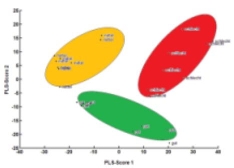
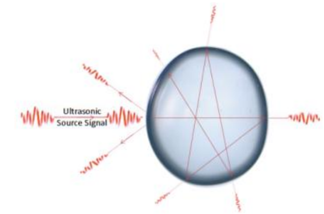
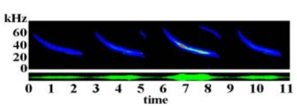
(Bio-) Process Technology, Process Analysis Technology

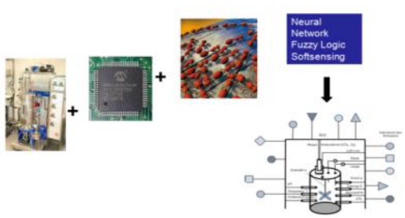
Hussein, M.

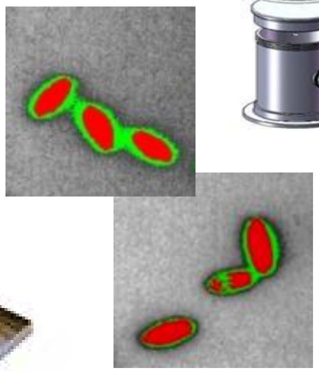


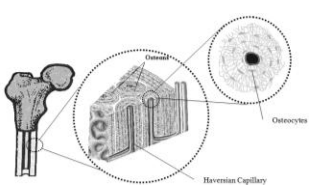


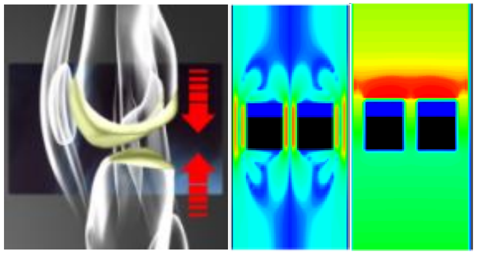
PAT
(Bio-) Process Analysis & Technology
Biotechnological Modeling Monitoring Sensing
CHEMOMETRICS
ULTRASONIC
SIGNAL PROCESSING
IMAGE PROCESSING
NUMERICAL MODELING
MICRO SENSING

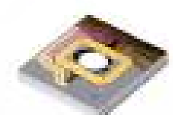












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Ultrasonic Measurement Techniques for Process Monitoring using the Example of Concentration Monitoring of Fermentation Fluids

Sven Hoche; M.A. Hussein; T.Becker

Requirements for industrial in-situ sensors

→ Requirements for process monitoring sensors:

- **robustness** (pressure, temperature, vibration, shock)
- high chemical **corrosion resistance**
- no **detrimental effect** on the process (contamination)
- **rapid response**
- **low investment and running costs** (minimal maintenance , energy consumption, …)
- easy **retrofit** for existing plants
- **standardized system integration** - mounting (flanges etc.), communication (output signal: 4–20 mA, Profibus, Fieldbus Foundation)
- conformity with standards (**hygienic standards, standards for electrical apparatus,…)**

Why ultrasonic techniques - the basics

→ Advantages:

- ultrasonic techniques are **non invasive**
- applicable **for in-line measurements, even in-situ**
- **low power consumption**,
- robust, **long-term stable** (maintenance free)
- **rapid response**, usually a fraction of a second
- **high resolution accuracy, high-precision measurement**
- no direct medium contact
  - → suitable for sterilisation and cleaning in process
  - → suitable for use in hazardous environments. . .
- no restrictions regarding optical or electrical properties of the medium
- works in a wide range of temperature an pressure

Why ultrasonic techniques - the basics

→ Disadvantages:

- **increase of effort in electronics for high accuracy** and information enhancement (this does not necessarily mean high costs)

- **acoustical transparency** of substances for transmission and some reflection techniques

- ultrasonic measurements are highly disturbed when gas bubbles in liquids are present – **noise, high signal variations**

- only **integral information** along the entire sound path is delivered

- ultrasonic signals tend to be complicated and need relatively **complex signal processing**

- knowledge of the **acoustic properties** of the substances through which the sound is travelling is often necessary

- **increase of the attenuation** of sound with frequency.

- **temperature and pressure dependent** measurement

The Options – Ultrasonic Sensor Types

- **Ultrasonic Distance Sensors**
  - distance, position, level
  - path
  - object structure
  - multidimensional recognition
  - presence of objects
  - process monitoring
  - motion, speed, path (Doppler effect)

- **Ultrasonic Propagation Path Sensor**
  - concentration of multicomponent systems
  - particle size distribution (suspensions, emulsions)
  - volume and mass flow velocity
  - transmission properties
  - temperature measurement
  - dynamic pressure measurement

- **Impedance Sensor**
  - density
  - acoustic impedance

- **Mass Sensitive Sensors**
  - mass
  - viscosity
  - specific chemical or biological species
  - multicomponent analysis

The Options – Industrial Applications

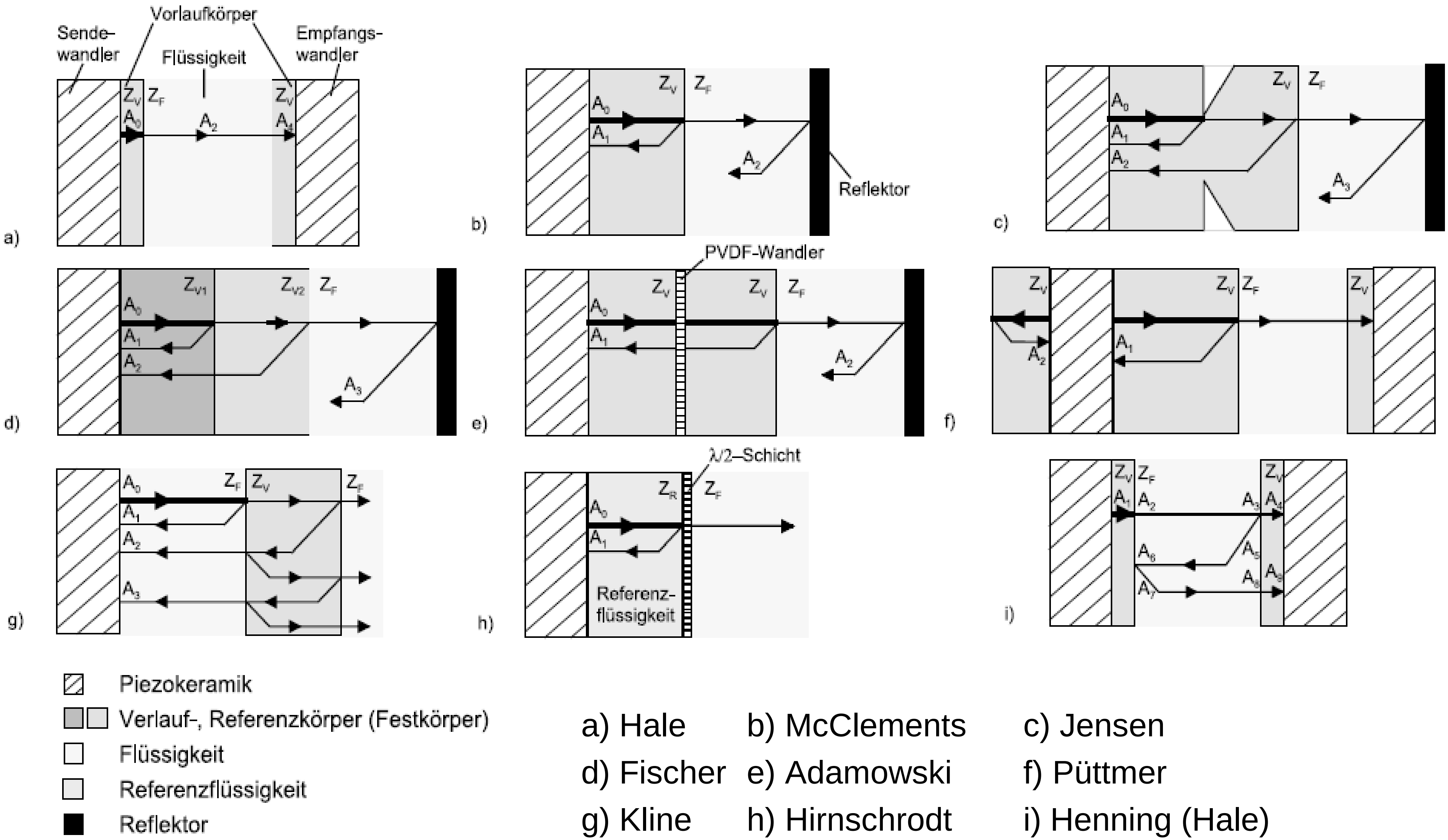
- **Chemical and pharmaceutical industry:**
  – Polymerisation
  – Galvanic
  – Lacquers and paints
  – Waste water treatment

- **Food industry:**
  – Beverage industry
  – Starch production
  – Margarine production
  – Dairy industry

- **Biotechnology:**
  – Alcoholic or lactic acid fermentation
  – Enzyme concentration

- **General other application**
  - particle/ bubble size
  - rheological properties

Ultrasonic Sensors for Concentration Measurement



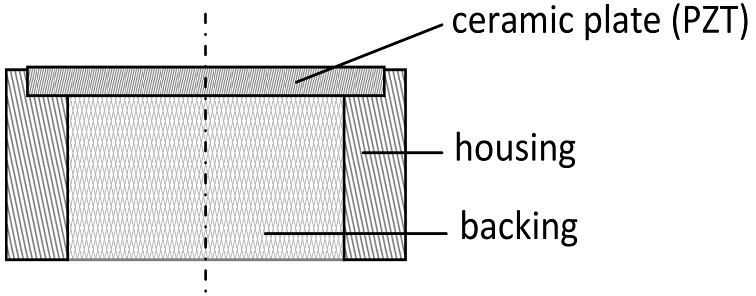
a) Hale	b) McClements	c) Jensen
d) Fischer	e) Adamowski	f) Püttmer
g) Kline	h) Hirnschrodt	i) Henning (Hale)

Alcoholic Yeast Fermentation Monitoring

- **In-Line-Monitoring (Control)** of sugar and ethanol concentration as important indicators for the status of alcoholic yeast fermentation processes.

- Current Work: Optimization and validation of an **ultrasound based inline sensor system.**

- **Principle**: pulse-echo; impulse generation (dirac impulse: 0-100 V, 2.5E-7s), signal processing and data transfer via data acquisition system.



System integration

Process Control System

Field Bus Communication
(PROFIBUS)

Process
Control Center

Sensor Settings
Signal Processing Settings
Process Information (T, TOF,…)

multichannel signal transformer

Microcontroller (Blackfin: 400 MHz, 2 MB Flash, 32 MB SDRAM)
→ firmware including signal processing algorithm

Ultrasonic Board (25 MHz signal resolution)

PT Board

Pulse – Echo – Configuration

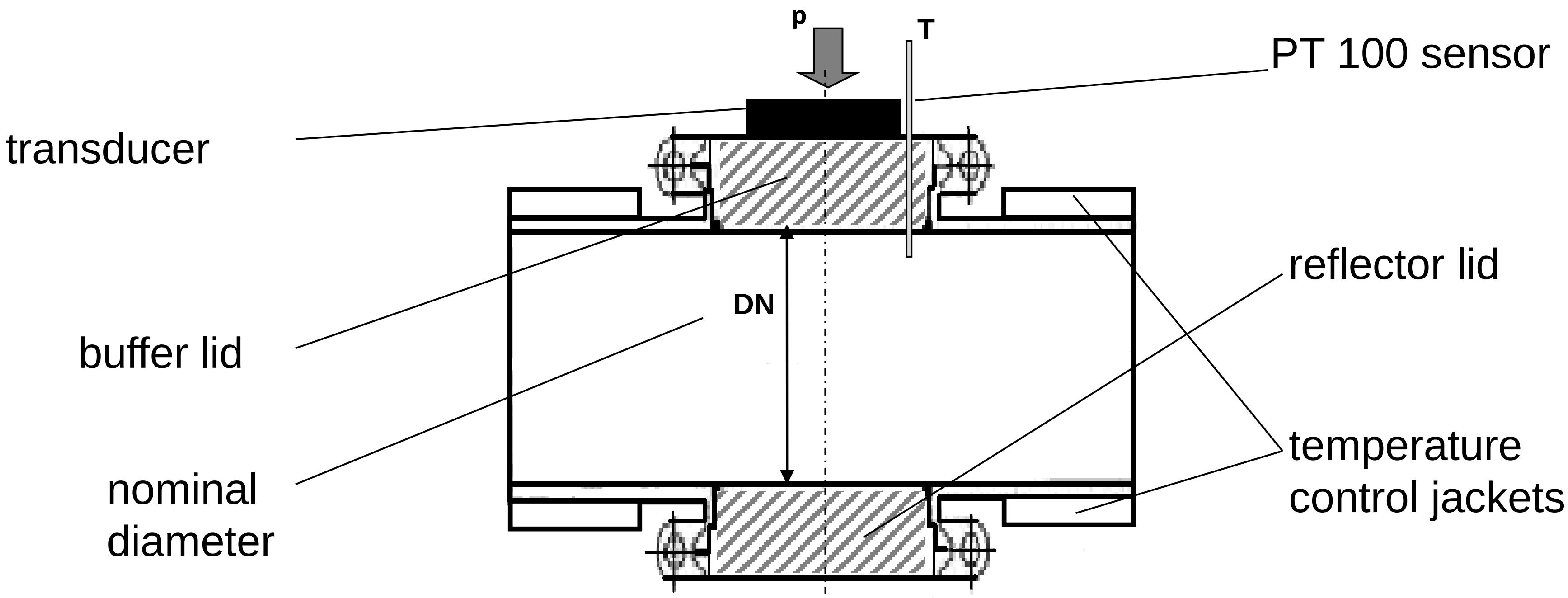
2 MHz PZT
Transducer

Temperature
Sensor (PT 100)

Laboratory Setup

→ pulse-echo setup based on a VARINLINE® access unit (typical process access for inline sensors in food industries, stainless steel)

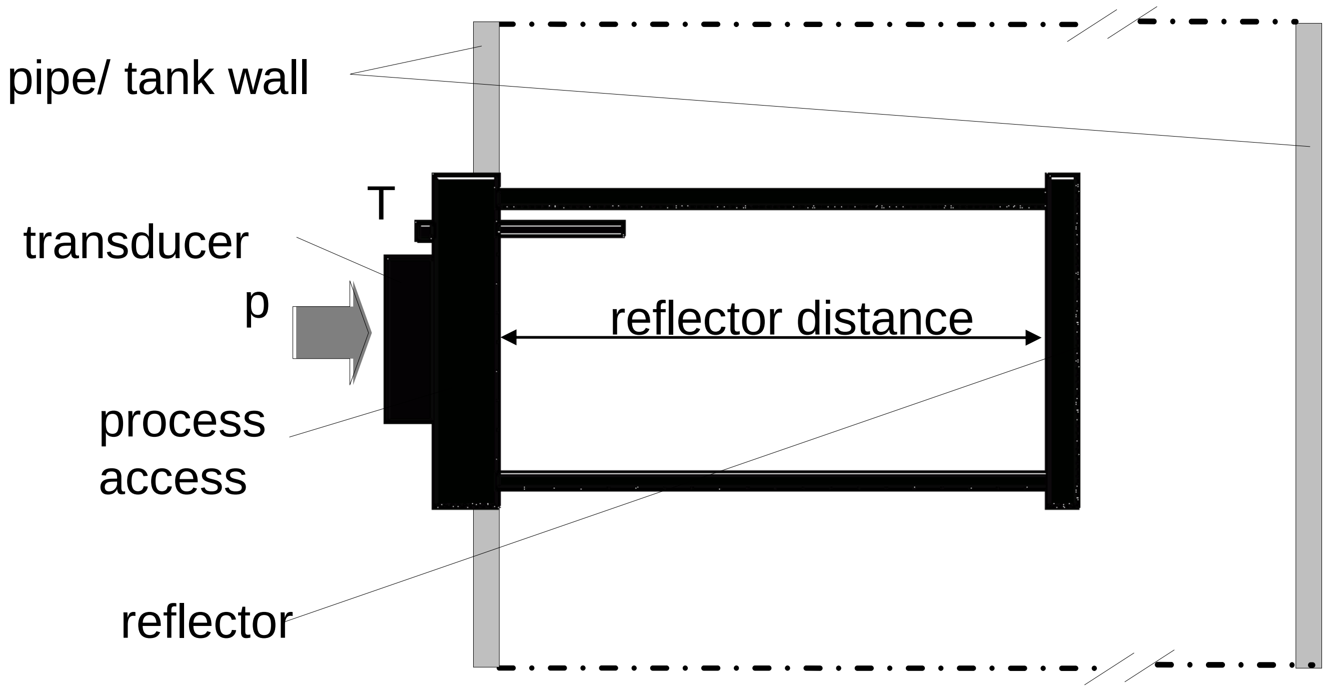
→ two nominal diameters were used, 50 mm (DN50) and 80 mm (DN80) (defines the approximate propagation path for the signal)



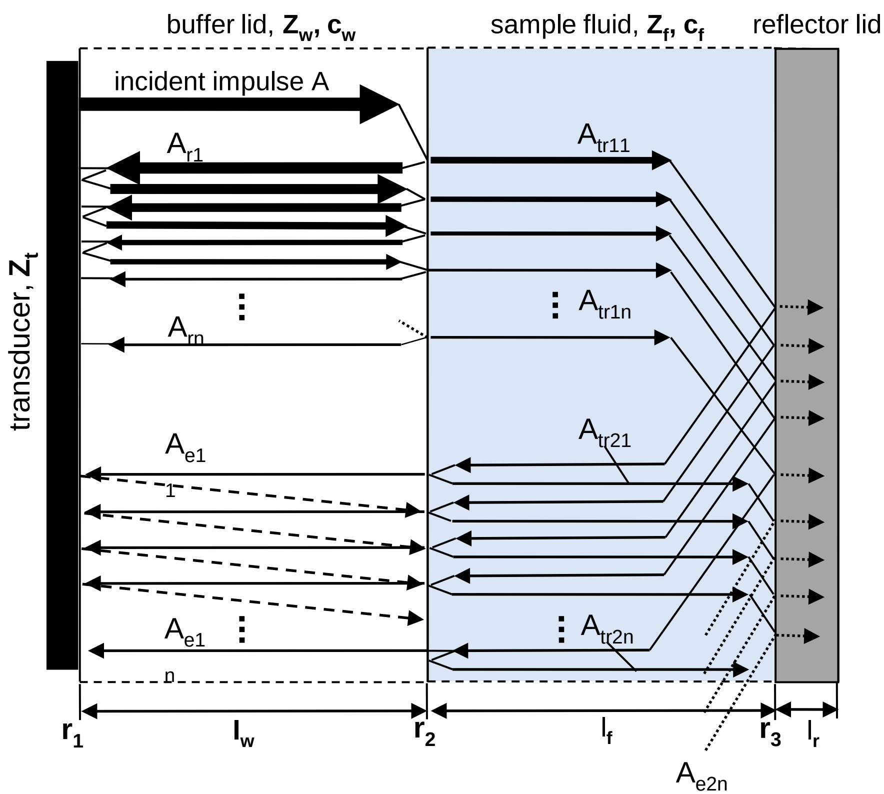
In-Site Setup

→ pulse-echo setup based on a VARINLINE® access unit

→ adjustable reflector distance, current 70 mm



Puls-Echo-Method



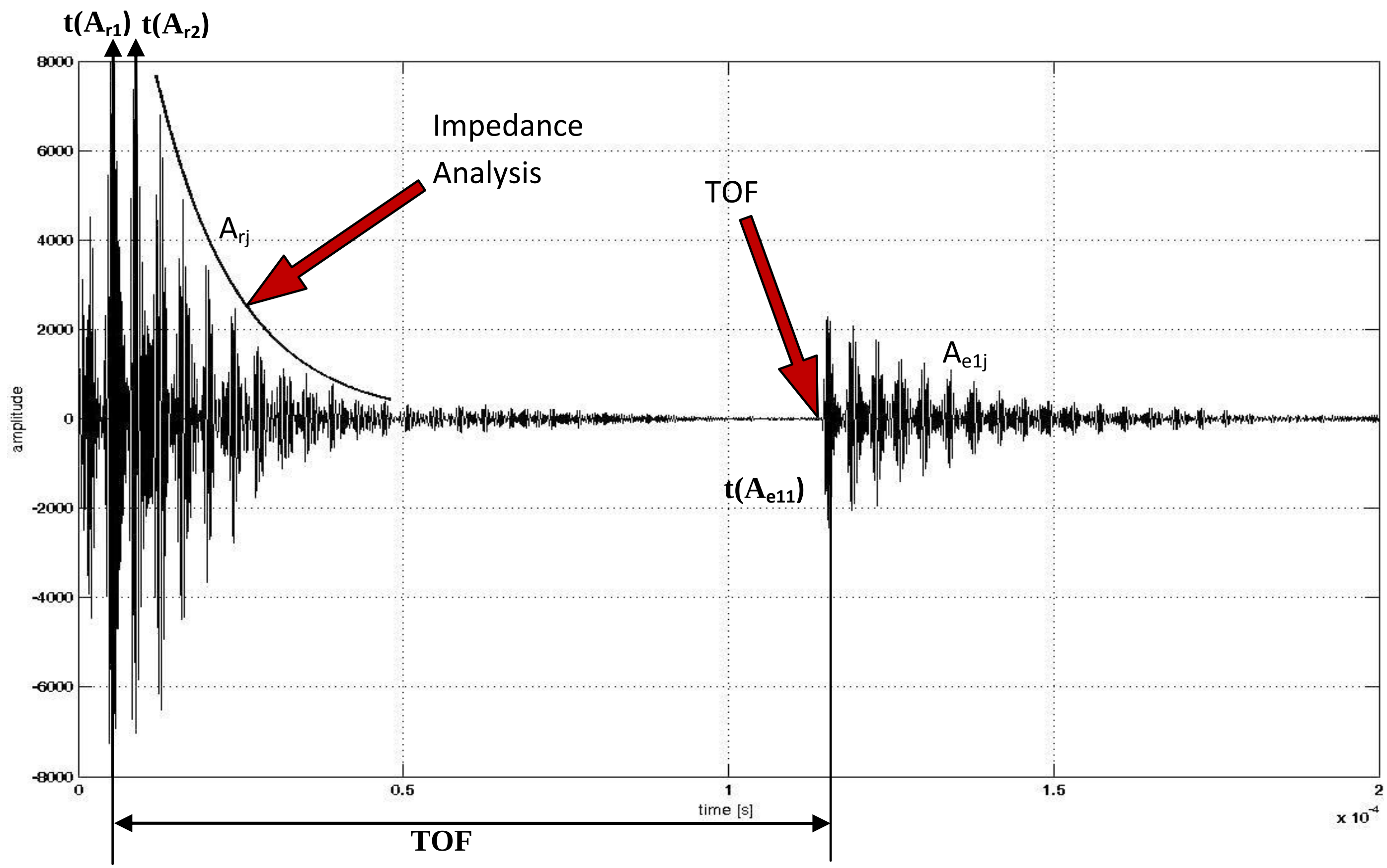
where:

A_{rn}:	from interface buffer fluid reflected impulse
A_{trjn}:	pulses, traveling from interface buffer-fluid to fluid-reflector
A_{ejn}:	echoes from fluid-reflector interface
Z:	medium impedance
c:	medium USV
r_1:	reflection coefficient of transducer-buffer interface
r_2:	reflection coefficient of buffer-fluid interface
r_3:	reflection coefficient of fluid-reflector interface
j:	index for the echo
n:	index for single pulses inside a set of multiple reflections

→ Impedance from reflections inside buffer

→ USV from echo (reflector)

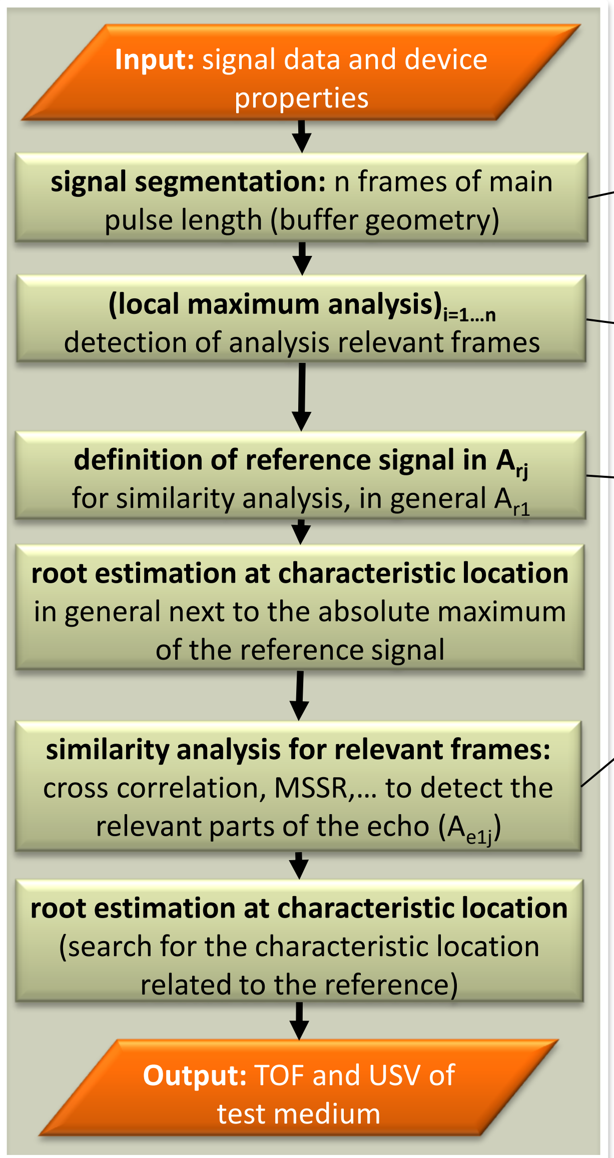
Example Signal



→ Exponential Decay of Buffer Reflections: $$A_n = A \cdot r_1^{\,n} \cdot r_2^{\,n-1} \cdot \exp(2 \cdot n \cdot \alpha_1 \cdot l_w)$$

→ Time of Flight (TOF): $$TOF = t(A_{e11}) - t(A_{r1})$$

TOF - Algorithm



frame definition due to known setup values
→ wall thickness (wt), wall material (wc)
frame time = sf·(2·wt)/wc   sf…safety factor (due to material variations)

frame wise maximum amplitude analysis

global threshold method to reduce analysis

constant constraints

Bivariate Time Series Analysis:

Mean Square Sum of Residuals

$$MSSR(k) = \frac{\sum_{i}^{n} \left| A_{reference}(k+i) - A_{frame}(k+i) \right|}{n}$$

Cross Correlation

$$corr(k) = \frac{\sum_{i}^{n} \left[A_{reference}(k+i) * A_{frame}(k+i) \right]}{n}$$

TOF - Algorithm

Input: signal data and device properties

↓

signal segmentation: n frames of main pulse length (buffer geometry)

↓

(local maximum analysis)$_{i=1...n}$
detection of analysis relevant frames

↓

definition of reference signal in A_{rj}
for similarity analysis, in general A_{r1}

↓

root estimation at characteristic location
in general next to the absolute maximum of the reference signal

↓

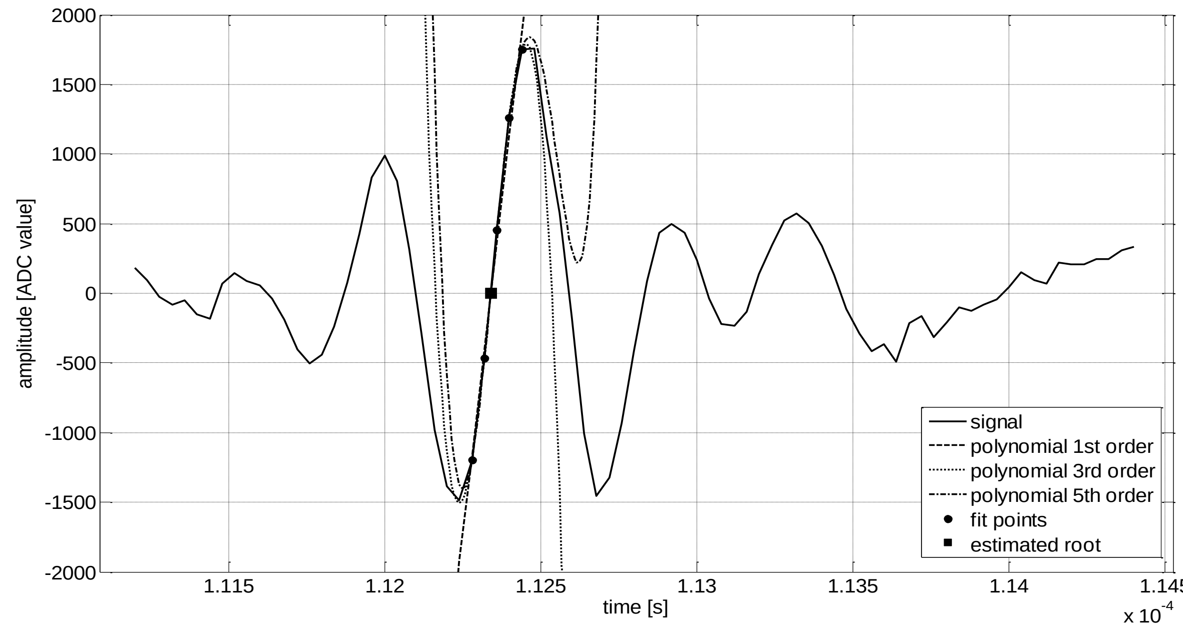
similarity analysis for relevant frames:
cross correlation, MSSR,... to detect the relevant parts of the echo (A_{e1j})

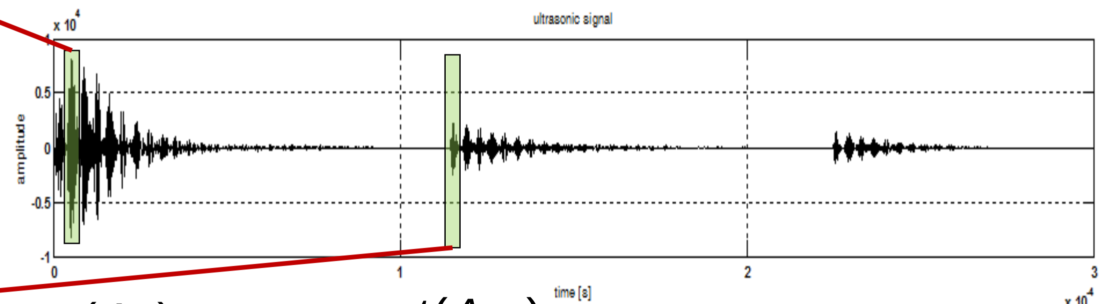
↓

root estimation at characteristic location
(search for the characteristic location related to the reference)

↓

Output: TOF and USV of test medium





$t(A_{r1})$ $t(A_{e11})$

$$TOF = t(A_{e11}) - t(A_{r1})$$

Impedance - Algorithm

Input: signal data and device properties

↓

signal segmentation: n frames of main pulse length (buffer geometry)

↓

definition of reference signal , A_{r1}

↓

Similarity analysis

cross correlation, MSSR, entropy

frame wise maximum search

↓

reflection analysis

maximum amplitude, energy

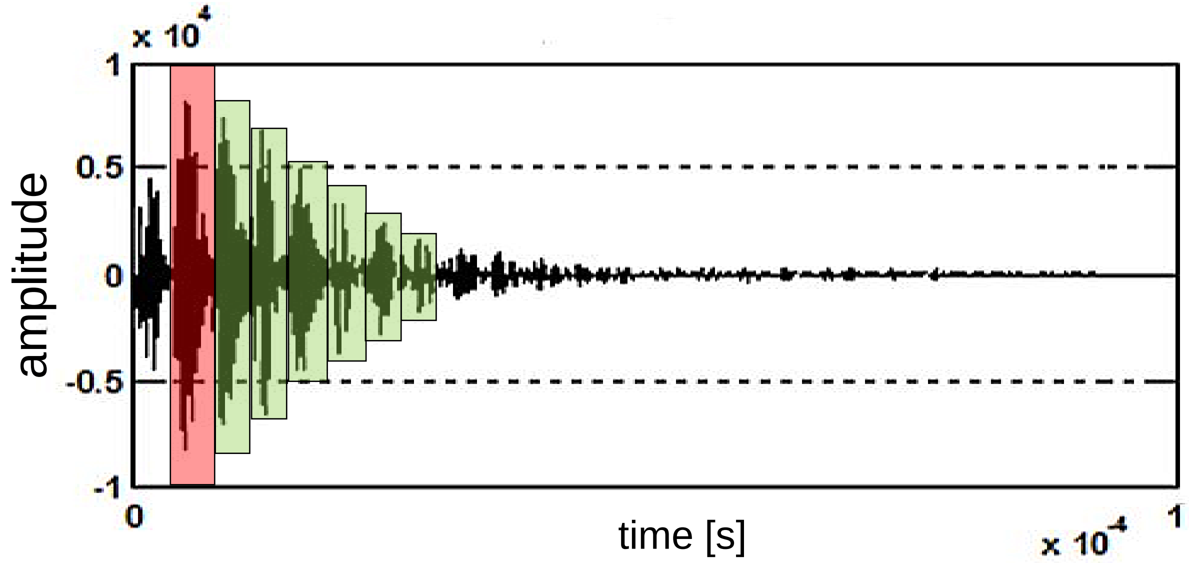
↓

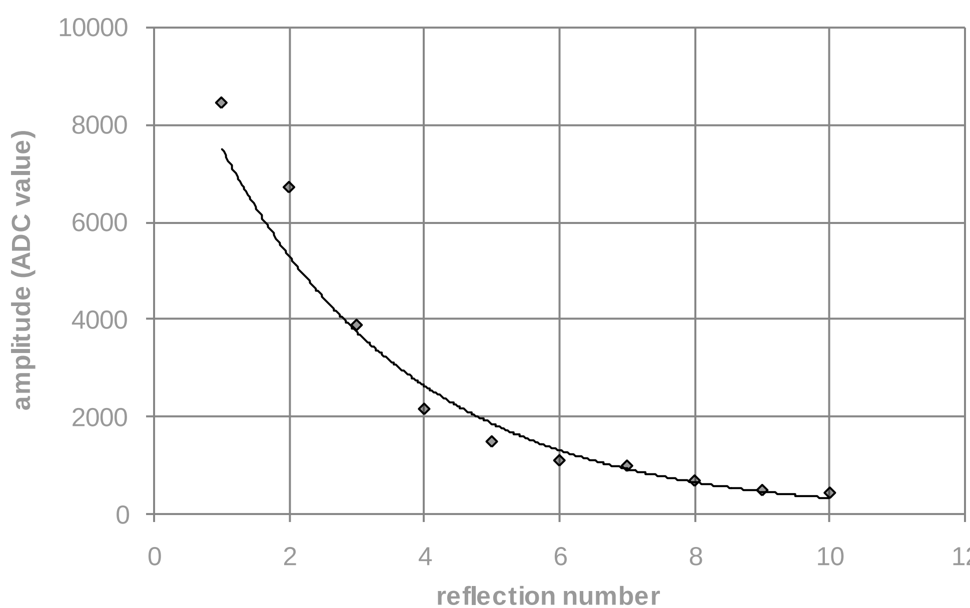
decay analysis

curve fitting for exponential decay

↓

Output: energy decay coefficient



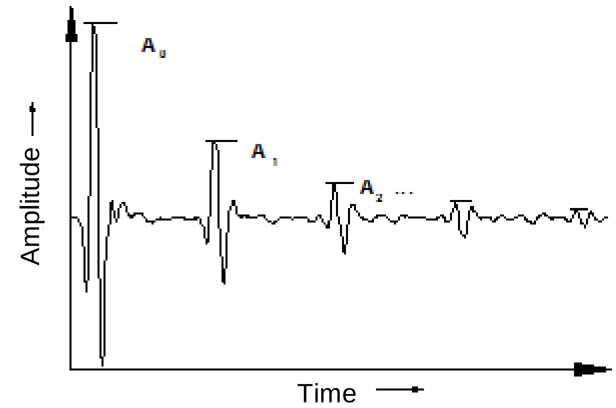


→ exponential decay

$$A_n = A \cdot r_1^{\,n} \cdot r_2^{\,n-1} \cdot \exp(2 \cdot n \cdot \alpha_1 \cdot l_w)$$

$$\ln A_n = \left[\ln r_1 + \ln r_2 + 2 \cdot \alpha_1 \cdot l_w\right] \cdot n + \left[\ln A - \ln r_2\right]$$

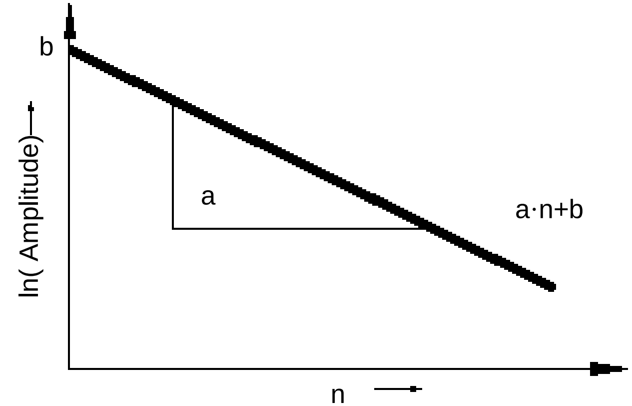
$$= a \cdot n + b$$



→ comparison of exponential slopes

$$\frac{e^{a_{sample}}}{e^{a_{reference}}} = \frac{\cancel{r_1} \cdot r_{sample} \cdot \cancel{e^{2 \cdot \alpha_1 \cdot l_w}}}{\cancel{r_1} \cdot r_{reference} \cdot \cancel{e^{2 \cdot \alpha_1 \cdot l_w}}}$$

$$r_{sample} = r_{reference} \cdot e^{\left(a_{sample} - a_{reference}\right)} = \frac{r_{reference}}{e^{\left(a_{reference} - a_{sample}\right)}}$$



→ impedance Z:

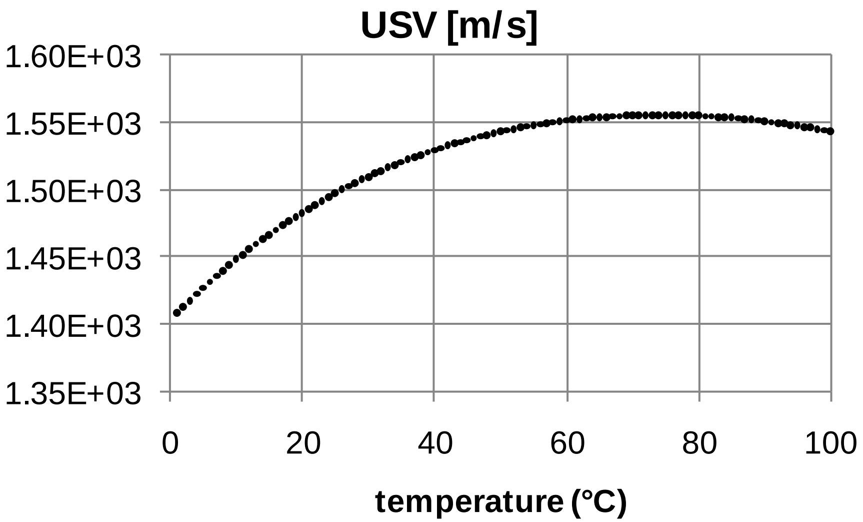
$$Z_{sample} = Z_{steel} \cdot \frac{1 - r_{sample}}{1 + r_{sample}}$$

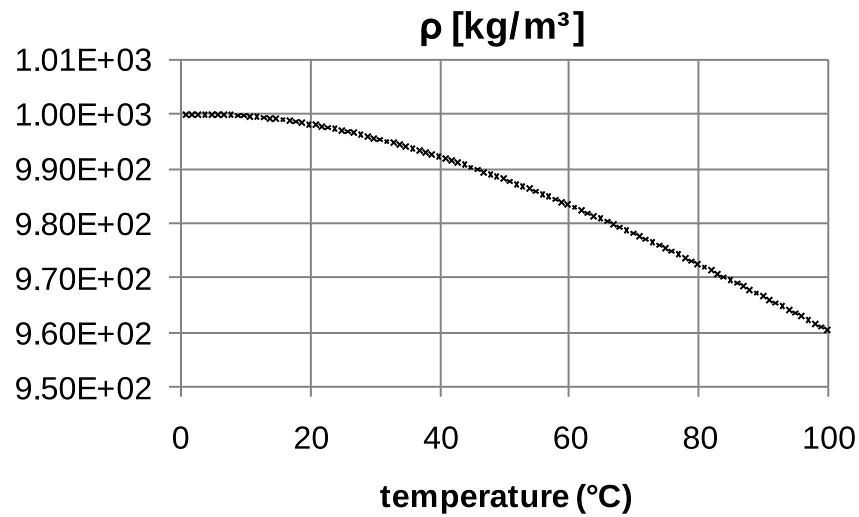
→ density ρ

$$\rho = \frac{Z}{USV}$$

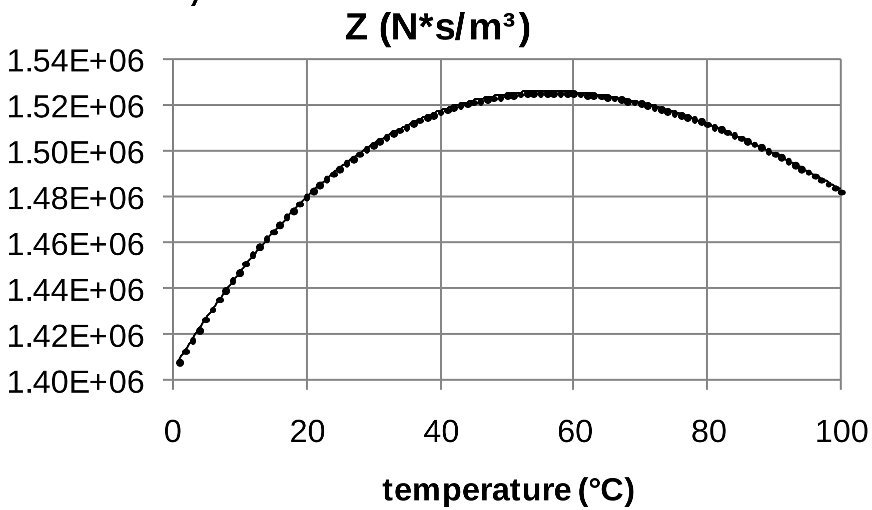
Signal Processing – Impedance Analysis

→ Ideal reference material is **demineralized, vented water**.

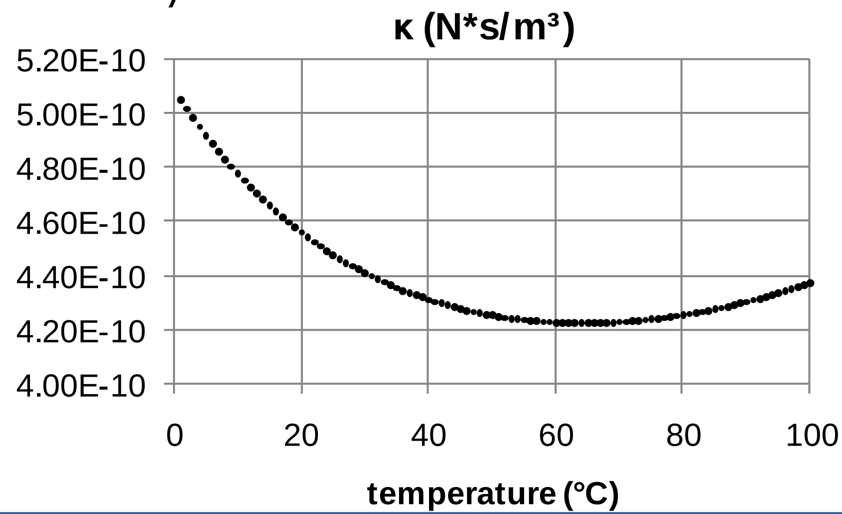




$$Z = USV \cdot \rho$$



$$\kappa = USV^{-2} \cdot \rho^{-1}$$



Basic Problems

→ For an **independent model** the TOF has to be related to the **passed distance in the medium (l_f)** and their changes due to **thermal expansion**.

→ Since t(A_{e11}) is determined from the **echo** the TOF-analysis is **influenced by signal variations** caused by disperse parts of the fermentation medium (bubbles of different size, disperse particles,…).

→ TOF-analysis accuracy is dependent on the **time resolution** of the captured data and the **stability** of the used methods to the signal variations.

Basic Problems

→ High **requirements** for accurate, high resolution concentration models for USV-measurements:

- Assuming a water-sugar-mixture of 10°C a sugar consistency change from 1% to 2% would cause an approximate **difference of 3 m/s in USV**
- This means, assuming an aimed process monitoring resolution of 0.01% sugar content a standard deviation less than **0.015 m/s USV** or approximate **1 ns TOF** should be achieved.

→ **Targets**: Sugar and ethanol concentration monitoring resolution of **0.05 %**:

- maximum USV standard deviation of **0.075 m/s**
- maximum TOF standard deviation of **3.5 ns (100 mm** distance)

Basic Problems – Monitoring Accuracy

$$\Delta c(USV,T) = \frac{\partial c}{\partial USV} \cdot \Delta USV + \frac{\partial c}{\partial T} \cdot \Delta T$$

$$\Delta USV(TOF,T) = \frac{\partial USV}{\partial TOF} \cdot \Delta TOF + \frac{\partial USV}{\partial T} \cdot \Delta T$$

$$USV(TOF,T) = \frac{l(T)}{TOF}$$

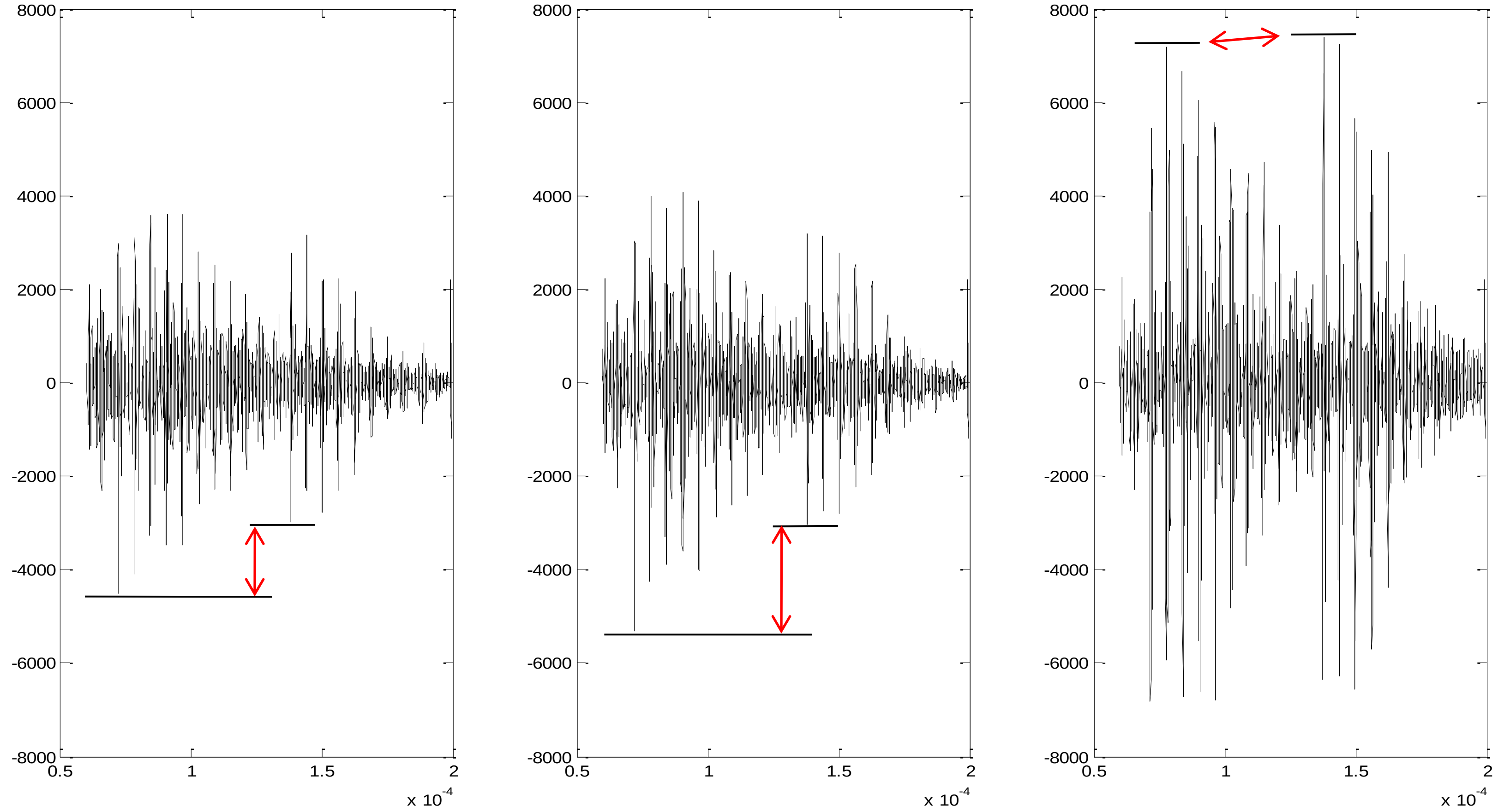
$$l = a + b \cdot T$$

$$\frac{\partial USV}{\partial TOF} = \frac{b}{TOF}$$

$$\frac{\partial USV}{\partial T} = \frac{\partial USV}{\partial l} \cdot \frac{\partial l}{\partial T}$$

$$\Delta USV(TOF,T) = \frac{a + b \cdot T}{TOF^2} \cdot \Delta TOF + \frac{b}{TOF} \cdot \Delta T$$

Basic Problem – Signal Variation



- → ultrasonic measurements are highly disturbed when gas bubbles in liquids are present – **variation of absolute value and order**
- → sampling frequency restriction: 40 ns

Basic Problem – Signal Time Resolution

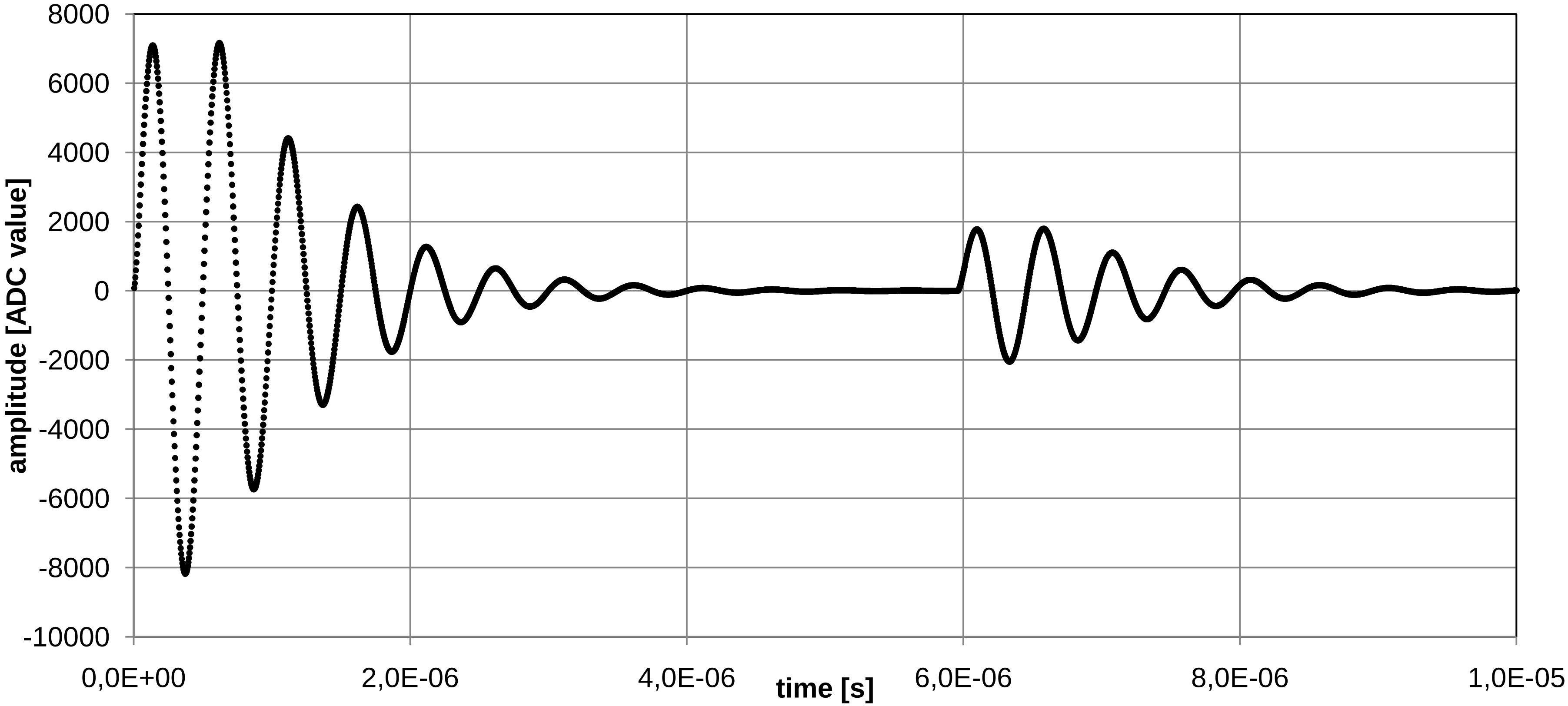
$$A_{exc}(t) = A_{exc0} \cdot e^{(-t/u)} \cdot t^{m} \cdot \sin(2 \cdot \pi \cdot f \cdot t + \varphi)$$

$$A_{echo}(t) = \alpha \cdot A_{exc}$$ → **shift of time axis** $t_{echo} = t + TOF$

$$A_{signal}\left[t_0, \ldots, TOF\right] = A_{exc}(t)$$

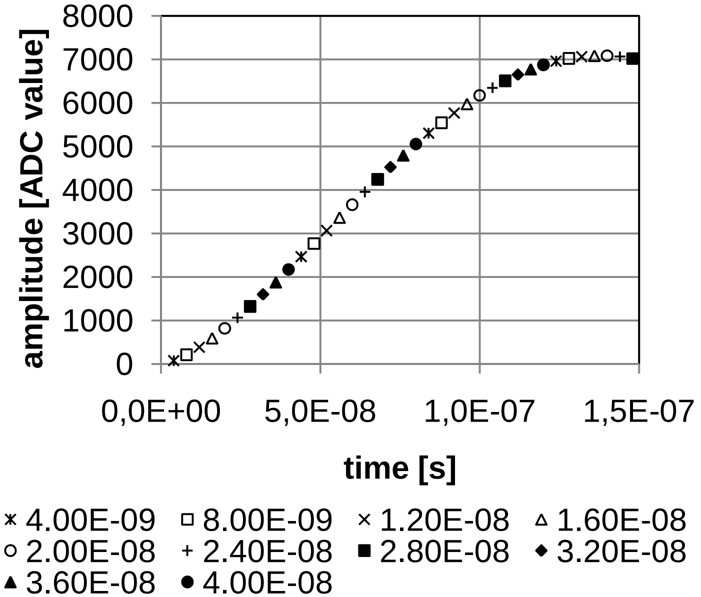
$$A_{signal}\left[TOF, \ldots, end\right] = A_{exc}(t) + A_{echo}(t - TOF)$$

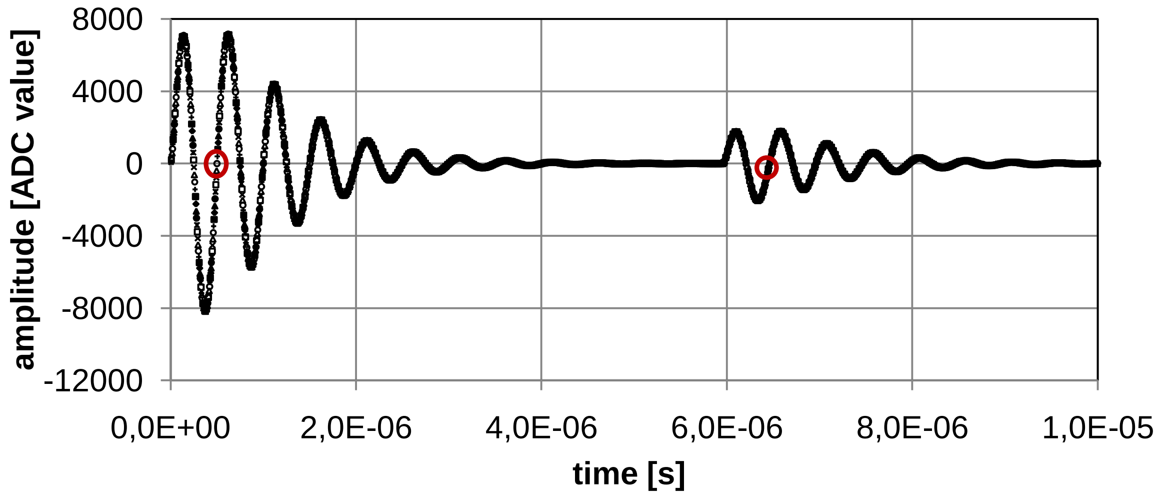
excitation amplitude	Ae	2.40E+07
excitation phase	phie	0
excitation frequency	fe	2.00E+06
% shape paramter	m	0.5
	u	16*4E-8
echo amplitude	Ar	6.00E+06
echo phase	phir	0



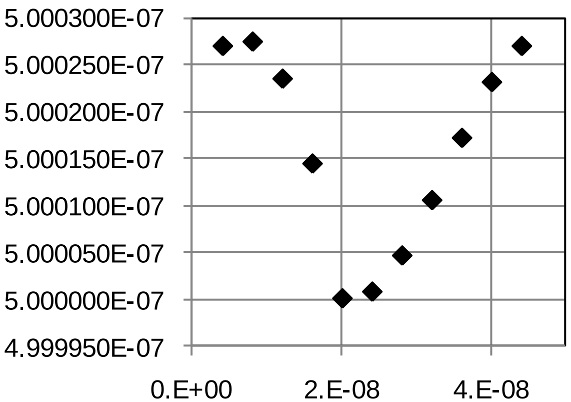
Basic Problem – Signal Time Resolution

→ 1st time resolution problem: jitter (max. 3 ns)

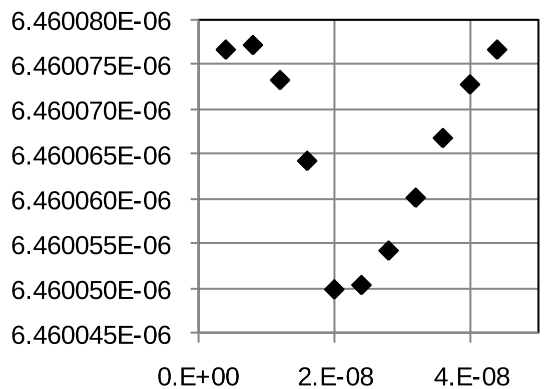




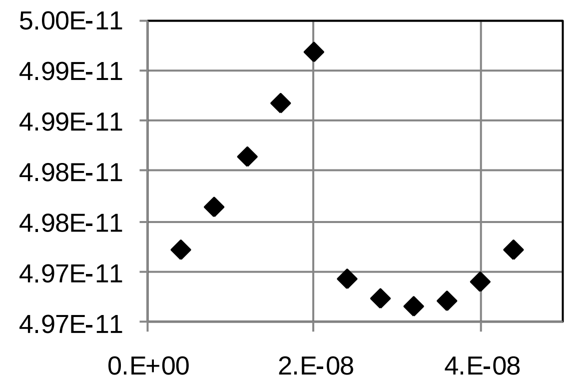
1st root



2nd root

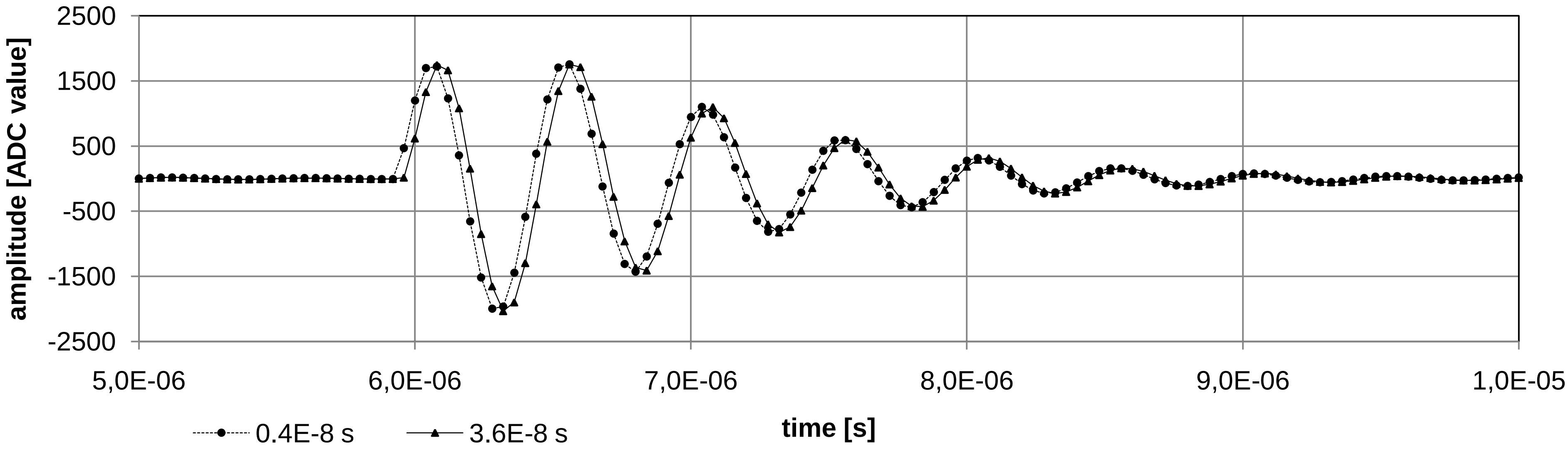


TOF - Variation

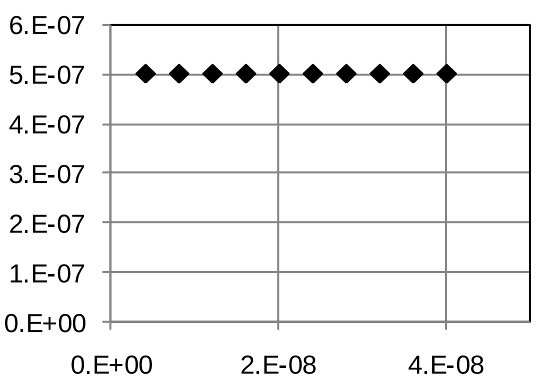


Basic Problem – Signal Time Resolution

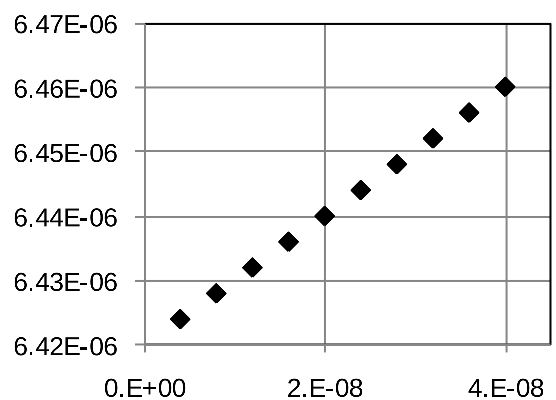
→ 2nd time resolution problem: change of echo



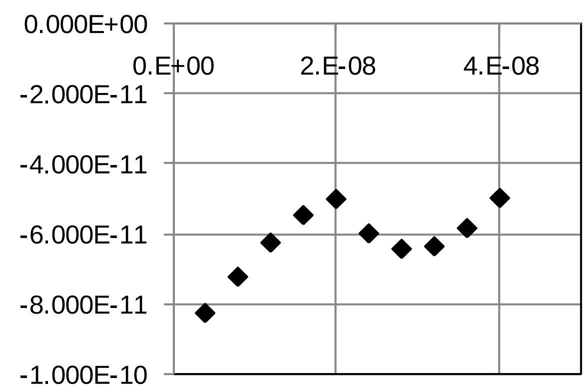
1st root



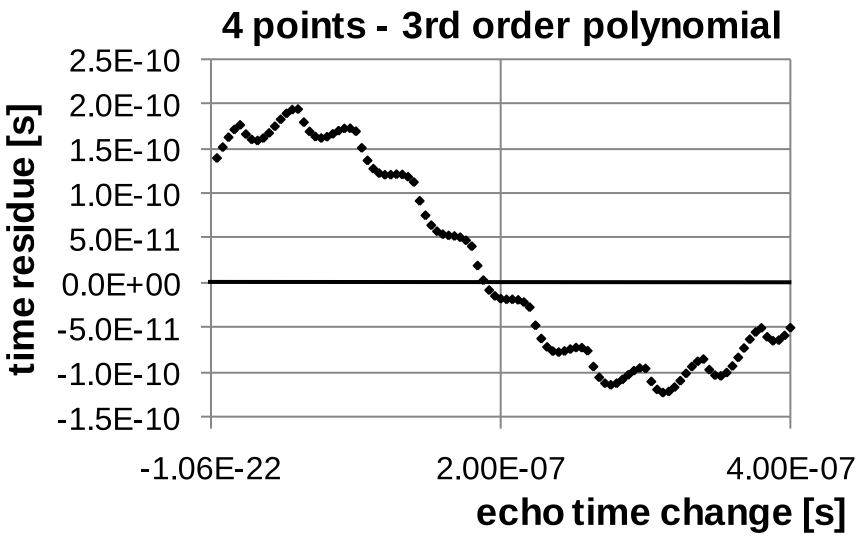
2nd root

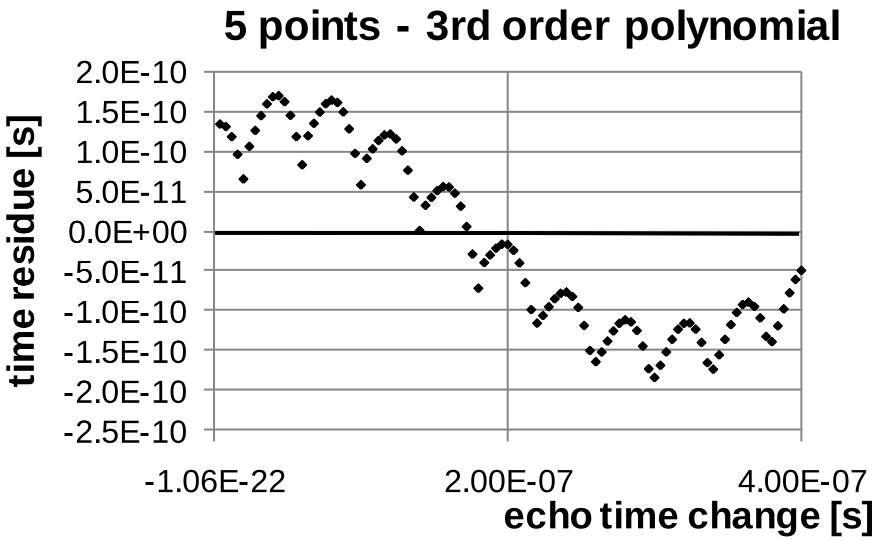


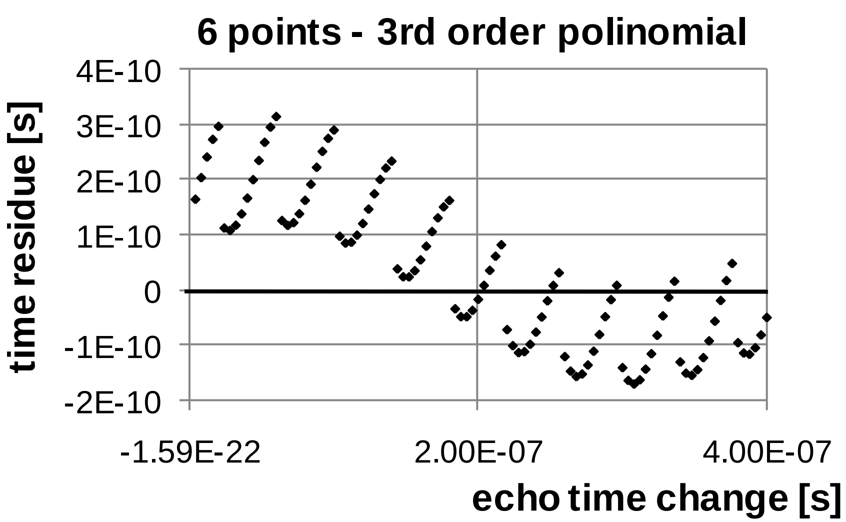
TOF - Variation

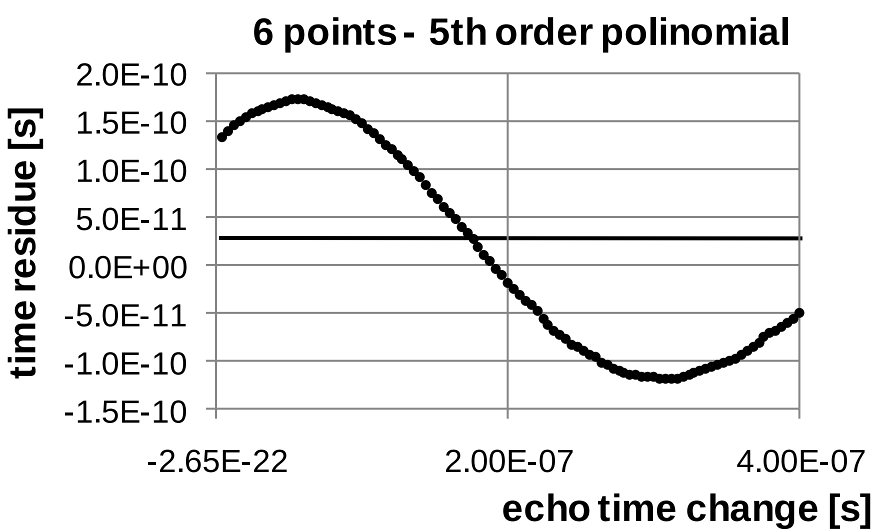


Basic Problem – Signal Time Resolution



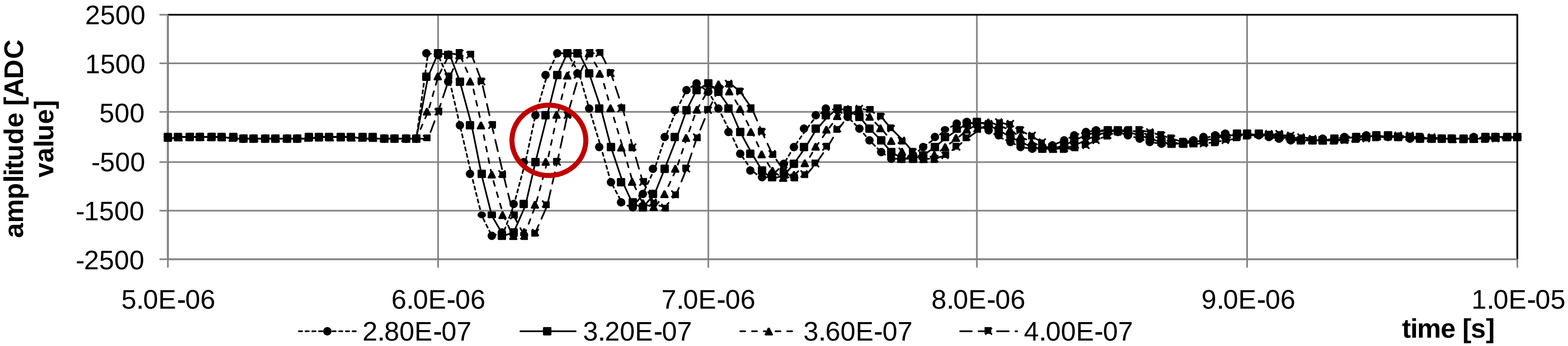


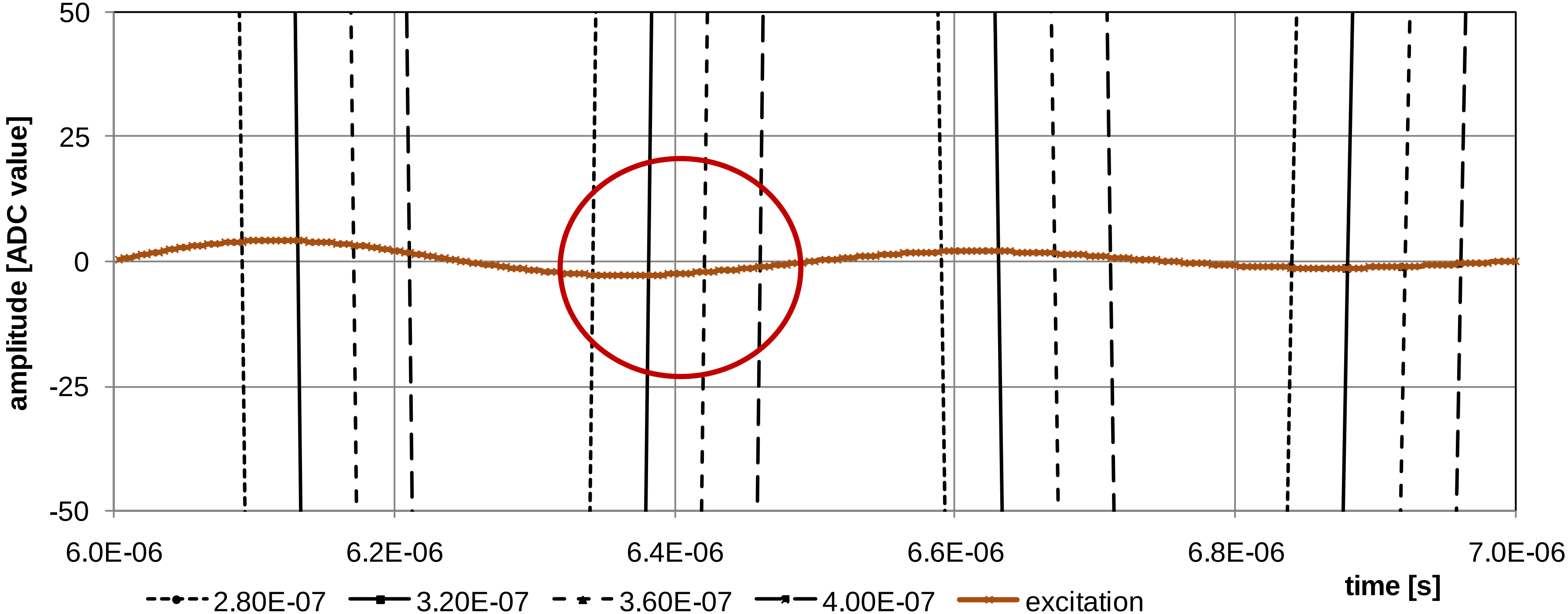




Basic Problem – Signal Time Resolution

→ 2nd time resolution problem: change of echo





Trial Results – Validation Analysis

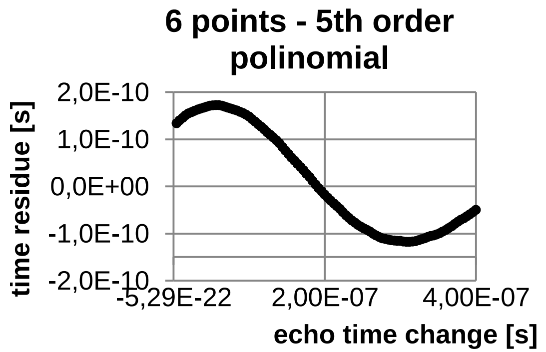
Prior Trial Targets:

→ **Absolute Error:** trials and analysis for a well known and elaborately analyzed material – WATER (Marczak, 1997)

→ **Stability Analysis** for real process data (yeast propagation) → malfunctions, outliers, standard deviation

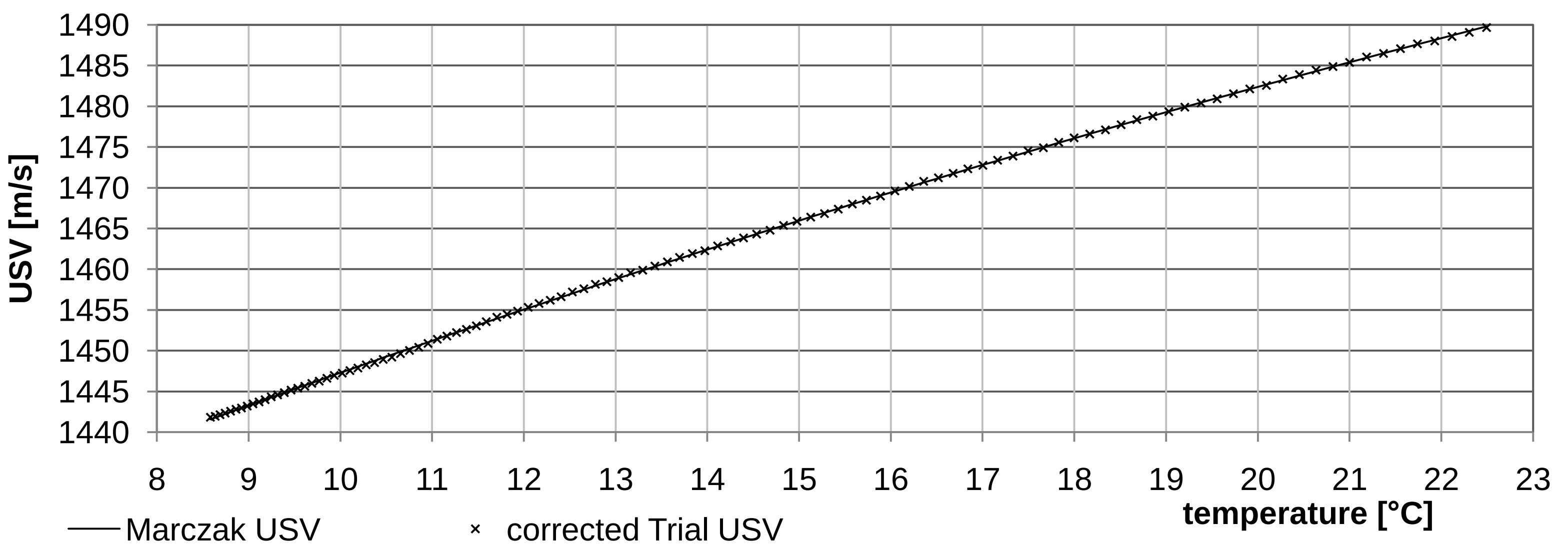
Trial Results – Validation Analysis

		laboratory, water 10°C	laboratory, water 15°C	laboratory, water 20°C	process, 110-119
TOF average	[s]	6.9069E-05	6.8179E-05	6.7416E-05	6.6346E-05
standard deviation	[s]	9.5716E-10	1.2457E-09	2.1864E-09	1.1653E-11
	[%]	1.39E-03	1.83E-03	3.24E-03	1.76E-05



→ Target of 3.5 ns TOF maximum standard deviation can be reached.

→ USV for DN50 setup - accurate reflector distance of 49.968 mm



Trial Results – Validation Analysis

Temperature	10°C		15°C		20°C	
	expansion corrected	constant distance	expansion corrected	constant distance	expansion corrected	constant distance
distance l_f [m]	4.9977E-02	4.9968E-02	4.9972E-02	4.9968E-02	4.9968E-02	4.9968E-02
TOF Trial Data [s]	6.9069E-05	6.9069E-05	6.8179E-05	6.8179E-05	6.7416E-05	6.7416E-05
USV Trial Data [m/s]	1447.152	1446.906	1465.922	1465.796	1482.384	1482.382
USV Marczak [m/s]	1447.291		1465.962		1482.382	
residues to Marczak [m/s]	0.139	0.385	0.040	0.166	-0.002	0.001
absolute error [%]	9.62E-03	2.66E-02	2.74E-03	1.13E-02	-1.20E-04	3.68E-05

$$USV(T) = \sum_{i=0}^{5} (P_i \cdot T^i)$$

	Konstanten
P0	1.4023875E+03
P1	5.0388130E+00
P2	-5.7991360E-02
P3	3.2871560E-04
P4	-1.3988450E-06
P5	2.7878600E-09

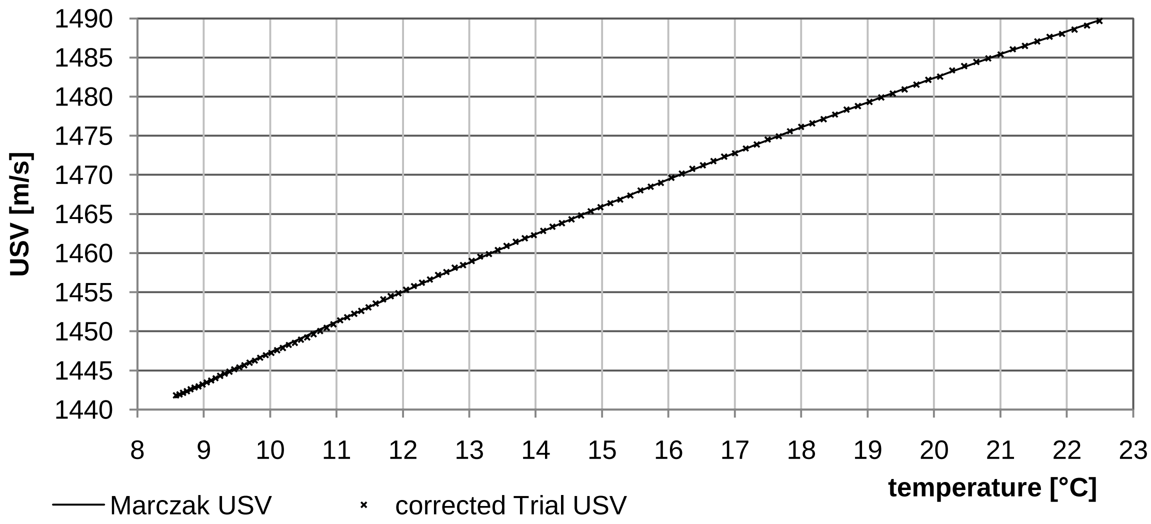
(Marczak, 1997)

→ *Theoretical, temperature generated error for $\Delta T = 0.1$ K:*

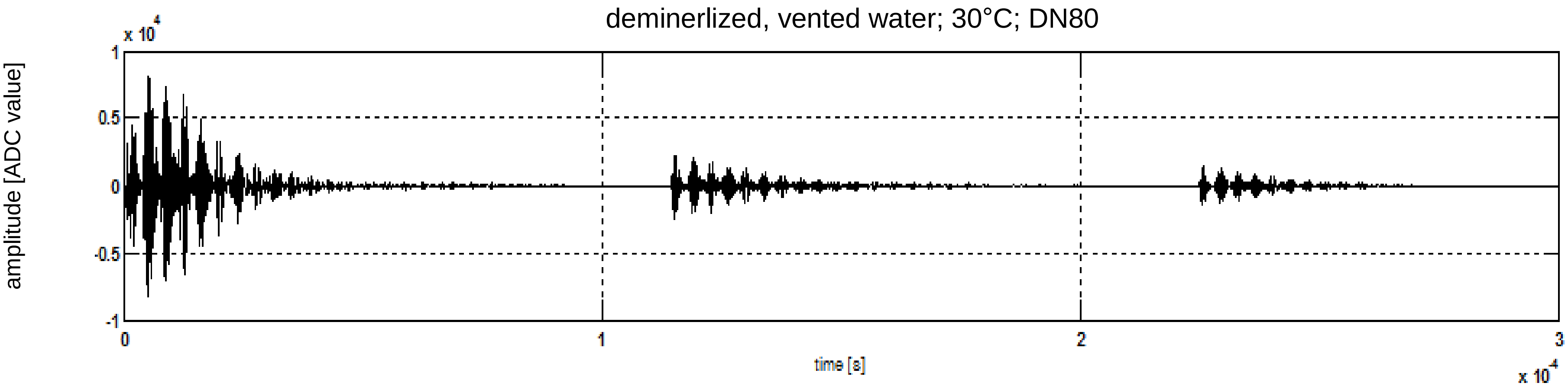
$$\Delta USV = \frac{\partial USV(T)}{\partial T} \Delta T$$

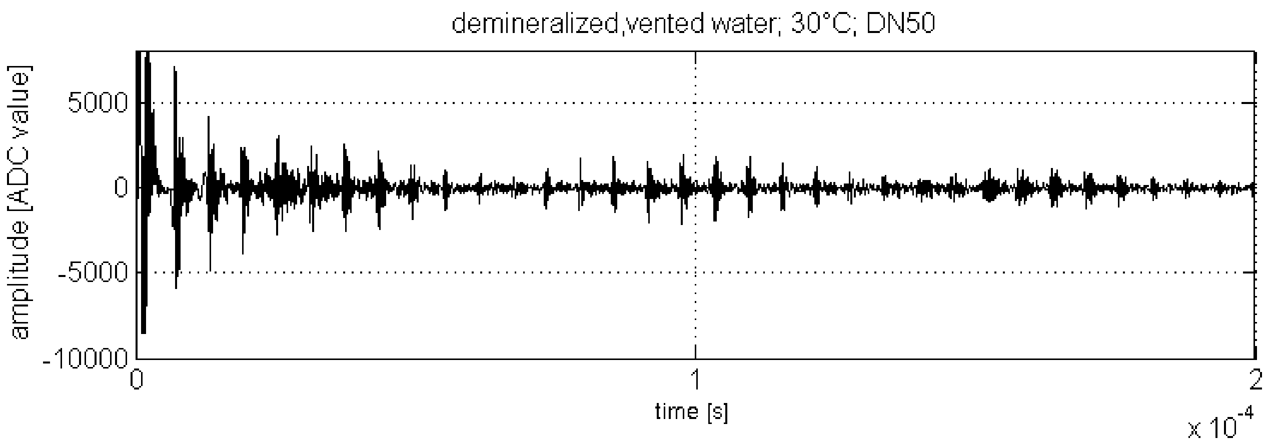
0.42 m/s (0.03%) for 8°C
0.29 m/s (0.02%) for 22°C

→ *Target: 0.075 m/s*



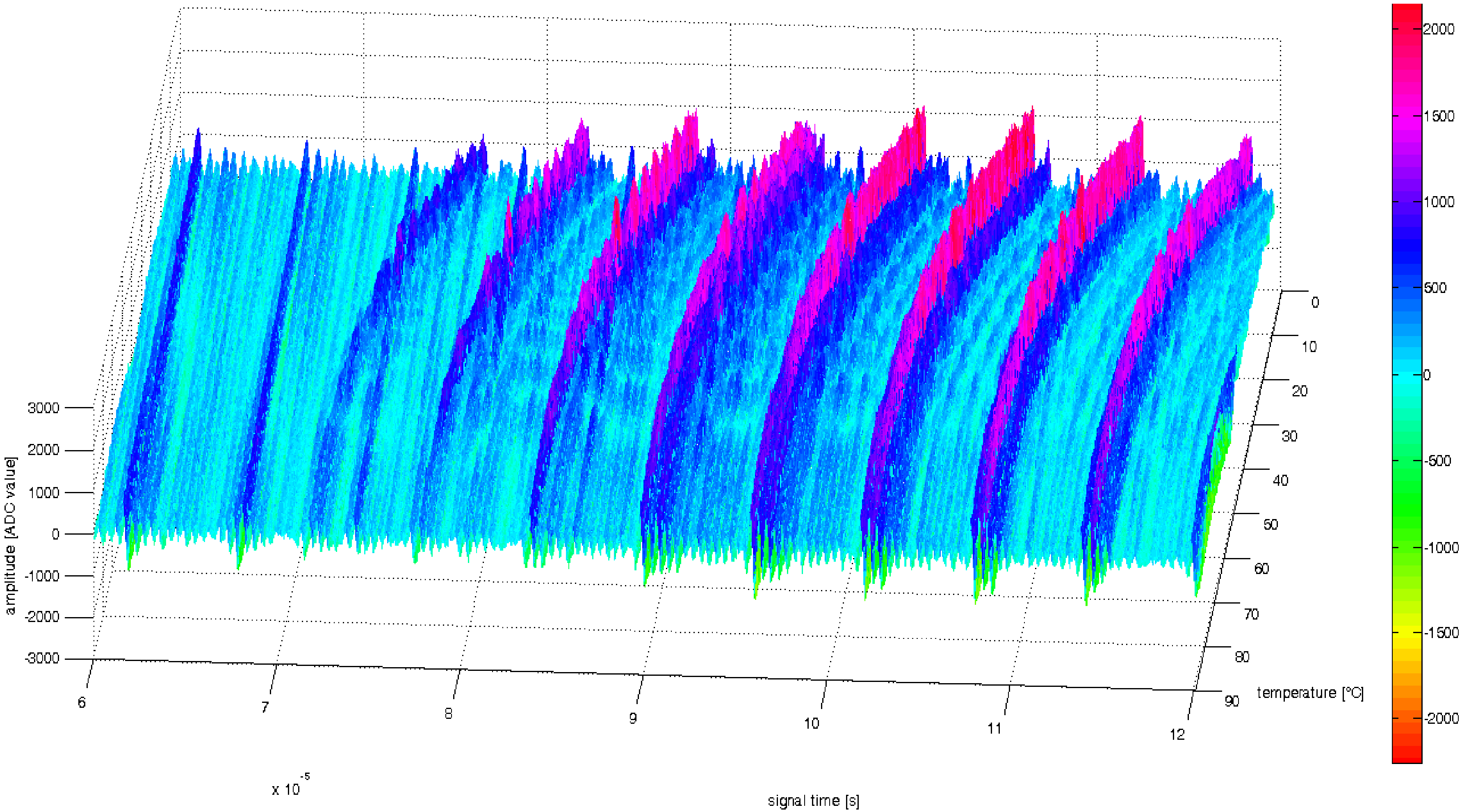
Trial Results – Stability Analysis





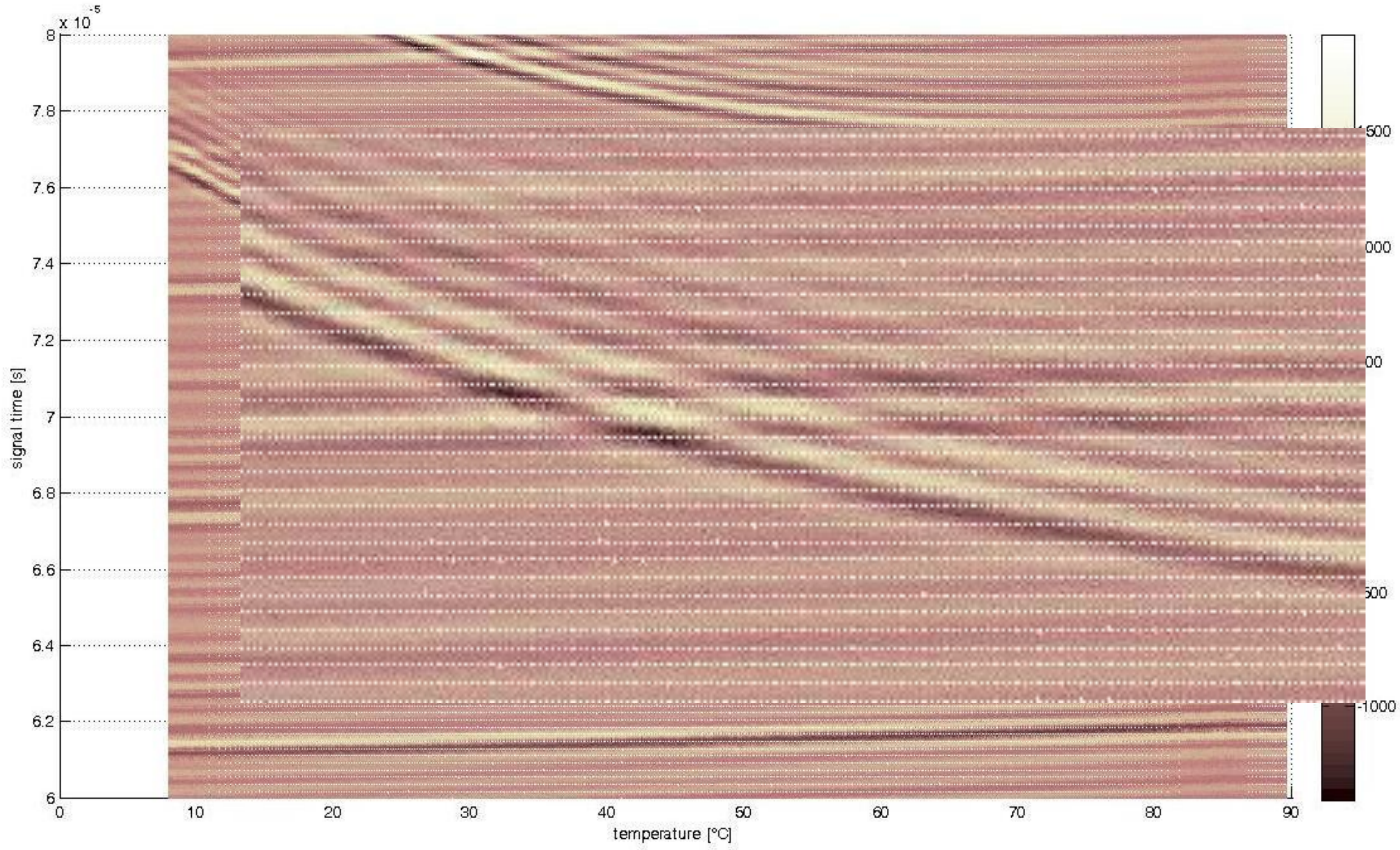
→ echo interferes with buffer reflections which causes, dependent on ultrasound velocity of medium c(T,Φ) different superposition phenomena

Trial Results – Stability Analysis - Overview



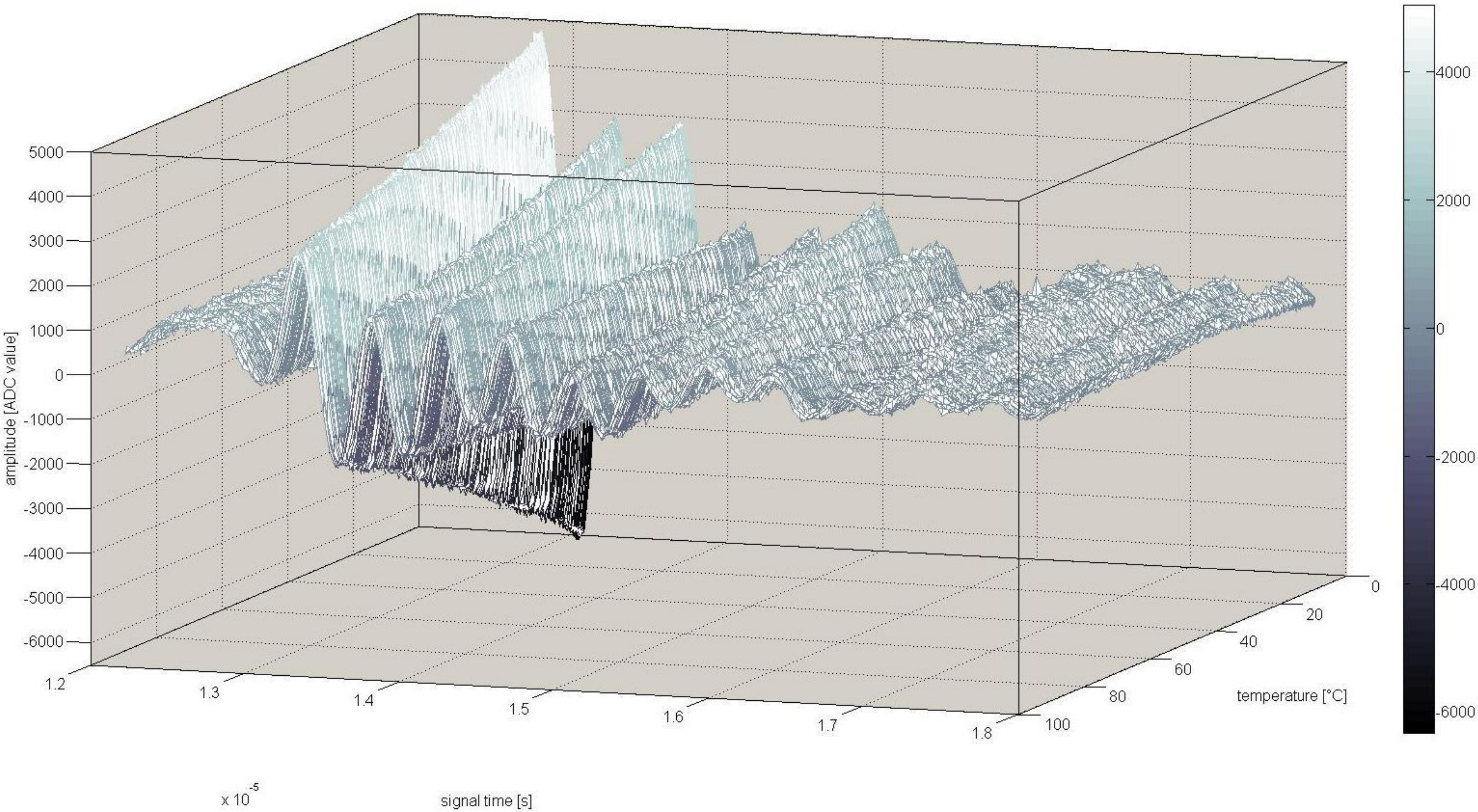
→ echo interferes with buffer reflections which causes, dependent on ultrasound velocity of medium c(T,Φ) different superposition phenomena

Trial Results – Stability Analysis – Echo



→ **constructive and destructive superposition of buffer reflections and echo changes signal and thus the correlation results**

Trial Results – Stability Analysis – Reflections

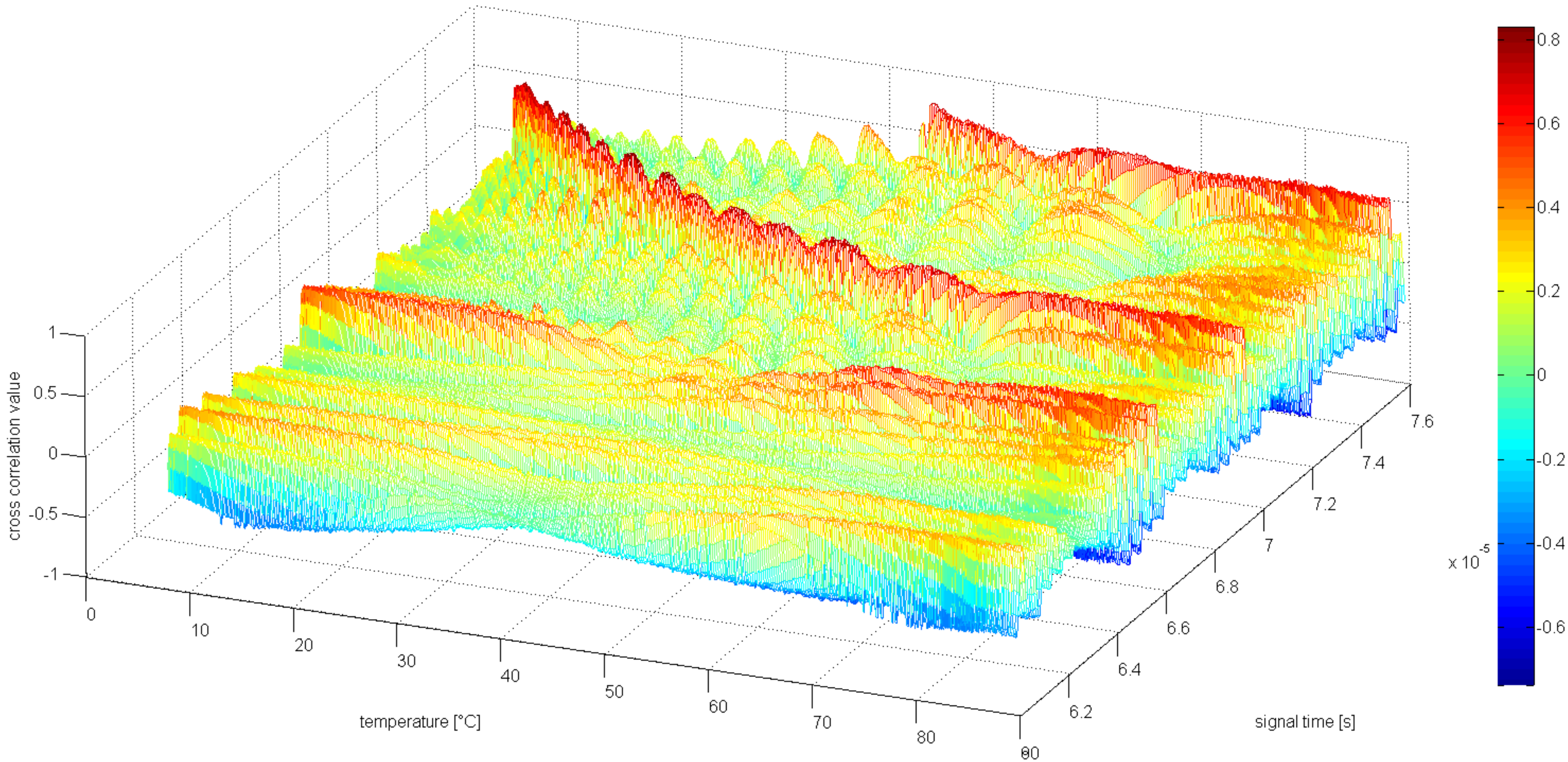


→ **time change due to temperature dependent thickness expansion of buffer**

→ **amplitude change due to temperature dependent absorption and reflection coefficients**

Trial Results – Stability Analysis – Correlation

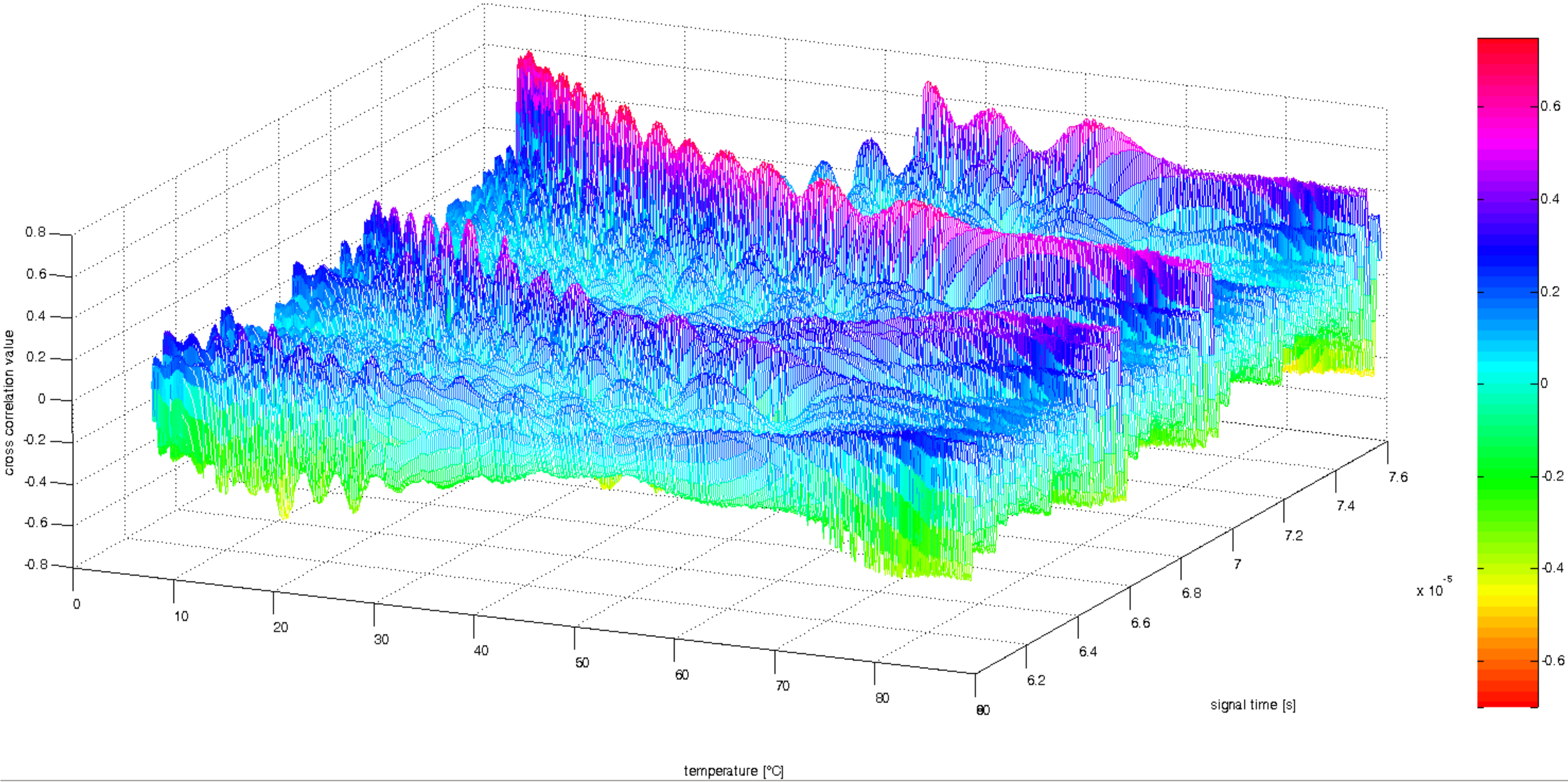
→ **cross correlation values for demineralized, vented water; reference signal of 1 frame length (150 points)**



→ **no stable correlation threshold for detection can be found**

Trial Results – Stability Analysis – Correlation

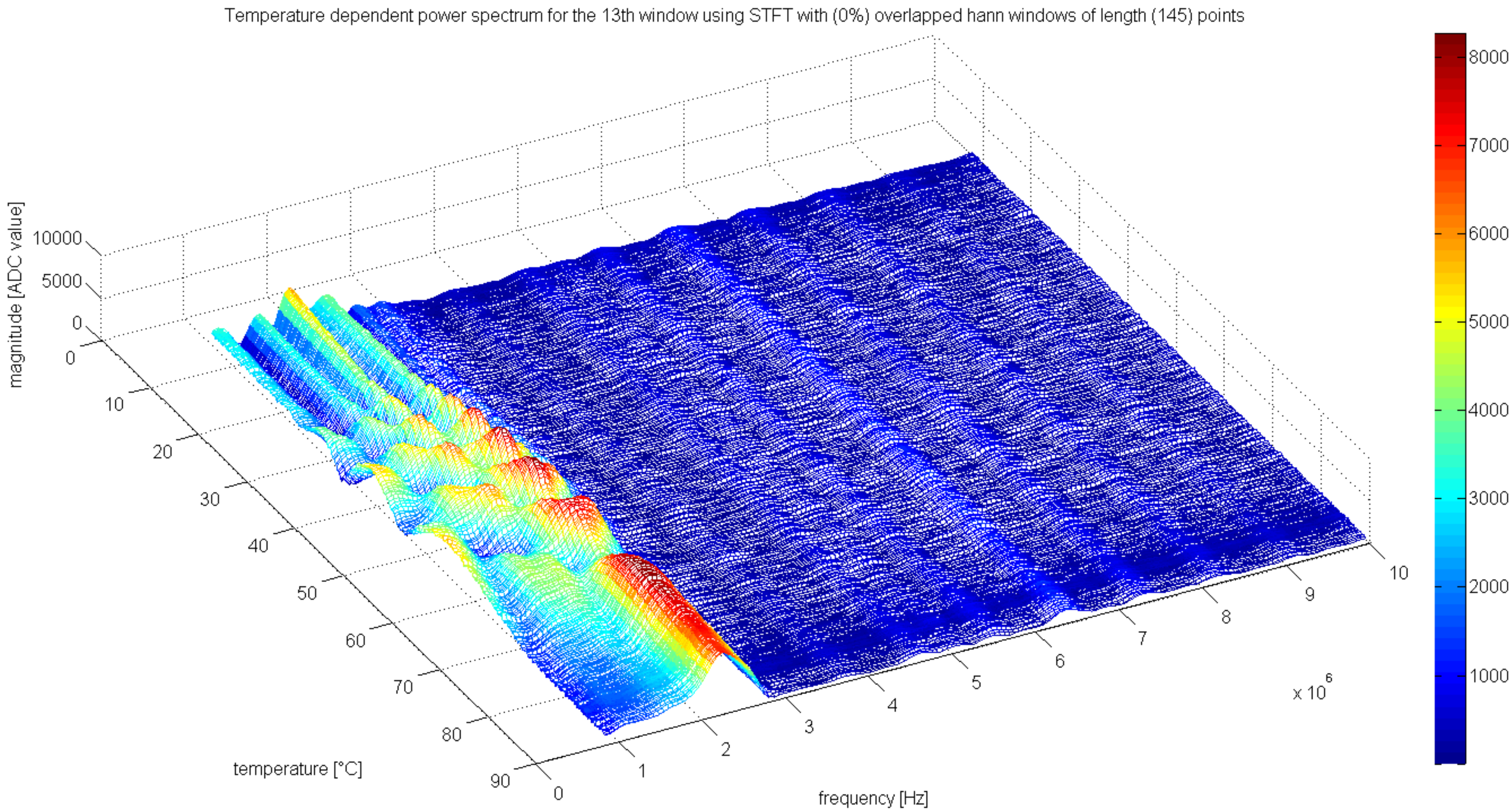
→ **cross correlation values for demineralized, vented water; reference signal of triple frame length (450 points)**



→ **same instability**

Trial Results – Stability Analysis – Frequency Domain

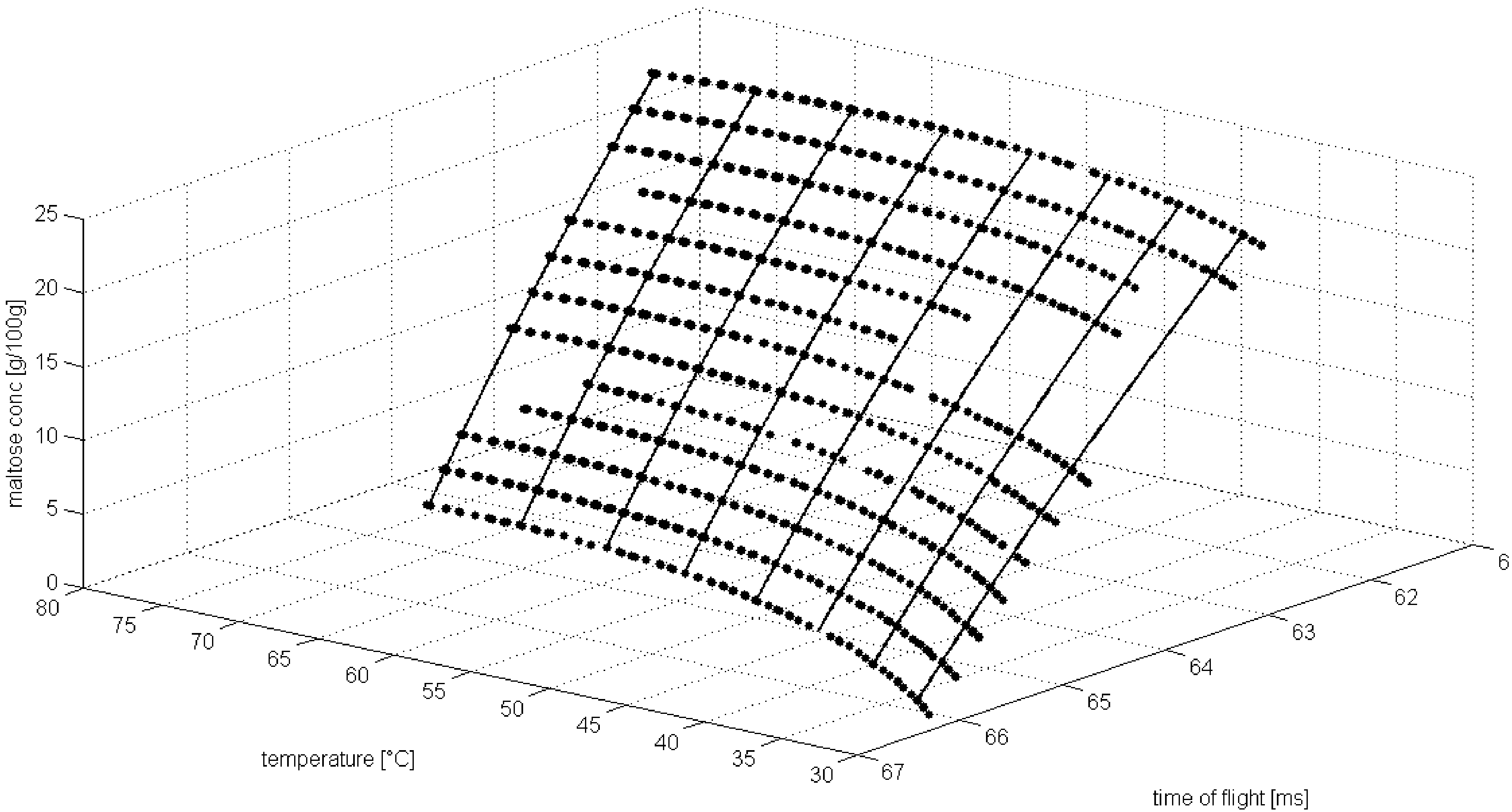
→demineralized, vented water



→ same up and down in the frequency domain

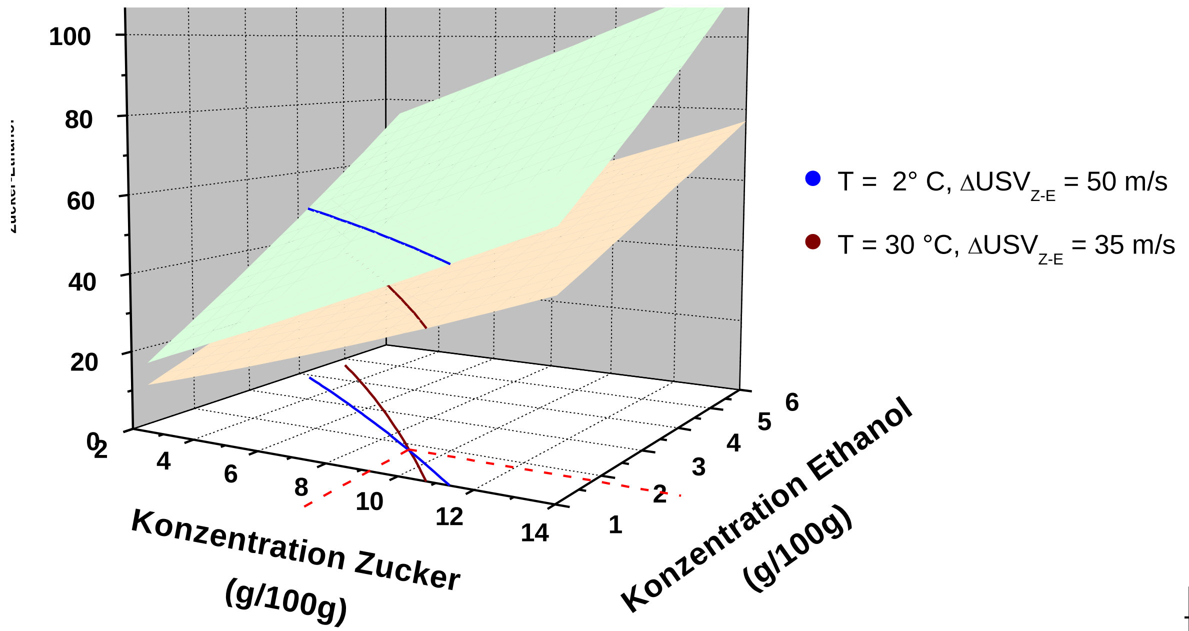
→ frequency domain data base will show dependency on concentration

Trial Results

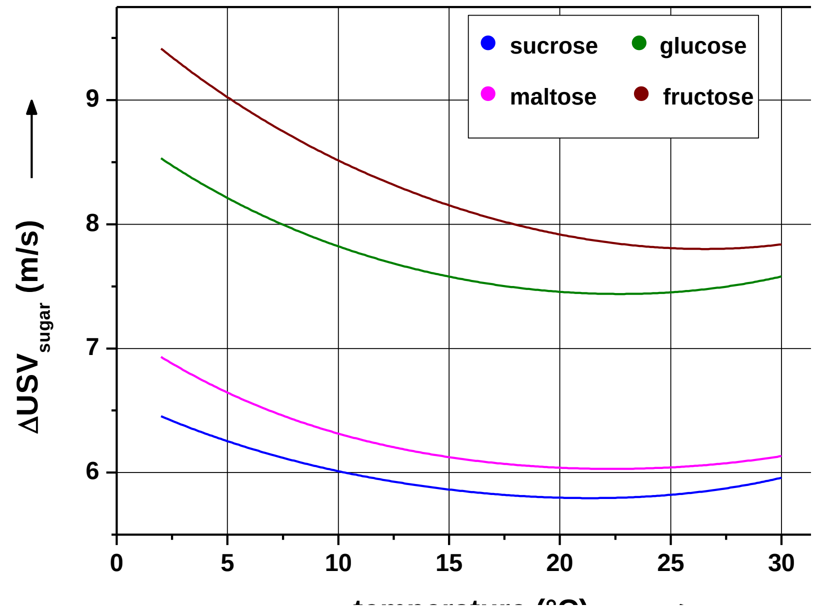


→ TOF for DN50 setup (2*49.968 mm propagation path)

Former Investigations



→ there is no unique correlation between one temperature/ USV and one ethanol-sugar combination



→ variance due to varying rare materials can be expected

Conclusions / Future Prospects

→ Process Monitoring for **Anaerobe** Yeast Fermentation:

- clear **stoichiometric ratio** between sugar and alcohol concentration (constant temperature) – model

- therefore clear concentration dependency for ultrasound velocity at one temperature

→ Process Monitoring for **Aerobe** Yeast Fermentation (Assimilation):

- modelling of stoichiometric ratio between sugar and alcohol concentration is **complex and based on experimental data**

- therefore a **third paramter** is necessary for accurate concentration determination (impedance, density, frequency domain)

Conclusions / Future Prospects

→ ultimate numerical solution (TOF determination) for DN50 or even less reflector distance in pulse-echo mode is quite hard; probably even impossible – **multivariate solutions** via elaborate data basis seems possible

→ compromise solutions:

- separation into relevant temperature ranges: 0-30°C, 30-70°C, 70-95°C
- temperature dependent time threshold (functionality depends on concentration range)

→ engineering solutions:

- buffer adaption: material, thickness,…
- reflector distance… DN80
- transmission mode

Thanks for your Attention

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