Application of Laser Ultrasonics for the evolution of microstructure in INCONEL 718 superalloy

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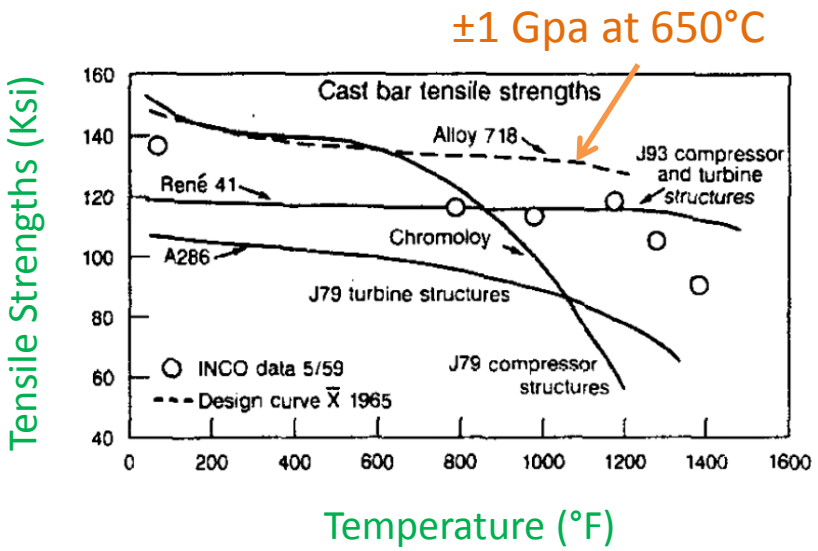
Motivation

- ✓ 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



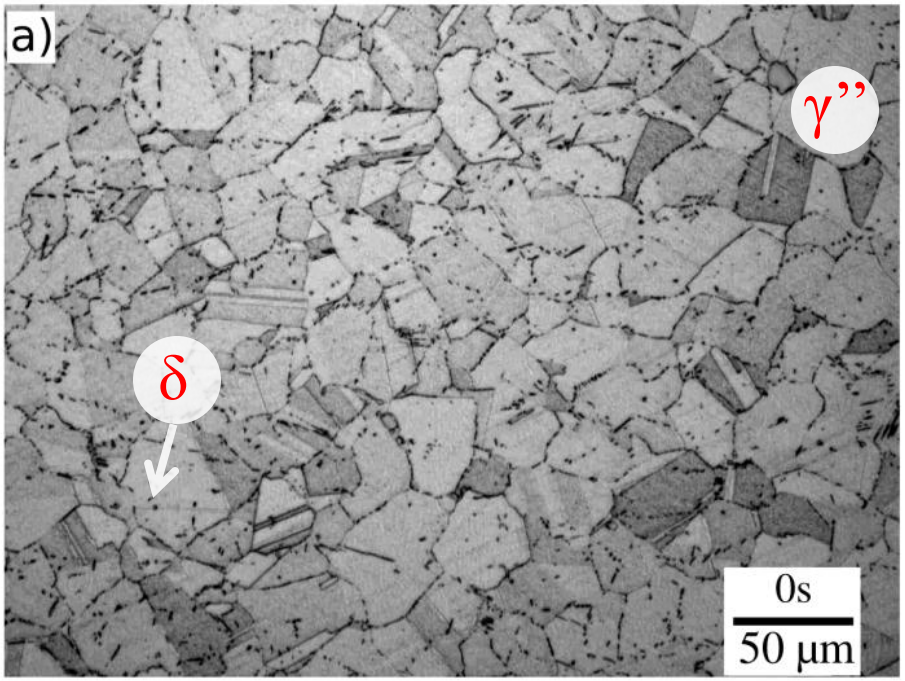
J. F. Barker
The initial years of alloy 718.
TMS Superalloys 718, 625, 706 and various derivatives (1989)

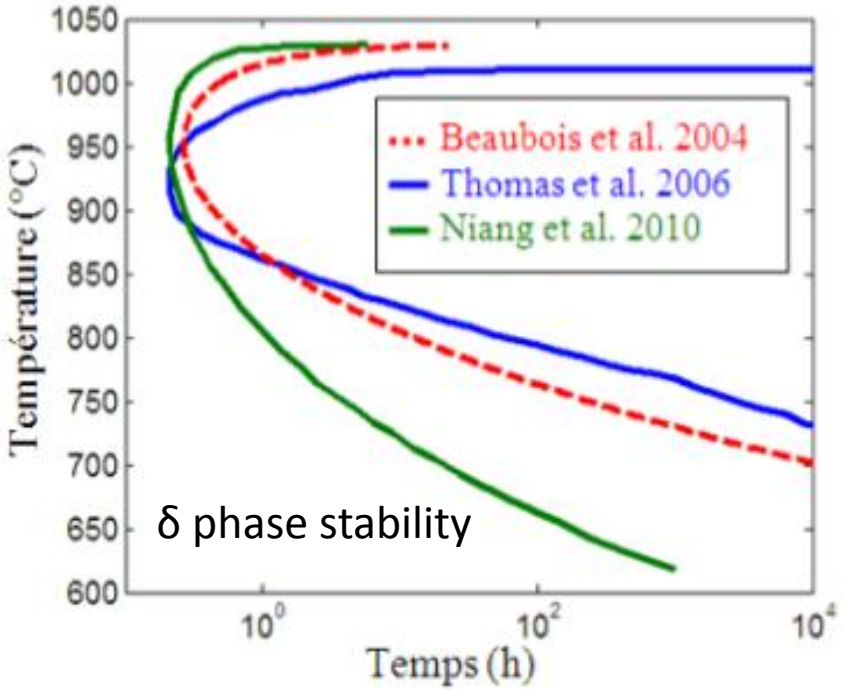
Material INCONEL 718

- ✓ Composition (Key elements wt.fraction)

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

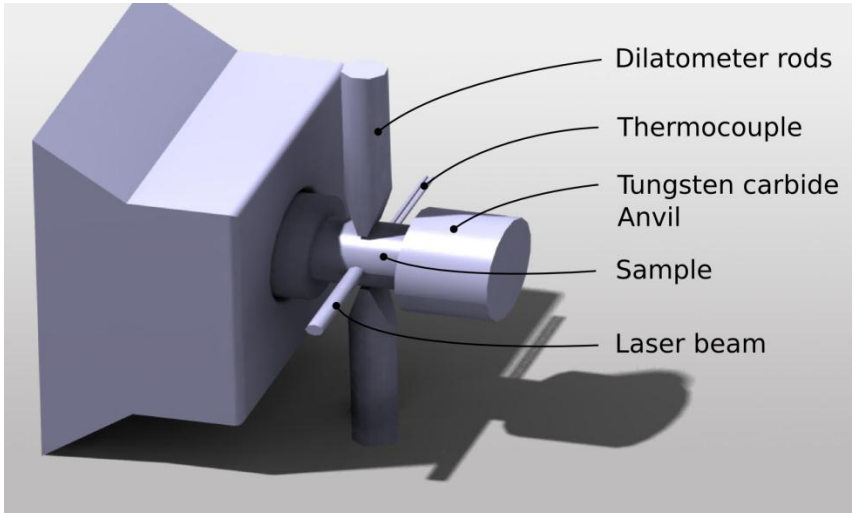
- ✓ Grain size 24 µm, globular delta phase precipitates

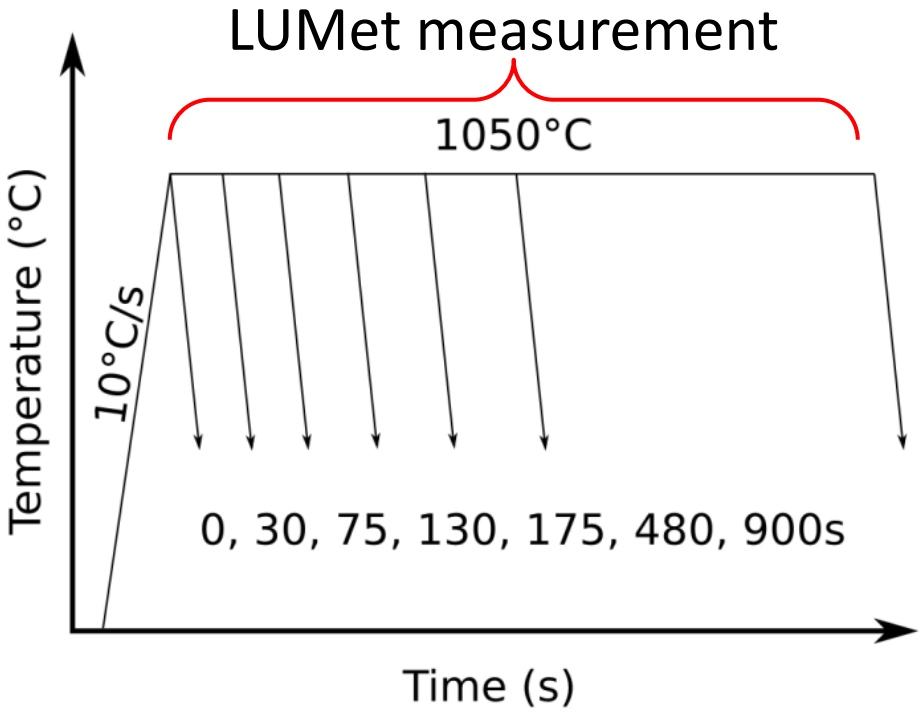




Experiments

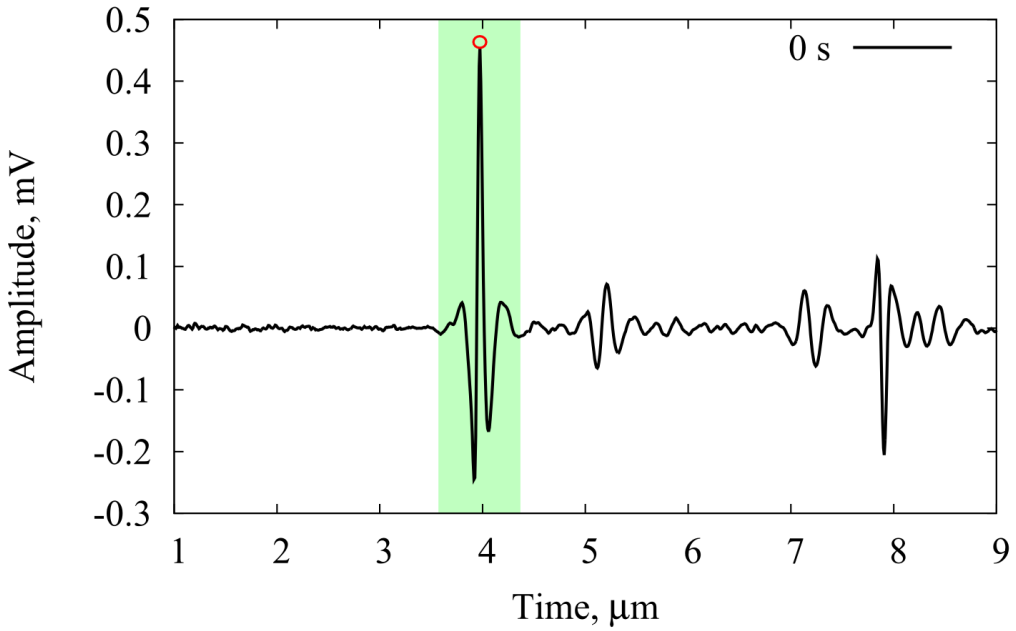
- ✓ Isothermal holding at 1050°C for various time
- ✓ Attenuation measurement under isothermal conditions
- ✓ Validation with metallography

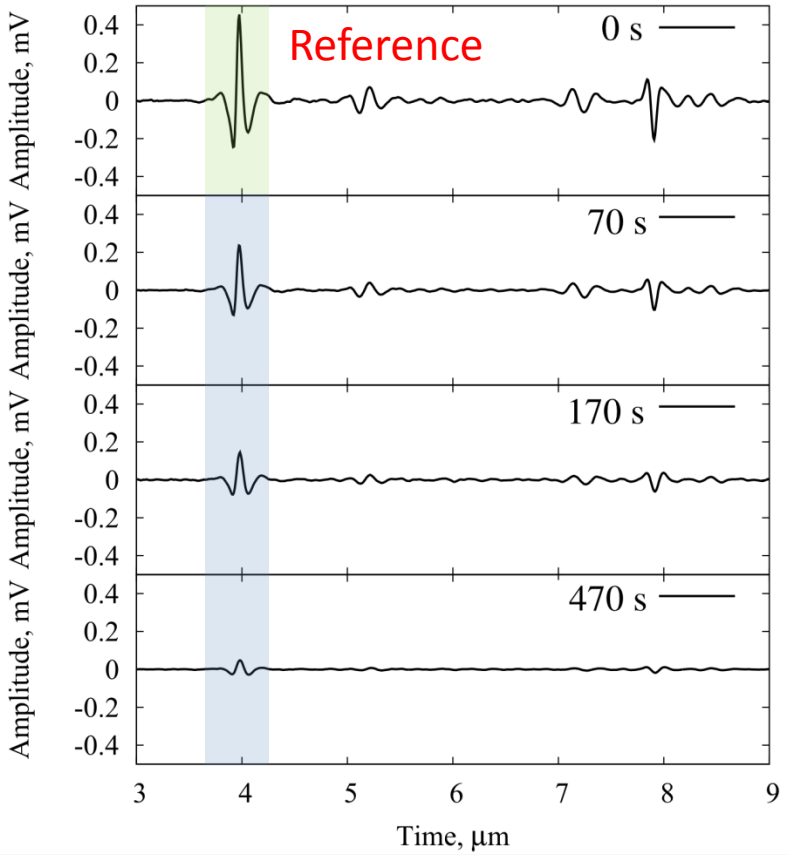




Ultrasound signal

- ✓ 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

$$\alpha(f) = -\frac{20}{2D} log10\left(\frac{g(f)A_{sc}(f)}{g(f)}\right) \begin{array}{l} \longleftarrow t \neq t_{ref} \\ \longleftarrow t = t_{ref} \end{array}$$

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

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

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

- ✓ Absolute grain size measurement

Modified single echo technique

- ✓ Here, reference has scattering contribution

$$\alpha(f) = -\frac{20}{2D} log10\left(\frac{g(f)A_{sc1}(f)}{g(f)A_{sc0}(f)}\right) \begin{array}{l} \longleftarrow t \neq t_{ref} \\ \longleftarrow t = t_{ref} \end{array}$$

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

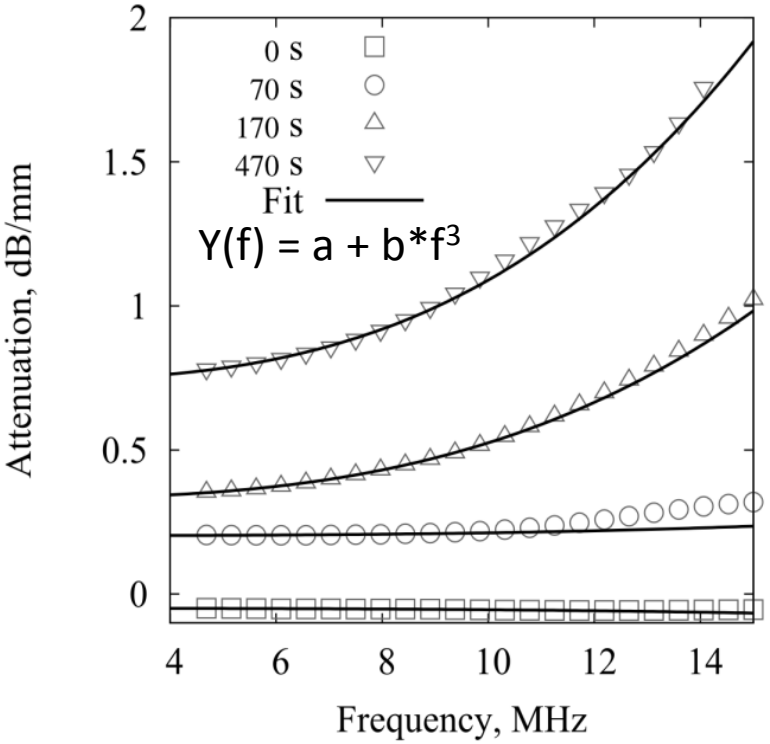
$$\alpha\,(f) = a \quad + C(D_1^{n-1} - D_0^{n-1})f^n$$

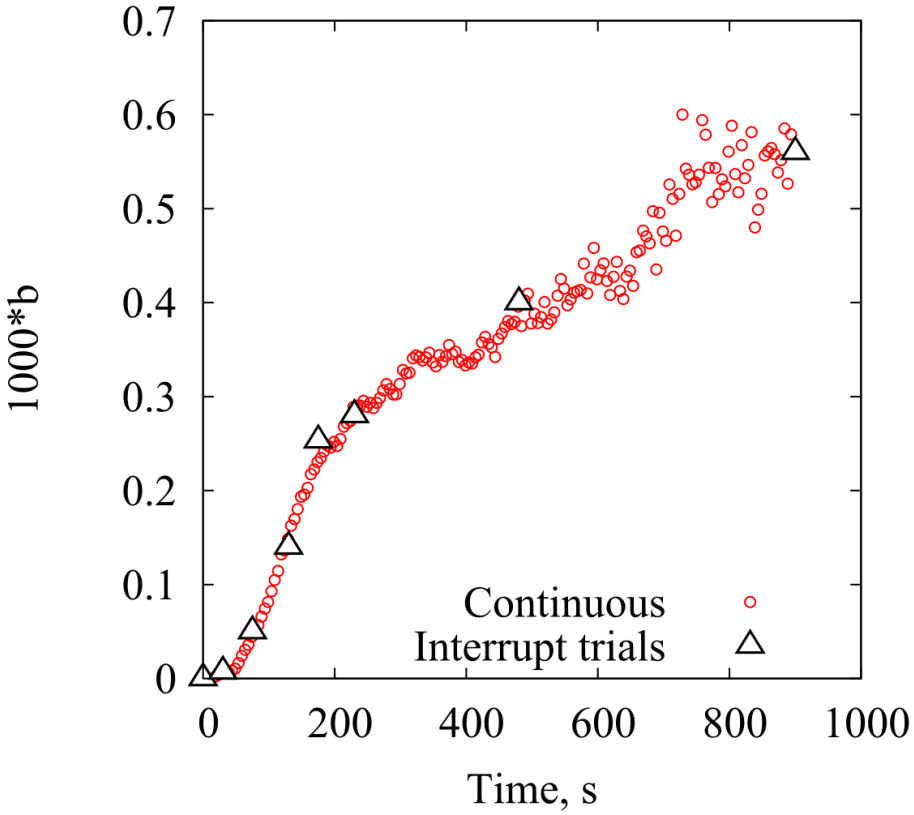
$$b \propto C\left({D_1}^2 - {D_0}^2\right)$$

- ✓ Relative change in grain size

Attenuation, grain size parameter

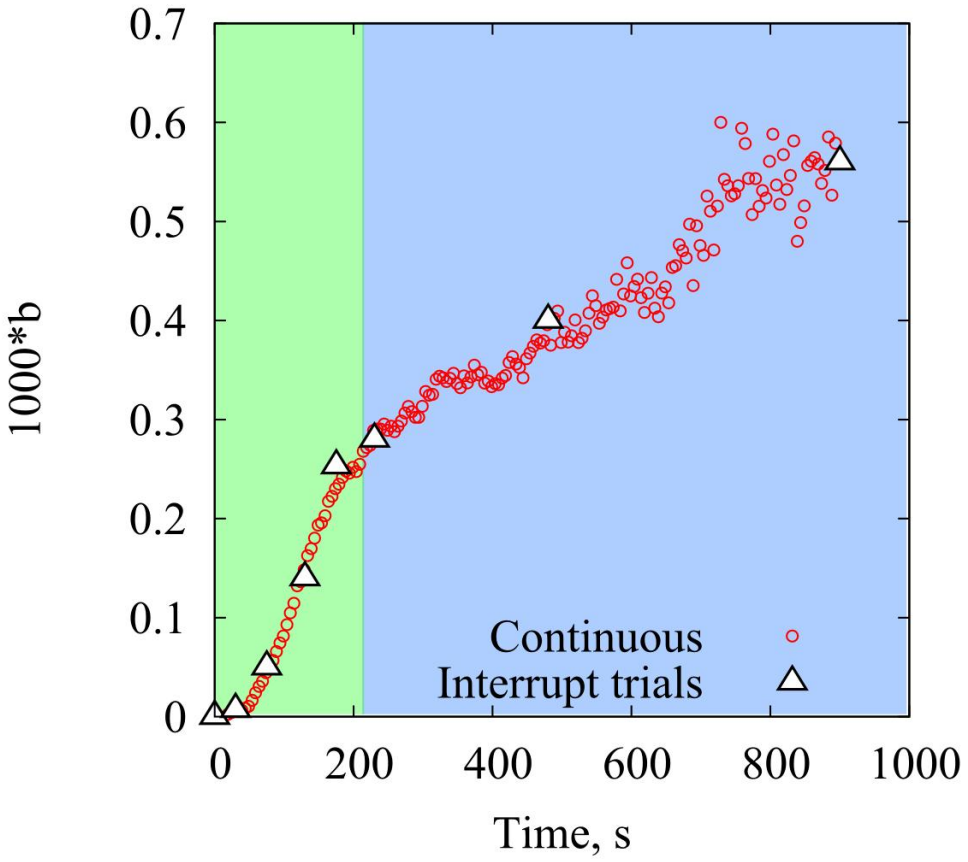
- ✓ Systematic evaluation of the grain size parameter b from the measured attenuation spectrum





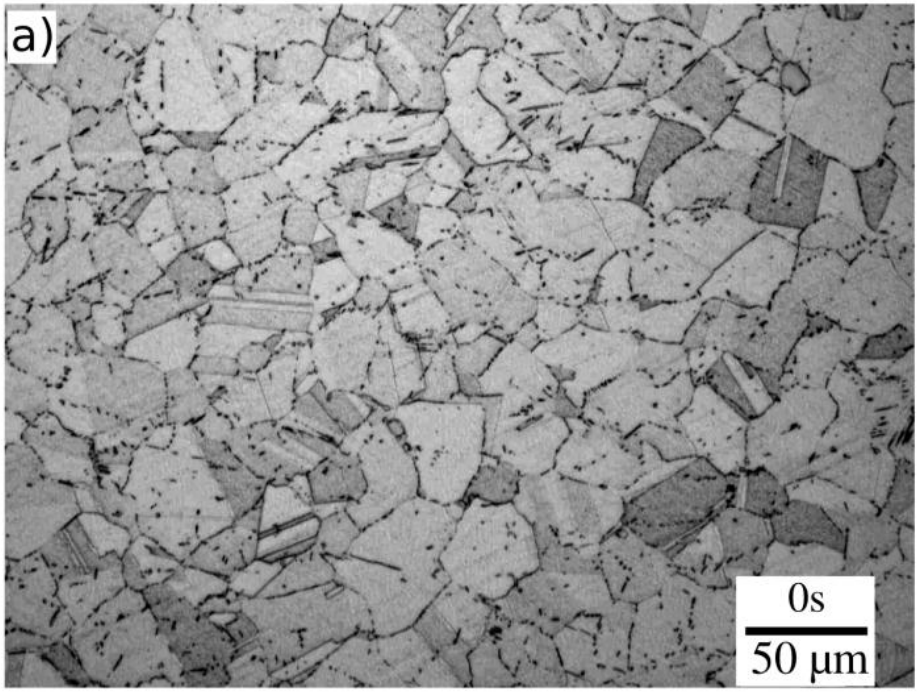
Attenuation, grain size parameter

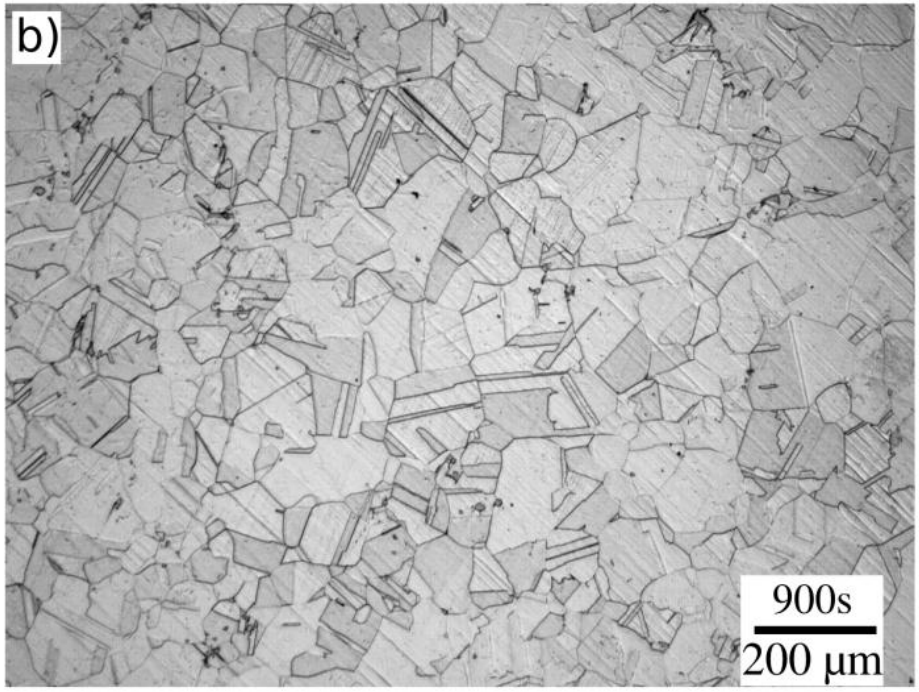
- ✓ Identification of two main stages
- ✓ 75s to 200s, rapid increase
- ✓ Above 200s, steady raise at slower rate
- ✓ Don’t reach a limiting value at 15 min



Initial and final stage

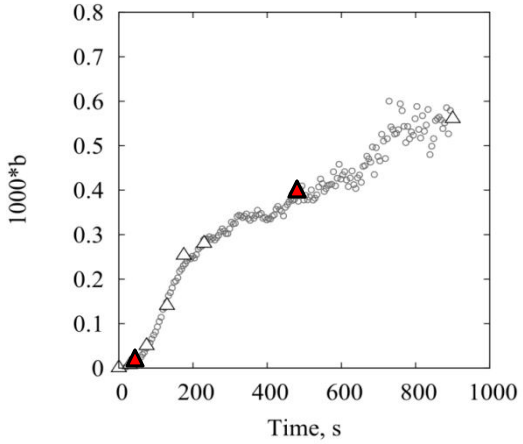
- ✓ Average grain size increases by a factor of 4 during the 15 mn annealing
- ✓ 900 s: Delta phase is almost fully dissolved
- ✓ 900 s: Formation of annealing twins

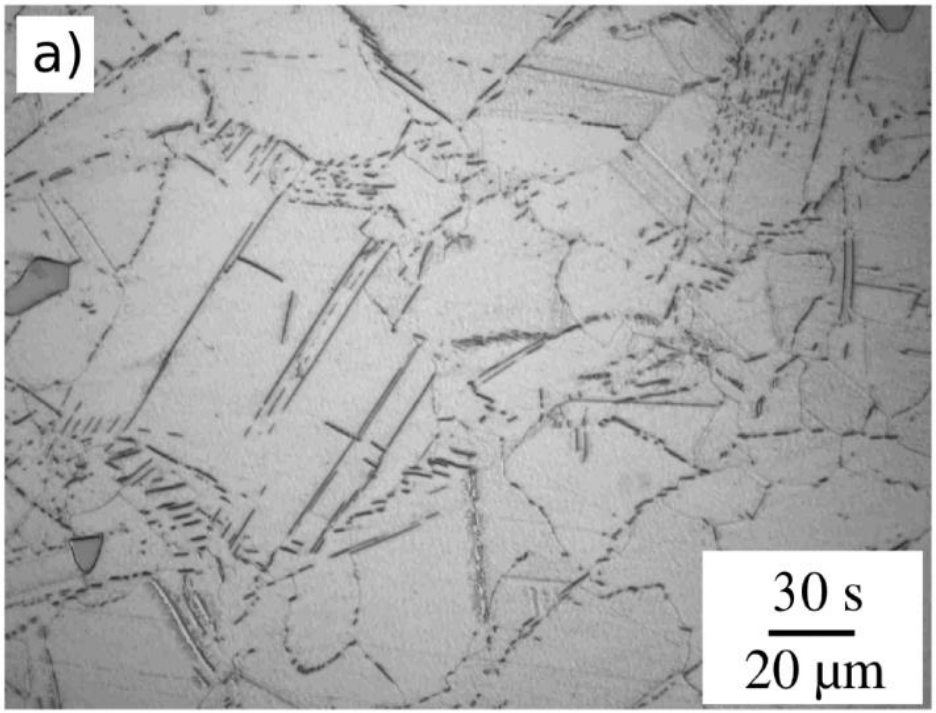


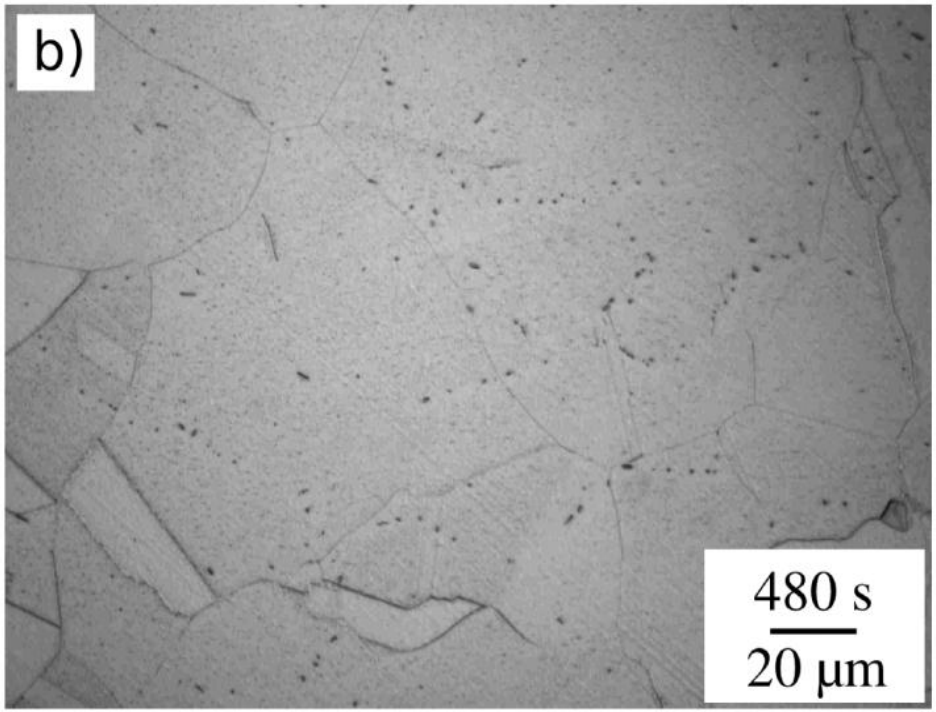


Evolution of Delta phase

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

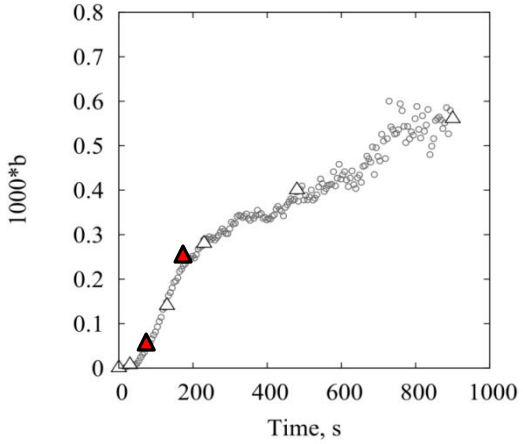


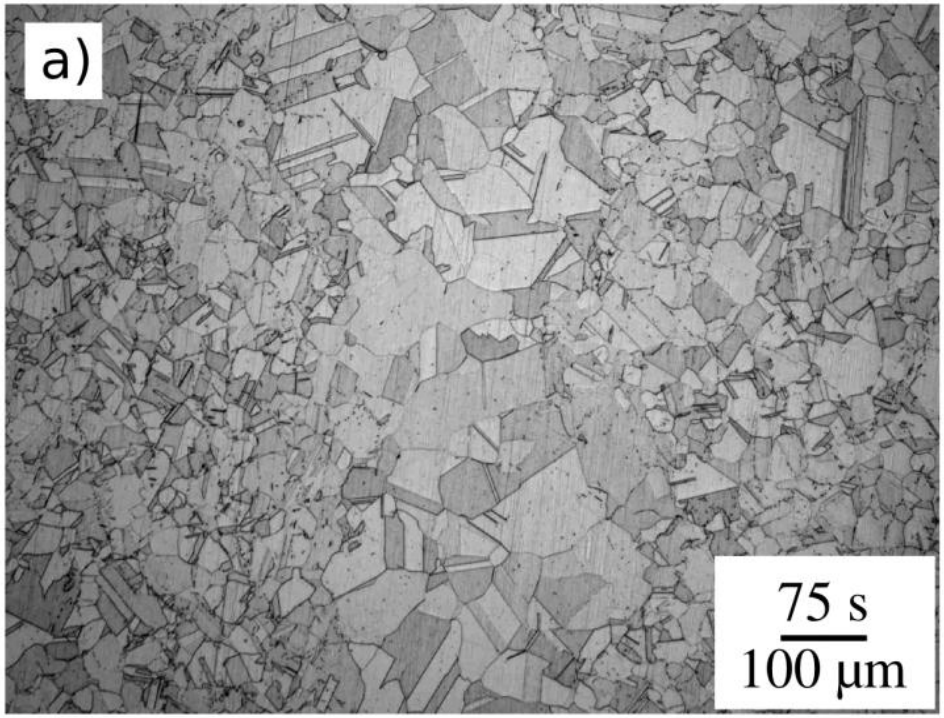


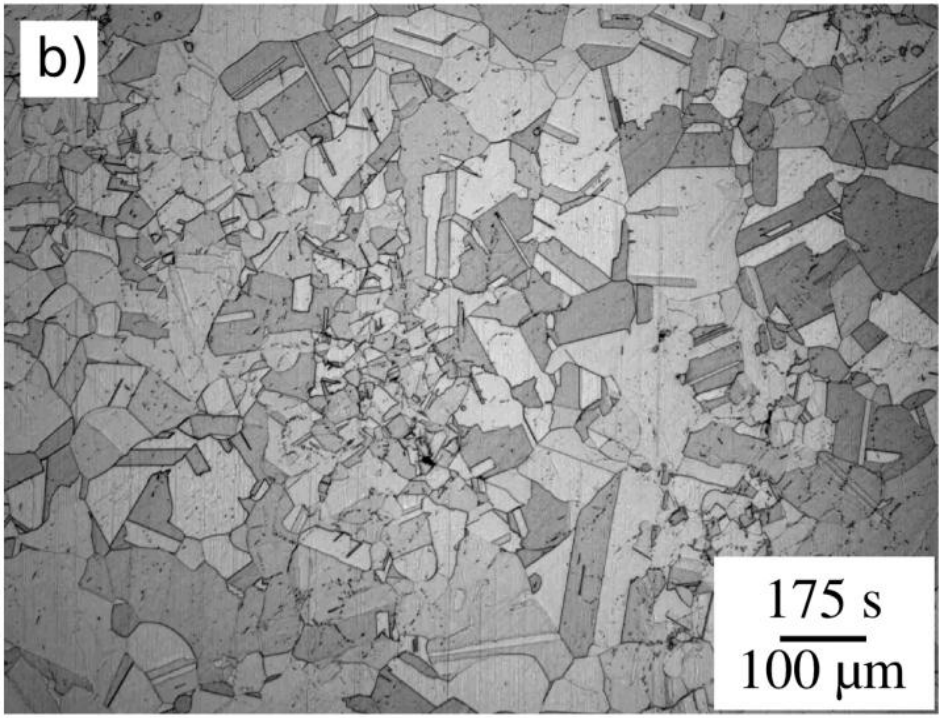


Heterogeneous grain structure

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







Mean grain size, distribution

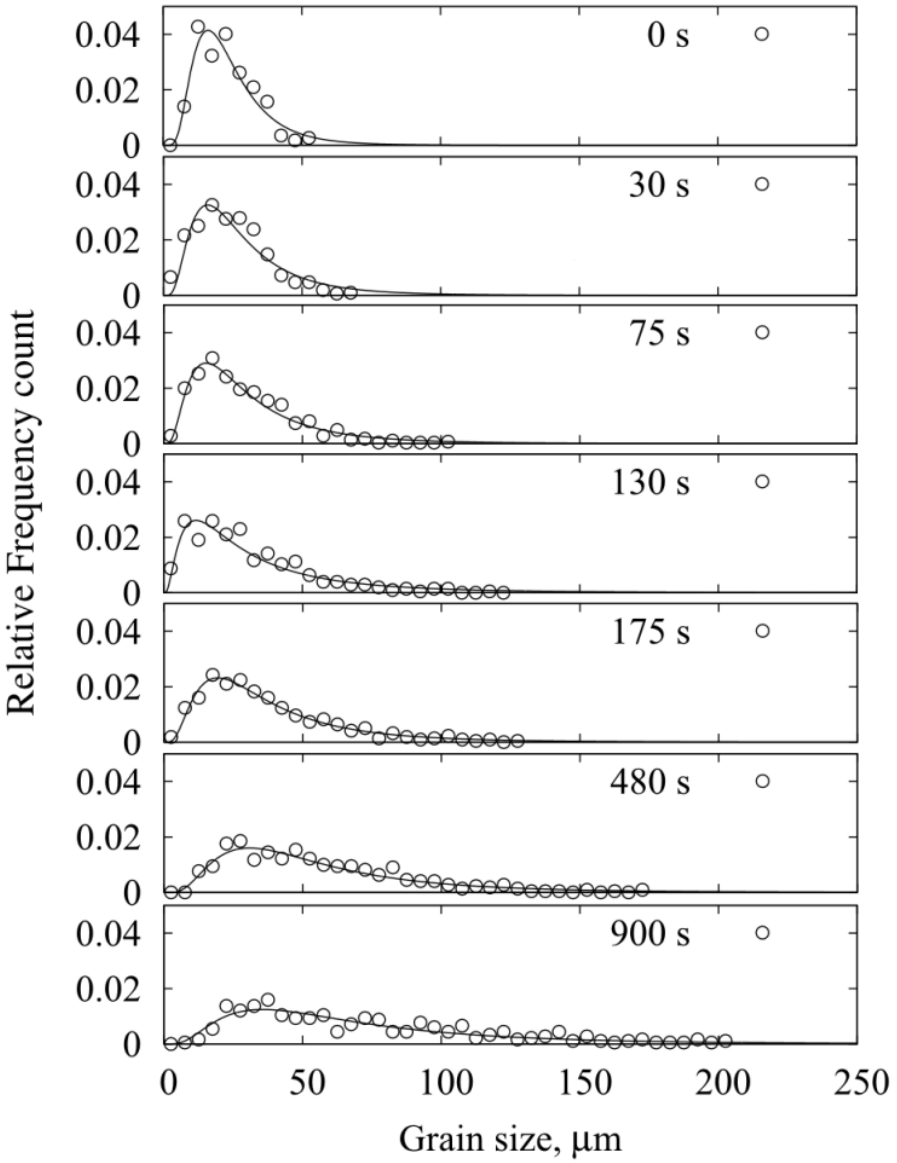
- ✓ Quantitative analysis of optically measured grain area

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

- ✓ Log normal distribution, M, S

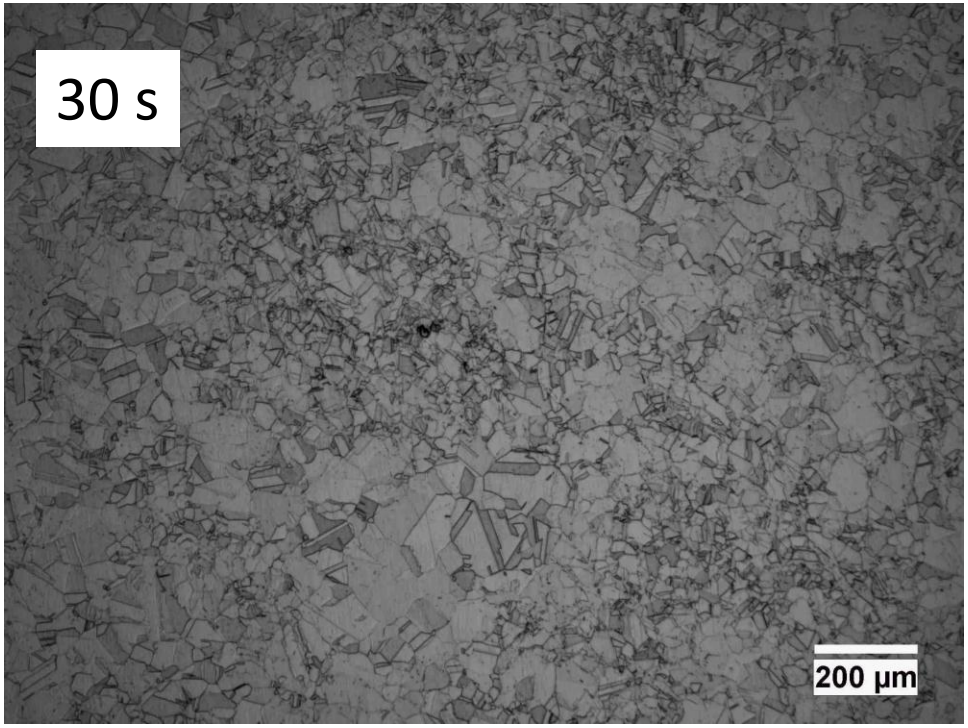
$$\mu = \exp(M + \frac{S^2}{2})$$

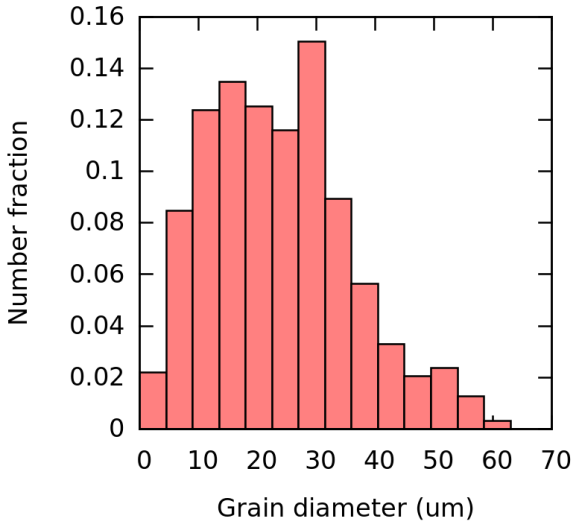
Time	EQAD	μ(M)	σ(S)
0	24	**24** (3.05)	**13** (0.52)
30	27	**29** (3.17)	**20** (0.63)
75	33	**32** (3.20)	**25** (0.70)
130	37	**38** (3.24)	**42** (0.89)
175	44	**40** (3.45)	**32** (0.70)
230	46	**43** (3.50)	**32** (0.68)
480	62	**59** (3.86)	**42** (0.65)
900	82	**74** (4.07)	**60** (0.70)

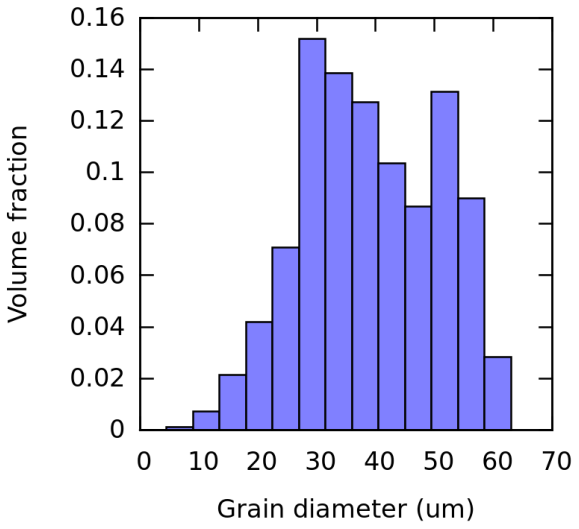


Volume fraction

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

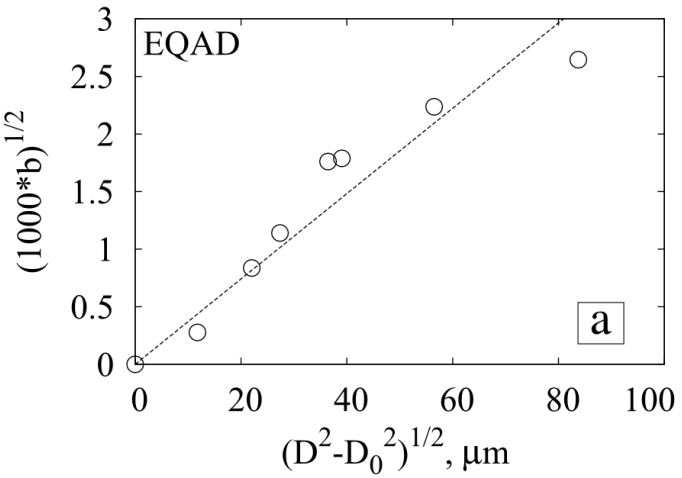


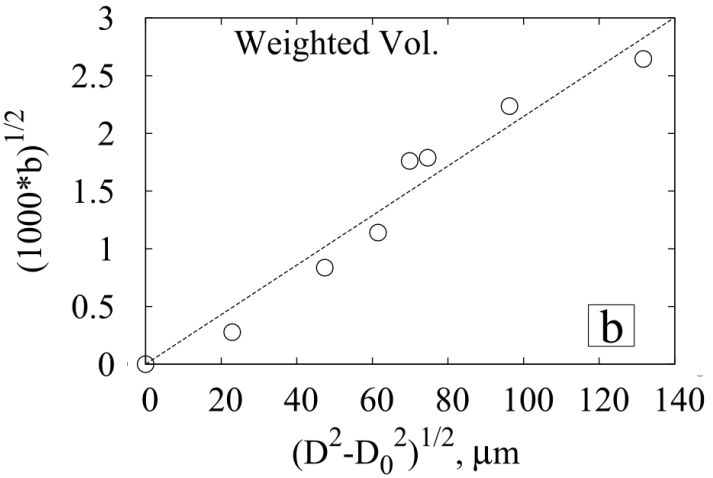




Attempt to build calibration

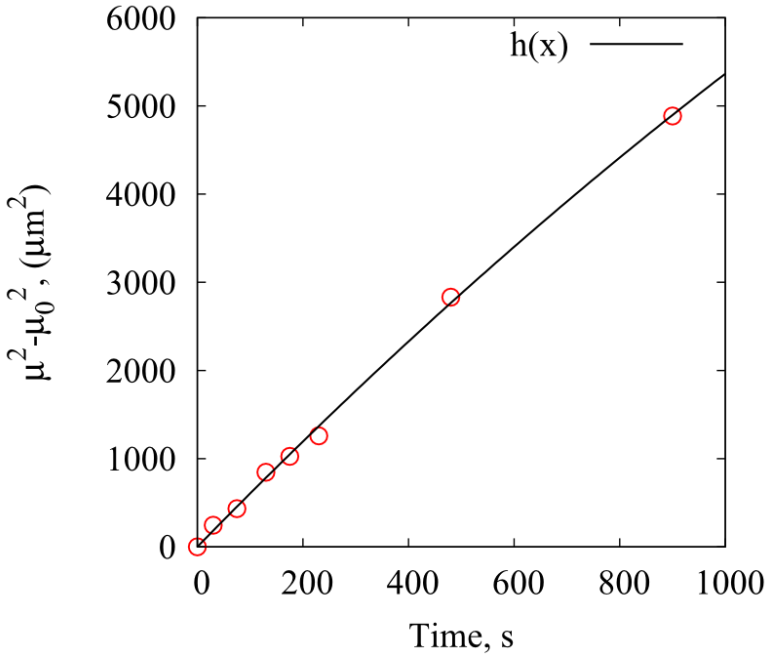
- ✓ Simple relation between mean grain size and grain size parameter ?
- ✓ Mean grain size only is not correctly describing the evolution of the size distribution.
- ✓ LUMet seems sensitive to variation in the distribution.
- ✓ Larger grains contribute more to the LU signal

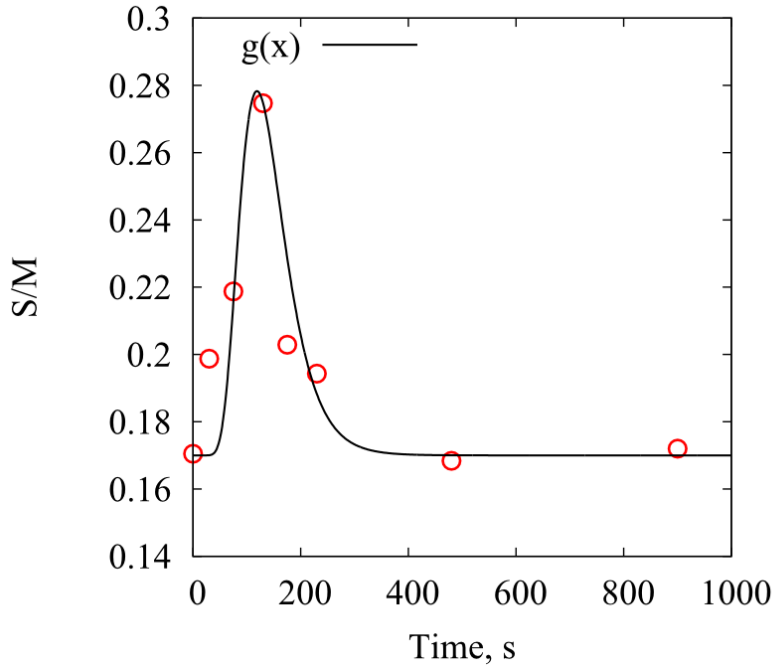




Attenuation and size distribution

- ✓ 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)



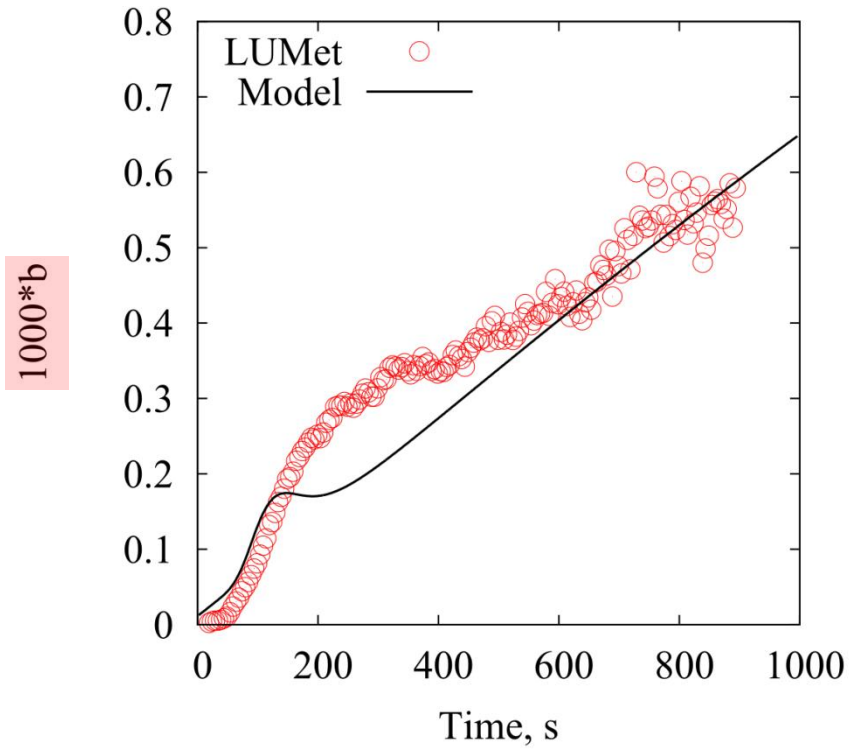


Prediction of the attenuation parameter

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

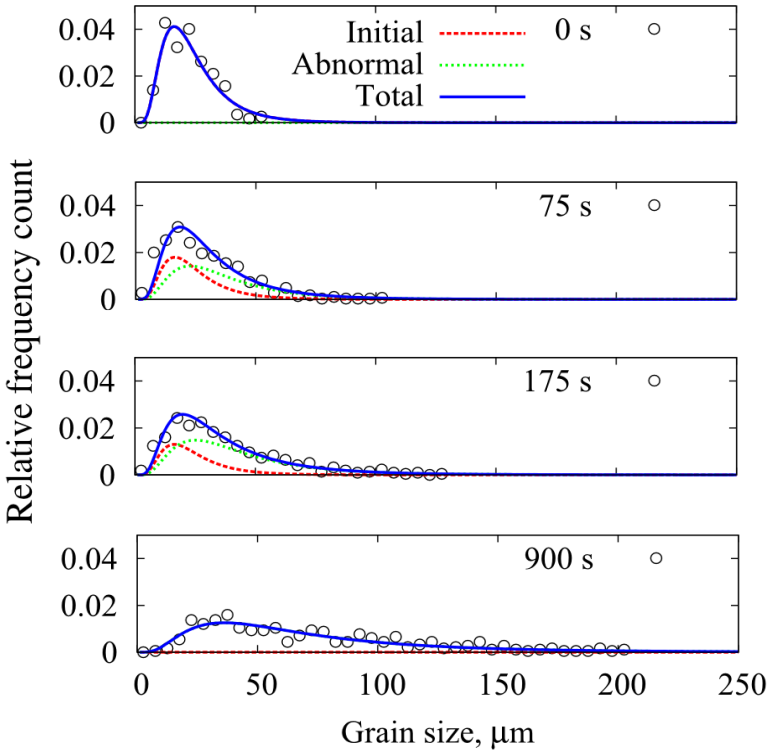
$$\alpha\left(f, F(D)\right) = Cst \cdot \sum_{i}^{n} F(D_i) {D_i}^2 f^3$$

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



Future work

- May be better to consider a bi-modal distribution composed of :
  - Initial distribution F_0 (M_0,S_0)
  - Distribution of larger grain growing F_L (M_L,S_L)
- What is the best manner to include the twins in the grain size statistic ?
- Can we define or extract a parameter related to the width of the distribution?



Conclusions

- ✓ Grain growth influenced by the heterogeneous dissolution of the delta phase
- ✓ Mean grain size not a sufficient parameter to construct the ultrasound calibration
- ✓ LUMet measurement may be capable of indicating the end of period of “abnormal” grain growth
- ✓ Can rapidly give important indication on the time required for annealing prior to forging.