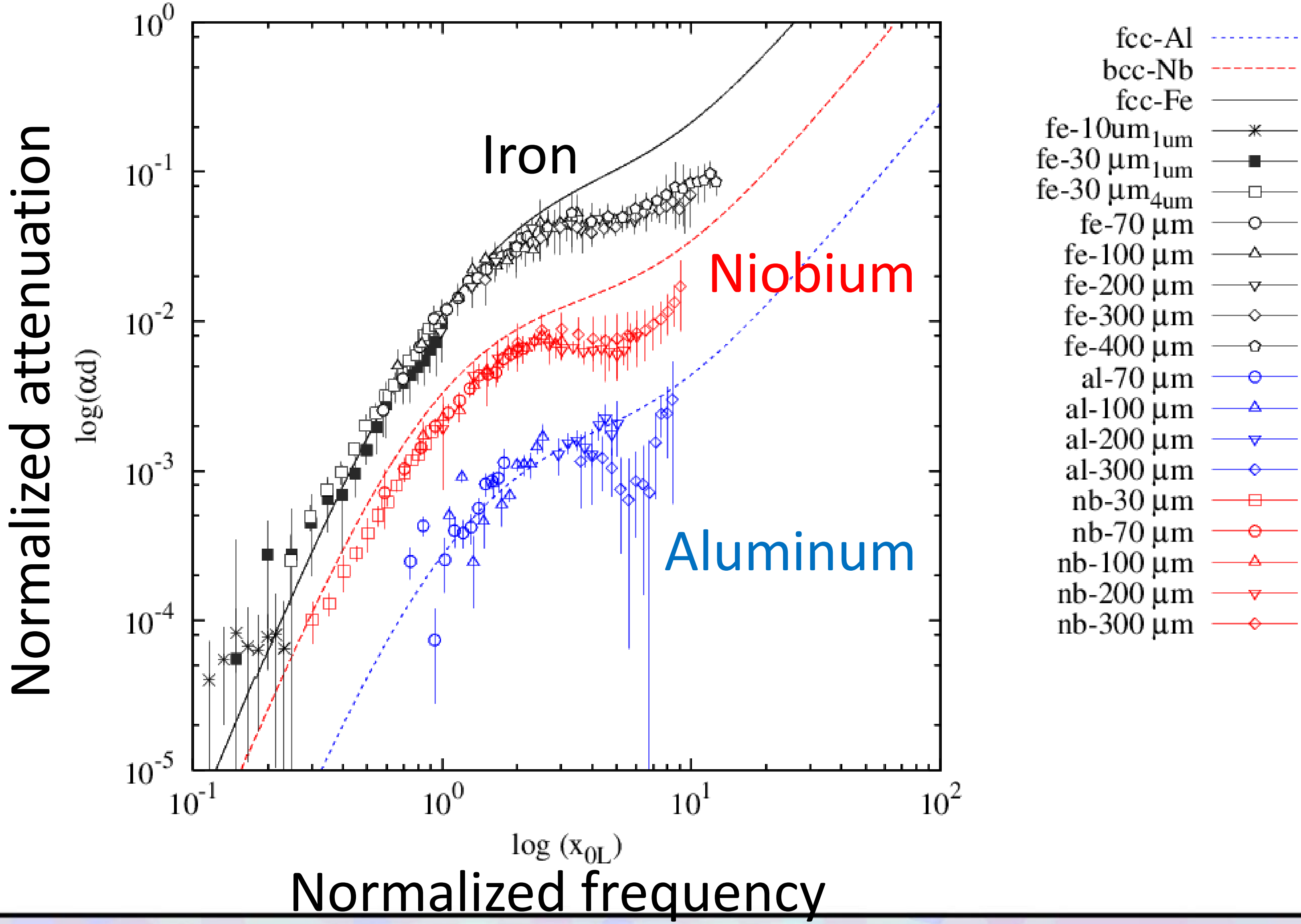
Validation using austenite calibration

- By selecting appropriate frequency range, the austenite calibration provide satisfying agreement with FEM generated attenuation spectrum



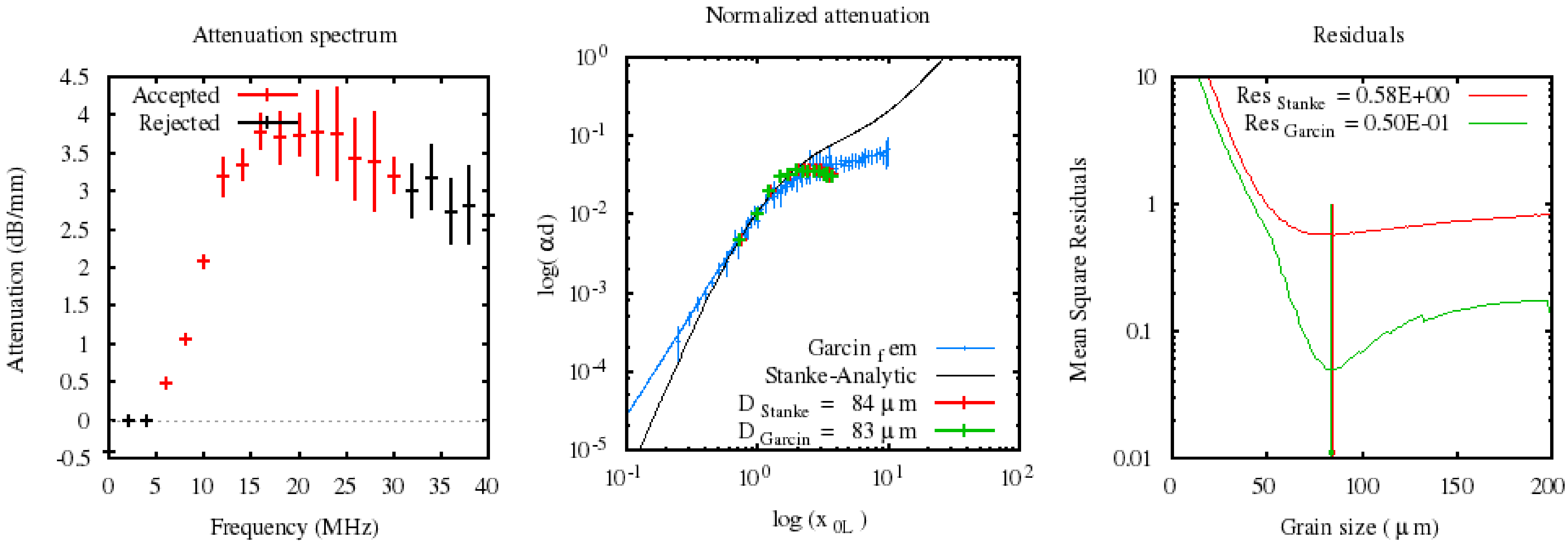


Validation with scattering theory



New approach for grain size measurements

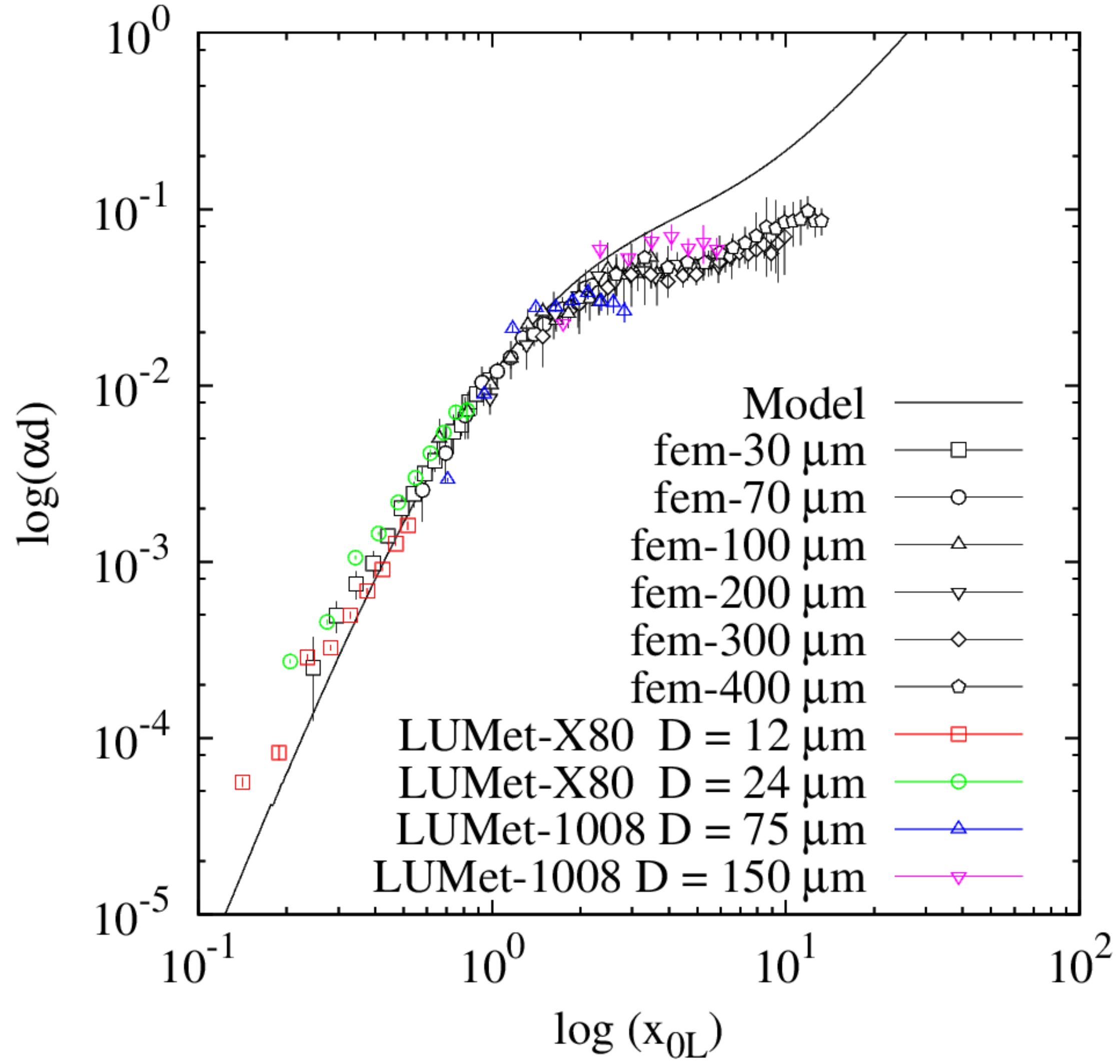
- Evaluation of grain size accounting for multiple regimes of scattering
- Using FEM simulated attenuation and/or scattering theory to predict grain size.



Quantitative tool to validate LUMet results

Example: Attenuation spectrum measured in austenite at high temperature.

Least square approach on FEM data provide quantitative estimate of the mean grain size.



Conclusions

- Ultrasonic attenuation can be sensitive to the self similarity of grain size distribution.
- FEM are integrated to simulate the wave propagation in anisotropic aggregate.
- Although in 2D (plain strain), it gives quantitative results.
- Empirical methodology (single scattering regime) have limitations in coarse grained structure.

Reference (isotropic) material

- Random ODF (Volume Fraction of orientation V)
- Weighted average on elastic tensor (T)

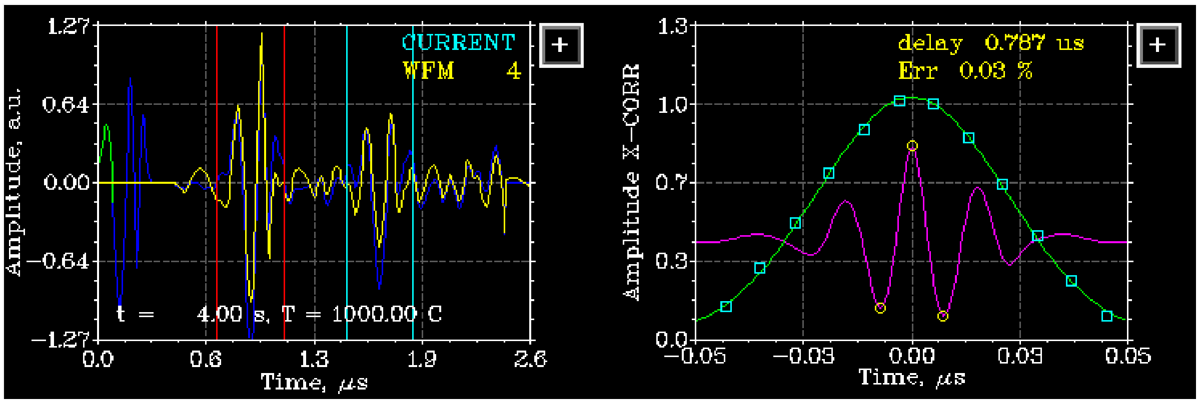
$$\langle T\rangle^{\text{Reuss}} = \left[\sum_{m=1}^{M} V_m T^{-1}(g_m^c)\right]^{-1}.$$

$$\langle T\rangle^{\text{Voigt}} = \sum_{m=1}^{M} V_m T(g_m^c).$$

```
#          Reuss (isoStress)           #             Hill (Average)            #          Voight (isoStrain)           #
#======================================#=======================================#=======================================#
#                                      #                                       #                                       #
#______Stiffness tensor (Unit : GPa)_____#_______________________________________#_______________________________________#
#                                      #                                       #                                       #
# | 173.6 112.0 112.4   2.1   1.1  -1.0| # | 188.3 104.6 105.1   2.3   1.2  -1.2| # | 202.9  97.2  97.9   2.6   1.4  -1.3| #
# | 112.0 173.9 112.2  -1.0  -2.1  -1.0| # | 104.6 188.5 104.9  -1.1  -2.4  -1.1| # |  97.2 203.2  97.5  -1.2  -2.6  -1.2| #
# | 112.4 112.2 173.4  -1.1   1.0   2.0| # | 105.1 104.9 188.0  -1.3   1.1   2.3| # |  97.9  97.5 202.6  -1.4   1.3   2.5| #
# |   2.1  -1.0  -1.1  30.8   1.9  -1.9| # |   2.3  -1.1  -1.3  41.7   2.2  -2.3| # |   2.6  -1.2  -1.4  52.5   2.5  -2.6| #
# |   1.1  -2.1   1.0   1.9  31.0   1.9| # |   1.2  -2.4   1.1   2.2  41.9   2.2| # |   1.4  -2.6   1.3   2.5  52.9   2.6| #
# |  -1.0  -1.0   2.0  -1.9   1.9  30.6| # |  -1.2  -1.1   2.3  -2.3   2.2  41.4| # |  -1.3  -1.2   2.5  -2.6   2.6  52.2| #
#                                      #                                       #                                       #
#_____Wave Velocity (mm/us)______________#_______________________________________#_______________________________________#
#                                      #                                       #                                       #
#    P-Wave   |    S1-Wave   |   S2-Wave   #    P-Wave   |    S1-Wave   |   S2-Wave   #    P-Wave   |    S1-Wave   |   S2-Wave   %
#-------------|--------------|-------------#-------------|--------------|-------------#-------------|--------------|-------------#
#------- X (RD) ---------------------------#-----------------------------------------#-----------------------------------------#
#    4.7787   |    2.0735    |   1.9493    #    4.9762   |    2.4032    |   2.2770    #    5.1662   |    2.6928    |   2.5631    #
#------- Y (TD) ---------------------------#-----------------------------------------#-----------------------------------------#
#    4.7820   |    2.0705    |   1.9441    #    4.9800   |    2.4003    |   2.2717    #    5.1703   |    2.6900    |   2.5577    #
#------- Z (ND) ---------------------------#-----------------------------------------#-----------------------------------------#
#    4.7755   |    2.0759    |   1.9542    #    4.9728   |    2.4053    |   2.2821    #    5.1625   |    2.6948    |   2.5685    #
```

Selection of appropriate averaging

- Velocity in the small grain size sample should be close to satisfy the isotropic condition



  - D = 30 um => V = 5.1035 ± 0.005
  - D = 100 um => V = 5.134 ± 0.048

	Reuss (isoStress)			Hill (Average)			Voight (isoStrain)		
	Wave Velocity (mm/us)								
	P-Wave	S1-Wave	S2-Wave	P-Wave	S1-Wave	S2-Wave	P-Wave	S1-Wave	S2-Wave
X (RD)	4.7787	2.0735	1.9493	4.9762	2.4032	2.2770	5.1662	2.6928	2.5631
Y (TD)									