Recent improvements in ultrasonic instrumentation for experimental studies of chemical reactions

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Structure of the presentation

- Introduction
  why ultrasound
  what developments were desirable
- Novel FPGA architectures for ultrasonic instruments
- Temperature compensation and its integrated sensing
- Summary and conclusions

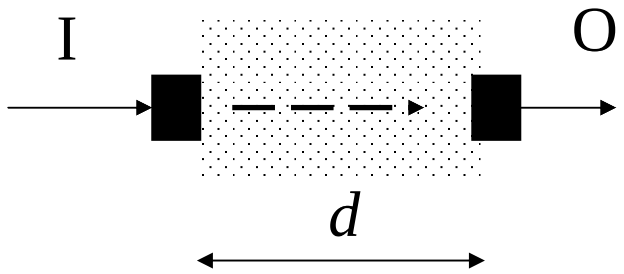
Why ultrasound?

- Applicable for opaque/conductive media
- High frequency (well above 1 MHz) thus small wavelengths (below 1 mm) became available
- Advances in electronics allow on line processing for process control and monitoring

Theoretical considerations

- Highly developed analytical models deal with concentration of a single chemical only
- Models representing different ingredients as separate layers are inaccurate
- Empirical models based on curve fitting require reliable experimental data

Early experimental approach

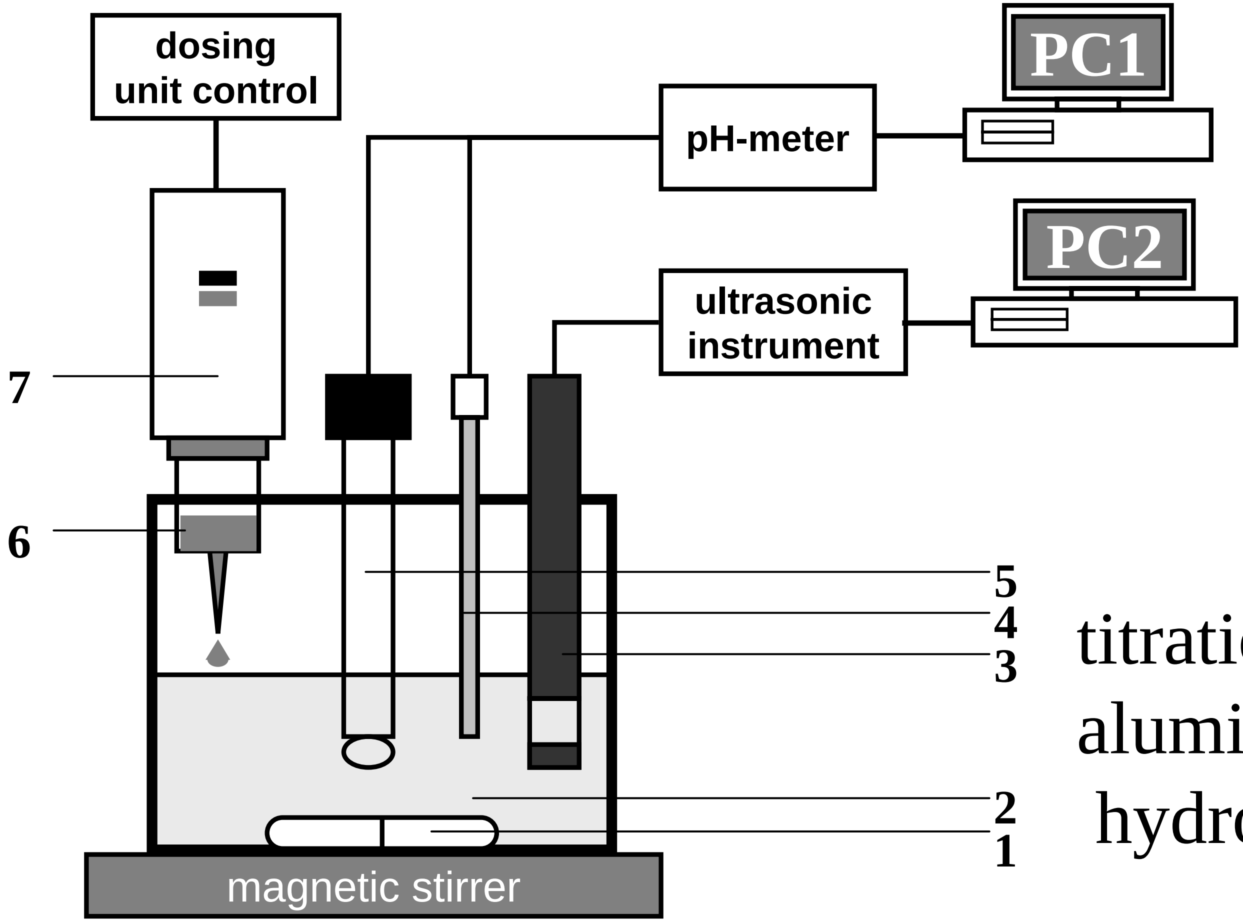
- 

- Absorption and velocity are measured
- Single frequency
- Gauge length *d* of few meters
- Temperature control was found essential

Modern approach

- Pulse excitation (many frequencies simultaneously)
- Software averaging to improve the signal-to-noise ratio (SNR) (requires several seconds)
- Use of high speed ADCs and digital signal processing (sampling at 100-300 MHz)
- PC connectivity

Setup for *in situ* measurements



Experiments were made with colleagues from the NTU Prof C.Perry Dr K.Shafran

titration of aluminium ions with hydroxide ions

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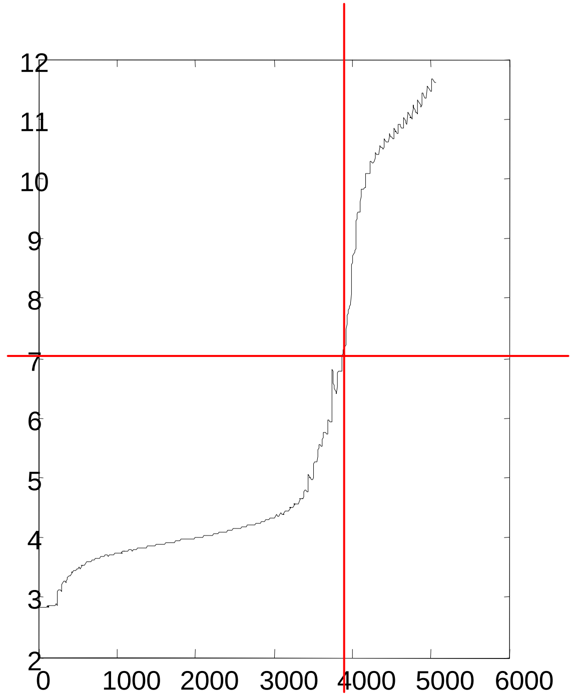
Description of the reaction

- First stage :
  $Al^{3+}(aq) + 3OH^{-}(aq) \rightarrow Al(OH)_3(s)$
  formation of a precipitate
- Second stage: $Al(OH)_3(s) + OH^{-}(aq) \rightarrow Al(OH)_3(aq)$
  dissolution of the precipitate



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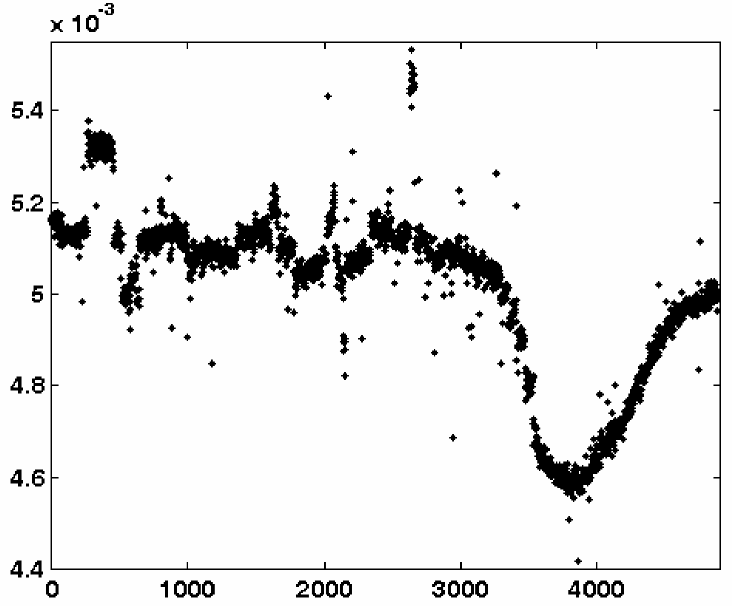
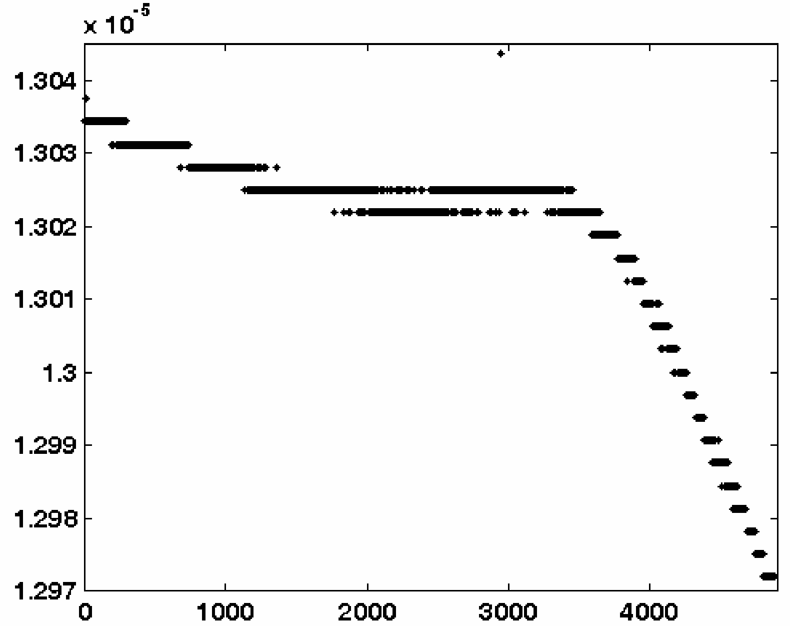
Experimental ultrasonic data



STD of a record



Delay of the max sample

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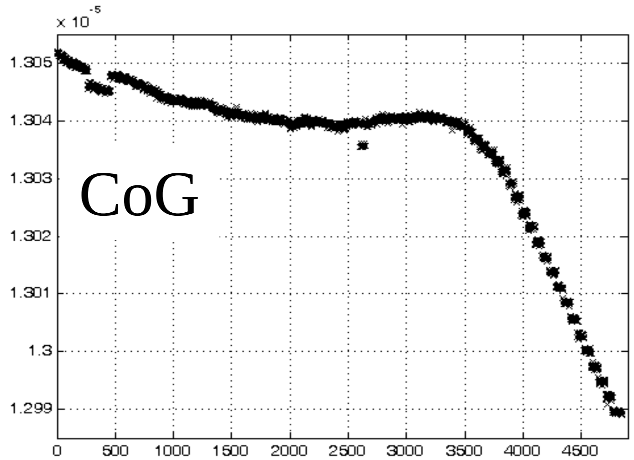
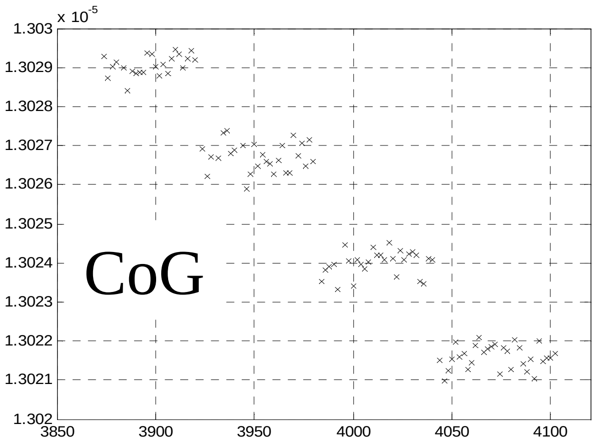
Centre-of-gravity (CoG) estimation

$$CoG = \frac{\int t s^2(t)\,dt}{\int s^2(t)\,dt}$$





Delay time gives better information, higher sampling frequencies required (IEEE Ultrasonics Symp, 2004)

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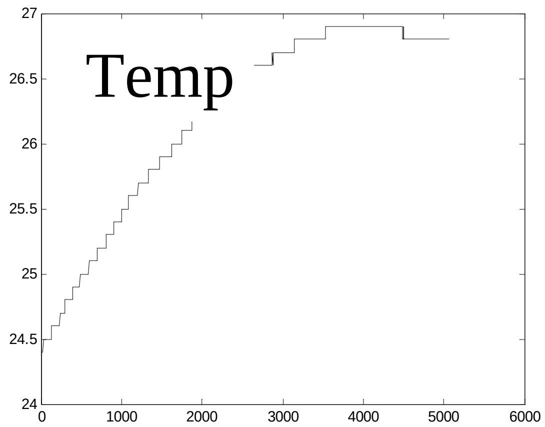
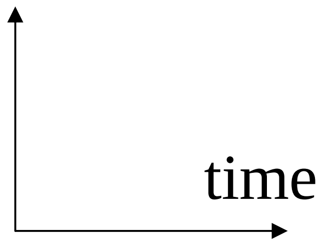
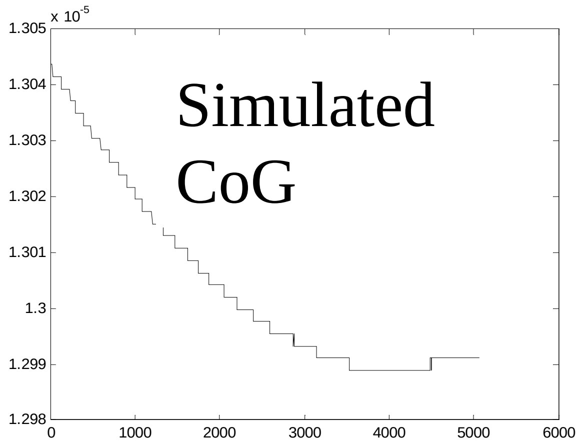
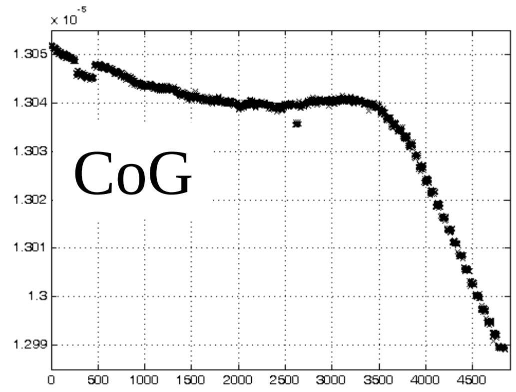
Influence of temperature







The amount of delay changes caused by temperature is compatible with changes in chemical composition



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Requirements for novel hardware

- Implement hardware averaging
- Provide higher sampling rates
- Reduce cost

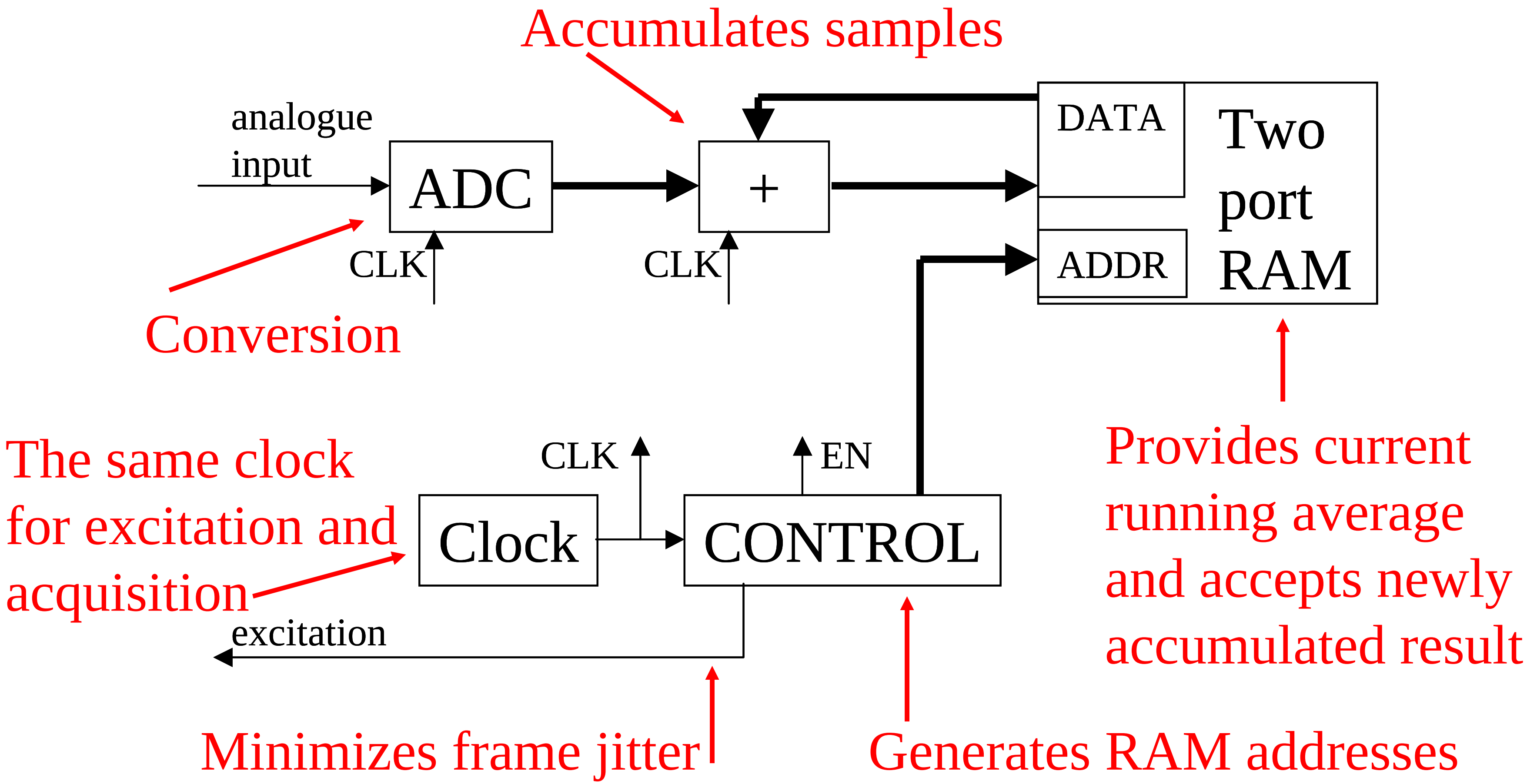
- EPSRC (UK) grant GR/S29713/01

- Temperature compensation

Choice of development platform

- Requirements:
  flexible
  upgradeable
  high level design tools
- Xilinx Xtreme DSP kit
  6,000,000 logic gates FPGA chip
  2 ADC and 2 DAC on board
  System Generator software

On-the-fly averaging –the principle



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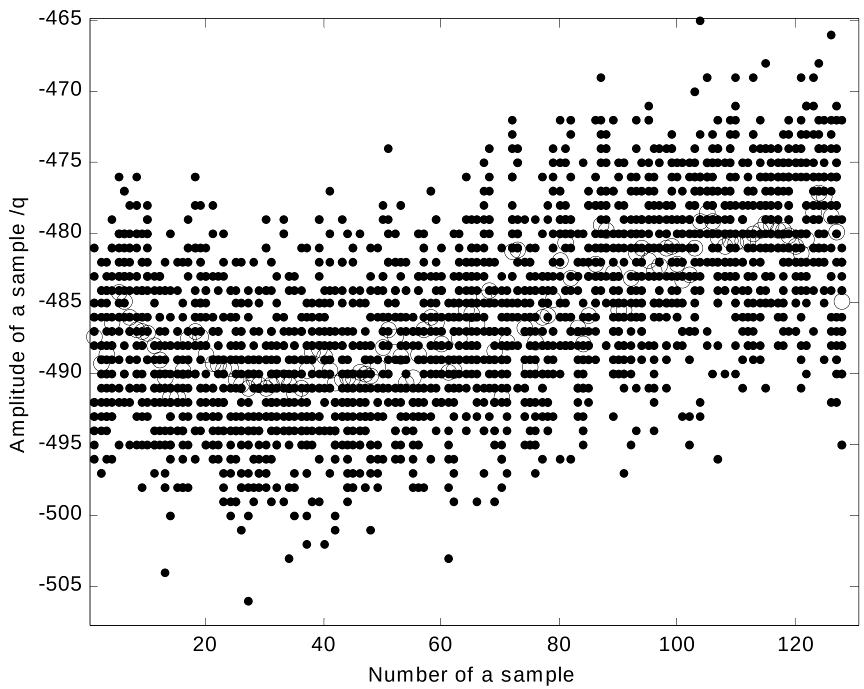
Examples of averaged waveforms



Dots – samples of recorded waveforms (two averages)
Circles – after 2048 averages

(IEEE WISP'2005)

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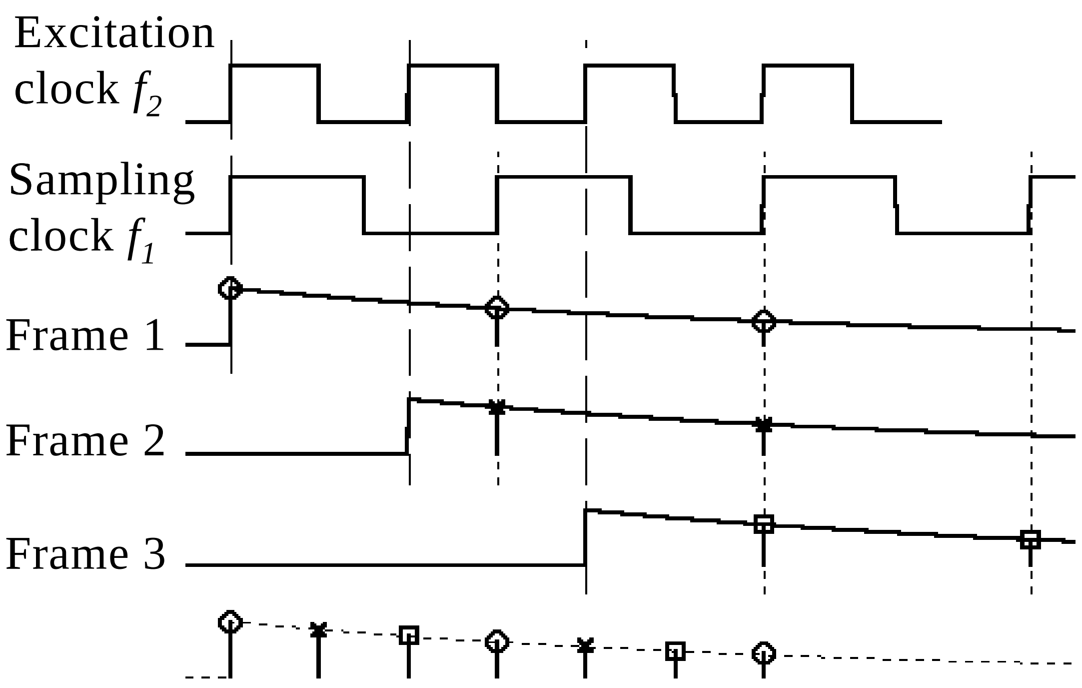
Two clock AIS – the principle



Interleaved digitised waveform

$$M_1 f_1 = M_2 f_2$$

$$M_1 = M_2 + 1$$

$$t_k = t_0 + k\frac{M_1}{f_2} = t_0 + k\frac{M_2}{f_1}$$

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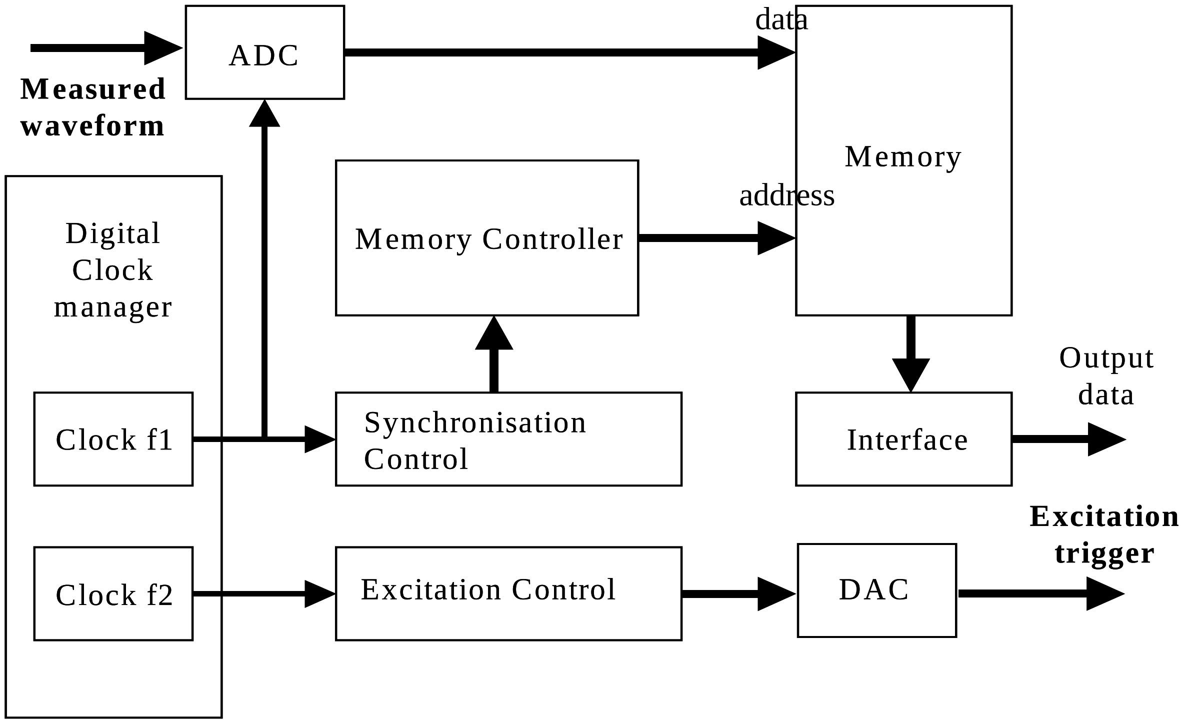
Two clock AIS – the schematic



This architecture is compatible with on-the-fly averaging

(IEEE IMTC-06)

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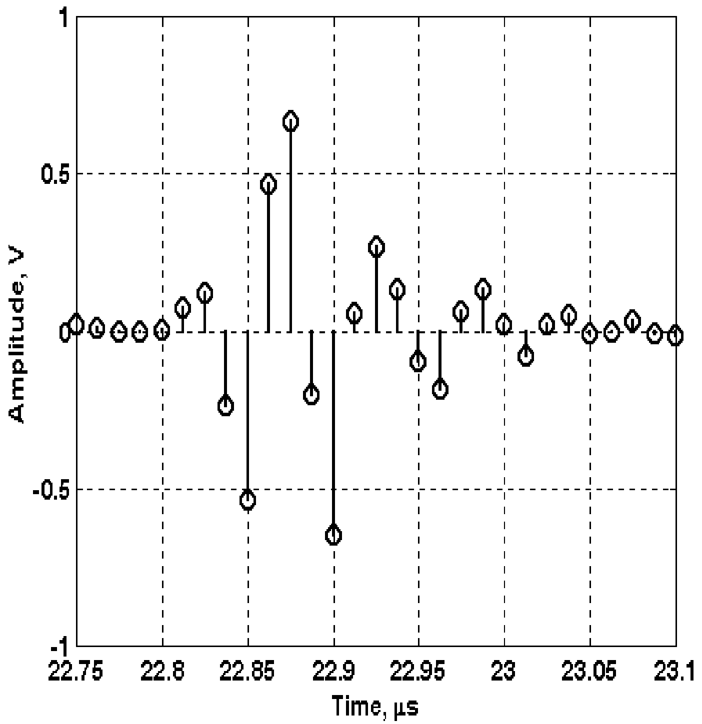
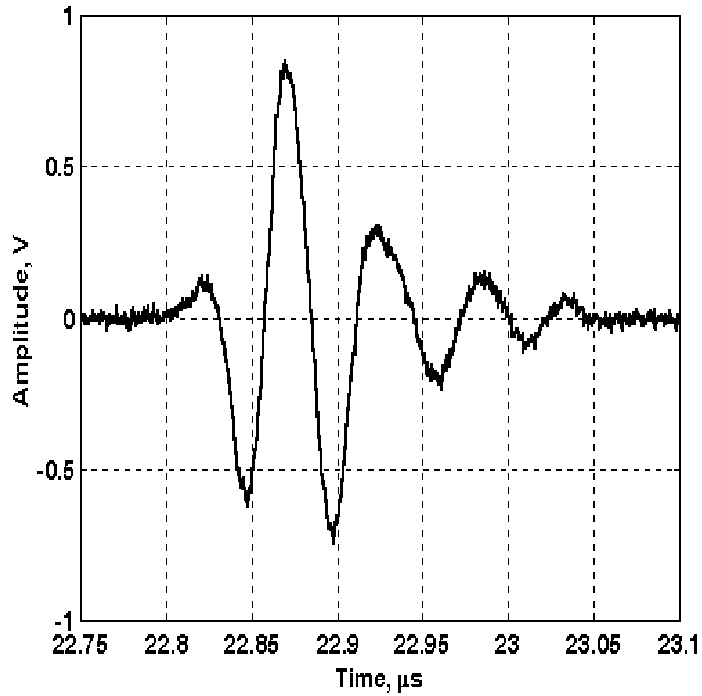
Waveforms (1) - interleaving





Single shot and interleaved waveforms

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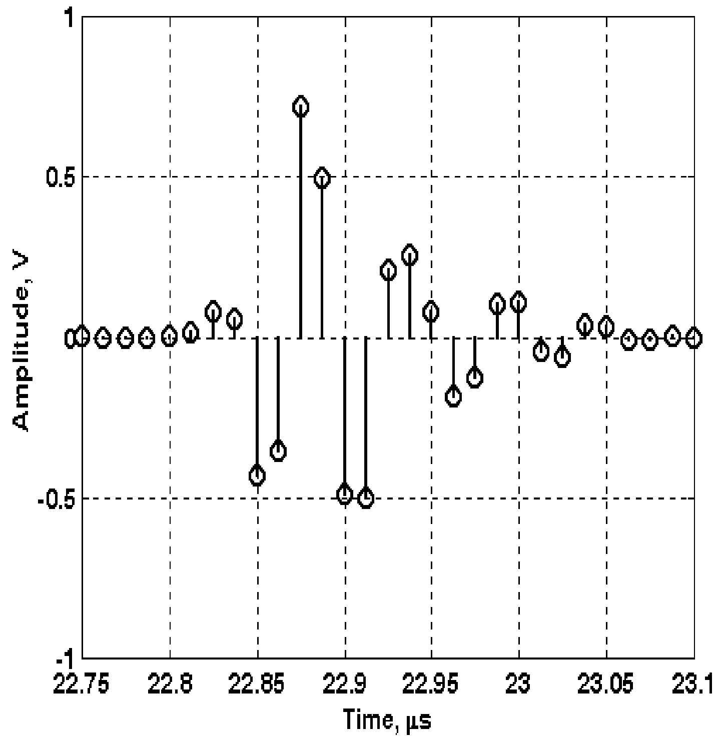
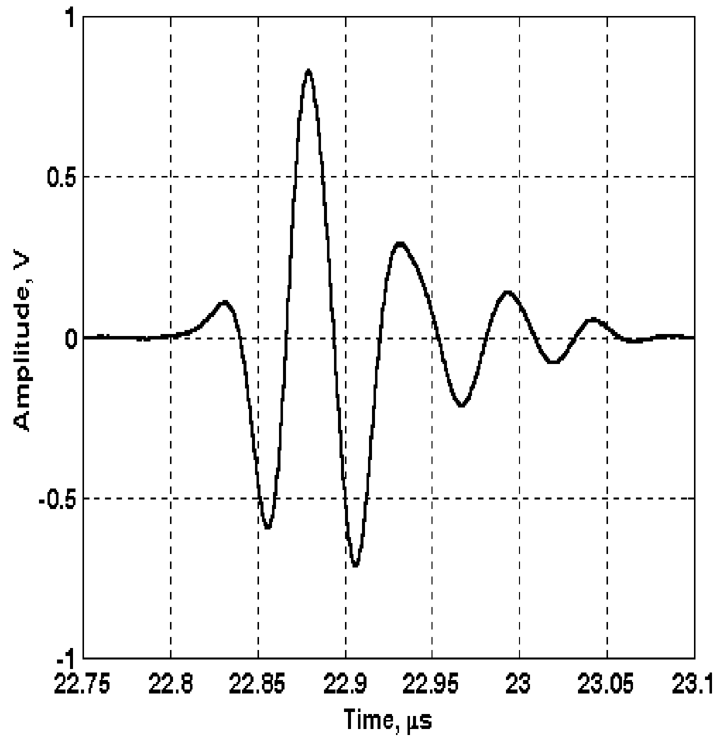
Waveform (2) - averaging





Single shot and interleaved waveforms

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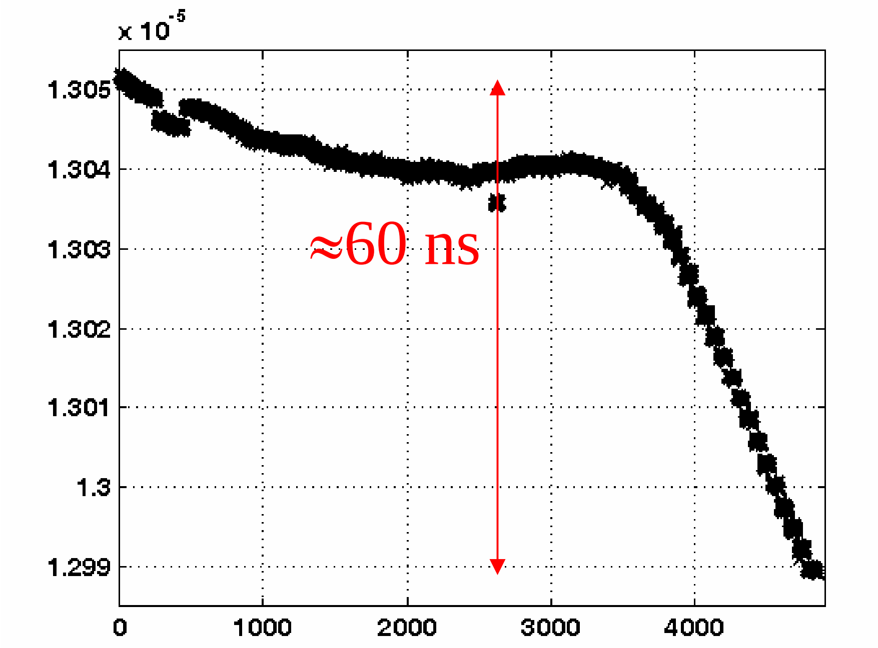
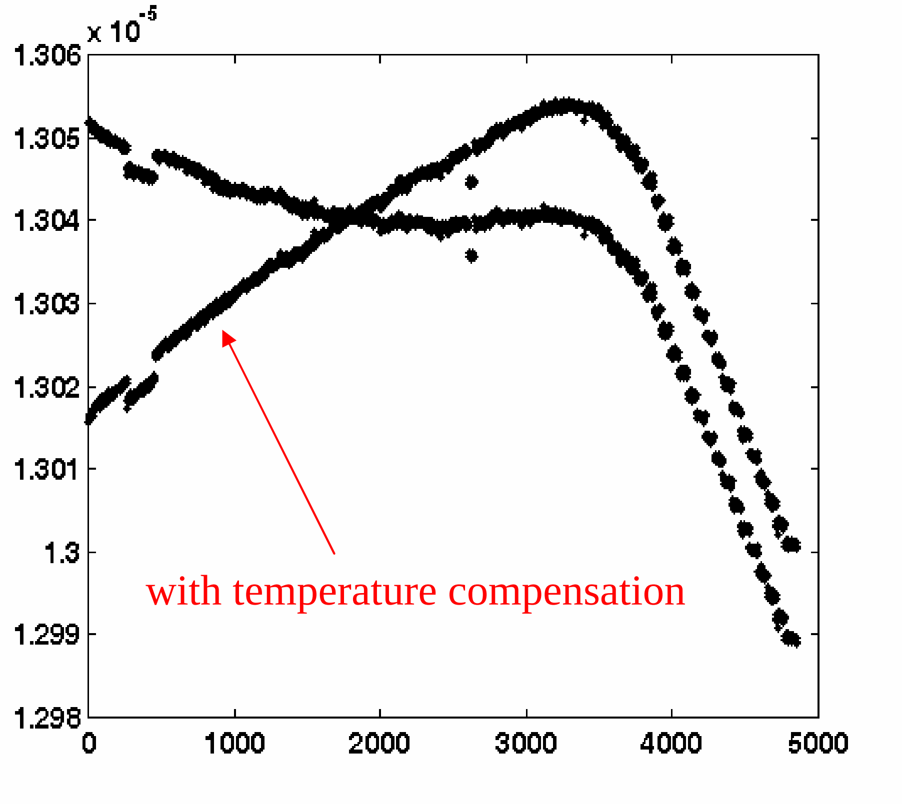
Compensation for temperature



Estimated delays of the US pulse



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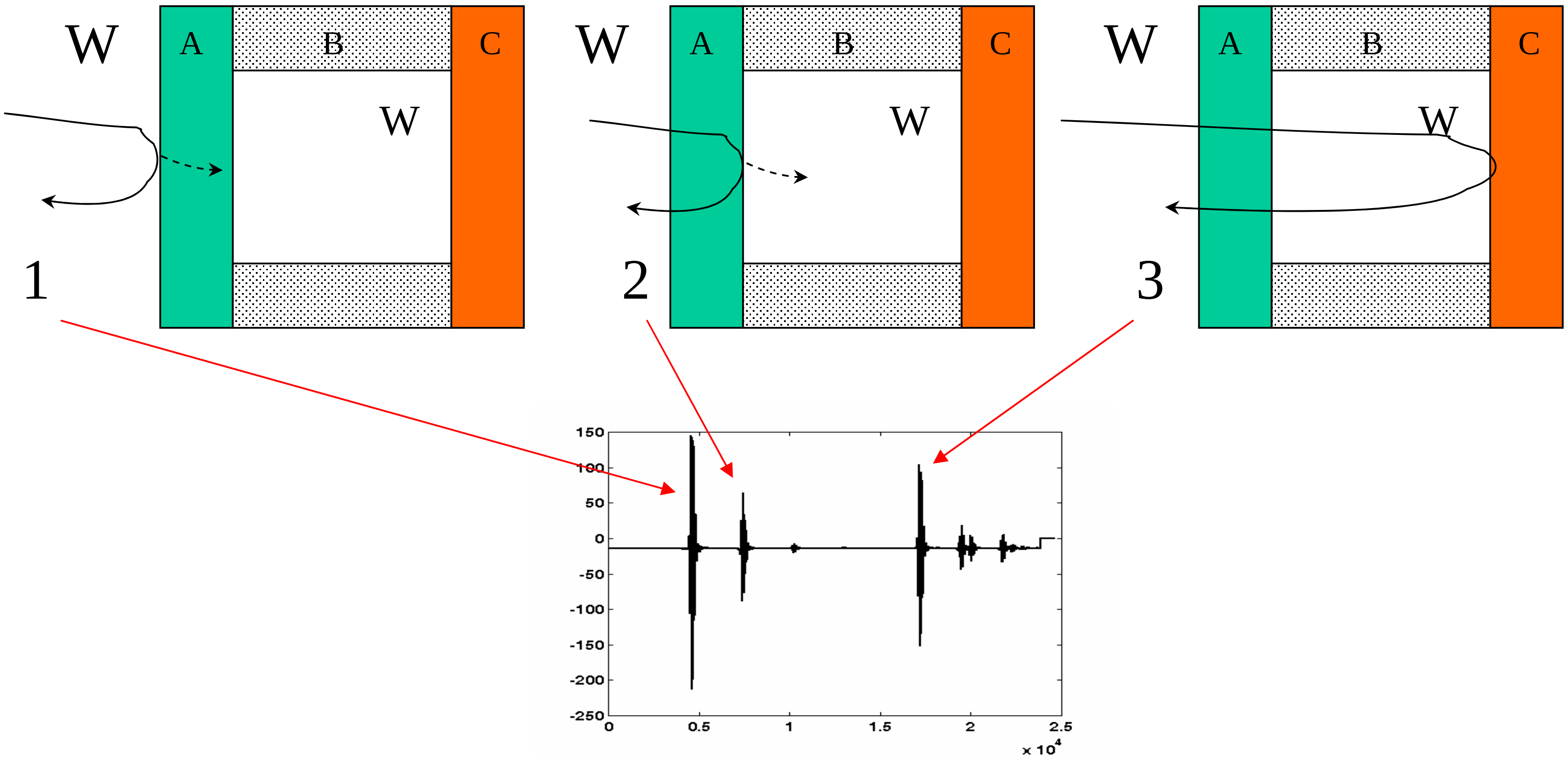
Integrated reflector



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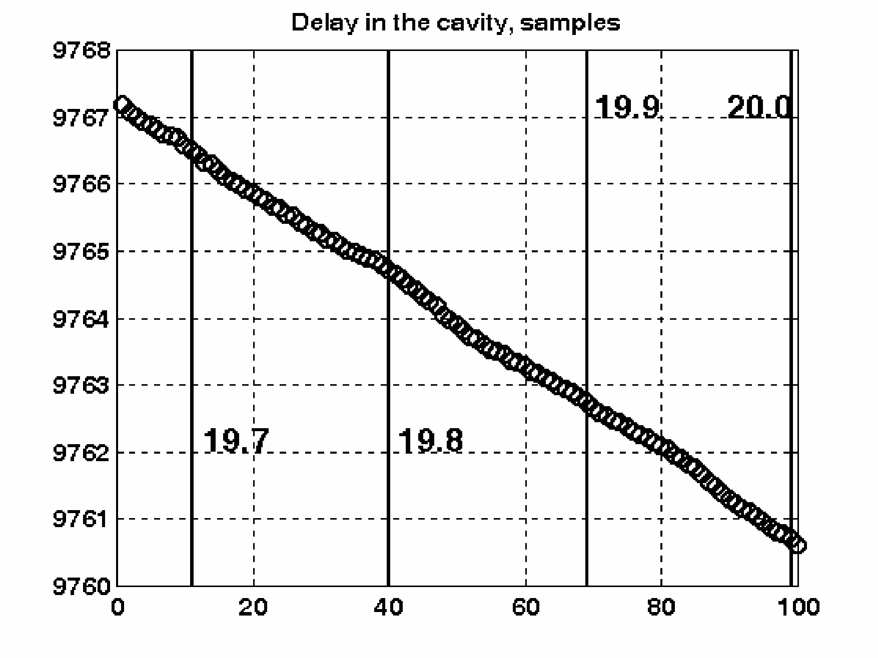
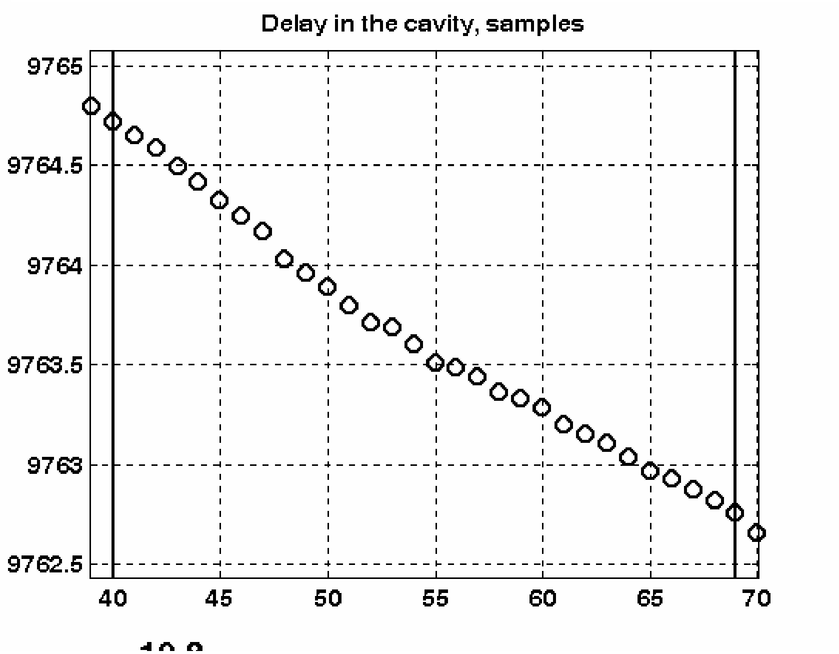
Experimental results





Temperature in the chemical reactor increased due to presence of a stirrer
Records were taken every 10 seconds (IEEE Ultrasonics Symp 2005)

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Summary

- Analysis of experimental data led to development of requirements to ultrasonic instruments
- These requirements were met by developing novel FPGA architectures that combine on-the-fly averaging and two clock accurate interleaved sampling
- Integrated reflector was developed that facilitates temperature monitoring

Conclusions

- Developed FPGA architectures provide high accuracy ultrasonic data acquisition
- Temperature information could be extracted from ultrasonic records purely if the integrated reflector is used
- Reported improvements allowed to track changes in chemical composition with a resolution of below 100 ppm