



Conseil national  
de recherches Canada

National Research  
Council Canada

Institut des matériaux industriels

Industrial Materials Institute

75, boulevard de Mortagne  
Boucherville, Québec  
J4B 6Y4

75 de Mortagne Boulevard  
Boucherville, Québec  
J4B 6Y4

# 2

---

## Laser-Ultrasonics for Metallurgy: Software and Applications

Prepared for Dynamic Systems Inc.

by André Moreau

May 2012

## Table of Contents

1	Introduction to Laser-Ultrasonics for Metallurgy (General overview) .....	4
1.1	<i>Everything</i> affects the elastic constants of metals .....	4
1.2	Laser-ultrasonic hardware.....	5
1.2.1	Optical generation of ultrasound pulses .....	5
1.2.2	Optical detection of ultrasound pulses.....	7
1.3	Ultrasound propagation in isotropic continuous media .....	8
1.3.1	Velocity and elastic moduli.....	8
1.3.2	Attenuation .....	10
1.3.3	Diffraction and Fresnel parameter.....	11
1.3.4	Scattering .....	12
1.3.5	Absorption & Internal friction .....	13
1.4	Ultrasound propagation in single crystals.....	14
1.5	Ultrasound propagation in polycrystalline aggregates .....	16
1.5.1	Elastic constants and texture .....	16
1.5.2	Scattering & grain size .....	17
1.5.3	Absorption, internal friction, and various phenomena.....	17
1.6	Thermo-mechanical processing .....	18
1.7	References .....	19
2	Introduction to LUMet software .....	20
2.1	Comparing 2 echoes – The basic LUMet paradigm.....	20
2.2	Overall software architecture.....	21
3	User Interface .....	23
3.1	Conventions:.....	23
3.2	General Features .....	24
3.3	Starting and Stopping the LUMet software .....	24
3.4	Front panel areas.....	25
3.5	Waveform Display and Navigation area .....	28
3.6	General Data Analysis area .....	32
3.7	Specific data analysis area .....	37
3.8	Result display area .....	41
3.9	Format of saved files.....	42
4	Methods and basic calculations .....	44
4.1	Echo localization .....	44
4.2	Materials properties .....	45
4.3	Noise reduction & estimation.....	46
4.3.1	Dead time .....	47
4.3.2	Filtering.....	47
4.3.3	Waveform averaging.....	48
4.3.4	Noise estimation method.....	49
4.3.5	Smoothing of results .....	49
4.4	Ultrasound Delay, velocity, thickness, and elastic constants .....	50

4.4.1	Velocity measurement when using different echoes from the same waveform.....	51
4.4.2	Velocity measurement when using two different waveforms .....	51
4.4.3	Elastic constants.....	52
4.4.4	Shear waves.....	53
4.4.5	Making accurate velocity measurements .....	54
4.5	Thickness.....	55
4.6	Amplitude, amplitude ratio, and attenuation spectra.....	56
4.7	Diffraction.....	59
4.7.1	Avoiding diffraction.....	59
4.7.2	Removing diffraction effect using model calculations .....	59
4.7.3	Removing diffraction effects empirically .....	59
4.8	Other sources of measurement errors.....	61
4.8.1	Shear waves, surface waves, sample width.....	61
4.8.2	Wedge sample.....	63
4.8.3	Surface roughness.....	63
4.8.4	Cylindrical sample.....	64
5	Advanced analysis.....	65
5.1	Grain size.....	65
5.1.1	Universal empirical calibration .....	67
5.1.2	Calibration 1 for austenite .....	70
5.1.3	Calibration 2 for austenite .....	71
5.2	Texture .....	72
5.2.1	General principles.....	72
5.2.2	Measuring texture changes.....	74
5.3	Isothermal processing (mostly recrystallization) .....	75
5.4	Non-isothermal processing .....	77
5.4.1	Recrystallization.....	77
5.4.2	Phase transformations and austenite decomposition .....	77
5.5	Internal friction .....	78
5.6	Review articles and other references .....	79

# 1 Introduction to Laser-Ultrasonics for Metallurgy (General overview)

Laser-ultrasonics is the generation and detection of ultrasound using lasers. The LUMet acronym stands from **L**aser-**U**ltrasonics for **M**etallurgy. In this section, basic ultrasonic and laser-ultrasonic concepts used are reviewed briefly. They will be used throughout this documentation.

The goal of this documentation is to introduce the LUMet user, typically a metallurgist with only a superficial knowledge of ultrasonics, to making *in-situ*, laser-ultrasonic measurement of ultrasound velocity, attenuation, elastic constants, grain size, texture phase transformations, and their kinetics during thermo mechanical processing of metals.

This documentation describes the basics of laser-ultrasonic technology, the accompanying LUMet software, and describes how various measurements can be made. Together, the documentation and the software are aimed at transmitting the knowledge that we have acquired at the Industrial Materials Institute of the National Research Council of Canada. To write everything in this one document would have been too large a task. Instead, this document presents a general overview and references are made to scientific papers we have published where more details can be found. A complete review of the literature would have been beyond the scope of this document and no attempt is made to reference papers published by others, although a few are cited when they are highly relevant to the subject being discussed. More complete references can be found within our published papers.

## 1.1 *Everything* affects the elastic constants of metals

Often engineers say that *nothing* affects the elastic constants of metals. When selecting a structural material, a 5% variation of elastic constants may seem completely irrelevant compared to a 100% variation in yield strength. But from an ultrasonic point of view *everything* affects the elastic constants of metals, if you can measure the elastic constants with sufficient precision.

The order-of-magnitude of the relative contributions of different factors affecting elastic constants is listed in Table 1.1 below. Laser-ultrasonics allows measuring the elastic constants of metals with an accuracy of order  $10^{-2}$  to  $10^{-3}$ , and with a precision sometimes exceeding  $10^{-4}$ , depending on how carefully the measurements and the analysis are made. Clearly, from a laser-ultrasonic point of view, nearly *everything* affects the elastic constant of metals. Fortunately, not *everything* changes simultaneously when materials are thermo-mechanically processed, and it is usually possible to identify which factor affects the elastic constants. And so, measuring the elastic properties of metals and alloys, and measuring how they vary during thermo-mechanical processing inside the Gleeble, can provide much information about the material, its microstructure, and its microstructure kinetics.

Table 1.1 orders of magnitude of the relative effects (fractional change) of various factors on the elastic constants and sound velocity of metals.

Factor	Relative effect
Material or Alloy	$10^{-1}$
Porosity	$10^{-1}$
Phase transformations	$10^{-1}$
Texture	$10^{-2}$
Grain size	$10^{-2}$
Internal friction	$10^{-3}$
Temperature	$10^{-4}/^{\circ}\text{C}$
Stress	$10^{-5}/\text{MPa}$

## 1.2 Laser-ultrasonic hardware

Laser-ultrasonics is the generation and detection of ultrasound using lasers.

### 1.2.1 Optical generation of ultrasound pulses

To generate ultrasound in metals, high energy pulsed lasers are used. These typically deliver hundreds of mJ of energy per pulse, each pulse having a duration of typically 5 to 10 ns (full width at half maximum, or fwhm). This represents a very high instantaneous power. For example, a 100 mJ pulse of 10 ns duration has an instantaneous power of 10 MW. Some of this light is absorbed in a very thin layer (of thickness comparable to the optical depth in metals) at the surface of the metallic sample. When the power density exceeds the ablation threshold or approximately  $10 \text{ MW}/\text{cm}^2$ , the metallic surface is ablated and plasma is formed at the surface. At power densities 10 to 100 times higher, the plasma creates a strong pressure. This pressure in turns causes a compression of the surface and launches a pressure pulse in the sample. The pressure pulse always travels in the direction normal to the surface where it is generated, although it will also travel with less amplitude in directions close to the normal direction. The pressure pulse is also called a pressure pulse or wave, or a longitudinal pulse or wave. The word "longitudinal" describes the fact that the motion of the atoms caused by the compression is in the same direction as the direction in which the pulse travels. This pulse is very short in duration, with a rise time comparable to the light pulse duration, that is a few ns. The frequency content of the pulse is therefore in the MHz range, well above the 20 kHz audible limit, and the pressure pulse is therefore an ultrasonic pulse.

Most of the time, the generation laser is focused to a circular spot. Within this spot, the light intensity is relatively constant. At the edge of the spot, it falls quickly to zero. This causes a plasma with a strong pressure gradient. This pressure gradient generates a shear pulse (also called transverse pulse or wave) that propagates roughly 45 degrees away from the surface normal direction, although the shear pulse will also travel with less amplitude in directions away from 45 degrees. The word "transverse" describes the fact that the motion of atoms caused by

the shear wave is in the direction perpendicular (or transverse) to the propagation direction. One special case of transverse waves is surface waves where the wave is confined to the surface of the sample. Surface waves travel at a speed slightly less than the speed of bulk transverse waves.

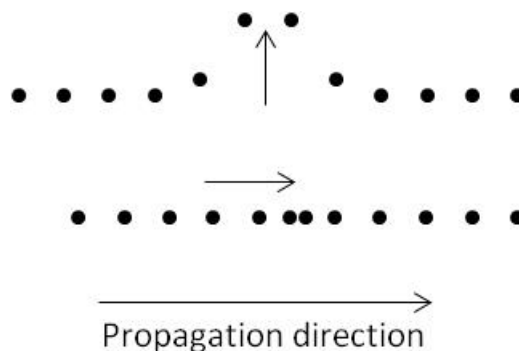


Figure 1.1: Schematic representation of the transverse (shear) pulse (top) and a longitudinal (compressive) pulse (bottom) propagating to the right in a one-dimensional array of atoms (fat dots). The motion of atoms is either transverse to (top) or in the same direction as (bottom) the propagation direction.

As the laser energy density, or fluence, is increased, the plasma generates higher and higher pressures until eventually the plasma generated in the first few ns blocks the laser radiation coming at later times. The laser radiation coming at later times only serves to heat the plasma further and creates a detonation wave in the air. The hot plasma can melt the metal surface, cause a lot of surface damage, and does not contribute to ultrasound generation. The importance of this phenomenon varies depending on the material (steel vs. Aluminum) and on the atmospheric conditions (air, vacuum, inert gas). This is why, for a given generation spot size and material, there is a maximum useful laser pulse energy for maximum ultrasound amplitude. Conversely, for a given generation laser energy and materials, there is an optimal spot size below which ultrasound amplitude will not increase further. It should be noted that the presence of an atmosphere provides larger signal amplitude than generation under vacuum.

The amount of metal ablated can be extremely small, or can be large enough to create a dip in the surface. As fluence (energy per unit area) is increased, the amount of material removed from the surface (before the hot plasma might melt the surface) increases. Eventually, this can cause a measurable amount of surface damage. Therefore, there is always a trade off between ultrasound amplitude and the number of times the measurement can be repeated. This trade off is controlled by laser fluence. Typically, a good compromise will generate ultrasound pulse of relatively large amplitude for more than 1000 times before the surface is damaged enough to affect the ultrasound.

In a typical laser-ultrasonic experiment in a flat sample, the generated ultrasound pulse is a compressive pulse traveling in the thickness direction away from the source. Upon reaching the opposite surface, the pulse is reflected back on itself and changes polarisation, i.e. the positive (compression) strain reverses itself into a negative (traction) strain. The pulse travels back to

the source and is reflected back into a compressive pulse. The process repeats itself until the ultrasound pulse decays below the noise level of the detector (Figure 1.2).

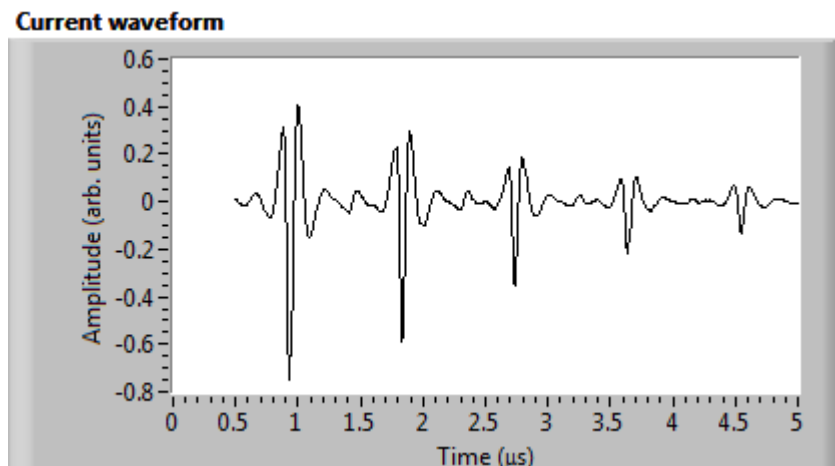


Figure 1.2 Successive compressive echoes observed at the point of generation of a compressive pulse for a flat steel sample of 2.21 mm thickness at 998 °C. The first three echoes can be seen near 0.9, 1.8, and 2.7  $\mu\text{s}$ , respectively. Small transverse pulses can also be seen near 1.5  $\mu\text{s}$  and 2.4  $\mu\text{s}$ .

### 1.2.2 Optical detection of ultrasound pulses

To detect these ultrasound pulses, a laser interferometer is utilized on either side of the sample. A laser illuminates a spot on the sample surface and the reflected light is collected by an optical system. The collected light is then analyzed in an interferometer. Either confocal Fabry Perot or photorefractive interferometers are used because they do not require the sample surface to be polished to a mirror finish. In other words, these two interferometers can utilize speckled light.

These laser interferometers sense the displacement of the sample surface along the normal direction. Therefore, they are very sensitive to the longitudinal waves that travel in the thickness direction. Conversely, they are not sensitive to transverse waves that travel in the thickness direction, and are weakly sensitive to transverse waves that travel close to the normal direction. On the other hand, transverse waves tend to have large amplitudes and the laser interferometer often will detect them.

The amplitude of the detected signal is proportional to the amount of light collected in the interferometer. Therefore, it is desirable to maximize the amount of collected light. On the other hands, un-polished samples have rough surfaces that diffuse light in all directions, and most of the light shone on the sample surface escapes the detection optics. Therefore it is desirable to have a powerful detection laser to avoid having to polish the sample's surface. Typically a long pulse (tens to hundreds of  $\mu\text{s}$  fwhm) laser is used to that effect. The duration of the pulse is long enough to observe the propagation and decay of an ultrasound pulse, but short enough to concentrate the pulse energy in time and so attain a high instantaneous power, typically of order 1 kW for a duration of tens of  $\mu\text{s}$ .

During an experiment in the Gleeble, it is often observed that the amplitude of the generated ultrasound pulse varies with temperature. This is normal because the material properties that affect ultrasound generation efficiency can change with temperature. Also, the amount of light collected can change because the surface may oxidize (if the vacuum is poor) or may be damaged or discolored by the generation laser. In addition, mechanical deformation affecting the shape of the sample will also change the amount of light reflected back in the collection optics. Therefore, in laser-ultrasonics, absolute signal amplitude is meaningless in the sense that it does not tell us any useful information about the sample being probed. However, signal amplitude must be maximized to improve signal-to-noise ratio (S/N). Having the best possible signal-to-noise ratio is key to obtaining accurate data and seeing subtle variations in materials' behaviour.

## 1.3 Ultrasound propagation in isotropic continuous media

### 1.3.1 Velocity and elastic moduli

In isotropic media, longitudinal waves travel with velocity,  $V_l$ , given by

$$V_l = \sqrt{\frac{K + \frac{4}{3}S}{\rho}}$$

where  $K$  is uniform compression modulus,  $S$  is shear modulus, and  $\rho$  is density of the material. Similarly, the transverse wave velocity,  $V_t$ , is given by

$$V_t = \sqrt{\frac{S}{\rho}}$$

These equations are valid for infinite media. They are also valid for waves traveling in the thickness direction of plates. However, these equations do not hold when the surfaces of the sample affect sound propagation. They do not hold for waves traveling at the surface (surface waves, also called Rayleigh waves), for waves traveling in the long directions of plates (plate waves, also called Lamb waves), or in the long direction of rods.

As these equations show, the sound velocities in isotropic media are simply related to the  $K, S$  pair of moduli. If the two velocities  $V_l$  and  $V_t$  are measured, then the two moduli can be estimated by inverting the above two equations. This  $K, S$  pair is often very useful because  $K$  describes mechanical deformations that affect the volume only, and  $S$  describes shear deformations that do not affect the volume at all. However, another pair is often used: Young's modulus,  $E$ , and Poisson's ratio,  $\sigma$ . They are related to  $K$  and  $S$  according to

$$E = \frac{9KS}{3K + S}$$

$$\sigma = \frac{1}{2} \left( \frac{3K - 2S}{3K + S} \right)$$



In laser-ultrasonics, the longitudinal wave velocity is much easier to measure than the shear wave velocity. If the sample is known to be elastically isotropic, and if Poisson's ratio is a known constant, an assumption commonly made, then Young's modulus is related to the longitudinal wave velocity according to

$$E = \frac{\rho(1 + \sigma)(1 - 2\sigma)V_l^2}{(1 - \sigma)}$$

The above equation is useful for providing an approximation of  $E$ . As we will see, laser-ultrasonic measurements of velocity are highly precise. They can easily detect variations in Poisson's ratio. Therefore, the assumption that Poisson's ratio is a constant is only an approximation. To obtain accurate estimates of  $E$ , it is necessary to measure both  $V_l$  and  $V_t$ , calculate  $K$  and  $S$ , and then calculate  $E$  and  $\sigma$ . To summarize, velocity measurements provide measurements of elastic moduli.

Velocity measurements are conceptually simple. In the case of an ultrasound pulse generated and detected on opposite sides of a flat sample, the sound velocity can be obtained simply by dividing the sample thickness by the arrival time of the first echo. When ultrasound is generated and detected on the same side of a flat sample, the sound velocity can be obtained simply by dividing twice the sample thickness by the arrival time of the first echo.

However, as can be observed in Figure 1.3, it is not clear exactly when the first echo arrives. Should the arrival time be measured as the leading edge of the pulse, or the time at maximum positive amplitude, or maximum negative amplitude, or the time at some zero crossing? Moreover, the origin of the time scale is related to some opto-electronic trigger and may not perfectly match the exact time of ultrasound generation. Finally, the laser generation process occurs on time scales of ns (Section 1.2.1), yet the travel time of acoustic pulses often can be measured to higher precision than 1 ns. So instead of measuring the arrival time of a single echo, velocity is typically measured by use of two successive echoes, provided that the two echoes have the same shape. In this case, velocity is measured according to

$$V = \frac{2(m - n)h}{(t_m - t_n)}$$

where  $V$  is velocity,  $h$  is thickness,  $m$  and  $n$  are the index number of the  $m^{th}$  and  $n^{th}$  echoes, and  $(t_m - t_n)$  is the time difference between the  $m^{th}$  and  $n^{th}$  echoes.

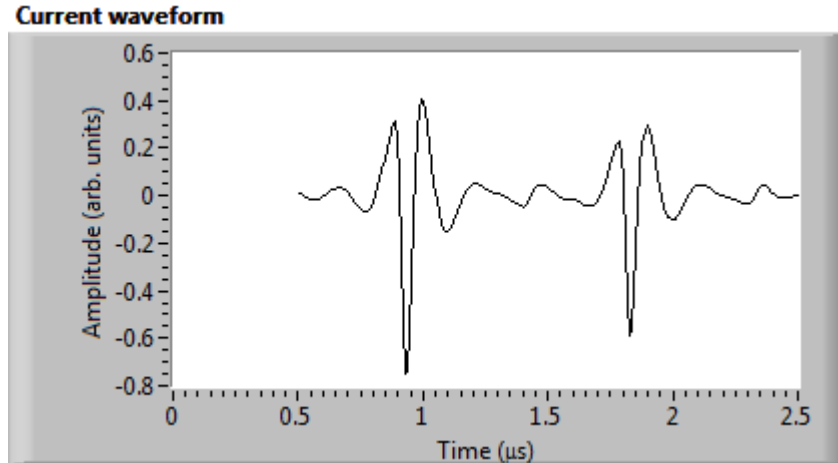


Figure 1.3 Enlargement of Fig. 1.2

The LUMet software utilizes a highly precise numerical cross-correlation method to estimate the time delay between two echoes. The precision of the measurement is much better than the sampling time interval of the digitizer. Velocity is typically measured with an accuracy (absolute error) equal to the accuracy of the thickness measurement, i.e. of about 1%. The precision (repeatability) of the measurement when it is done multiple times at the same location on a sample, such as when the temperature dependence of velocity is measured, can be as good as or better than 0.01%. Because time delay can be measured so accurately, and because it is usually not too difficult to measure or estimate density, the best measurements of elastic moduli are usually done by measuring ultrasound velocity.

### 1.3.2 Attenuation

Attenuation is defined as the loss of ultrasound amplitude as a function of propagation distance. In Section 1.2.2, it was mentioned that the absolute amplitude of echoes was meaningless because signal amplitude depends on changing experimental conditions; only the relative amplitude of echoes is meaningful. The amplitude ratio of the  $m^{th}$  and  $n^{th}$  echoes is defined as  $A_m/A_n$ . It has no units. The attenuation,  $\alpha$ , of an ultrasound pulse is the amplitude ratio of two echoes, converted to decibels, and divided by the propagation distance:

$$\alpha = \frac{20}{2(m-n)h} \log \frac{A_n}{A_m}$$

Attenuation is highly dependent on ultrasound frequency,  $f$ . Therefore, it is important to always specify the frequency at which it is measured. Laser-ultrasonic pulses contain a broad spectrum of frequencies. The signal bandwidth usually extends from zero (or rather the low frequency cut-off of the interferometer) to tens of MHz. Therefore, we can highlight this frequency dependence in the previous equation, by writing

$$\alpha(f) = \frac{20}{2(m-n)h} \log \frac{A_n(f)}{A_m(f)}$$

where  $A_n(f)$  is the amplitude spectrum of the  $n^{\text{th}}$  echo, and  $\alpha(f)$  is the attenuation spectrum. The absolute amplitude of echoes is never used to measure attenuation. It is imperative to use amplitude spectra of pulses. The LUMet software measures  $A_n(f)$  and  $\alpha(f)$ .

Attenuation is caused by three distinct phenomena: Diffraction, scattering, and absorption.

### 1.3.3 Diffraction and Fresnel parameter

Just as light waves traveling through a small hole diffract, ultrasound waves traveling through holes diffract also. Moreover, there is no difference between a wave traveling through a hole and a wave generated at the hole: both will diffract in exactly the same way.

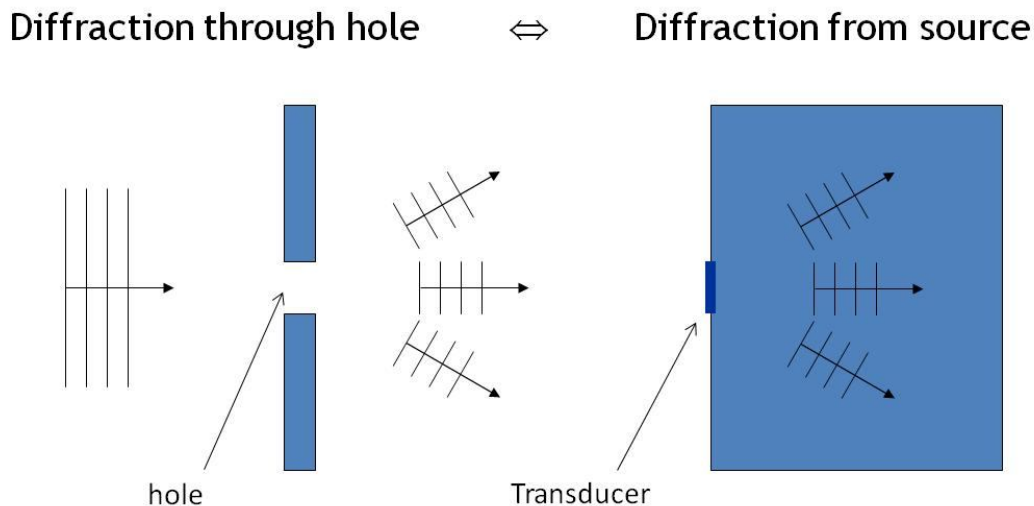


Figure 1.4: Wave traveling through a hole produces the same diffraction pattern as a wave emitted by a transducer of the same dimensions.

Diffraction is thus caused and determined by the geometry of the source. The reason we need to worry about diffraction is that it affects the velocity and attenuation measurements. For most purposes, the following discussion will suffice.

Diffraction can be described as occurring in three stages: the near, intermediate, and far fields. The near field occurs when the source is large, ultrasonic wavelength is short, and the propagation distance is short. In the near field, the ultrasound pulse behaves like a plane wave. Diffraction is of no consequences.

In the far field, the source is small and the propagation distance is large. The ultrasound amplitude is proportional to  $1/r$ , where  $r$  is propagation distance. Therefore, the contribution of diffraction to ultrasound attenuation is a constant that depends of the propagation distance of the two echoes being compared. For example, when generation and detection are on the same side of a flat sample, and when the first and second echoes are measured, the diffraction contribution to the total attenuation is, in the far field,

$$\alpha(f) = \frac{20}{2(2-1)h} \log \left( \frac{1/2h}{1/4h} \right) = \frac{20 \log(2)}{2h} = \frac{6 \text{ dB}}{2h}$$

That is, the contribution of attenuation is to reduce the pulse amplitude of the second echo to one half the amplitude of the first echo, at all frequencies. In-between the near and the far field, that is in the intermediate field, the contribution of diffraction to ultrasonic measurements is complex. It is best to avoid doing measurements, especially attenuation measurements, in the intermediate field.

For a source with the geometry of a circular disc, the Fresnel parameter,  $S$ , is defined as

$$S = \frac{\lambda z}{a^2}$$

where  $\lambda$  is ultrasound wavelength,  $z$  is propagation distance, and  $a$  is the radius of the source. The definitions of the near, intermediate, and far fields and their effects on ultrasound are described in the table below.

Table 1.2 Description of the near, intermediate and far acoustic fields for a circular transducer.

Fresnel parameter	Field	Wave characteristics
$S < 0.1$	Near	Plane waves
$0.1 < S < 10$	Intermediate	Complicated diffraction patterns
$10 < S$	Far	Amplitude decreases as $1/r$

Unfortunately, measurements are often made in the intermediate field. In such conditions, diffraction will cause sizeable measurement errors. However, in the intermediate field, the effects of diffraction are greatly reduced when the source and the detector have the same dimensions. For this reason, it is recommended that the LUMet always be operated using generation and detection spots of the same size. More details can be found in Section 4.7.

### 1.3.4 Scattering

This section addresses homogeneous media and no scattering should occur in a homogenous medium. However, if inhomogeneities such as defects, precipitates or inclusions are present in the otherwise homogeneous medium, then scattering can occur. Scattering redirects the ultrasound energy in random directions. This contributes to the overall attenuation of the (unscattered) ultrasound pulse.

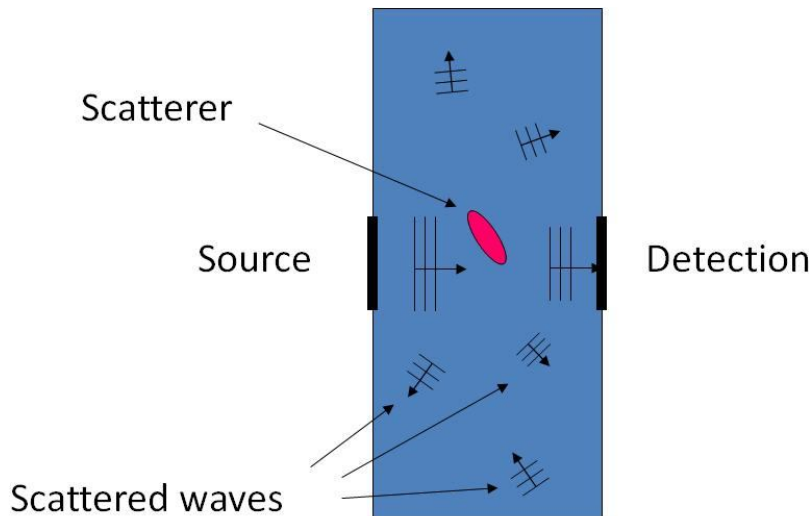


Figure 1.4 A wave traveling from the source to the detection area is scattered. Less energy reaches the detector.

Scattering occurs when the acoustic impedance of the material changes. The acoustic impedance is the ultrasonic equivalent of the index of refraction for light. When light propagates through a boundary between two materials of different indices of refraction, it is reflected and/or refracted at the boundary. The same occurs for ultrasonic waves propagating between two materials of different acoustic impedances. The acoustic impedance,  $Z$ , is defined as  $Z = \rho V$  where  $\rho$  is density and  $V$  is ultrasound velocity.

When the scatterers are much smaller than the ultrasonic wavelength or when the difference in acoustic impedance is small, scattering is weak. For weak scattering, scattering increases linearly with the number of scatterers. Also, when the scatterers are much smaller than the ultrasonic wavelength, scattering increases as the sixth power of their size. At 10 MHz, in a typical metal having a sound velocity of 6000 m/s, sub-micron precipitates are much smaller than the ultrasonic wavelength of 600  $\mu\text{m}$  do not scatter ultrasound measurably, even if their acoustic impedance is very different. However, defects, porosity, precipitates or inclusions in the range of 10 to 100 microns (or larger) can contribute to scattering.

### 1.3.5 Absorption & Internal friction

Absorption is the conversion of the ultrasound energy into heat energy. Ultrasound absorption multiplied by some constant and divided by frequency is called internal friction, or  $Q^{-1}$ . Because of this simple relationship between absorption and internal friction, the two names are often used interchangeably.

$$Q^{-1} = \frac{0.115}{\pi} \times \frac{\alpha [\text{dB}/\mu\text{s}]}{f [\text{MHz}]}$$

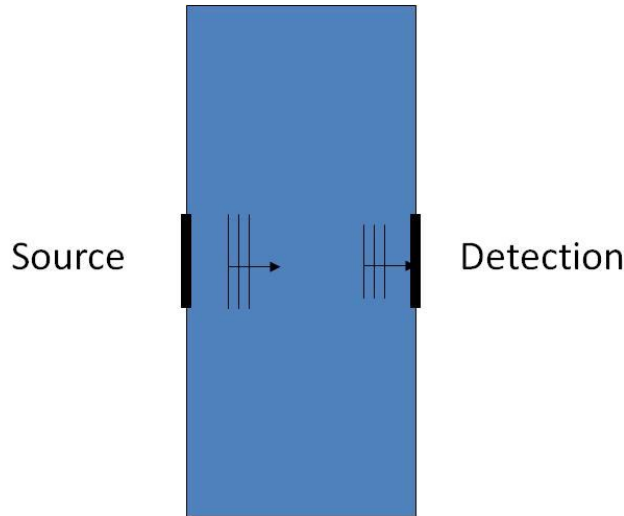


Figure 1.5 A wave traveling from the source loses mechanical energy to heat energy before reaching the detector.

Traditionally, the field of internal friction, also called mechanical spectroscopy, describes how mechanical vibrations are damped. The vibrations may have any frequency, from the mHz range up some hundreds of kHz. However, ultrasound is also a mechanical vibration, and ultrasonics allows to extend the field of internal frictions to frequencies up to tens or even hundreds of MHz. Laser-ultrasonics is ideally suited for internal friction measurements at ultrasonic frequencies because, unlike for contact transducers, no ultrasonic energy is absorbed in the transducer or in the bond between the transducer and the sample.

Absorption can be caused by a large variety of phenomena. For homogeneous media, it can be a measure of viscosity. However, the effect of absorption in ultrasonics for metallurgy is described more appropriately in the section on ultrasonic propagation in polycrystalline aggregates (Section 1.5).

## 1.4 Ultrasound propagation in single crystals

Single crystals are not elastically isotropic. The relationship between stress and strain is described by Hooke's law:

$$\sigma_{ij} = c_{ijkl} \varepsilon_{kl}$$

where  $\sigma_{ij}$  is the second rank stress tensor,  $\varepsilon_{kl}$  is the second rank strain tensor, and  $c_{ijkl}$  is the fourth rank elastic stiffness tensor. In this notation, the indices run from 1 to 3, and there is an implied summation over repeated indices. The elastic stiffness tensor contains 81 entries (3 to the fourth power), of which at most 21 are independent. For materials with orthorhombic symmetry, only 9 entries are independent. For materials with hexagonal, cubic, or isotropic symmetries, only 5, 3, or 2 entries are independent, respectively. Because of this high degree of

redundancy, and because fourth rank tensors are difficult to represent on a sheet of paper, it is customary in ultrasonics to utilize an alternate representation of Hooke's law,

$$T_i = c_{ij}S_j$$

where  $T$  and  $S$  are the stress and strain pseudo-vectors, respectively, and  $c$  is the elastic stiffness pseudo-tensor of second rank. In this notation, the indices run from 1 to 6, and there is an implied summation over repeated indices. The pseudo vectors are defined as:

$$T = \begin{pmatrix} T_1 \\ T_2 \\ T_3 \\ T_4 \\ T_5 \\ T_6 \end{pmatrix} = \begin{pmatrix} \sigma_{xx} \\ \sigma_{yy} \\ \sigma_{zz} \\ \sigma_{yz} \\ \sigma_{zx} \\ \sigma_{xy} \end{pmatrix}, \quad S = \begin{pmatrix} S_1 \\ S_2 \\ S_3 \\ S_4 \\ S_5 \\ S_6 \end{pmatrix} = \begin{pmatrix} \varepsilon_{xx} \\ \varepsilon_{yy} \\ \varepsilon_{zz} \\ \varepsilon_{yz} \\ \varepsilon_{zx} \\ \varepsilon_{xy} \end{pmatrix}$$

They are called pseudo tensors and pseudo vectors because their mathematical properties are a little different from those of true tensors. For example, they have a different rotation operator. However, within the scope of this document, the differences are not important. In particular, matrix multiplications are not affected.

Manufacturing processes typically impart orthotropic (the symmetry of a rectangular parallelepiped) or higher symmetry on the macroscopic elastic constants. In this case, the elastic stiffness tensor is

$$c = \begin{pmatrix} c_{11} & c_{12} & c_{13} & 0 & 0 & 0 \\ c_{12} & c_{22} & c_{23} & 0 & 0 & 0 \\ c_{13} & c_{23} & c_{33} & 0 & 0 & 0 \\ 0 & 0 & 0 & c_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & c_{55} & 0 \\ 0 & 0 & 0 & 0 & 0 & c_{66} \end{pmatrix}$$

The nine independent constants are the 6 diagonal element and 3 off-diagonal elements. The six diagonal elements are simply related to 6 ultrasound velocities, namely

$$v_{ii} = \sqrt{\frac{c_{ii}}{\rho}}$$

When the index  $i$  is equal to 1 to 3,  $V_{ii}$  is the longitudinal velocity in the 1 to 3, or  $x$ ,  $y$ ,  $z$  directions, respectively.  $c_{44}$  is the shear modulus corresponding to waves propagating in the  $y$  direction and polarized in the  $z$  direction.  $c_{55}$  is the shear modulus corresponding to waves propagating in the  $z$  direction and polarized in the  $x$  direction.  $c_{66}$  is the shear modulus corresponding to waves propagating in the  $x$  direction and polarized in the  $y$  direction.

More generally, the relationship between the elastic stiffness tensor and the ultrasound velocity in any direction is given by the Christoffel equation. The off-diagonal elements are difficult to measure.

The symmetry properties of the elastic stiffness tensor, the simple expressions for ultrasound velocities in high symmetry directions, and the Christoffel equation are described in Auld's or Royer and Dieulesaint's textbooks listed in reference below.

## 1.5 Ultrasound propagation in polycrystalline aggregates

Usually, metals are polycrystalline aggregates. That is they are made of a collection of crystallites, also called grains. Polycrystalline aggregates are characterized, among other things, by the mean size and the size distribution of the grains, and by the crystallographic orientation distribution (also called crystallographic texture, or texture) of the grains. Measuring ultrasound velocity can give information about texture. And measuring ultrasound attenuation can give information about the mean grain size.

### 1.5.1 Elastic constants and texture

The overall properties of metals are generally not isotropic, in spite of the fact that isotropic behaviour is often assumed. Typical manufacturing steps, such as rolling, forging, and elongating, cause the development of crystallographic texture, and this in turns causes the anisotropy of elastic constant. In general, however, the overall anisotropy retains orthorhombic or higher symmetry. Therefore, the elastic and ultrasonic behaviour of metals are described using the same equations as those for single crystals. However, to distinguish the single crystal elastic stiffness constants  $c_{ij}$ , from the elastic stiffness constant of the aggregate, the elastic constants of the aggregate will be written  $C_{ij}$  (with a capital letter "C"). Therefore, Hooke's law for polycrystalline aggregates is written

$$T_i = C_{ij}S_j$$

The elastic constants of the aggregate,  $C_{ij}$  can be calculated from the elastic constants of the single crystals,  $c_{ij}$ , and from the orientation distribution function (ODF). Therefore, measuring the sound velocity of polycrystalline aggregates can give information about texture. It is even possible, in some cases, to solve the inverse problem and to calculate some of the coefficients of the ODF.

Note that to help avoid the potential confusion between single crystal and aggregate properties, the single crystal properties are written with lower case letters, while aggregate properties are written with upper case letters. Therefore, the longitudinal sound velocity relationship to elastic constants is written

$$v_{ii} = \sqrt{\frac{c_{ii}}{\rho}}$$

for a single crystal, and

$$V_{ii} = \sqrt{\frac{C_{ii}}{\rho}}$$



for a polycrystalline aggregate such as a steel sample.

### 1.5.2 Scattering & grain size

In section 1.4, it was discussed that the ultrasound velocity varies according to the propagation direction in single crystals. In polycrystalline aggregates, the crystallites, or grains, are randomly oriented (even in the presence of a preferential alignment, or texture). Therefore, as the ultrasound pulse propagates, it keeps changing velocity.

When light travels through an interface between two transparent materials that differ in their index of refraction, it is partially reflected and refracted by the interface. In ultrasonics, the equivalent of the index of refraction is the acoustic impedance,  $Z$ , defined as

$$Z = \rho V$$

In a polycrystalline aggregate, the density,  $\rho$ , is constant while the velocity,  $V$ , varies according to the orientation of the grains (Section 0). Therefore the ultrasound pulse sees varying acoustic impedance. The variation may be small or large, depending on the materials (e.g. it is relatively small for Al and large for Fe). In analogy with optical materials, this causes reflection and refraction at grain boundaries, and thus some scattering of the ultrasound energy, and a reduction in the amplitude of the unscattered pulse. That is, it causes an observable attenuation of the ultrasound pulse. This scattering can be shown to increase with the ratio of grain size to ultrasound wavelength, as discussed for precipitates in Section 1.3.4. However grains tend to be much larger than precipitates and, usually, ultrasound at 10 MHz is scattered measurably by grains in the 10 to 100 microns (or larger) range. Consequently, a measurement of ultrasound attenuation can provide information about the mean grain size. This will be discussed further in Section 5.1.

### 1.5.3 Absorption, internal friction, and various phenomena

Various phenomena can cause ultrasonic absorption. In general, these phenomena are described by anelastic relaxation theory, i.e. they involve a mechanism whereby the material, after being mechanically deformed, returns to its equilibrium state only after some time has elapsed. Plastic or viscoelastic processes (that cause permanent deformation) are rarely involved in laser-ultrasonic propagation. (Two exceptions are laser-peening and spallation of coatings. Both require very high energy lasers.) The following anelastic phenomena have been observed to cause ultrasound absorption using laser-ultrasonics:

Compressive stresses can cause magnetic domains to re-orient themselves. As they re-orient, the changing magnetic field induces electrical eddy currents that dissipate energy through ohmic resistance. Thus, laser-ultrasonics can be used to obtain information about magnetic domains and magnetic domain pinning.

Compressive stresses can cause interstitial atoms to hop from one interstitial site to another. The Snoek relaxation describes this process. Thus, laser-ultrasonics can be used to obtain information about interstitial atoms.

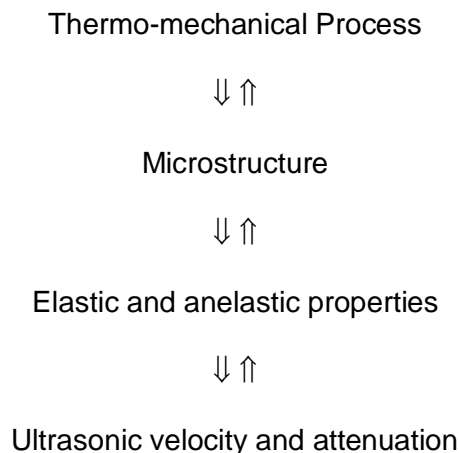
Stresses can also make dislocations move, or at least bend between two pinning sites. Thus, laser-ultrasonics can be used to obtain information about dislocations, such as during recovery.

However, the effect of absorption on velocity and attenuation is small compared to the effect of phase transformations, texture, and grain size. Absorption can be neglected in first approximation most of the time. However, when a small and un-explained variation in velocity or attenuation is observed to depend on recovery, precipitation, magnetic effects, or dislocation behaviour, it is possible that the internal friction contribution is changing significantly.

## 1.6 Thermo-mechanical processing

In the previous sections, we saw that laser-ultrasonics can be used to obtain information about the microstructure: this information includes information about phase, texture, grain size, magnetic domains, dislocations, and interstitial elements. When a sample is processed thermo-mechanically in the Gleeble, phase transformations, recrystallization, grain growth, precipitation, and other phenomena can occur. All of these processes modify the microstructure, and can therefore be monitored using laser-ultrasonics.

In summary, laser-ultrasonics measures ultrasound velocity and attenuation as a function of frequency, and as a function of processing parameters such as temperature, time, and applied deformation. The sound velocity changes because the elastic constants of the material are affected by every aspect of the microstructure. And the microstructure changes because of the applied thermo-mechanical process. This relationship is illustrated below.



Although the task of figuring out what affects the elastic properties of the materials may seem daunting at first, usually only one or two microstructural parameters are changing at a time, and the metallurgist has a fair idea of what might be going on. Phase transformations (structural phase transformations such as alpha to gamma iron, not precipitation), texture and grain size effects should always be considered first.

## 1.7 References

### Textbook on ultrasonics and laser-ultrasonics:

B. A. Auld, Acoustic Fields and Waves in Solids, Second ed. (Krieger Publishing Company, Malabar, 1990).

D. Royer and E. Dieulesaint, Elastic Waves in Solids, Translated by D. P. Morgan (Springer-Verlag, Berlin, 2000).

C. B. Scruby and L. E. Drain, *Laser Ultrasonics* (Adam Hilger, Bristol, 1990)

### Diffraction

J.-D. Aussel and J.-P. Monchalin. "Measurement of Ultrasound Attenuation by Laser Ultrasonics" Journal of Applied Physics, Vol. 65, No. 8, 15 April 1989, p. 2918-2922.

G. S. Kino. "Acoustic waves, devices, imaging, & analog signal processing" (Englewood Cliffs NJ, Prentice-Hall, 1987) Chapter 3.

### Textbooks on Internal friction:

A. S. Nowick and B. S. Berry, Anelastic relaxation in crystalline solids, 1st ed. (Academic Press, New York, 1972).

Mechanical Spectroscopy  $Q^{-1}$  2001. Editors: R. Schaller, G. Fantozzi and G. Gremaud. (Trans Tech Publications, Zuerich, 2001).

## 2 Introduction to LUMet software

### 2.1 Comparing 2 echoes – The basic LUMet paradigm

As discussed in Section 1.2, the exact ultrasonic generation depends on the material properties, on temperature, and on laser pulse energy and duration. In addition, the gain of the detection laser interferometer is proportional to the amount of collected light, and this depends on surface condition and sample geometry. Therefore, the detected ultrasonic pulse shape and amplitude depends on many experimental conditions.

These conditions are far too complex to be modeled for anything but the simplest materials. Even then, many assumptions must be made. However, once a pulse is generated, it travels back and forth in the sample thickness and is observed as multiple echoes. Moreover, the detection conditions are unlikely to change within the few microseconds during which time the ultrasound pulse can be observed. Therefore, the pulse can be compared with itself, that is two different echoes can be compared to each other. This allows for much more accurate delay and amplitude measurements than if one attempted using a single echo (Section 1.3.1). **Therefore, except in special circumstances, all measurements should be made by comparing two echoes.** This is the basic LUMet paradigm. And the LUMet software is designed to compare two echoes.

As a LUMet convention, the two echoes are called the Current echo and the Reference echo. The Current echo is selected in the Current waveform and the Reference echo is selected in the Reference waveform. The Current and Reference echoes must be different. They can be:

- 1) Two different echoes (successive or not, but with different echo numbers) within a single waveform, or
- 2) Echoes with the same echo number but from two different waveforms. The echoes should be obtained in similar experimental conditions, on the same sample or on two similar samples, and preferably at the same temperature.

In general, it does not make sense to deviate from either of these two conditions. This is why the LUMet software has the following restrictions:

- 1) When comparing two echoes from the Same waveform, the Reference echo is assumed to be an earlier echo of the same waveform, and the Echo # of the Current waveform must be greater than the Echo # of the Reference waveform.
- 2) When comparing two echoes from two different waveforms, the Reference echo # is set to the same value as the Current echo #.

If the two echoes are from the Same waveform and therefore have different numbers, it is assumed that the two echoes are from the same ultrasound pulse, and that the pulse has travelled different path lengths in the sample. The difference in path length is used to calculate velocity from the time delay between the two echoes, and to calculate attenuation. If the two

echoes are from different waveforms and therefore have the same number, it is assumed that the two echoes have propagated the same path length in the sample.

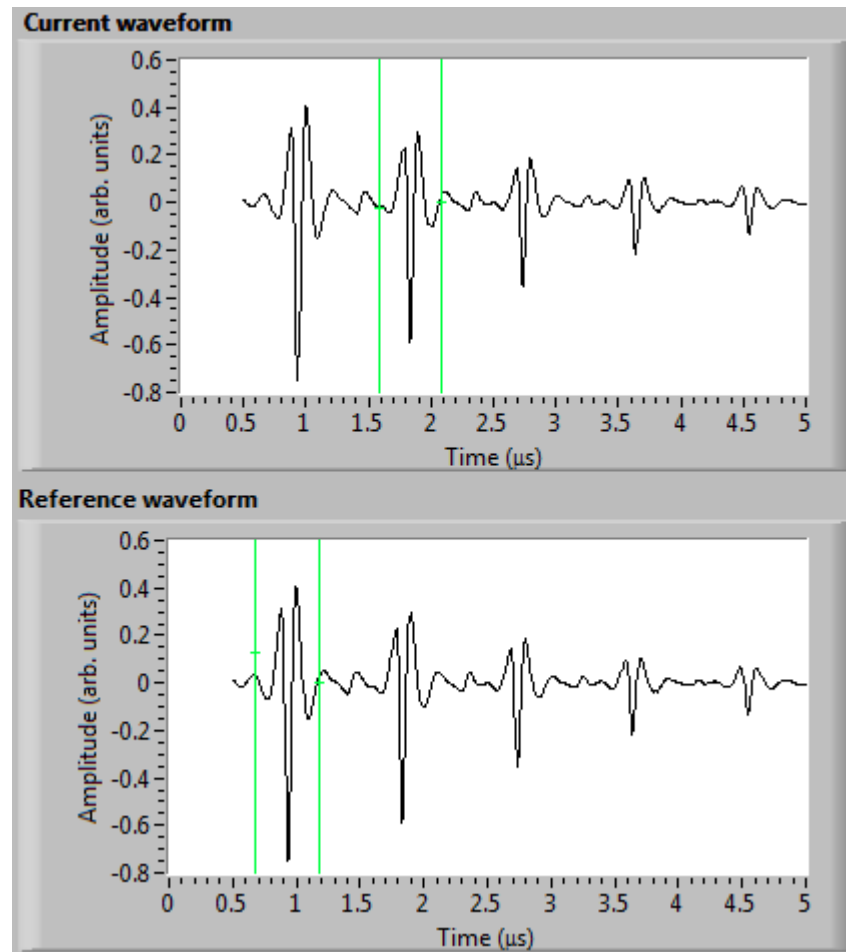


Figure 2.1 Current and Reference waveforms. Here they are chosen to be the Same waveform. The Current echo is the second echo of the Current waveform and the Reference echo is the first echo of the Reference waveform. The selected echoes are surrounded by a pair of vertical cursors.

## 2.2 Overall software architecture.

The LUMet software provides a general framework to analyze ultrasonic data. The analysis always follows the following sequence:

- 1) Display waveform(s).
- 2) Estimate and remove noise.
- 3) Select two echoes.
- 4) Compare the two echoes to estimate the time delay between them, and to estimate their amplitude ratio as a function of frequency.

- 5) Use the obtained time delays and amplitude ratios, together with thickness information to estimate ultrasound velocity and attenuation.
- 6) Use the ultrasound velocity and attenuation to calculate other materials properties.
- 7) Display, plot, and save the results of the analysis.
- 8) Optionally select other waveforms. Return to Step 1

These data analysis steps are detailed in Sections 4 and 5. Section 3, which follows, is a reference section detailing the functionality of each control and indicator of the LUMet user interface.

### 3 User Interface

This Chapter is intended as a reference area to document every control and indicator and how they operate. It can also be used as a "How-to" guide. Detailed information on the methods used is delayed to the next Chapter.

#### 3.1 Conventions:

The first letter of the name of a control or indicator is capitalized, on the LUMet user interface, and in this LUMet documentation.

Controls: various buttons or boxes containing numbers or information used to set various parameters for data analysis. All controls except Tab Controls have a white background.

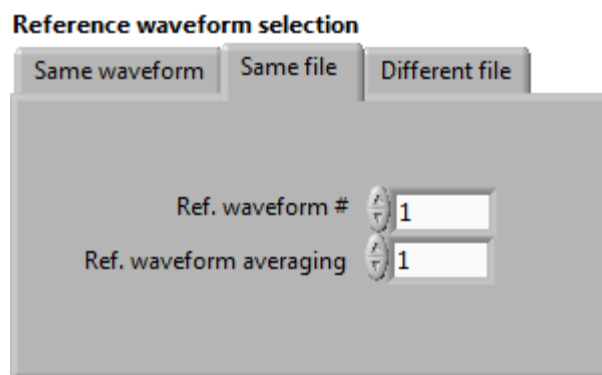
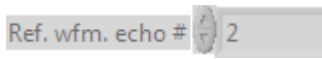


Figure        Greyed Tab control with 3 tabs ("Same waveform", "Same file", and "Different file"). The Same file tab contains two controls ("Ref waveform #" and "Ref. waveform averaging")

Indicators: Various ASCII or number boxes and graphics. They display the value of parameters related to the data or the data analysis. Non-graphics indicators have a grey background. Graphics have a white background to improve the looks when screen captures are pasted into reports. Values displayed by indicators cannot be changed by the user but the LUMet software will change them if the user modifies the analysis.

Greyed controls indicate that the control is disabled. This happens when the control is ignored. For example, the control Ref. wfm. echo # is ignored when the Reference waveform is different from the Current waveform because, in this case, the Reference waveform echo # must be the same as the Current wfm. echo # (Section 2.1) and the LUMet software sets it automatically.

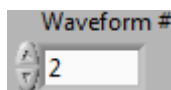
Example of a greyed and disabled control: 

Definitions: When a control or indicator or a term is defined in the text, it is underlined. This allows for quick scanning of this manual when searching for a specific functionality or definition.

## 3.2 General Features

### *Control buttons*

have a pair of little arrows on the left. These can be used to increment or decrement the control value. Alternatively, any valid value may be entered in the control. If a value cannot be entered in a control, then it is not allowed because the LUMet software believes it would cause some error. For example, Waveform # cannot be negative or zero.



### *XY Plots (Graphs)*


can be modified in different ways. Right clicking on the axes brings a context menu that contains various options.

Right clicking on the graph and selecting "AutoScale X" or "AutoScale Y" toggles between setting the X and Y scales automatically (AutoScale) or manually. When not in autoscale, the scale is simply adjusted by selecting and typing new values of the minimum and maximum values on the scales themselves, next to the X and Y axes. Major intervals can also be adjusted to some extent by selecting one of them and typing a new value.

Right clicking on the graph and selecting "Copy Data" puts a copy (image) of the XY plot on the clipboard. The plot can then be pasted into other software such as Word or Power Point.

Right clicking on the graph and selecting "Export" allows exporting the data contained in the plot to the Clipboard or to an Excel spreadsheet. The plot can then be remade using Excel or publication quality software.

When right clicking and selecting "Visible Items", only the "Graph Palette" option is supported.


This option displays (or hides) a set of 3 tools: . When the graph is not in Auto-scale, the second tool allows for easy zooming of a portion of a graph while the third tool allows for easy translations. The first tool is not used.

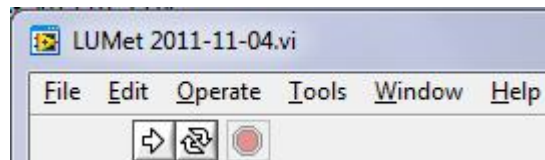
## 3.3 Starting and Stopping the LUMet software

When the LUMet software is launched (from the Windows Start menu or otherwise), it automatically prompts to user to open two files. The first file must be a file with an extension ".ascan". This file contains the LUMet ultrasonic waveforms related. The second file must be a file with an extension ".dtXX" where XX is a two digit number. This file contains the time and temperature information corresponding to the \*.ascan file. The LUMet software verifies that the two files are compatible by checking, among other things, that they contain the same number of measurements. If they are incompatible, a warning is displayed. The software may or may not continue working as if there were no errors, depending on the content of the \*.dtXX file. IF YOU



IGNORE THIS WARNING, YOUR ANALYSIS MAY BE MEANINGLESS. The reason the software does not stop completely is that, sometimes, looking at the waveforms without proper time and temperature information may be a useful debugging tool.

To stop the execution of the LUMet software, simply press the Stop button . To restart, press the right arrow in the third horizontal heading bar. The software will ask again for two new files. Starting and stopping the software is the only way to change files.






The top of the LUMet window contains 3 horizontal bars.

Top bar: This is a Windows information and menu bar containing the LUMet program name, hide button, maximize button, and close button.

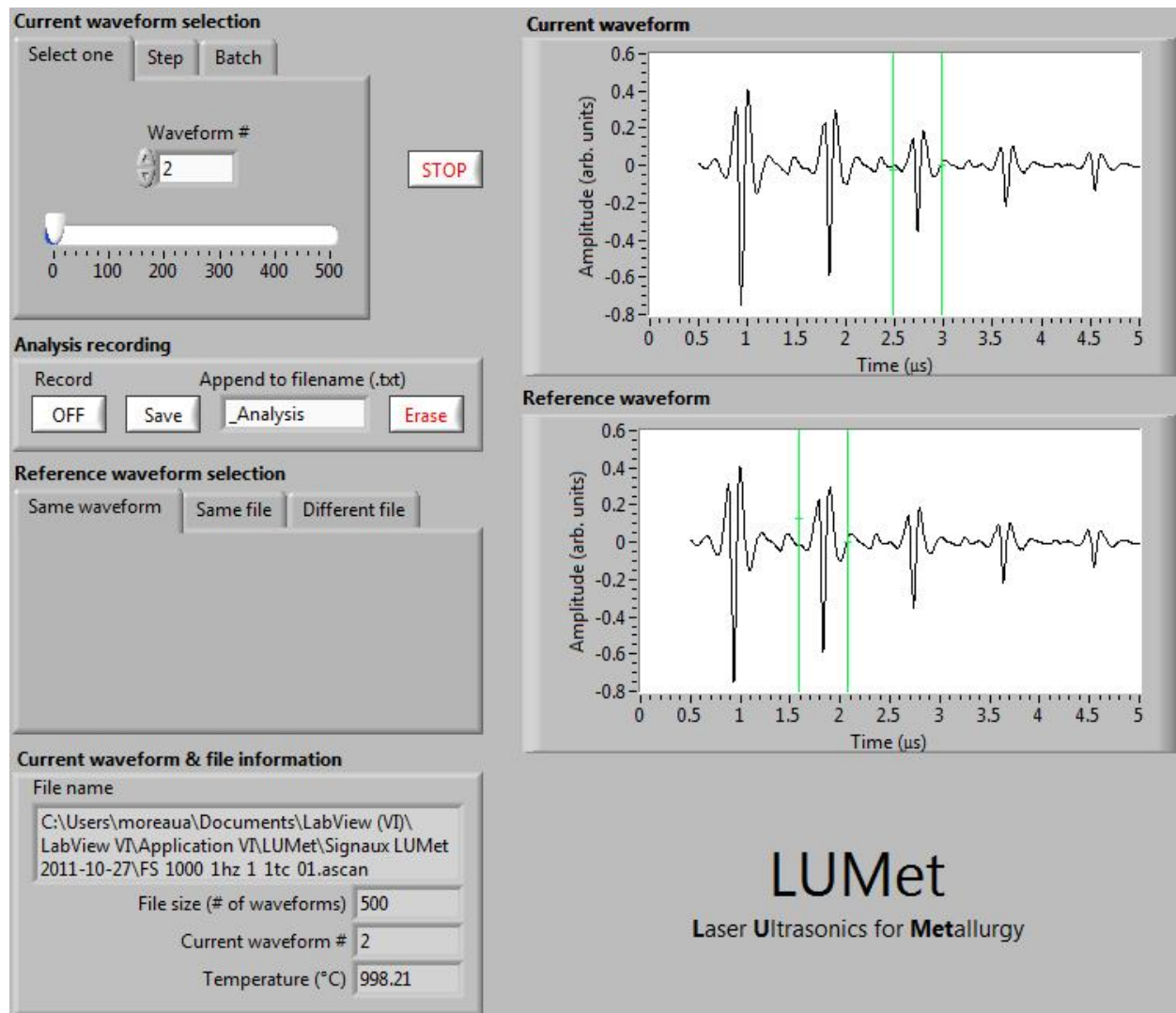
Middle bar: The LUMet software is written in the LabView programming language. The middle bar is a standard LabView menu bar. It contains functionality implemented by LabView and not documented in this manual. This middle bar is not required to use the LUMet software.

However, it contains two useful features: Print Window in the File menus and Reinitialize Values to Default in the Edit menu. Print Window is useful for documenting the parameters used in the analysis.

Bottom bar: This bar contains on 3 icons. The Right arrow  is used to restart the LUMet software after the Stop button has been pressed. The Double arrow  automatically restarts the LUMet software after the Stop button has been pressed. When the double arrow is active, the Stop button becomes equivalent to "Close data files and open new ones". To Stop the program when the double arrow is pressed, one must press the double arrow a second time to de-activate the feature, and then press Stop. The Abort button  aborts the program in an uncontrolled manner. It should only be used as a last resort should the program not stop when the Stop button is pressed (and the double arrow is not activated). As of this writing, it has never been necessary to use the Abort button. The Abort button is not active when the program is not running.

### 3.4 Front panel areas

The user interface is made of several areas: the Waveform display and navigation area,



the General data analysis area,



the Application-specific data analysis area

**Analysis**

Delay   Attenuation   Grain size

What do you want to measure?

Velocity at known thickness

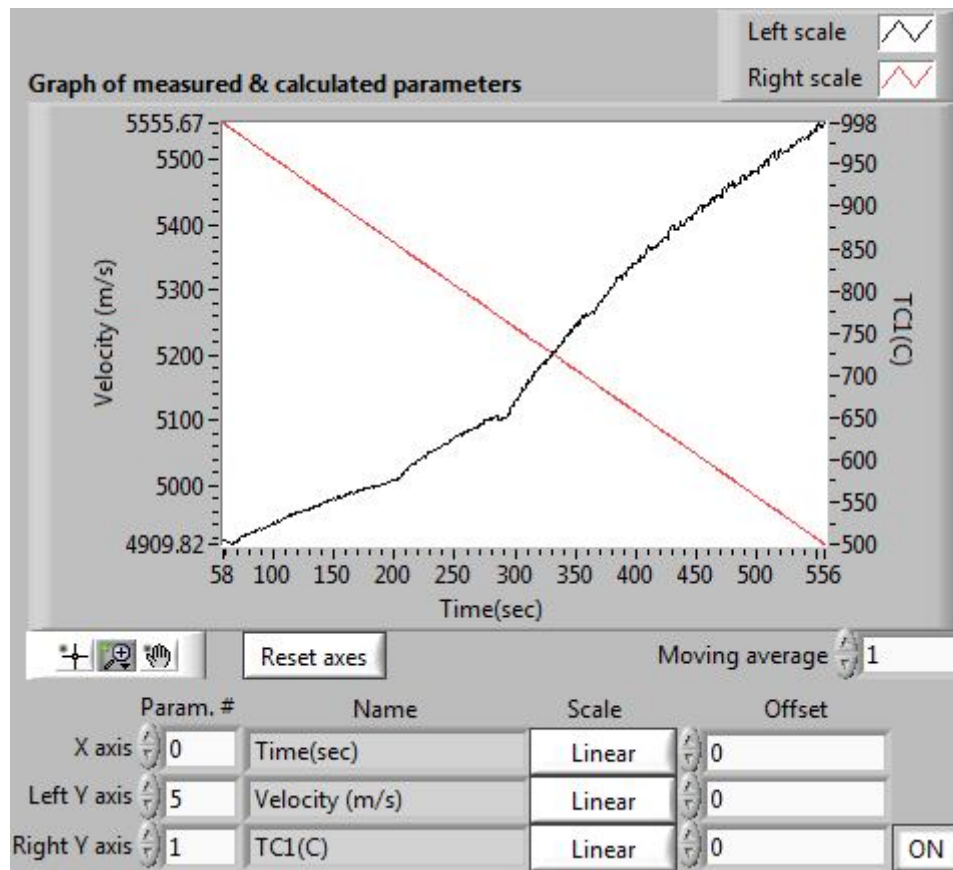
Delay ( $\mu\text{s}$ )   Velocity (m/s)   C33 (GPa)

3.98635   5089.37   195.039

Reference time estimation

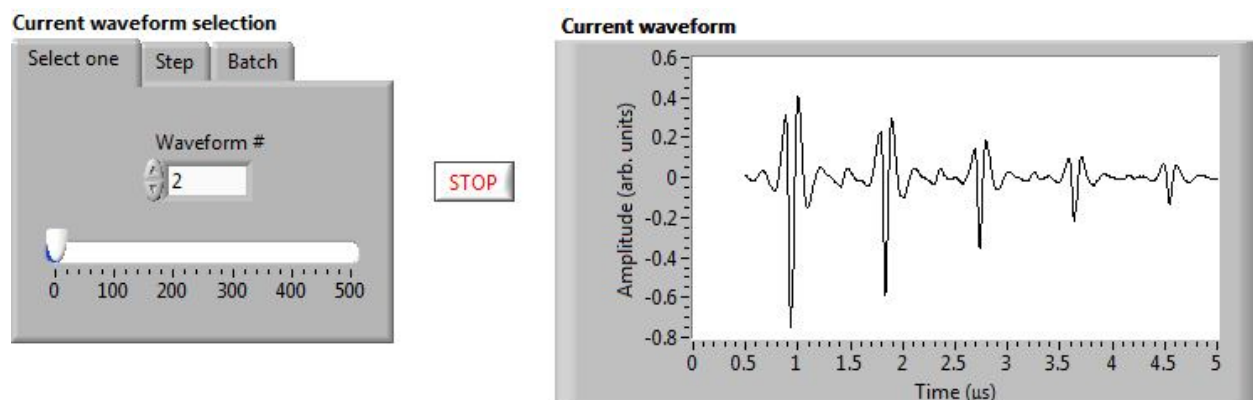
Method	Reference time
Window centering ▼	4.10237
Reference waveform, Second Echo #	Inverted 2nd echo
2	No

and the Result display area,



### 3.5 Waveform Display and Navigation area

The top part of the Waveform display and navigation area,



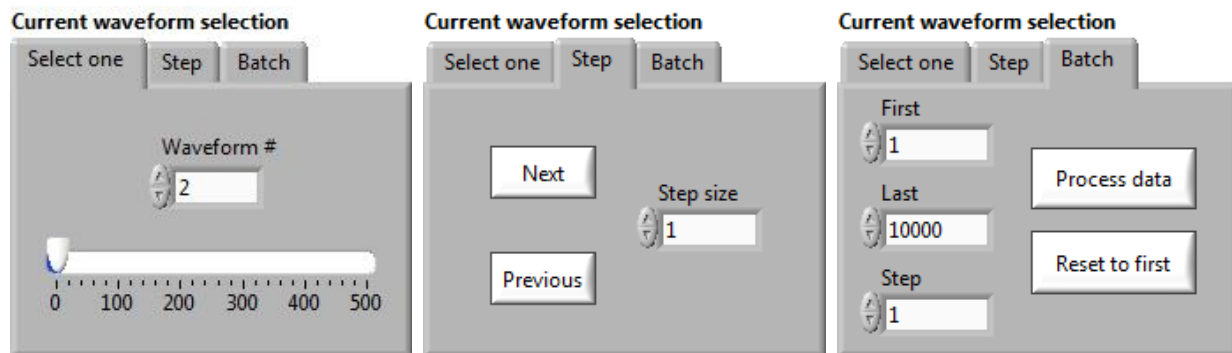
shows, on the right, a graph of the Current waveform, and on the left, a Current waveform selection control. In-between, the Stop button ends the program.

Current waveform selection control: This tab control is used to select the Current waveform. It contains 3 tabs:

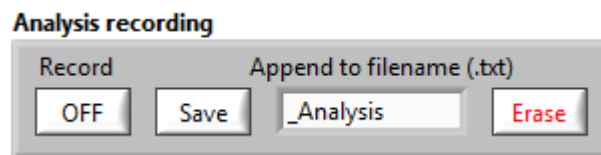
**Select one:** When this tab is selected, the current waveform is set to the value contained in the control Waveform #. A number may be entered or the slider bar may be used to scan the file quickly. These two controls are redundant and one can use either one. The limits of that control are set to the first and last waveform of the file.

**Step:** This tab provides two convenient large buttons (Next and Previous) to step through the file either one waveform at a time, or in steps of several waveforms as determined by Step size.

**Batch:** This tab allows for Batch processing, i.e. it allows stepping automatically through the file from the First to Last waveform # entered. The control Step is used to skip a number of waveforms. For example, a Step of 1 indicates that every waveform is to be analyzed, while a Step of 10 indicates that every tenth waveform is to be analyzed. The control Process Data starts and stops the automatic processing. The control "Reset to first" sets the Waveform # to First.



Below the Current waveform selection control panel there is an Analysis recording panel. This panel contains the controls Record, Save, Erase and Append to filename. These controls are used as follows:



**Record:** When pressed, the results of the analysis are recorded into temporary electronic memory. Every time the waveform number changes, if the record button is ON, the LUMet software creates a new entry containing the last analysis for the waveform being displayed. If several analyses are made on the same waveform by changing various parameters, only the last analysis is retained. All parameters that can be displayed in the Result display area are recorded, as well as one spectrum selected by the Y-scale control found in the Attenuation tab of the Analysis tab control.

**Save:** When pressed, the information recorded in the temporary electronic memory is saved in a file onto disk. The file name is based on the file name of the Current waveform (minus the extension) to which is appended the character string contained in the control Append to filename. The file name extension is set to ".txt". The data is saved onto disk in a tab delimited

ASCII file (text file) that can be read with spreadsheet software. For example, if the Current waveform is from the file named "MySteelTest at 1000C.ascan", the Append to filename control is set to "\_Analysis", then the analysis will be saved to disk in the same directory as "MySteelTest at 1000C.ascan" under the file name "MySteelTest at 1000C\_Analysis.txt".

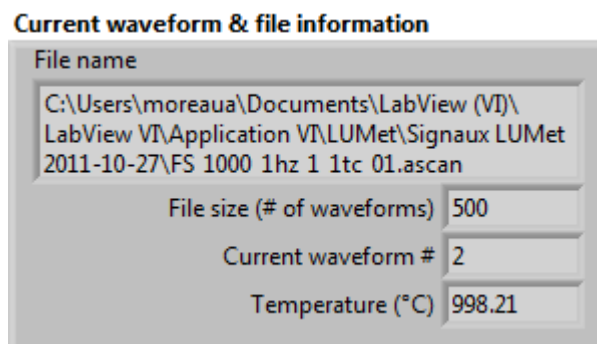
**Beware:** The Save button will save the recorded spectral information only if the Attenuation tab of the Analysis tab control is selected. This is because the spectral information takes more space on disk and is only used by ultrasonics specialists. More details are provided in Section 3.9.

Erase: When pressed, the information contained in the temporary electronic memory is erased. It does not erase any information saved on disk.

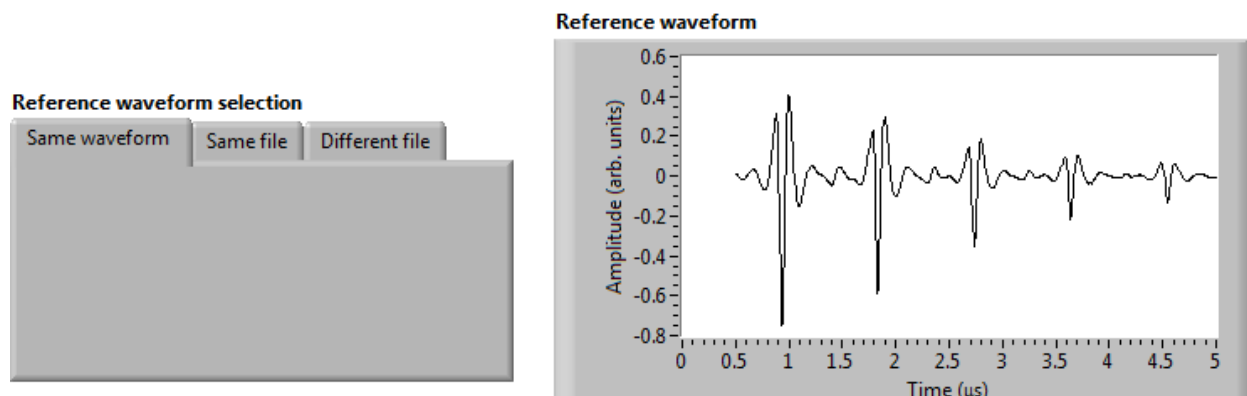
Practical tip:

The Current waveform selection and the Analysis recording panels are made to be used together to allow easy batch analysis and recording. To analyze a file, adjust the various analysis parameters on a few waveforms selected using the tabs "Select one" or "Step" of the Current waveform selection control panel. Then select the tab "Batch", set the First and Last waveform numbers. Press Reset to first to set the Current waveform # to First. Press Record (on the Analysis recording panel). Press Process Data and watch the analysis being made. Once the analysis is completed, press Record again to stop recording and press Save to save the results onto disk. Press Erase before beginning a new analysis. The results of the analysis are shown as the calculations are being made in the Graph of measured & calculated parameters (Section 3.8).

Current waveform & file information panel: Contains information about the file and Current waveform being analyzed.



The middle part of the Waveform Display and Navigation area contains, on the right, a graph of the Reference waveform and contains, on the left, the Reference waveform selection control panel with 3 tabs.



Same waveform: When selected, this tab sets the Reference waveform to the same waveform as the Current waveform. This tab contains no controls. Therefore, the Reference and Current waveforms being displayed are identical.

Same file: When selected, this tab allows setting the Reference waveform to any waveform in the same file as the file containing the Current waveform. The Reference waveform can be an average of any number of waveforms starting with the waveform Ref. Waveform # (see Section 4.3.3). The control Ref. waveform averaging sets the number of waveforms to be averaged together. If no averaging is desired, then Ref. waveform averaging must be set to 1.

Different file: When selected, this allows setting the Reference waveform to any waveform in a file different from the file containing the Current waveform. The Reference waveform can be an average of any number of waveforms starting with the waveform Ref. Waveform # (see Section 4.3.3). The control Ref. waveform averaging sets the number of waveforms to be averaged together. If no averaging is desired, then Ref. waveform averaging must be set to 1. When this tab is selected for the first time, the LUMet software automatically asks which file should be open. Later, to select a different file, press the control button New file. The name and size of the reference file are displayed at the bottom of the tab.



**Reference waveform selection**

Same waveform Same file Different file

Ref. waveform # 1

Ref. waveform averaging 1

**Reference waveform selection**

Same waveform Same file Different file

Ref. waveform # 2 1

Ref. waveform averaging 2 1

Ref. file size (# of waveforms) 0

New file

### 3.6 General Data Analysis area

The General data analysis area contains the Sample and Material properties panel. The LUMet software requires some information about the sample and its material properties to provide accurate results. In addition, the information provided is used to locate the echoes contained in the waveforms. This information is provided by the Sample and Material properties panel.

**Sample and Material properties**

**Sample properties**

Material Fe

Thickness (mm) 2.21

Auto velocity OFF

**Material properties**

Temperature (°C) 998.21

Density (kg/m3) 7523.92

Thermal exp. coeff. (x 10-6 /°C) 15

Velocity (m/s) 5007.79

dV/dT (m/s°C) -1

**Sample and Material properties**

**Sample properties**

Material User defined

Thickness (mm) 2.21

Auto velocity OFF

**User-defined material properties**

Reference temperature (°C) 20

Density (kg/m3) 7860

Thermal exp. coeff. (x 10-6 /°C) 0  
(Set to 0 for apparent vel. or att.)

Velocity (m/s) 5986

dV/dT (m/s°C) -1



### Sample properties panel:

Material: This control tells the LUMet software what is the approximate composition of the sample (such as iron (Fe), aluminium (Al), or User defined material). It also controls which of the Material properties indicator panel or User-defined material properties control panel is displayed.

The choice of a specific material is often not critical. A few choices are provided and the user may select "User defined" to enter the material properties of his sample. If the material's approximate composition is one of the pre-programmed compositions, the Material properties indicator panel is displayed and the properties are those of the selected composition. If User defined is selected, the User-defined material properties control panel is displayed.

Thickness: The exact value of sample thickness must always be entered otherwise the LUMet software may be unstable or give erroneous results. In most cases thickness should be measured at room temperature. Typically, the accuracy of velocity and elastic stiffness constants is limited by the accuracy of the thickness measurement. Therefore, a room temperature measurement of thickness accurate to 1% or better is required usually.

Auto Velocity: When this button is On, the LUMet software estimates automatically and replaces the Approximate velocity displayed in the Material properties panel or in the User-defined material properties panel. The algorithm used works well if the waveform contains multiple echoes, otherwise it may fail.

### Material properties panel:

Temperature (°C): Temperature recorded with the Current waveform. This temperature is used to calculate the other properties contained in the Material properties panel.

Density: Calculated density from a reference value, from the indicated Temperature, and from the indicated Thermal exp. Coeff. This value is used in the calculation of elastic constants. An error of 1% in the entered value will result in an error 1% in the estimate of the elastic constants.

Thermal exp. Coeff. ( $\times 10^{-6} / ^\circ\text{C}$ ): Linear thermal expansion coefficient. This value is used to estimate the temperature dependence of thickness and density. It affects also the measured sound velocity and elastic constants because these values depend on thickness and density. This constant can be set to zero or to the true value for the material. If it is set to zero, the LUMet software will calculate "apparent" velocities and elastic constants, as opposed to the true values, corrected for thermal expansion. Apparent values are adequate for many applications. The LUMet software only considers thermal expansions that are linear in temperature. To account for non-linearities or phase transformations with volume changes, one should set the Thermal expansion coefficient to zero and make the correction later on the output data files saved to disk.

Velocity: Approximate velocity of the ultrasound pulse. If Auto velocity is set to On, then Velocity is the velocity estimated by the automatic algorithm. If Auto velocity is set to Off, then Velocity is the velocity at the indicated Temperature estimated using some reference velocity and the velocity change with temperature  $dV/dT$ . This value is only used to locate the ultrasonic echoes. The exact sound velocity is calculated from the ultrasonic data. As long as the echoes are correctly identified, the precise value of Velocity is irrelevant.

DV/dT (m/s°C): The variation in ultrasound velocity in m/s per degree Celsius. This value is used to make a temperature correction on the above approximate Velocity. This helps better locate the echoes when velocity changes with temperatures. Usually, this value is not known *a priori*. It may be sensitive to the composition of alloys and can be estimated during the data analysis.

User-defined material properties panel:

Reference temperature (°C): Temperature at which the other values of the User-defined material properties are defined. Note that this differs from the Temperature indicator of the (non user-defined) Material properties.

Density: Density at Reference Temperature. This value is used in the calculation of elastic constants. An error of 1% in the entered value will result in an error 1% in the estimate of the elastic constants.

Thermal exp. Coeff. ( $\times 10^{-6}$  / °C): Linear thermal expansion coefficient at Reference temperature. This value is used to estimate the temperature dependence of thickness and density. It affects also the measured sound velocity and elastic constants because these values depend on thickness and density. This constant can be set to zero or to the true value for the material. If it is set to zero, the LUMet software will calculate "apparent" velocities and elastic constants, as opposed to the true values, corrected for thermal expansion. Apparent values are adequate for many applications. The LUMet software only considers thermal expansions that are linear in temperature. To account for non-linearities or phase transformations with volume changes, one should set the Thermal expansion coefficient to zero and make the correction later on the output data files saved to disk.

Velocity: If Auto velocity is set to On, then Velocity is the velocity estimated by the automatic algorithm. If Auto velocity is set to Off, then Velocity is the ultrasound velocity at Reference temperature. This value is only used to locate the ultrasonic echoes. The exact sound velocity is calculated from the ultrasonic data. As long as the echoes are correctly identified, the precise value of Velocity is irrelevant.

DV/dT (m/s°C): The variation in ultrasound velocity in m/s per degree Celsius. This value is used to make a temperature correction on the above Velocity when Auto velocity is Off. This helps better locate the echoes when velocity changes with temperatures. Usually, this value is

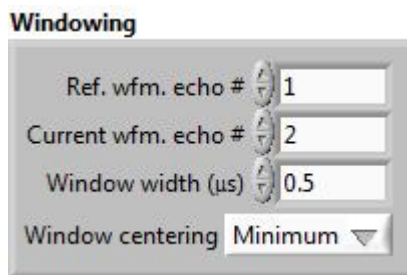
not known *a priori*. It may be sensitive to the composition of alloys and can be estimated during the data analysis.

Practical tip:

Adequate User-defined values of Temperature, Velocity, and  $dV/dT$  may be obtained by using the Auto-Velocity, or an "eye-ball" estimate at 20 °C and at some other convenient temperature. An eye-ball estimate can be made by dividing twice the thickness by the approximate time delay between two echoes.  $dV/dT$  is simply the slope of the variation between the two measurements at the two temperatures. For example, if the eyeball estimate of velocity for some steel is 6000 m/s at 20 °C and 5000 m/s at 1000 °C, then one would set Reference Temperature = 20 °C, Velocity = 6000 m/s, and  $dV/dT = -1$  m/s°C (-1 is close to 1000 m/s divided by 980 °C). Deviations from linearity and inaccuracies of order 10% in  $dV/dT$  are usually of no consequences for the purpose of identifying echoes.

Windowing panel:

This panel is used to select (to window) the acoustic echoes to be analyzed. One echo is selected from the Current waveform and one from the Reference waveform. Two cursors are drawn on the Waveform display to indicate the portion of the waveform that is selected (windowed). The window width can be specified, as well as whether the window is to be centered on the maximum or the minimum of the echo. When comparing echoes from different waveforms, the Reference waveform Echo # is greyed (disabled) and set to the same value as the Current waveform Echo #.



Ref. wfm. Echo #: Echo number of the echo selected in the Reference waveform. This number corresponds to the number time the ultrasound pulse has traveled back and forth through the sample thickness.

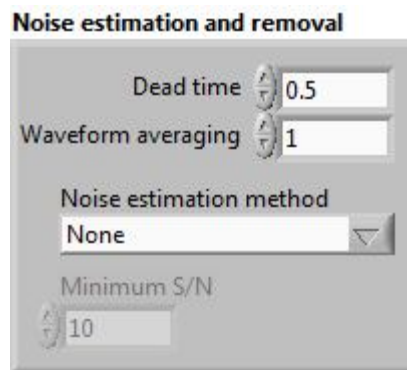
Current. wfm. Echo #: Echo number of the echo selected in the Current waveform. This number corresponds to the number time the ultrasound pulse has traveled back and forth through the sample thickness.

Window width ( $\mu\text{s}$ ): Width of the window in microseconds. The width should be selected such that only one single pulse is contained within the window. The beginning or end of another pressure pulse or of a shear pulse should be excluded from the window.

Window centering: The window should be centered on the largest positive or negative peak, that is on the maximum or minimum of the pulse. Usually, the Reference and Current echoes have the same shape, but sometimes (especially for cylindrical samples) they are inverted. In such cases the "Max / Min" or "Min / Max" options should be selected. These options refer to the "Current / Reference" echoes, respectively.

Noise estimation and removal panel:

This panel contains tools to estimate and remove noise from the data. It is also used to specify how many waveforms should be averaged together to improve the signal-to-noise ratio (S/N).



Dead time: Removes all data up to the time specified. It is useful to eliminate noise that occurs at time zero and shortly thereafter. This noise is unavoidable and is caused by multiple noise sources. More details are provided in Section 4.3.1.

Waveform averaging: The waveform being analyzed and is averaged with the following waveforms of the file. The total number of waveforms to be averaged is selected by the "Waveform averaging" control for the Current waveform, and by Ref. waveform averaging for the Reference waveform. Temperatures measurements made at the same time are averaged also. This control controls only the averaging of the Current waveform. If the Reference waveform is the same as the Current waveform, then the Reference waveform is also the same averaged waveform. However, if the Reference waveform is a different waveform obtained from the same or a different file, then averaging of the Reference waveform is controlled within the Same file or Different file tabs of the Reference waveform selection tab control. More details are provided in Section 4.3.3,

Noise estimation method: Used to estimate the noise content of the ultrasonic data. More details are provided in Section 4.3.4.

**Minimum S/N:** Minimum signal-to-noise ratio for valid ultrasonic data. A noise estimation method other than "None" must be selected otherwise this control has no effect on the analysis. More details are provided in Section 4.3.4.

### 3.7 Specific data analysis area

The Specific data analysis, or simply Analysis area contains three tabs: Delay, Attenuation, and Grain Size used for different types of data analyses and different applications to metallurgy.

**Analysis**

Delay Attenuation Grain size

What do you want to measure?

Velocity at known thickness

Delay (µs) Thickness (mm) C33 (GPa)

3.98635 5089.37 195.039

Reference time estimation

Method Reference time

Window centering 4.10237

Reference waveform, Second Echo # Inverted 2nd echo

2 No

#### Delay tab:

The analysis corresponding to this tab is always executed, even when this tab is hidden, and the results are used by the other tabs if they are selected. This tab is used to estimate ultrasonic propagation delay, ultrasound velocity, sample thickness and the elastic modulus derived from the velocity measurement.

**What do you want to measure?:** Velocity is obtained by dividing the distance traveled (a multiple of sample thickness) by the time delay. Therefore, thickness must be known to measure

velocity. On the other hand if the velocity is known, thickness can be measured. This control button selects whether one wishes to measure velocity for a sample of known thickness, or thickness for a sample of known ultrasound velocity. The option to measure velocity is useful when no mechanical deformation is applied, while the option to measure thickness is useful for monitoring mechanical deformations. The latter option is equivalent to using a strain gauge to measure changes in thickness (as long as Velocity is constant). More details are provided in Sections 4.4 and 4.5.

Delay ( $\mu$ s): Measured delay between the Current echo and the Reference echo.

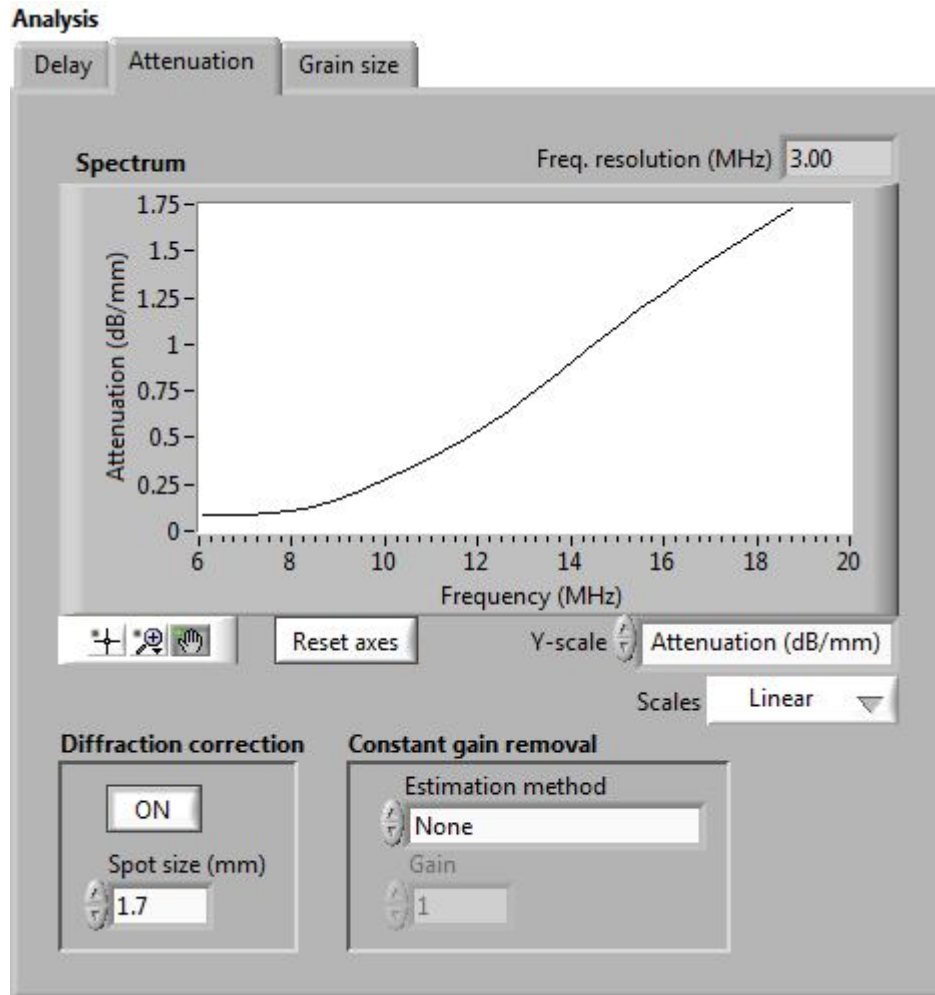
Velocity (m/s) or Thickness (mm): These superimposed indicator (only one is visible at a time when the software is running) indicate the velocity or thickness calculated from the measured Delay and from either the Thickness or Velocity information provided in the Sample and Materials properties panel. More details are provided in Sections 4.4 and 4.5.

C33 (GPa): This indicator is valid only when the control What do you want to measure? is set to Velocity at known thickness. It displays the value of the C33 elastic stiffness constant calculated from Delay, Thickness, Density, and Thermal exp. coeff. More details are provided in Section 4.4.3.

Reference time estimation: This panel is used when the Reference waveform is different from the Current waveform. In this case, the LUMet software forces the Current and Reference echo # to be the same and the distance traveled by the ultrasound pulse is the same (to within a thermal expansion effect) for the two waveforms. The Delay indicator measures the delay between the current and reference echoes. Suppose that the only difference between the Current and Reference waveforms is temperature, then Delay only indicates the relative delay change, not the absolute delay. This panel is used to estimate the absolute value of the Delay. Three methods are available (Window centering, Automatic, and Second Echo). The Reference time indicator indicates the estimated absolute arrival time of the Reference echo. More details are provided in Section 4.4.2.

Attenuation tab:

This tab is used to calculate Amplitude spectra of single echoes, ratios of two amplitude spectra, and attenuation spectra. These spectra are displayed in an XY graph. This tab also contains controls to perform a simple diffraction correction and controls to remove the effect of a constant gain from the amplitude ratio spectra and attenuation spectra. For Amplitude spectra, the "Constant gain removal" control is de-activated and greyed. Detailed information regarding this tab can be found in Sections 4.6 and 4.7.



**Spectrum:** XY plot of the calculated Amplitude, Amplitude ratio, or Attenuation spectrum.

**Y-scale:** Control menu used to select which spectrum is calculated and displayed.

**Scales:** Control used to select whether the Spectrum plot is scaled on a Linear, Semi-log, or Log-log scale.

**Reset axes:** When pressed, this buttons adjusts the scales as if the axes of the plot were in AutoScale mode (Section 3.2), but immediately reverts to non AutoScale.

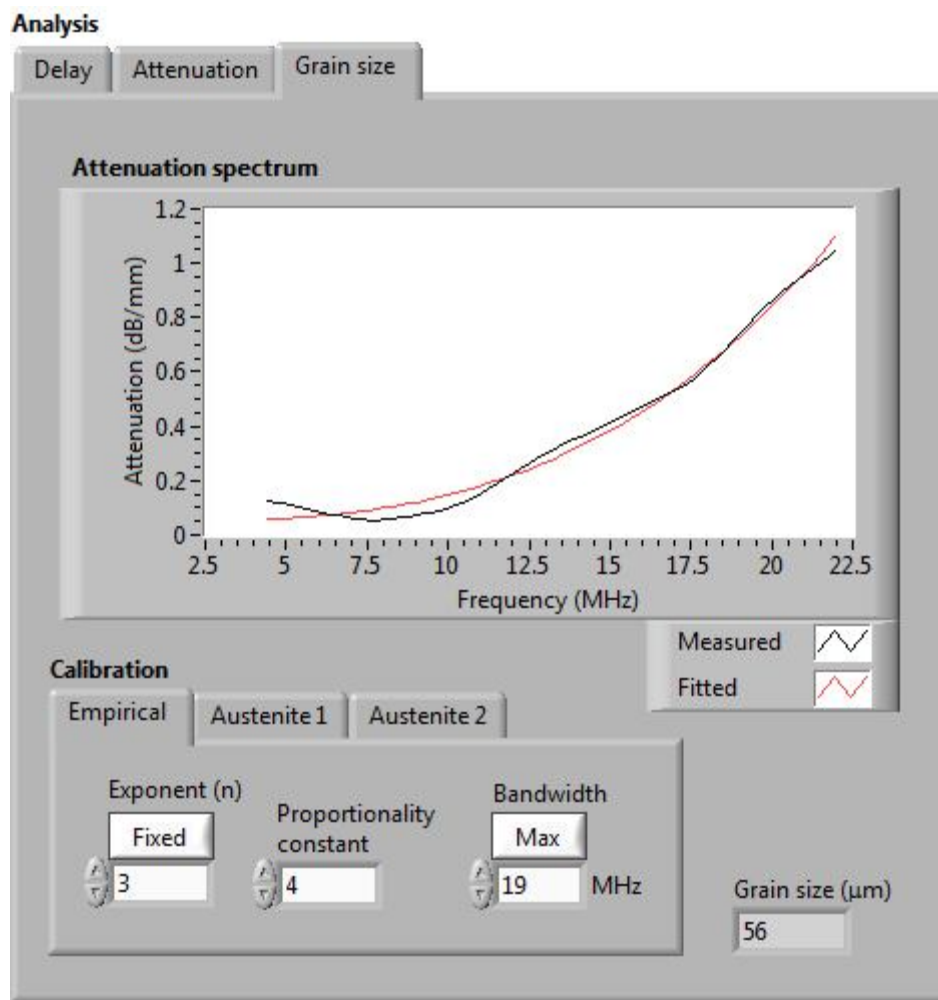
**Freq. resolution (MHz):** Frequency resolution of the displayed Spectrum. It is defined as the -3 dB bandwidth of the spectrum that would be calculated should the waveform be a perfect single frequency. In practice two different spectral features will be observable only if they are separated in frequency by at least the value of Freq. Resolution. Frequency resolution improves when wider windows are used (See Windowing panel).

**Diffraction correction:** This panel is used to apply a simple diffraction correction to the spectra. More details are provided in Section 4.7.

**Constant gain removal:** This panel is used to remove a frequency-independent gain from the spectra. More details are provided in Section 4.6.

**Grain Size tab:**

This tab is used to estimate grain size based on the attenuation spectra calculated in the Attenuation tab. Therefore, it is important to properly adjust controls of the Attenuation tab before using the Grain Size tab. Details about grain size measurements are described in Section 5.1.



**Attenuation spectrum:** The black curve displays the same attenuation spectrum as that calculated in the Attenuation tab. In some cases, the measured attenuation spectrum is fitted to a model. In such cases, the fitted model is displayed by a red curve. A legend is provided at the bottom right of the graph as a reminder.

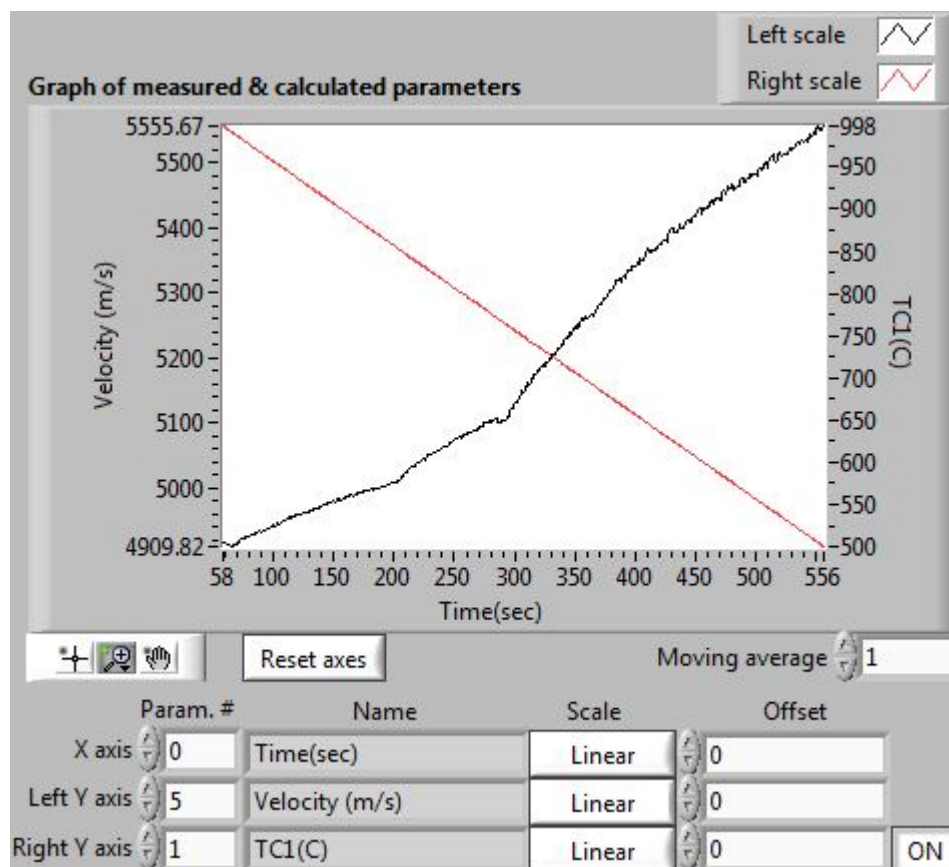
**Calibration:** Different calibrations are provided to estimate grain size from the attenuation spectrum. More details are provided in Section 5.1



Grain size ( $\mu\text{m}$ ): This indicator indicates the estimated grain size for the Current waveform.

### 3.8 Result display area

The Result Display area, is a convenient means to display the calculated results during the data analysis. It is meant to allow rapid visualization of the data to quickly and easily detect whether the analysis makes sense or whether there are problems to be addressed. It is not a publication-quality tool. For high quality graphics, the calculated data should be saved to file using the procedure described in Section 3.2 and plotting software should be used.



When, in the Analysis recording panel, Record is ON, the results of the analysis are stored in temporary electronic memory. These results are plotted in the Result display area. For each analysis of the ultrasonic waveforms, the following parameters are recorded: Time, temperature (displayed as TC1, TC2, TC3 or TC4 depending on which thermocouple is used) and waveform number of the recorded Current waveform. The following parameters estimated from the ultrasonic data are also recorded displayed: Delay, amplitude, velocity (or thickness), C33. In addition, other parameters such as grain size may be recorded and displayed. All parameters may be plotted vs. each other. Note that the amplitude information is qualitative and depends on generation efficiency and detections sensitivity, and is a global estimate for all frequencies. Specifically, the amplitude information is the amplitude of the maximum of the cross-correlation used to estimate the delay.

Graph of measured and calculated parameters: XY plot of any two or three of the parameters stored in temporary electronic memory. One parameter must be assigned to the x axis, and one parameter must be assigned to the y axis. Optionally, a third parameter may be assigned to the second y axis (to the right of the plot). A legend is provided at the top right of the graph to indicate that the black curve corresponds to the left y axis and the red curve correspond to the right y axis.

Reset axes: When pressed, this buttons adjusts the scales as if the axes of the plot were in AutoScale mode (Section 3.2), but immediately reverts to non AutoScale.

Moving average: Performs a simple moving average of all parameters simultaneously before plotting, in the order they have been recorded. . For example, during a temperature ramp, with a moving average of 10, the average temperature of 10 successive waveforms, the average measurement time of the same 10 waveforms, and the average velocity of the same 10 waveforms would be used simultaneously to form a plot velocity and temperature vs. time. When a moving average is made, it only affects the display of the results. When the results are saved using the Save button of the Analysis recording panel, the moving average is not applied to the saved results. This moving average is a display tool, not a data analysis tool.

The bottom section of the Result display area is a table of controls and indicators with three rows and five columns. The first, second and third rows are labeled X axis, Left Y axis, and Right Y axis, respectively. Each control or indicator on a specific row refers to the corresponding axis. The first four columns are labeled Param. #, Name, Scale, and Offset. The Param # column allows selecting which parameter is to be plotted on the corresponding axis. The Name column displays the name of the parameter. The same name is also used in the plot as an axis label. The scale column allows selecting whether the corresponding axis is linear or logarithmic. Finally, the offset column allows subtracting a constant value (an offset) from the displayed parameter. This offset is often used to reset time to zero at a particular point in an experiment. When the results are saved using the Save button of the Analysis recording panel, the offset is not applied to the saved results. This offset is a display tool, not a data analysis tool. The last column only contains one ON/OFF button to turn the On and Off the Right Y axis.

### 3.9 Format of saved files

When the Save button of the Analysis recording panel is pressed, the data store in temporary memory is saved to disk. Two formats are used. The two formats are nearly identical. The following discussion applies to both formats unless specifically noted.

All files are stored as a tab-delimited ASCII file that can be read easily by spreadsheet software. The data is stored as a table where each row corresponds to the analysis of one Current waveform and each column correspond to a different parameter. The order of the rows follows the order of the data analysis. The first row of the table is used to store tab-delimited labels for each column. Each such label corresponds to a parameter plotted in the Result display area, and all parameters that can be displayed are saved.

The Moving average and the Offset applied in the Result display area have no effect on the saved data. These are display tools and do not affect the results that are saved in temporary memory or on disk.

The only deviation from this format occurs when saving Amplitude, Amplitude ratio, or Attenuation spectra. These results are not a single number and do not fit into the above format. Only one of these spectra may be recorded and saved for each waveform that is analyzed. The analysis has to be made a second time to record and save a different spectrum. Spectra are recorded whenever the Record button is On, but they are saved only when the Attenuation tab of Analysis tab control is selected. This is because the spectral information takes more space on disk and is only used by ultrasonics specialists.

When spectra are saved, three more labels are added to the first row: "f0", "Delta f", and the name of the last spectrum analyzed (the name displayed in the Y-scale control of the Attenuation tab). If this control is changed during the analysis, there will be no record of that change. The last value of the Y-scale control will be displayed as a label.

The x value the spectral data is always equally spaced. Therefore, it is only sufficient to record the lowest frequency (f0) and the interval (Delta f). The x axis can be reconstructed from these two data. The y values corresponding to f0 are saved in the column immediately following the Delta f column. The next column contains the y values corresponding to the frequency  $f_0 + \Delta f$ . The next column contains the y values corresponding to the frequency  $f_0 + 2 * \Delta f$ . And so on.

As an example, let's consider the portion of the saved file shown below. The spectral information begins after the C33 column. The lowest frequency is 6.09 MHz (rounding to 2 decimals to lighten the text) and the frequency interval is 0.47 MHz. Amplitude spectra of the Current waveform are saved. The amplitude of the first waveform at 6.09 MHz is 0.576. The amplitude at  $6.09 + 0.47 = 6.56$  MHz is 0.5485. The amplitude at  $6.09 + 2 * 0.47 = 7.03$  MHz is 0.510. The amplitude at  $6.09 + 3 * 0.47 = 7.50$  MHz is 0.4655.

Velocity (m/s)	C33 (GPa)	f0 (MHz)	Delta f (MHz)	Amplitude spectrum, Current waveform			
5239.319663	215.7607	6.09375	0.46875	0.576326	0.54853	0.510364	0.465541
4917.503675	190.0693	6.09375	0.46875	0.359889	0.343892	0.321938	0.296149
4916.395192	189.9836	6.09375	0.46875	0.317836	0.303877	0.284598	0.26188

## 4 Methods and basic calculations

Readers may wish to re-read Section 2.2 before reading this Chapter.

### 4.1 Echo localization

The Sample and Materials properties and Windowing blocks are used to locate echoes on the Current and Reference waveforms. This is done as follows:

- 1) The sound velocity at the temperature of the measurement is estimated using the Velocity,  $dV/dT$ , Thermal expansion coefficient, Reference temperature, and Temperature information.
- 2) The approximate arrival time of the ultrasonic echoes is calculated from the calculated Velocity, Sample thickness, and Echo #.
- 3) Depending on the setting of Window centering, the software looks for either the time at Minimum or Maximum signal amplitude within a time interval centered on the estimated arrival time, and of width equal to Window width.
- 4) The window is then re-centered on the time at Minimum or Maximum signal amplitude.

This algorithm can fail if

- The values of Material properties (Usually Velocity or  $dV/dT$ ) are too far from the real values.
- The sample Thickness is wrong.
- The Window width is very narrow.

It is usually easy to detect a failure of the Echo localization algorithm: The echo one intends to select will not have its minimum or maximum centered between the two cursors. If the algorithm fails, there is no other choice but entering more accurate values of Material properties or thickness. Fortunately, if the Materials properties are unknown, they are easy to estimate from the data and can be entered in the User-defined material properties panel.

**Sample and Material properties**

**Sample properties**

Material

Thickness (mm)

Auto velocity

**Material properties**

Temperature (°C)

Density (kg/m<sup>3</sup>)

Thermal exp. coeff. (x 10<sup>-6</sup> /°C)

Velocity (m/s)

dV/dT (m/s°C)

**Sample and Material properties**

**Sample properties**

Material

Thickness (mm)

Auto velocity

**User-defined material properties**

Reference temperature (°C)

Density (kg/m<sup>3</sup>)

Thermal exp. coeff. (x 10<sup>-6</sup> /°C)  
(Set to 0 for apparent vel. or att.)

Velocity (m/s)

dV/dT (m/s°C)

**Windowing**

Ref. wfm. echo #

Current wfm. echo #

Window width (μs)

Window centering

## 4.2 Materials properties

When selecting a User-defined Material in the Sample properties panel, the properties displayed in the Material properties panel can be modified at will. The Reference temperature,  $T_{Ref}$ , is the temperature at which the properties are defined.

When selecting a Material other than User-defined in the Sample properties panel, the Material properties are defined internally at some Reference temperature. These values are taken from a variety of references. Material properties displayed in the Material properties panel are calculated for the displayed Temperature,  $T$ , which corresponds to the temperature of the laser-ultrasonic measurement of the Current waveform. These values are calculated according to:

$$h(T) = h_{Ref}(1 + \alpha_l(T - T_{Ref}))$$

$$\rho(T) = \frac{\rho_{Ref}}{(1 + \alpha_l(T - T_{Ref}))^3}$$

$$V(T) = V(T_{Ref}) + (T - T_{Ref})dV/dT$$

where  $h$  is thickness,  $\alpha_l$  is the linear coefficient of thermal expansion,  $\rho$  is density, and  $V$  is ultrasound velocity for the longitudinal wave. In all LUMet calculations, whether the Material properties are User-defined or not, the values of  $h$ ,  $\rho$  and  $V$  are taken at the measurement temperature  $T$  according to the above equations. The temperature correction on  $h$ , and  $\rho$  can be over-ridden by using the User-defined material properties and setting  $\alpha_l = 0$  or any other value.

The coefficient of linear thermal expansion is assumed to be constant. The selected constants are shown in the Table below. This allows a first order correction to the calculation of sound velocity and elastic moduli. More accurate corrections must be done manually as described in Section 3.6.

Table            Approximate coefficient of linear thermal expansion in the temperature range of 0-1000 °C.

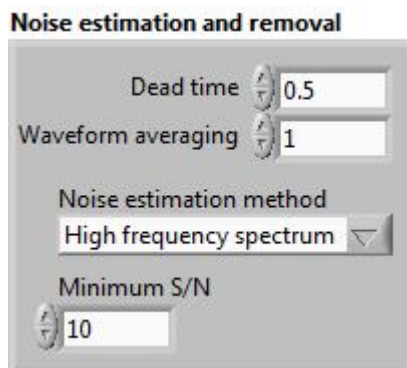
Material	$\alpha_l$ ( $\times 10^{-6}/^{\circ}\text{C}$ )
Fe- $\alpha$	15
Al	25

#### Reference

Xian-Gang Lu, Malin Selleby, Bo Sundman. Assessment of molar volume and thermal expansion for selected bcc, fcc, and hcp metallic elements. Computer Coupling of Phase Diagrams and Thermochemistry **29** (2005) 68–89.

### 4.3 Noise reduction & estimation

The Current waveform and Reference waveform graphs display waveforms acquired with the LUMet system. To improve the signal-to-noise ratio (S/N) of the signal, various numerical treatments are applied, some of which can be controlled by the user in the Noise estimation and removal block shown in the Figure below.



### 4.3.1 Dead time

At time  $t = 0$ , there is what seems to be an ultrasonic signal. However, this signal is caused by the interaction of the detection laser light with the surface plasma and by the thermal expansion of the sample surface. This spurious signal should never be included in any of the windows set around ultrasonic echoes. The easiest way to eliminate that spurious signal is to discard it. This is done by setting the Dead time, i.e. the time before which all signal is discarded, to the desired value, typically from a few tenths of a microsecond up to about  $1\ \mu\text{s}$ . The effect of Dead time is shown in the figure below. An added advantage of properly setting the Dead time is that the horizontal and vertical scale, when set to Autoscale, are magnified to better show the ultrasonic echoes.

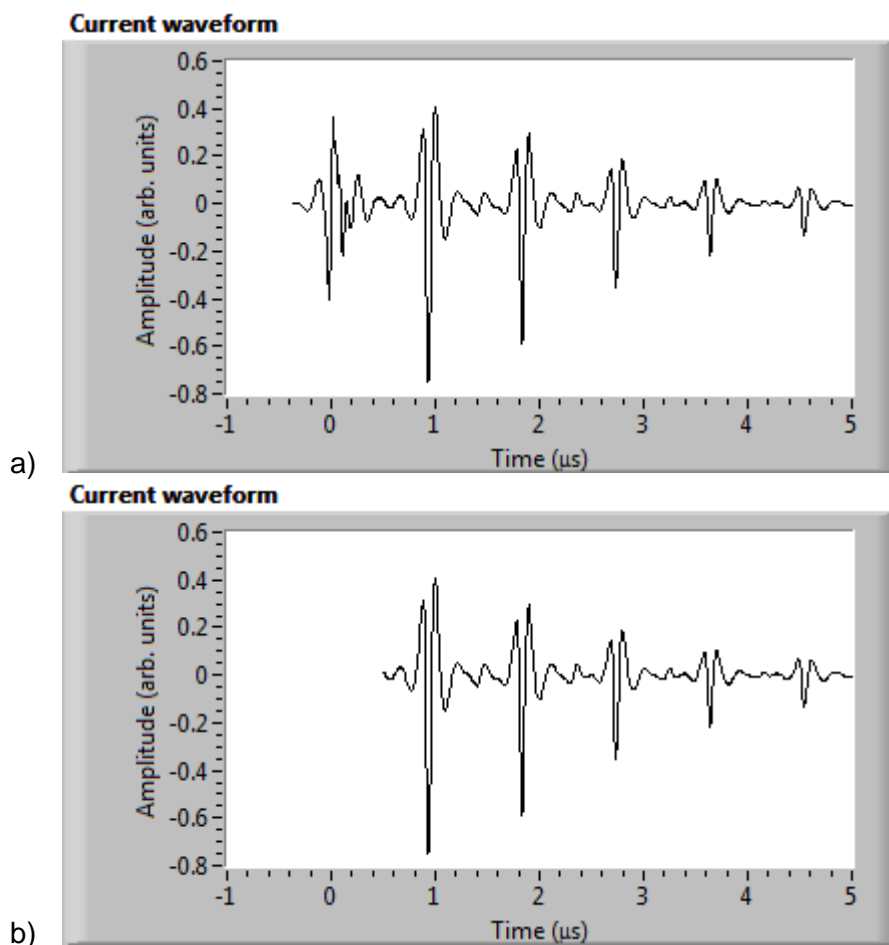


Figure caption: Ultrasonic waveform acquired with the LUMet system: a) raw waveform, b) with an applied Dead time of  $0.5\ \mu\text{s}$ .

### 4.3.2 Filtering

The laser-ultrasonic hardware has built-in low-pass and high-pass filters. In addition, the LUMet software adds its own numerical filter to the data. The filter's cut-off frequency is set automatically depending of the data analysis. The filter is weak in the sense that the user will not notice it most of the time. However, one may notice that the waveform may change its

appearance, especially when the window width is small (0.5 microseconds or less). This is normal and it does not affect adversely the final results.

#### 4.3.3 Waveform averaging

Signal averaging can dramatically improve S/N. Signal averaging can be done either with the ultrasonic waveforms, or with the estimated parameters (delay, velocity, grain size, etc...). Here, we discuss Waveform averaging. The averaging of the final results will be discussed in Section 4.3.5.

Waveform averaging should only be done if the waveforms do not vary (except for the noise component) from shot to shot. The Waveform averaging control determines how many waveforms are averaged together. These waveforms begin at the Waveform number of the Current waveform and include the immediately following waveforms. For example, if the Current waveform # is 100 and the Waveform averaging control is set to 10, the waveforms 100 to 109 are averaged together. Whatever varies between the waveforms, like electronic noise, but also any waveform variation caused by thermo-mechanical processing, is averaged out. If  $n$  waveforms are averaged together, the random noise will be reduced by a factor of  $\sqrt{n}$ , while leaving the signal unchanged. Therefore, the S/N improves by a factor of  $\sqrt{n}$ . When Waveform averaging is set to  $n = 1$ , no averaging is made.

Because Waveform averaging involves measurements made over some time duration (as opposed to the single waveform measurement which can be considered to be nearly instantaneous), the time and temperature parameters that are stored with the waveform are averaged also.

The averaged waveform is then displayed and used for all subsequent calculations.

##### Practical tip:

Because Waveform averaging of  $n$  waveforms, where  $n$  is more than a few waveforms, should only be done when the signal does not change significantly during the  $n$  measurements and, consequently, because the average does not change significantly when the Current waveform # is increased by 1 when  $n \gg 1$ , the Step value in the Batch processing can be set in the range of  $n/4$  to  $n/2$  without affecting the quality of the analysis.

##### Practical tip:

If the Reference waveform is not the Same waveform as the Current waveform, such as when the Reference waveform is the first or last waveform of an experiment, it is a good idea to acquire the Reference waveform many times and to use Waveform averaging on the Reference waveform. The reason for that is that the Reference waveform will be used many times and it should ideally have a noise level less than that of the Current waveform. A Reference waveform averaging value of 10 is often good enough since it decreases the noise amplitude by a factor of more than 3, which is usually enough to make it negligible in the calculations.



#### 4.3.4 Noise estimation method

The LUMet software can estimate and remove the noise level in the signal. It is a general feature of laser-ultrasonics that the amplitude spectrum of an ultrasound pulse decreases with frequency. At high enough a frequency, there is only noise. The LUMet software can estimate the noise level and at what frequency the S/N falls below some value. How is does that is determined by the Noise estimation method control button. All data that does not satisfy the S/N ratio indicated by the Minimum S/N control is rejected. In addition, the noise level can be subtracted from the data.

There are 3 choices for the noise estimation method.

When the Noise estimation method is set to None, only the high frequency content where no signal is present is removed. The noise level is not estimated and not subtracted from the signal.

When the Noise estimation method is set to High frequency spectrum, the noise is assumed to be white (i.e. uniform as a function of frequency). The high frequency component of the signal is estimated and used as an approximation of the noise level at all frequencies. This noise level is then removed from the data. This approximation usually under-estimates the noise level, especially at low frequencies. Nevertheless, this estimate is very useful because it is more accurate at high frequencies where the S/N is not as good and where noise subtraction is more useful. Following the estimation, a low-pass filter is applied to the signal at the frequency where the S/N ratio drops to the value selected by the Minimum S/N control. The estimated noise level is subtracted from the data.

To set Noise subtraction method to Waveform averaging, the Waveform averaging value must be set to 2 or more. In addition, if a different waveform is used as a Reference waveform, the Ref. waveform averaging must also be set to 2 or more. If it is set to 1, than the Reference waveform Noise subtraction method defaults to None and no warning is issued. The signal averaging process is used to estimate the noise level at all frequencies. This noise level is then subtracted (averaged out) from the signal. All frequency components where the signal does not meet the Minimum S/N are discarded for further data analysis.

The control Minimum S/N is used with the Noise estimation method to eliminate all frequency components that do not meet the required S/N. This affects the data analysis but does not affect the display of the waveforms. When no Noise estimation method is used, the Minimum S/N is greyed and not used.

#### 4.3.5 Smoothing of results

As discussed in Section 3.8, a simple moving average can be applied to the data being plotted in the Graph of measured & calculated parameters. The moving average is applied to all parameters. When the calculated parameters are Saved to disk, the original values, not the moving averages, are saved to disk.

**Practical tip:** Moving averages can dramatically improve the look of graphs. However, while the data scatter is reduced by the moving average, the time resolution is also reduced and fine details may become hidden. Moving averages must be used sparingly and carefully.

**Practical tip:** Which of Waveform averaging or Moving averages of the final data should be used preferably? Waveform averaging improves the S/N of waveforms, increasing the usable bandwidth of the signal. Therefore, in situations of poor S/N ratio, waveforms should be averaged. On the other hand, if the S/N ratio is good but for some reason the final results have considerable scatter, or if the waveforms change so rapidly with processing conditions that Waveform averaging actually decreases the usable bandwidth, then averaging the final results is preferable.

#### 4.4 Ultrasound Delay, velocity, thickness, and elastic constants

The measurements of ultrasound delay, velocity, sample thickness, and elastic constants are controlled and displayed in the Delay tab of the Analysis panel.

**Analysis**

Delay   Attenuation   Grain size

What do you want to measure?

Velocity at known thickness

Delay ( $\mu$ s)   Thickness (mm)   C33 (GPa)

3.98635   5089.37   195.039

Reference time estimation

Method: Window centering

Reference time: 4.10237

Reference waveform, Second Echo #: 2

Inverted 2nd echo: No

The ultrasound Delay between the echoes selected in the Current waveform and the Reference waveform,  $\Delta t = t_C - t_R$ , is estimated using cross-correlation methods. Given a sufficiently good S/N, the algorithm allows delay measurements to a precision much higher than the time base of the digitizer. It should be noted here that the amplitude of the maximum of the cross-correlation is reported as an estimate of signal amplitude. This amplitude can be selected and displayed in the Result display. However, a much better measurement of signal amplitude is the provided by the amplitude spectra in the Attenuation tab of the Analysis tab control.

The measured Delay is interpreted in two different ways depending on whether one is comparing two different echoes from the same waveform, or comparing two echoes with the same Echo # from different waveforms.

#### 4.4.1 Velocity measurement when using different echoes from the same waveform

When comparing two different echoes from the same waveform, Delay,  $\Delta t$ , and Velocity,  $V$ , are related to each other by the difference in propagation paths,  $\Delta z$ , by

$$V = \frac{\Delta z}{\Delta t} = \frac{2(m - n)h}{\Delta t}$$

where  $m$  and  $n$  are the two different Echo #,  $m > n$ , and  $h$  is thickness corrected for thermal expansion, as described in Section 4.2.

#### 4.4.2 Velocity measurement when using two different waveforms

When comparing two echoes with the same Echo # but from different waveforms, the difference in propagation distance,  $\Delta z$  is zero (assuming the same amount of thermal expansion between the two waveforms), and the above equation cannot be used. Instead, the velocity is calculated using the equation

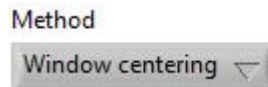
$$V = \frac{z}{t_R + \Delta t} = \frac{2nh}{t_R + \Delta t}$$

where  $z$  is propagation distance,  $t_R$  is the arrival time of the Reference echo, and  $h$  is sample thickness. An absolute value measurement of  $t_R$  is required to use this equation. This is problematic because absolute value measurements are much harder to make than relative measurements, as discussed in Section 1.3.1.

To measure  $t_R$  accurately, one needs to know exactly when the ultrasound pulse is generated and to know what feature of the pulse should be considered. This is not easy to determine. To begin, we do not know the precise moment of ultrasound generation. There are various unknown experimental optical and electronic delays that can be significant. After all, the speed of light in vacuum is about 30 cm per ns, and we are attempting to measure ultrasonic times to better than 1 ns. So at the very least, we would have to measure optical paths precisely. Also, there are electronic delays and delays caused by the ultrasound generation mechanism, and these are hard to measure. In addition, the relationship between the generation laser pulse shape and the ultrasound pulse shape is not obvious, and it varies with laser pulse fluence.

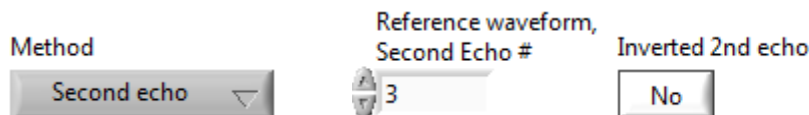
Therefore, it is not clear what feature (maximum, minimum, leading edge, ...) of the pulse represents the true arrival time of the pulse.

The first Method to measure  $t_R$  is not a very good one. It is called Window centering. Depending on the setting of Window centering control button in the Windowing panel, the time at the maximum or minimum amplitude of the Reference waveform is used to estimate  $t_R$ . (The method is called Window centering because it uses the Window centering button to select the time at minimum or maximum. The method could have been called the Min/Max method.) To use this method, simply set the Reference time method to Window centering.



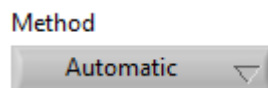
The advantage of this method is that it always works. Often, there is a need to measure velocity changes precisely, but there is no need to obtain accurate absolute values. In such cases, the Window centering method may be sufficient.

A better method to estimate  $t_R$  is to use two echoes of the Reference waveform and to accurately measure the time delay between the two echoes using cross-correlation methods. This time delay can then be used to estimate  $t_R$ . To use this method, one sets the Reference time method to Second echo and select a Reference waveform Second Echo # that is different from the Reference waveform Echo # (the LUMet software does not allow the Second Echo # to be the same as the Reference Echo #). If the Second echo of the Reference waveform is inverted with respect to the Reference Echo, the control button Inverted 2<sup>nd</sup> echo should be set to Yes.



The advantage of this method is that it is very accurate. The disadvantage is that the Reference waveform must have at least two usable echoes.

An Automatic Method is provided also. This method is as accurate as using a Second echo and more convenient. However, it requires multiple echoes to work properly.



#### 4.4.3 Elastic constants

As discussed in Section 1.5.1, the elastic constant  $C_{33}$  is estimated from velocity and density according to the equation

$$V_{ii} = \sqrt{\frac{C_{ii}}{\rho}}$$

which can also be re-written

$$C_{33} = \rho V_{33}^2$$

where the "3" direction is the direction normal to the sample surface, i.e. the thickness direction. Here, we have used the two-index notation where the indices go from 1 to 6 (Section 1.4).

#### 4.4.4 Shear waves

Although this is outside of the scope of what the LUMet software was designed to do, it is possible to use the software to detect and measure shear wave velocity. When the generation laser launches a pressure pulse, it also launches a shear pulse. This pulse is usually observable as a single echo between the first and second compression echoes. To measure the shear wave velocity, one needs compare the Current waveform to a fixed Reference waveform and measure the delay between the shear pulse in the Current waveform and the shear pulse in the Reference waveform. This provides for a precise measurement of the velocity variations. The absolute value can be estimated the Reference time (the time of arrival of the shear pulse in the Reference waveform) using the Window centering method. The procedure is described in the references below. Also, to locate the shear pulse echo, the User-defined Materials properties data must be selected and modified to provide representative values for shear waves.

The measured shear wave velocity and shear elastic moduli are related to each other by the relation

$$V_{44} = V_{55} = \sqrt{\frac{C_{44}}{\rho}} = \sqrt{\frac{C_{55}}{\rho}}$$

for a sample with orthotropic symmetry (see Section 1.5.1). In other words, this allows measuring the two shear wave velocities propagating in the thickness direction and having polarization in the two main direction of the plane. However, these two elastic constants are usually close to each other and this measurement method would not allow distinguishing the two.

Shear waves are also observable using the patented Laser-Ultrasound Resonance Spectroscopy technique. This technique is very precise and allows measuring the difference between  $C_{44}$  and  $C_{55}$  in most materials. The LUMet software does not provide the analysis tools for that technique.

#### References

André Moreau and John T. Jones. "Laser-ultrasonics Assess High Temperature Elastic Properties." *Ceramic Industry* **144**, 3 (March 1995) p. 45-46.

A. Moreau and F. Taheri. "Elastic Moduli Measurements of SiC Reinforce Alumina at High Temperatures Using Laser-Ultrasonics." *Nondestructive Characterization of Materials VII*. Edited by Anthony L. Bartos, Robert E. Green, Jr., and Clayton O. Ruud (Transtec Publications,

Switzerland, 1996). p. 235-242. Re-published in *Materials Science Forum* **210-213** p. 235-242 (<http://www.scientific.net>).

A. Moreau, D. Lévesque, M. Lord, M. Dubois, J.-P. Monchalin, C. Padioleau, and J. F. Bussière. "On-line Measurement of Texture, Thickness, and Plastic Strain Ratio Using Laser-Ultrasound Resonance Spectroscopy." *Ultrasonics* **40**, (2002) p. 1047-1056.

D. Lévesque, A. Moreau, M. Lord, C. Padioleau, M. Dubois, J.-P. Monchalin and J. F. Bussière. "Laser-Ultrasound Spectroscopy Apparatus and Method with Detection of Shear Resonances for Measuring Anisotropy, Thickness, and other Properties." US Patent # 6,057,927 (2000).

#### **4.4.5 Making accurate velocity measurements**

In general, the time delay between two echoes can be measured with great accuracy, sometimes to better than one part in  $10^4$ . The errors on velocity measurements usually arise from other sources:

##### *Thickness*

Thickness is usually measured to an accuracy of about one part in 1000 (1 micron accuracy for a 1 mm thickness). In addition, the linear correction to thermal expansion is only an approximation. More accurate velocities can be calculated by setting the thermal expansion value to zero (in the User-defined material parameters) and calculating the velocity from the delay information in a spreadsheet.

##### *Diffraction*

Diffraction (Sections 1.3.3 & 4.7) can affect velocity measurements by about 1%. Therefore, for maximum accuracy, it is important that the two selected echoes be either both in the near acoustic field ( $S \leq 0.1$ ) or both in the far acoustic field ( $S \geq 10$ ). Because the echoes have a wide frequency bandwidth, different frequency components have different values of  $S$ . Therefore, one should insure that the frequency components of largest amplitude satisfy either the near field or far field criterion. To observe the frequency range of largest amplitude, one can display the Amplitude spectra of the Current and Reference waveforms using the Y-scale control of the Attenuation tab in the Analysis tab control (Section 4.6).

##### *Scattering by grain size or second phases*

Grain size and scattering by second phases can affect the measured velocity by an amount that can be as high as about 1%.

##### *Static vs. dynamic moduli*

The measured moduli are dynamic moduli. They can differ from static moduli due to various phenomena related to anelasticity, or internal friction. Also, ultrasonic waves are adiabatic and so provide an estimate of adiabatic moduli, while static moduli are usually isothermal. In most cases, this distinction is ignored but it could cause an error of order 1%.

*In summary*

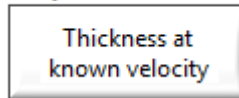
Numerous factors influence the absolute value of the measured velocity and moduli. As a rule of thumb, the accuracy (error on absolute value) is about  $\pm 1\%$  and could be as high as several percent in the worst cases. Precision (relative errors on repeated measurements) depends only on the precision of the delay measurement and can be as good as 0.01%, i.e.  $10^{-4}$ .

## 4.5 Thickness

The LUMet software can also be used to measure sample thickness when ultrasound velocity is constant. This could be used, for example, to monitor sample thickness during mechanical deformation. In this case, proceed as follows:

- 1) Select a waveform before any change in sample thickness occurs. Measure the ultrasound velocity as described in Section 4.4.
- 2) In the Sample properties, under Material, select User-defined and overwrite the values for Velocity and Reference Temperature with the measured values.
- 3) On the control button What do you want to measure?, select Thickness at known velocity.

What do you want to measure?



- 4) The Delay tab of the Analysis panel now estimates sample thickness instead of Velocity.

The Reference waveform can be either same waveform or a different waveform. When the Same waveform is used, thickness is estimated using

$$h = \frac{V \Delta t}{2(m - n)}$$

where  $V$  is the velocity indicated in the Materials properties. When two different waveforms are used, thickness is estimated using

$$h = \frac{V(t_R + \Delta t)}{2n}$$

The reference time,  $t_R$ , is estimated as before (Section 4.4.2).

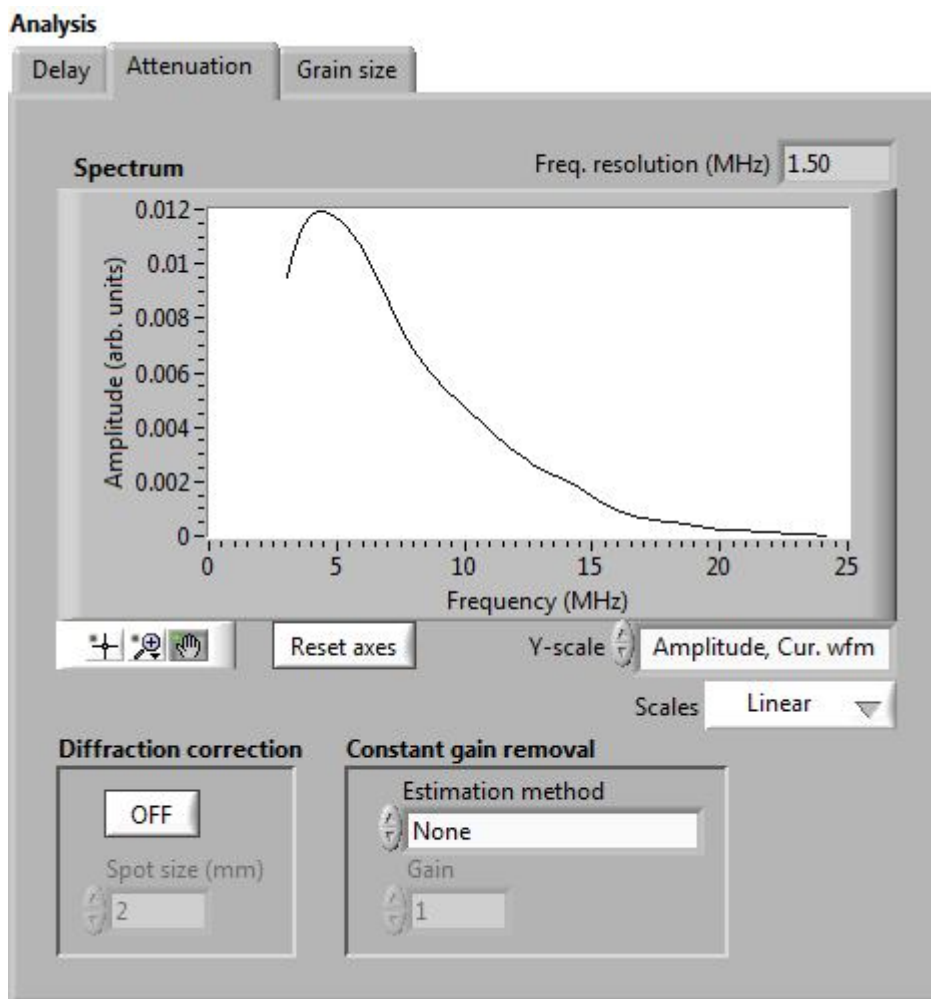
### Practical tip:

To measure sample thickness variations in a hot deformation test, do the following:

- 1) Set the control What do you want to measure? to Velocity at known thickness.
- 2) Use the LUMet software to calculate the ultrasound velocity up until a short time before the mechanical deformation and stop the analysis. Note the temperature and velocity.
- 3) Select User-defined Material properties and enter the measurement temperature and velocity.
- 4) Set the control What do you want to measure? to Thickness at known velocity.
- 5) Erase any previously recorded data if any. Set the "Record" control to "On". Analyse the data during the hot deformation. Set the "Record" control to "Off".
- 6) In the Result display area, display Thickness as a function of Time.

## 4.6 Amplitude, amplitude ratio, and attenuation spectra

The amplitude spectrum (Section 1.3.2) of either the Current or Reference echo is displayed by selecting the appropriate choice for the Y-scale in the Attenuation tab of the Analysis tab control.





Note that these amplitude spectra can be saved to file. The Result display, however, does not provide for a means to plot the amplitude at a specific frequency as a function of other measurement parameters. This must be done in a spreadsheet or plotting software. The only amplitude information that can be plotted is the amplitude of the cross-correlation used to calculate the time delay. This latter information is often misleading. The information obtained from the spectra is much more reliable and representative of the material.

The Amplitude ratio spectrum,  $R(f)$  is simply the ratio of the Current to Reference amplitude spectra.

$$R(f) = \frac{A_C(f)}{A_R(f)}$$

The Amplitude ratio has no units. If the Reference waveform is the Same waveform as the Current waveform, then the Attenuation spectrum is defined as

$$\alpha(f) = \frac{20}{2(m-n)h} \log \frac{A_C(f)}{A_R(f)}$$

where  $m$  and  $n$  are the two echo numbers. It has the units of dB/mm. If the two echoes are similar echoes from two different waveforms, then

$$\alpha(f) = \frac{20}{2nh} \log \frac{A_C(f)}{A_R(f)}$$

A difficulty arises when the two echoes are from different waveforms: the two waveforms are unlikely to have the same gain. The reason for this is that the ultrasound generation efficiency depends on temperature and sample surface conditions, and the detection sensitivity depends on sample surface reflectivity, which can vary. These variations are not the exception, they are almost always present. However, the gain fluctuations of the detection interferometer are independent of frequency. The gain fluctuations of the laser-generation process might depend on frequency but, if the samples of the Reference and Current waveform are either the same sample or very similar samples at the same temperature, the gain fluctuations can be assumed to be independent of frequency.

Let's denote the frequency-independent component of the overall system gain,  $g$ , so that  $g_R$  and  $g_C$  are the frequency-independent gains of the Reference and Current waveforms, respectively. Then, the measured attenuation is

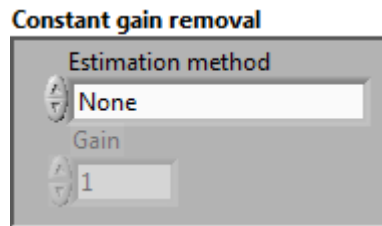
$$\alpha(f) = \frac{20}{2nh} \log \frac{g_C A_C(f)}{g_R A_R(f)}$$

$$\alpha(f) = \frac{20}{2nh} \left( \log \frac{A_C(f)}{A_R(f)} + \log \frac{g_C}{g_R} \right)$$

The second term in the parentheses is a constant. The effect of not knowing the gain of the LUMet system is to add or subtract an unknown constant to the attenuation spectrum. This constant can cause difficulties in subsequent analyses. Therefore, several empirical algorithms

are provided to estimate and remove this constant. These algorithms are provided in the Constant gain removal panel.

The Estimation method control is used to select which algorithm is used. In all cases, the algorithm sets  $g_R = 1$ . The selected method sets the value  $g_C$ . This value of  $g_C$  is displayed in the Gain control, even if it is disabled. The value of  $g_C$  affects the Amplitude spectrum of the Current waveform, the Amplitude ratio spectrum, and the Attenuation spectrum. The Amplitude spectrum of the Reference waveform is never affected.



The choices of Estimation method are:

None: No constant is removed from the attenuation spectrum. This is equivalent to assuming that  $g_R = g_C = 1$ . This option should be selected when the Reference waveform is the Same waveform as the Current waveform. The gain control is disabled and displays a value of 1.

User-defined constant: The Gain control is enabled and sets the value  $g_C$ . This may be useful to remove a known geometrical gain constant.

Maximum of amplitude ratio: The maximum of the amplitude ratio spectrum is set to 1. This is equivalent to setting the minimum value of the attenuation spectrum to zero. The gain control is disabled and displays the estimated value of  $g_C$ .

Amplitude ratio at lowest frequency: Sometimes, it is known or assumed that the attenuation tends towards zero as frequency tends towards zero. Here, the gain is adjusted so that the amplitude ratio at the lowest measured frequency is equal to 1. This is equivalent to setting the attenuation at the lowest measured frequency equal to zero. The gain control is disabled and displays the estimated value of  $g_C$ .

Alpha =  $a_0 + a_1 f^n$ : Sometimes, it is known or assumed that the attenuation tends towards zero as frequency tends towards zero. In particular, many models predict that the attenuation spectrum should behave according to

$$\alpha(f) = a_0 + a_1 f^n$$

where  $a_0$  and  $a_1$  are calculated or fitted parameters, and  $n$  depends on the model. Usually, the models predict that  $a_0 = 0$ . When this option is selected, the attenuation spectrum,  $\alpha(f)$ , is fitted to the above equation using a non-linear least squares fit. Then precisely,  $g_C$  is adjusted so that  $a_0 = 0$ . The gain control is disabled and displays the estimated value of  $g_C$ .

## 4.7 Diffraction

### 4.7.1 Avoiding diffraction

Diffraction is discussed last because it is a complex subject. Whenever possible, it is strongly recommended to do measurements either in the near field ( $S < 0.1$ ) or in the far field ( $S > 10$ ). As discussed in Section 1.3.3, in the near field, the ultrasound pulse is a plane wave and diffraction is negligible. In the far field, the ultrasound pulse decreases as the inverse propagation distance and the contribution to attenuation is a constant independent of frequency, which can be removed either using the Diffraction correction (which works well in this case) or using the Constant gain removal algorithms described in Section 4.6.

Also, it is recommended to always make measurements with generation and detection spots of equal dimensions. When the spots are of equal dimensions, diffraction effects are minimized. When the spots have different dimensions, it is for all practical purposes impossible to model the diffraction effects because too many variables are involved.

One reason for this is that it is difficult to know precisely the dimensions of the generation laser spot. The ablation mechanism involves a threshold in fluence, and the effective diameter of the generation spot can vary with laser power, laser "hot spots" (non-uniformity in spatial distribution of energy), atmospheric conditions (vacuum, air, inert gas, pressure), and sample conditions (surface reflectivity, surface roughness, temperature).

### 4.7.2 Removing diffraction effect using model calculations

For all the reasons cited in Section 4.7.1, diffraction corrections should be avoided. However, a correction is provided whereby only one variable is made adjustable: the diameter of the generation and detection spots, which are assumed to be equal. This provides a first order correction to diffraction while avoiding the pitfalls caused by too many adjustable variables. The model calculation used in the LUMet software is the exact solution to the circular piston-piston model, whereby the generation and detection transducers are circular and have a uniform gain over their active area.

The diffraction correction (if used) is applied before the constant gain removal described in Section 4.6.

### References

J.-D. Aussel and J.-P. Monchalín. "Measurement of Ultrasound Attenuation by Laser Ultrasonics" *Journal of Applied Physics*, Vol. 65, No. 8, 15 April 1989, p. 2918-2922.

G. S. Kino. "Acoustic waves, devices, imaging, & analog signal processing" (Englewood Cliffs NJ, Prentice-Hall, 1987) Chapter 3.

### 4.7.3 Removing diffraction effects empirically

The effect of diffraction is to introduce a frequency-dependent gain  $g(f)$  to the Amplitude spectrum.

$$\alpha(f) = \frac{20}{2nh} \log \frac{g_C(f)A_C(f)}{g_R(f)A_R(f)}$$

$$\alpha(f) = \frac{20}{2nh} \left( \log \frac{A_C(f)}{A_R(f)} + \log \frac{g_C(f)}{g_R(f)} \right)$$

Therefore, the total attenuation  $\alpha(f)$  can be written

$$\alpha(f) = \alpha_i(f) + \alpha_D(f)$$

Where  $\alpha_i$  is the attenuation intrinsic to the sample, and  $\alpha_D$  is the diffraction contribution. In other words, the intrinsic and the diffraction contributions are additive. One way to remove the diffraction contribution would be to measure it on a sample with zero intrinsic attenuation, and then to subtract this contribution from the total attenuation spectrum. This can be done, but it is a relatively complex procedure.

In the far field, the diffraction contribution can be reduced to a frequency independent constant,  $C$ , and the condition

$$g_C(f) = C g_R(f)$$

is satisfied. In such a case, the Constant gain removal methods discussed in Section 4.6 can be applied.

In general, however, diffraction depends on ultrasonic frequency, the dimensions of the source and detection area, velocity, and propagation distance. Typically, the source and detection dimensions are fixed by the measurement instrument. Velocity can be constant if the Reference waveform comes from the same material (preferably at the same temperature) as the Current waveform. And propagation distance can be made constant by comparing the same Echo # for two samples of the same thickness. In such a case, the diffraction effect is the same on both waveforms, to within a constant gain factor.

This special case occurs relatively frequently. It happens, for example, when two samples of the same material have received different thermo mechanical treatments, and the Reference sample has near zero intrinsic attenuation. The Reference sample can often be the same sample before and after thermal processing, whereby the Reference sample has a fine microstructure (with low intrinsic scattering and attenuation) while the measurements at temperature involve substantial attenuation due to scattering by a coarse microstructure. Moreover, it turns out that the dependence of diffraction on small changes in velocity is weak. Therefore, it is even possible to compare two samples at different temperatures.

The frequency-dependent gains depend on ultrasound generation. Therefore, it is important to use the same material in both cases. It would not be a good idea to compare echoes from aluminium and iron samples, for example. Temperature can have an effect on ultrasound generation amplitude, but experience has shown that this effect can be assumed to be independent of frequency.

**Practical tip:** To remove the effect of diffraction empirically, compare the same Echo # for two different Current and Reference waveforms. The Reference waveform must be obtained from a sample of the same material, same thickness, and negligible intrinsic attenuation. The sample used for the Reference waveform is often the same sample as for the Current waveform. Negligible intrinsic attenuation can be obtained by changing the sample's microstructure with the Gleeble™ thermo mechanical simulator. The Reference waveform can be obtained either before or after the main experiment.

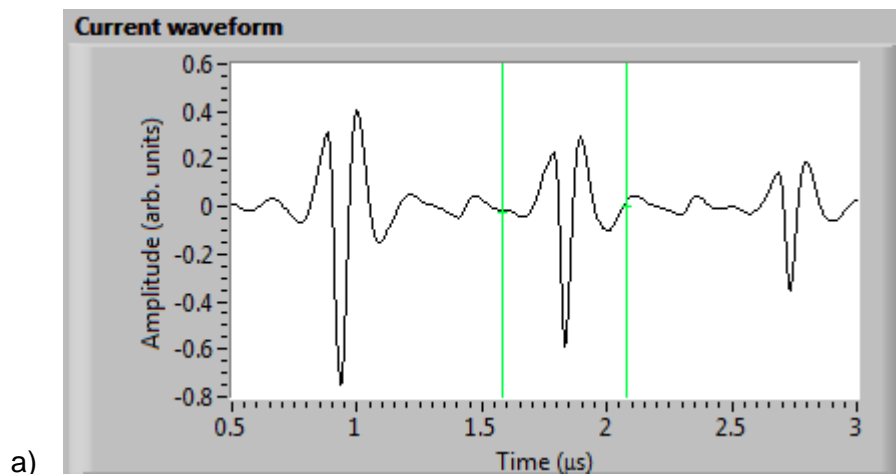
Reference:

S. Sarkar, A. Moreau, M. Militzer, and W. J. Poole. "The evolution of austenite recrystallization and grain growth using laser-ultrasonics" *Metallurgical and Materials Transactions A* **39** (April 2008) p. 897-907.

## 4.8 Other sources of measurement errors

### 4.8.1 Shear waves, surface waves, sample width

It is important to exclude shear waves from the windows around the echoes of interest. The figure below shows the first three echoes in a flat steel sample of 2.21 mm thickness at 1000°C. Shear waves pulses are observed at approximately 1.5 and 2.3 microseconds as a small positive pulse. If they are included in the windows surrounding the pressure pulses, they will affect their amplitude spectra which, in turn, will produce erroneous attenuation spectra.



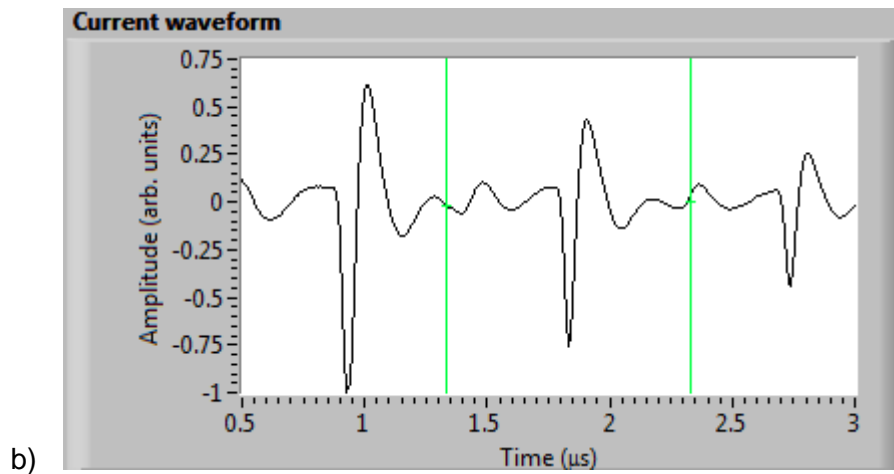
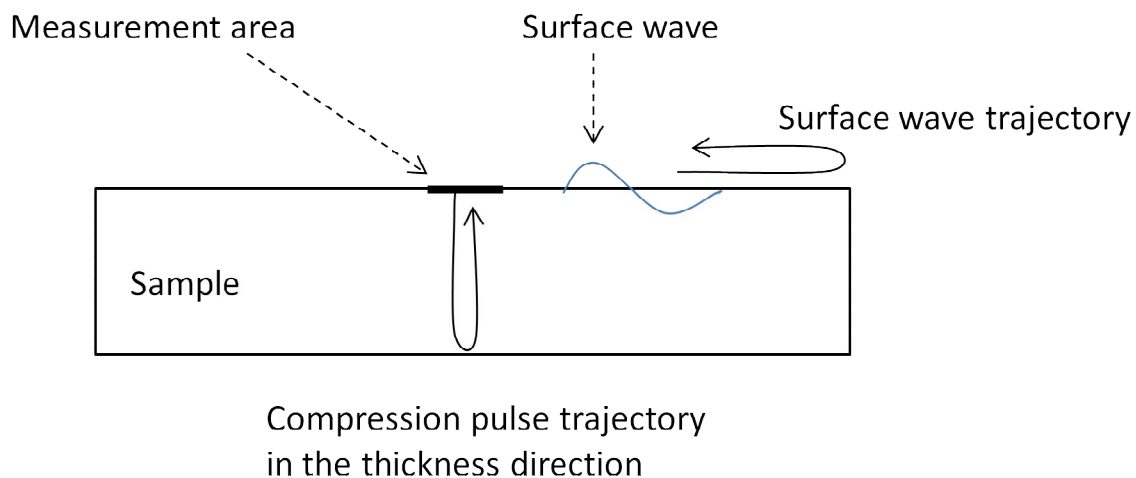


Figure caption: First three echoes in a flat steel sample of 2.21 mm thickness at 1000°C. a) Window large enough to enclose the entire ultrasound pulse but narrow enough to not enclose the shear pulses. b) Window too wide because it encloses the shear pulse near 1.5  $\mu\text{s}$  and the beginning of the shear pulse near 2.3  $\mu\text{s}$ . Notice that the width of the window affects the shape of the sound pulses because internal filters are adjusted according to the window width.

Other ultrasound pulses or signals may be found between the pressure pulses. One must be aware that although the LUMet system is somewhat optimized to generate and detect pressure pulses in the thickness direction, it also generates and detects all types of waves in all directions. One must be careful to keep the sample geometry simple and avoid reflections by the sample boundaries, reflections that can propagate back to the detection spot.

With flat samples, surface waves can be generated at the generation area, travel to the edge of the sample, and be reflected back to the detection area. This is illustrated in the Figure below. Surface waves are a kind of shear wave where the displacement is mostly perpendicular to the surface. This wave always travels at a velocity slightly less than the velocity of the shear wave in the bulk. It decreases in amplitude as the square root of the propagation distance. So the further the edges are, the lower is the amplitude of the wave when it comes back.



To avoid any potential superimposition of the surface wave onto the compression echoes of interest, the simplest method is usually to make sure that the sample is wide enough so that the surface wave returns to the measurement location later than the arrival time of the end of the latest pressure pulse of interest. For example, in the first Figure of this section, if only the last Echo of interest is Echo #2 is, then the surface wave should arrive later than about  $2.3 \mu\text{s}$  because Echo #2 ends near  $2.1 \mu\text{s}$ . For steels, shear wave velocity is around 2700 m/s at room temperature. The surface wave velocity is approximately 10% less and so would be 2400 m/s. This means that the distance between the edge of the measurement spot and the edge of the sample width should be more than  $0.5 * 2400 \text{ m/s} * 2.3 \mu\text{s} = 3 \text{ mm}$ . If the measurement spot diameter is 2 mm, the sample should be at least 8 mm wide ( $3 + 2 + 3 \text{ mm}$ ). This is barely sufficient so one would choose at least a 10 mm sample width.

Because the shear wave velocity is about half the velocity of compression wave, a simple rule of thumb, the minimum sample width should be more than twice the thickness times the (Echo # +1) of the latest echo of interest plus the size of the measurement area. "One " is added to the Echo # because the beginning Echo  $n+1$  is near the end of Echo  $n$ . Applying this rule to the above example, the sample width should be at least  $3 * 2.21 \text{ mm} + 2 \text{ mm} = 8.6 \text{ mm}$ . This should insure that surface waves do not have time to travel to the edge of the sample and back to the detection area before the last through thickness echo of interest has arrived.

#### 4.8.2 Wedge sample

Flat samples must have flat and parallel faces. Wedge samples will produce erroneous attenuation spectra. As a rule of thumb, samples that are milled flat by a milling machine, or sheet or plate metal rolled at the factory are flat enough to allow measurements on the first few echoes, but not flat enough for high Echo #.

An improved rule of thumb is that the total thickness variation over the dimensions of the measurement area should be much less than  $\lambda/10 * 2n$ , where  $\lambda$  is the ultrasonic wavelength at the highest frequency of interest, and  $n$  is the largest Echo #. A detailed analysis is found in the reference below.

#### Reference:

R. Truell, C. Elbaum, and B. B. Chick, *Ultrasonic Methods in Solid State Physics* (Academic Press, New York, San Francisco, London, 1969). p. 107-121.

#### 4.8.3 Surface roughness

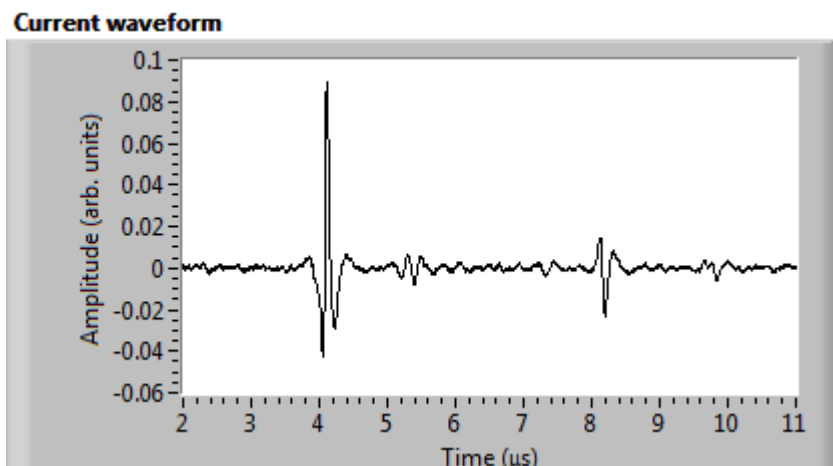
Sample surfaces must be machined to a smooth finish otherwise the attenuation spectra may be erroneous. It is not necessary to polish sample surfaces. The last machining (milling) pass should be light. Commercial sheet metal products (like automotive steel sheets) often have a surface roughness of about one micron  $R_A$  (about the same as 1 micron root-mean-square deviation). This level of surface roughness will not affect the spectra significantly.

On the other hand, polishing sample surfaces will increase the reflectivity of the surface and improve the sensitivity of the detector, if the main reflection goes back into the detecting optics.

At the extreme, when the surface is close to mirror like, the amount of light collected can be high enough to damage the photo-detector. Mirror-like samples should not be used.

#### 4.8.4 Cylindrical sample

Cylindrical samples usually show echoes that alternate in polarity. This is illustrated below for a cylindrical steel sample of 10 mm diameter, at 993°C. In such cases, if two successive echoes are selected, it is important to set the Window centering to either "Max / Min" or "Min / Max".



Cylindrical samples often present many echoes caused by multiple reflections inside the sample. One such echo can be seen clearly near 5.3  $\mu\text{s}$  in the figure above. However, most of the little oscillations between the two main echoes are not random noise and are probably caused by multiple reflections of some kind. Such echoes must be excluded (as much as possible) from the windows set around the echoes of interest. The complexity of the multiple echoes increases with time (i.e. with higher Echo numbers). Therefore, it is best to use either the first two echoes of a single waveform, or the first echo of two different waveforms.



## 5 Advanced analysis

In this section, the measurements of grain size (Section 5.1) and texture (Section 5.2) are given special attention because these two microstructural parameters have a significant effect on ultrasonic attenuation and velocity, respectively. Following these two sections, references are made to published papers that discuss in details how the LUMet technology can be used to monitor various phenomena occurring during thermo mechanical processing.

### 5.1 Grain size

The laser-ultrasonic measurement of grain size is based on the principle that the larger are the grains, the more ultrasound is scattered by them, and the larger is the ultrasonic attenuation. The Grain size scattering equation may be written

$$\alpha_{sc}(f, T) = K(T)D^{n-1}f^n$$

where  $\alpha_{sc}$  is attenuation caused by grain scattering,  $f$  is frequency,  $T$  is temperature,  $D$  is grain diameter, and  $K(T)$  is a constant that depends on temperature. It is known from theoretical considerations that  $0 \leq n \leq 4$  depending on the ratio of acoustic wavelength to grain diameter. In the limit of wavelengths much larger than  $2\pi D$ ,  $n$  tends towards 4. And in the limit of wavelengths much smaller than  $2\pi D$ ,  $n$  tends towards 0. In practice, at very long wavelengths, the total attenuation is small and difficult to measure, so  $n \approx 4$  is rarely observed. Conversely, at very short wavelengths, the attenuation is so strong that the ultrasound is completely scattered by a few grains, and the unscattered signal is very weak. Therefore,  $n < 1$  is rarely observed.

In all cases,  $n$  varies only slowly with frequency. For example, in stainless steel, a log-log plot of attenuation vs. frequency shows an almost linear behaviour when frequency changes by a factor of about 5 (at constant grain size), and  $n$  varies from 3.3 to 2.0 when grain size varies from 10 to 57  $\mu\text{m}$  (at roughly constant frequency). So, in practice,  $n$  can be considered essentially constant and its value is usually somewhere between 1.5 and 3.5.

However, other important phenomena contribute to the total attenuation. In particular, diffraction can affect signal amplitude and the apparent attenuation. Also, internal friction often contributes to the intrinsic attenuation of the material. Therefore, the total attenuation  $\alpha$  can be written

$$\alpha(f, T) = \alpha_{sc}(f, T) + \alpha_{IF}(f, T) + \alpha_D(f, T)$$

Where  $\alpha_{IF}$  is the contribution of internal friction, and  $\alpha_D$  is the contribution of diffraction. Section 4.7 already discussed how diffraction can be made negligible or removed from the attenuation spectrum. Therefore, the attenuation spectrum provided by appropriate experimental procedures and the LUMet software is

$$\alpha(f, T) = \alpha_{sc}(f, T) + \alpha_{IF}(f, T)$$

At this point, not much can be done to separate the two contributions. Sometimes, the internal friction component (or a portion of the internal friction component) can be assumed to be independent of frequency, and so the Constant gain removal described in Section 4.6 will remove that component.

Often, however, the scattering contribution dominates and the internal friction component can be neglected. Equation 5.1.1 indicates that scattering increases with grain size and frequency. Fortunately, laser-ultrasonics provides ultrasound with a wide bandwidth and ultrasonic attenuation can be measured at relatively high frequencies (tens of MHz).

The proportionality constant  $K(T)$  of the Grain size scattering equation depends strongly on the single crystal elastic anisotropy. As discussed in Section 1.5.2, scattering occurs when the ultrasound pulse travels through grains having different orientations, and thus different ultrasonic impedances. The difference in impedance is larger if the grains are more anisotropic. A few values of the single crystal anisotropy factor,  $a$ , whereby

$$a = \frac{2c_{44}}{c_{11} - c_{12}}$$

(for cubic materials) are given in the Table below. When  $a$  is close to 1, the material has little anisotropy and scattering is weak. When  $a$  is large, scattering is large. In simple scattering models,  $K$  is proportional to  $a^2$ . Clearly, scattering is much higher in iron and copper than it is in aluminium. In fact, scattering becomes dominant in aluminium only for very large grain sizes (hundreds of microns), while scattering can be the dominant contribution for iron for grain sizes smaller than ten microns.

Table caption: The anisotropy factor of a few cubic elements at room temperature.

Element	$a$
Al	1.22
Fe (ferrite)	2.42
Cu	3.21

When the attenuation spectrum measured according to any of the methods of Section 4.6 is assumed to be caused by scattering, the LUMet software provides three calibrations to relate the attenuation to grain size.

#### References:

This reference gives a good experimental review of the dependence of ultrasonic attenuation on grain size: E.P. Papadakis, "Revised Grain Scattering Formulas and Tables", Journal of the Acoustical Society of America Vol. 37, 1965, 703-710.

This reference is the starting point of modern grain size scattering theories: Fred. E. Stanke and G. S. Kino. "A unified theory for elastic wave propagation in polycrystalline materials" J. Acoust. Soc. Am. Vol. 75, No. 3, March 1984. p. 665-681.

This reference and several subsequent publications by the same author provide different grain size scattering theories for weakly scattering materials: S. Hirsekorn, The scattering of ultrasonic waves by polycrystals, J. Acoust. Soc. Am., 1982; 72, 1021-1031.

This paper is the first known (to us) publication of the first equation of this section. M. Dubois, M. Militzer, A. Moreau, and J. F. Bussière, "A New Technique for the Quantitative Real-Time Monitoring of Austenite Grain Growth in Steel", *Scripta Materialia* **42**, 9 (2000) p. 867-874.

This paper illustrates the slow variation of  $n$  with frequency: Bruce W. Krakauer and André Moreau. "Application of Laser Ultrasonics to the Measurement of Grain Size." *Advanced Sensors for Metals Processing*. Proceedings of the International Symposium on Advanced Sensors for Metals Processing, Quebec City, Canada, 22-26 August 1999. Edited by B. W. Brusey, J. F. Bussière, M. Dubois, and A. Moreau. Canadian Institute of Mining, Metallurgy and Petroleum, Montreal, 1999. p. 41-52.

Scattered ultrasonic waves are studied in this paper: Silvio E. Kruger, Andre Moreau, Daniel Lévesque and Martin Lord. "Laser Ultrasonic Measurements of Scattered Waves in Steel". *Review of Progress in Quantitative Nondestructive Evaluation* **20B**. Edited by D. O. Thompson and D. E. Chimenti (American Institute of Physics, New York, 2001). p. 1298-1305.

### 5.1.1 Universal empirical calibration

To relate ultrasonic attenuation to grain size, we consider that the attenuation caused by scattering is given by

$$\alpha_{sc}(f, T) = K(T) D^{n-1} f^n$$

Scattering theory usually describes the frequency dependence in terms of the wavevector  $k = 2\pi/\lambda = 2\pi f/v$ , instead of frequency. This is because the product  $kD$  is dimensionless, and because scattering is often described as depending on the ratio  $D/\lambda$ . The above equation can be re-written

$$\alpha_{sc}(f, T) = K'(T) D^{n-1} k^n$$

where  $K' = K(T) (v/2\pi)^n$ . In this form,  $K'$  is a number (it has no units) because the attenuation is expressed as a number (dB) per unit distance. Empirically, the measured attenuation is fitted to the equation

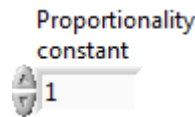
$$\alpha = a + bk^n$$

where  $a$  and  $b$  are parameters fitted to the ultrasonic data. We recall here that  $a$  is equivalent to a change in gain between the two echoes. It can also account for other sources of attenuation if

these are frequency-independent. The parameter  $n$  is best left fixed to a value representative of the data ( $n = 3$  is often good). The grain size  $D$  is related to the fitted parameter  $b$  according to

$$D \propto b^{1/(n-1)}$$

The LUMet software calculates  $b^{1/(n-1)}$ . It is up to the user to find the proportionality constant (equal to  $K^{1/(n-1)}$ ) by comparing the LUMet empirical grain size number to metallographic grain sizes. Note that a new calibration constant must be found for each temperature. If the calibration constant is known, it can be entered in the control entitled "Proportionality constant", and the LUMet software will make the multiplication.



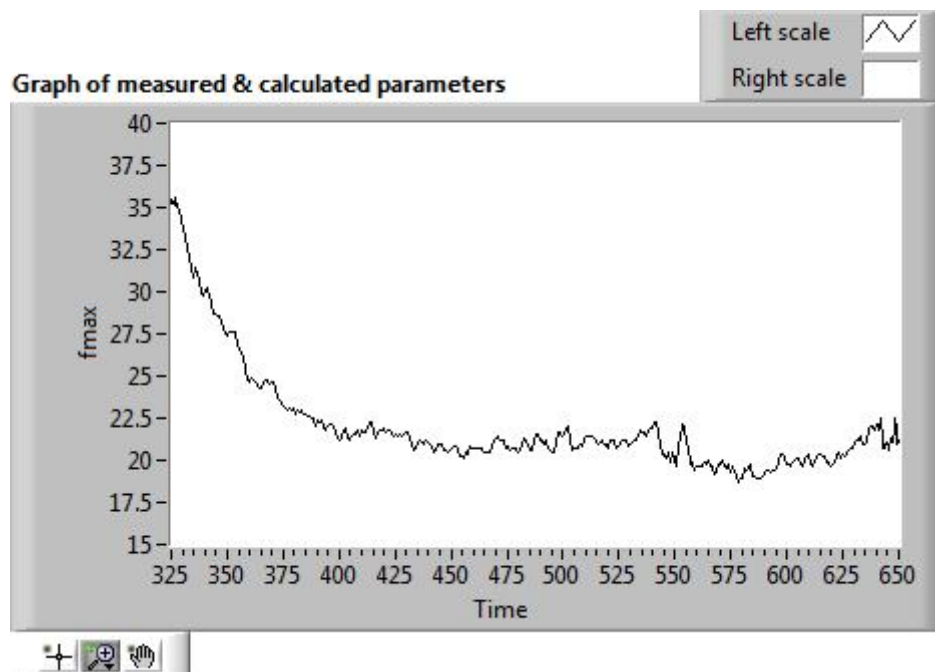
The exponent,  $n$ , can be fitted automatically. This is provided as a convenience to estimate what a good value of  $n$  might be. However, it should always be set to a fixed value when estimating grain size otherwise unstable values of grain size will be obtained. Experience has shown that a value of 3 is often good.

Also note that because the parameter  $a$  is fitted to the data, the constant gain removal algorithm used in the Attenuation tab has no effect on the calculated grain size number.

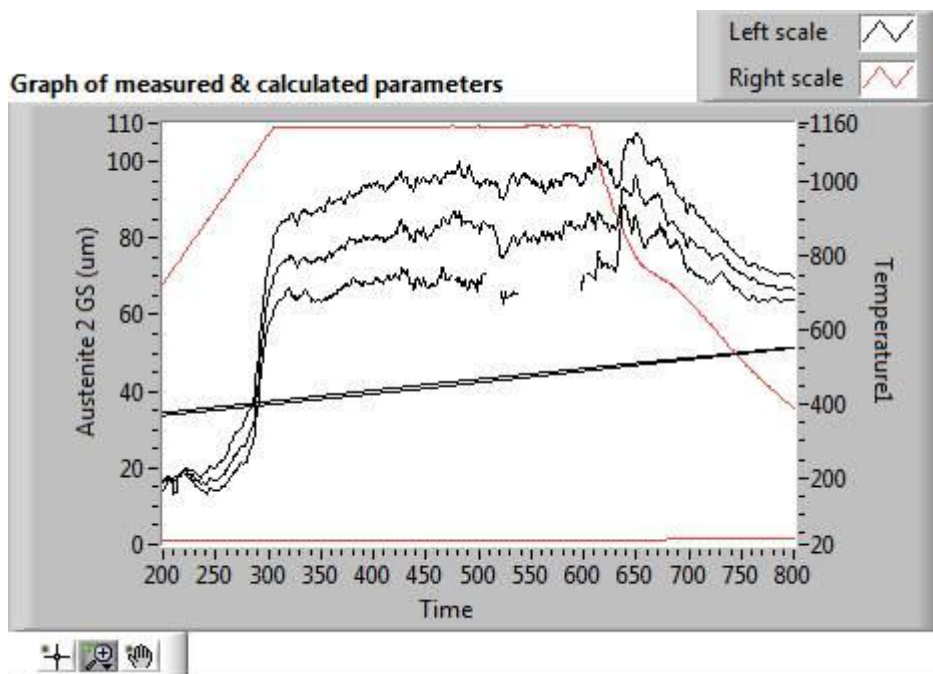
The attenuation spectra only include data whose signal-to-noise ratio exceeds a threshold chosen by the user, as discussed in Section 4.3.4. However, the maximum frequency of the signal affects the exponent,  $n$ . When  $n$  is fixed, changes in signal bandwidth affect the estimate of grain size. Selecting a smaller bandwidth produces systematically higher grain sizes. The effect is not large, but it is measurable. The bandwidth changes because the attenuation changes in the material.

When the software automatically selects the signal bandwidth, the bandwidth fluctuations from one waveform to the next cause fluctuations in the estimated grain size. It is therefore better to limit the bandwidth to stabilize the estimates of grain size. As a rule of thumb, if the highest frequency of each attenuation spectrum fluctuates between  $f_1$  and  $f_2$ ,  $f_1$  being lower than  $f_2$ , then the Bandwidth limit should be set between  $f_1$  and  $f_2$ , close to  $f_1$ . To keep the entire signal bandwidth, one simply sets the Bandwidth button to Max. To keep the bandwidth fixed, one sets the Bandwidth button to Fitted and selects a bandwidth in the control immediately below. The attenuation spectrum will be truncated to that fixed frequency for the purpose of estimating grain size only. If the attenuation spectrum has a lower bandwidth than requested, then grain size is not calculated and the value NaN (not a number) is displayed.

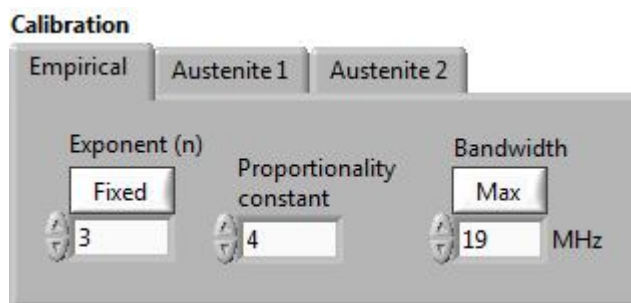
To help in selecting the appropriate bandwidth, one can run the analysis a first time and then plot a graph of the maximum frequency of the spectra as a function of time (or any other convenient parameter). In the example below, a fixed frequency of 20 MHz would be a good choice because most of the data has a bandwidth exceeding 20 MHz. The only exception is near 580 seconds on the time scale.



When the data of that particular experiment is analyzed for austenite grain size during a plateau at 1150 °C (the data before 300 seconds and after about 625 seconds should be discarded because the steel was not in the austenitic phase), the effect of selecting a 16 (top), 18 (middle), or 20 MHz (bottom) bandwidth is clear. Note the missing data in the bottom curve near 280 seconds. In this figure, the data was analyzed 3 times while leaving the Record button On. (Note: The nearly horizontal lines are linking the last data point to the first data point. These lines are not a software bug. The graph can plot any series of data points, in the order that the data are recorded. Remember that these graphs are provided for convenience, not as a publication tool.) Because bandwidth has an effect on estimated grain size, one may choose to always use the same bandwidth. Alternately, the proportionality constant could be adjusted for frequency bandwidth.



Below is a description of the buttons found in the Empirical Calibration.



**Exponent (n):** Can be either Fitted to the data or Fixed. If fixed, one can select any value greater or equal to 0 and less than or equal to 4. In most cases, it is recommended to use a fixed value of 3 as displayed above.

**Proportionality constant:** Proportionality constant that converts  $b^{1/(n-1)}$  into  $D$ .

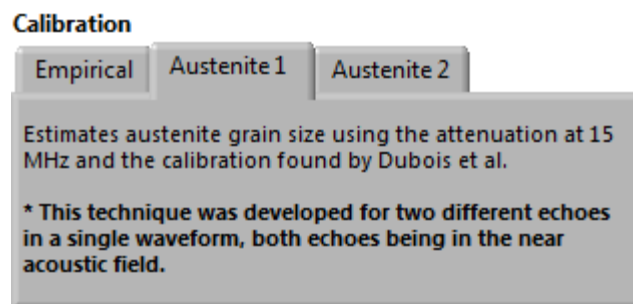
**Bandwidth:** Can be either the maximum (Max) bandwidth that satisfies the minimum S/N criterion (Section 4.3.4) or Fixed. If fixed, one can select any frequency. The attenuation spectrum will be truncated to that fixed frequency for the purpose of estimating grain size only. If the attenuation spectrum has a lower bandwidth than requested, then grain size is not calculated and the value NaN (not a number) is displayed.

### 5.1.2 Calibration 1 for austenite

A simpler method to estimate grain size is to relate attenuation at a single frequency to grain size. This is the method used by most authors. Going back to the equation

$$\alpha_{sc}(f, T) = K(T) D^{n-1} f^n$$

One simply finds the value of  $K(T)$  for a particular ultrasonic frequency. This was done for austenite at high temperature and presented in a paper by Dubois et al. It was found the frequency of 15 MHz worked best. The calibration found in that paper is used together with the attenuation value measured at 15 MHz. The main difficulty with this calibration method is that the absolute value of attenuation must be used. Therefore, it is extremely important that the constant gain removal be used appropriately. Better still, it is best if this method is used in the near acoustic field using the first two echoes of a single waveform (the Reference waveform is the Same waveform) so that there is no diffraction correction and no need for a constant gain removal. The technique should also work well in the far acoustic field because it is easy to calculate the diffraction correction (see Section 4.7) in the far field. However, S/N is often not as good in the far field. Because the technique relies on the measurement at a single frequency, it does not depend on the measurement bandwidth like the other two calibrations. If the attenuation spectrum does not reach 15 MHz, it is extrapolated to 15 MHz from the available data.



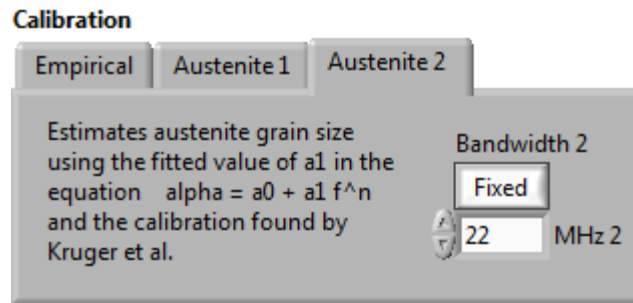
## References

- M. Dubois, M. Militzer, A. Moreau, and J. F. Bussière, "A New Technique for the Quantitative Real-Time Monitoring of Austenite Grain Growth in Steel", *Scripta Materialia* **42**, 9 (2000) p. 867-874.
- S. E. Kruger, A. Moreau and G. Lamouche. "Monitoring Austenitic Grain Growth Kinetics by Laser-Ultrasonics". *Proceedings of the First Joint International Conference on recrystallization and Grain Growth*. Edited by G. Gottstein and D. Molodov (Springer, 2001). p. 583-588.
- M. Dubois, M. Militzer, A. Moreau, and J.F. Bussière. "Real-Time Monitoring of Austenite Grain Growth in Steel Using Laser-Ultrasonics." *Grain Growth in Polycrystalline Materials III*, Proceedings of Third International Conference on Grain Growth, Pittsburgh, PA, 14-19 June 1998. Edited by H. Weiland, B. L. Adams and A. D. Rollett. p. 593-598.

### 5.1.3 Calibration 2 for austenite

This calibration method is similar to the empirical calibration (Section 5.1.1) except that the calibration constant (and its temperature dependence) has been measured for a broad range of steels in the austenitic phase at high temperatures.

Also note that, as for the empirical calibration, the constant gain removal algorithm used to estimate the attenuation spectrum has no effect on the calculated grain size.



## References

S. Sarkar, M. Militzer, W. J. Poole and A. Moreau. "Austenite Grain Size Measurements by Laser-Ultrasonics on a Modern Low Carbon Steel" Proceedings of the International Symposium on Advanced Steels, Edited by J. A. Szpunar and H. Li, Canadian Institute of Mining, Metallurgy and Petroleum, Montreal, QC, Canada, 1-4 October 2006, p. 119-130.

Silvio Kruger, Guy Lamouche, Jean-Pierre Monchalain, Robert Kolarik II Gerald Jeskey, Marc Choquet. "On-line Monitoring of Wall Thickness and Austenite Grain Size on a Seamless Tubing Production Line at the Timken Company" AISTech 2005 Proceedings - Volume II p.553-560.

## 5.2 Texture

### 5.2.1 General principles

The table below shows the ultrasound velocity for a pressure pulse traveling in the [111], [110], and [100] principal directions of single crystals of iron and aluminum. Clearly, the velocity varies with direction, and more so for iron than aluminium. However, even for aluminum, those differences are easy to measure: A difference of 200 m/s on an average velocity of 6450 m/s is a difference of 3%. This is well within the measurement precision of the LUMet. For iron, the difference is 5 times as large.

Table Velocity, in m/s, of the pressure pulse traveling in different directions of single crystals of iron and aluminum

Direction	Fe	Al
100	5432	6336
110	6183	6482
111	6414	6530

In metallurgy, the word "texture" can have several meanings. Here, it signifies "crystallographic" texture or, more precisely, the crystallographic orientation distribution (COD).



Suppose that in some steel alloy, there is a perfect [111] fiber texture, i.e. all crystallites have their 111 axes pointing in the thickness direction of a flat sample. Then, the ultrasound velocity is 6414 m/s. Conversely, if the alloy has a perfect [100] fiber texture, then the ultrasound velocity will be 5432 m/s. In practice, there is no such thing as a perfect texture, except for single crystals. In polycrystalline aggregates, there is always a random distribution of orientations around the preferential orientation. This distribution can be broad, with significant probabilities of finding, say the [111] axis, far away from the normal direction. Or it can be sharp, with very low probability of finding the axis far away from the normal direction. Accordingly, the velocity will be more or less close to 6414 m/s. This example illustrates that when texture changes, velocity is likely to change too.

The orientation of a single crystal is given by 3 Euler's angles. Therefore, the COD is really a combination of 3 different distributions, one for each of the 3 Euler's angles. Although the COD affects the sound velocity, the measurement of a single velocity in the thickness direction is insufficient to determine the 3 distributions.

Therefore, in its simplest use, the LUMet velocity measurement can be used to monitor a texture change, but it does not tell how the texture changes.

Many models have been developed to calculate the effective elastic constants of a polycrystalline aggregate,  $C_{ij}$ , based on the elastic constants of the single crystals and on the COD. The COD is expressed as an orientation distribution function (ODF) that is a series expansion of basis functions, and where the coefficients of the basis functions are called the orientation distribution coefficients (ODC). The effective elastic constants are then estimated by calculating the volume average of the elastic constants of the crystallites rotated over all possible orientations and weighted by the ODCs. There are several ways of conducting these averages (the Voigt, Reuss, or Hill averages) and there are even alternative theories based on the variational principle (Hashin, Shtrikman, Bunge) or even more sophisticated theories. In the end, all these theories produce a result (either the exact result for the specific model or the first approximation of the model) that have the same overall form. For the Voigt, Reuss, and Hill models applied to orthotropic aggregates of cubic crystallites, the  $C_{ij}$  of interest to the LUMet, in the Voigt, Reuss, and Hill models, have the form

$$\begin{aligned} C_{33} &= A + BW_{400} \\ C_{44} &= C - \frac{B}{2}(W_{400} - \sqrt{5/2}W_{420}) \\ C_{55} &= C - \frac{B}{2}(W_{400} + \sqrt{5/2}W_{420}) \end{aligned}$$

where  $W_{400}$  and  $W_{420}$  are two ODCs, and A, B, and C are constants that depend on the single crystal elastic constants and the model used.<sup>1</sup> In practice, A, B, and C are fitted to the data

---

<sup>1</sup> There are two standard series expansion of the ODF, those of Roe and Bunge. In ultrasonics it is customary to use Roe's. In metallurgy, it is customary to use Bunge's. It is sufficient here to state that the  $W_{400}$  coefficient for aggregates of cubic crystallites in Roe's notation is proportional to the  $C_4^{11}$  coefficient in Bunge's notation.

because the model predictions are not sufficiently accurate. As a reminder (Section 1.4),  $C_{33}$  is related to the velocity of the pressure pulse traveling in the thickness direction; and  $C_{44}$  and  $C_{55}$  are related to the velocity of the shear pulses traveling in the thickness direction with polarization in the two principal directions. These two shear pulses have velocities that are only slightly different and cannot be separated unless laser-ultrasound spectroscopy or other measurements techniques are used. So, in practice, both the compression and shear pulses are sensitive to the  $W_{400}$  texture coefficient. The pressure pulse, however, is twice as sensitive and much easier to measure.

Therefore, the LUMet velocity measurement can be used to monitor texture changes that affect the  $W_{400}$  texture coefficient. With a suitable calibration, (by measuring the velocity in two different samples of the same material that have different but known textures), the LUMet can be used to monitor the value of  $W_{400}$  during thermo mechanical processing.

For orthotropic aggregates of hexagonal crystallites, Voigt, Reuss and Hill models predict that

$$C_{33} = D + EW_{200} + FW_{400}$$

where D, E, and F are also fitted to the data in practice. In this case, the two texture coefficients,  $W_{200}$  and  $W_{400}$  have a simple physical meaning: they represent the probability of finding the single crystal c axis pointing the in thickness direction. The  $W_{200}$  coefficient represents a broader distribution (more scatter around the thickness direction) than the  $W_{400}$  coefficient.

### 5.2.2 Measuring texture changes

To measure texture changes in cubic or hexagonal materials, it is necessary that the single crystal be anisotropic so that velocity changes with propagation direction (tungsten is a rare case where the anisotropy is near zero) and that the texture change affects either the  $W_{200}$  (for hexagonal materials) or  $W_{400}$  texture coefficients.

The "fractional change" in texture, can then be measured from the fractional change in  $C_{33}$ , i.e. from the fractional change from the initial value to the final value of  $C_{33}$ . Let  $C_{33}^i$  and  $C_{33}^f$  be the initial and final values of  $C_{33}$ , respectively, then

$$\text{Fractional texture change} = (C_{33} - C_{33}^i) / (C_{33}^f - C_{33}^i)$$

It is up to the LUMet user to determine if this concept is meaningful in the context of his application. However, any time that the change in the  $W_{400}$  (or  $W_{200}$ ) coefficient is proportional to the volume fraction that is transformed, then the Fractional texture change defined above is meaningful.

In general,  $BW_{400} \ll A$ ,  $BW_{400} \ll C$ ,  $EW_{420} \ll D$ , and  $FW_{400} \ll D$ . Because velocity is proportional to the square of  $C_{33}$ , a simple Taylor's expansion of the above formula shows that the fractional texture change can also be written

$$\text{Fractional texture change} = (V_{33} - V_{33}^i) / (V_{33}^f - V_{33}^i),$$

where  $V_{33}^i$  and  $V_{33}^f$  be the initial and final values of  $V_{33}$ , respectively.

## References

Ryong-Joon Roe. Description of Crystallite Orientation in Polycrystalline Materials III. J. Appl. Phys. **36** (6) 2024-2031, 1965.

H.-J. Bunge. Texture Analysis in Materials Science. Translated by P. R. Morris. Butterworths, London, 1982.

Voigt averages for orthotropic aggregates of cubic crystallites: C. M. Sayers. J. Phys. D: Appl. Phys. **15** (1982) 2157-2167.

Reuss and Hill averages for orthotropic aggregates of cubic crystallites: A. J. Allen et al. J. Appl. Phys. **54** (2) Feb. 1983, 555-560.

Voigt, Reuss, Hill averages for orthotropic aggregates of hexagonal crystallites: Y. Li and R. B. Thompson. J. Appl. Phys. **67** (5) 1 March 1990 p. 2663-2664.

Technique to measure the  $C_{44}$  and  $C_{55}$  elastic constants: A. Moreau, D. Lévesque, M. Lord, M. Dubois, J.-P. Monchalán, C. Padioleau, and J. F. Bussière. "On-line Measurement of Texture, Thickness, and Plastic Strain Ratio Using Laser-Ultrasound Resonance Spectroscopy." *Ultrasonics* **40**, (2002) p. 1047-1056.

Review of ultrasonic measurements of texture in cubic materials: R. B Thompson et al. Metall. Trans. **20A**, Nov. 1989, 2431.

Ultrasonic measurement of texture in Zr (a hexagonal material). Also contains a graphical representation of the meaning of texture coefficients for hexagonal materials: A. Moreau, P. J. Kielczynski, J. F. Bussière, and J. H. Root. "Ultrasonic Characterization of Texture in Pure and Alloyed Zirconium." *Nondestructive Characterization of Materials VI*. Edited by Robert E. Green, Jr., Krzysztof J. Kozaczek, and Clayton O. Ruud. (Plenum Press, New York, 1994) p. 119-127.

Ultrasonic measurement of texture in hexagonal material: P. J. Kielczynski, A. Moreau and J. F. Bussière. "Determination of Texture Coefficients in Hexagonal Polycrystalline Aggregates with Orthorhombic Symmetry Using Ultrasounds." *Journal of the Acoustical Society of America* **95**, 2 (February 1994) p. 813-827.

## **5.3 Isothermal processing (mostly recrystallization)**

Isothermal processing differs from non-isothermal processing in that it usually does not involve phase transformations other than the precipitation of some minor phase. Isothermal processing can be monitored either by monitoring grain growth or texture changes. However, internal friction, annealing, or precipitation can also affect velocity and attenuation measurements.

## References

M. Maalekian, R. Radis, M. Militzer, A. Moreau, W. J. Poole. "In situ measurement and modeling of austenite grain growth in Ti/Nb-microalloyed steel." *Acta Materialia* **60** (2012) p.1015-1026.

S. Sarkar, A. Moreau, M. Militzer, and W. J. Poole. "The evolution of austenite recrystallization and grain growth using laser-ultrasonics" *Metallurgical and Materials Transactions A* **39** (April 2008) p. 897-907.

A. Moreau, C. Bescond, S. Bolognini, M. Lord, S. Kruger, and C.-S. Man. "Inline Measurements of Texture and Recrystallization on Aluminum Alloys" *Proceedings of the Materials Science and Technology 2005 Conference*, Pittsburgh, 25-28 September 2005, p. 3-10. CD-ROM. Also published in *Proceedings of the World Conference on Nondestructive Testing 2004*. Montreal QC, 30 August – 3 September 2004. CD-ROM.

S. E. Kruger, G. Lamouche, A. Moreau, and M. Militzer. "Laser Ultrasonic Monitoring of Recrystallization of Steels" *Proceedings of the Materials Science and Technology 2004 Conference*, p. 809-812. CD-ROM.

André Moreau, Christophe Bescond, Stéphane Bolognini, Martin Lord, Silvio Kruger. "Inline Measurements of Texture and Recrystallization at Commonwealth Aluminum Corporation" in *Textures in Strip-Cast Aluminum Alloys: Their On-Line Monitoring and Quantitative Effects on Formability*. Final DOE Technical Report DE-FC07-00ID13902. July 27, 2003.

S. E. Kruger, A. Moreau, M. Militzer and T. Biggs. "In-Situ, Laser-Ultrasonic Monitoring of the Recrystallization of Aluminum Alloys" *Thermec 2003 International Conference on Processing & Manufacturing of Advanced Materials*, Part 1. Edited by T. Chandra, J. M. Torralba and T. Sakai. (Trans Tech Publications Ltd., Uetikon-Zurich, 2003). p. 483-488. Re-published in *Materials Science Forum* 426-432. (<http://www.scientific.net>).

G. Lamouche, S. E. Kruger, L. Gille, N. Giguère, S. Bolognini and A. Moreau. « Laser-Ultrasonic Characterization of the Annealing Process of Low-Carbon Steel" *Review of Progress in Quantitative Nondestructive Evaluation* **22B**. Edited by D. O. Thompson and D. E. Chimenti (American Institute of Physics, Melville NY, 2003). p. 1681-1688.

S. E. Kruger, A. Moreau, M. Militzer, and T. Biggs. "Monitoring the Annealing of an Al-Mg Alloy by Laser-ultrasonics." *Light Metals 2002 Métaux Légers*. Proceedings for the 2002 Light Metals Symposium of the Conference of Metallurgists of the CIM, Montreal, Canada, 11-15 August 2002. Edited by T. Lewis. Canadian Institute of Mining, Metallurgy and Petroleum, Montreal, 2002. p. 161-171.

M. Dubois, M. Militzer, A. Moreau, and J. F. Bussière, "A New Technique for the Quantitative Real-Time Monitoring of Austenite Grain Growth in Steel", *Scripta Materialia* **42**, 9 (2000) p. 867-874.

A. Moreau, D. Lévesque, M. Lord, M. Dubois, J.-P. Monchalain, C. Padioleau, and J. F. Bussière. "On-line Measurement of Texture and Plastic Strain Ratio on Steel Sheets Using Laser-Ultrasonics." *Proceedings of the Twelfth International Conference on Textures of Materials*,

Montreal, Canada, 9-13 August 1999. Edited by Jerzy A. Szpunar (NRC Research Press, Ottawa, 1999). p. 493-498.

M. Dubois, M. Militzer, A. Moreau, and J.F. Bussière. "Real-Time Monitoring of Austenite Grain Growth in Steel Using Laser-Ultrasonics." *Grain Growth in Polycrystalline Materials III*, Proceedings of Third International Conference on Grain Growth, Pittsburgh, PA, 14-19 June 1998. Edited by H. Weiland, B. L. Adams and A. D. Rollett. p. 593-598.

## 5.4 Non-isothermal processing

Non-isothermal processing often involves phase transformations. Phase transformations are observed usually by a sudden change in ultrasound velocity because the two phases do not have the same elastic constants or because of a sudden modification of the lattice spacing which will have a measurable effect on sample thickness.

On phenomena, such as recrystallization or grain growth can be monitored either by monitoring grain growth or texture changes. However, internal friction, annealing, or precipitation can also affect velocity and attenuation measurements.

### 5.4.1 Recrystallization

#### References

S. Sarkar, M. Militzer, W. J. Poole and A. Moreau. "Austenite Grain Size Measurements by Laser-Ultrasonics on a Modern Low Carbon Steel" Proceedings of the International Symposium on Advanced Steels, Edited by J. A. Szpunar and H. Li, Canadian Institute of Mining, Metallurgy and Petroleum, Montreal, QC, Canada, 1-4 October 2006, p. 119-130.

A. Moreau, C. Bescond, S. Bolognini, M. Lord, S. Kruger, and C.-S. Man. "Inline Measurements of Texture and Recrystallization on Aluminum Alloys" *Proceedings of the World Conference on Nondestructive Testing 2004*. Montreal QC, 30 August – 3 September 2004. CD-ROM. Republished in *Proceedings of the Materials Science and Technology 2005 Conference*, Pittsburgh, 25-28 September 2005, p. 3-10. CD-ROM.

A. Moreau, C. Bescond, S. Bolognini, M. Lord, S. E. Kruger. "Inline Measurements of Texture and Recrystallization at Commonwealth Aluminum Corporation" in *Textures in Strip-Cast Aluminum Alloys: Their On-Line Monitoring and Quantitative Effects on Formability*. Final DOE Technical Report DE-FC07-00ID13902. July 27, 2003.

### 5.4.2 Phase transformations and austenite decomposition

#### References

S. E. Kruger, A. Moreau, and J.-P. Monchalín. "Laser-Ultrasonic Monitoring of Metallurgical Transformations in Advanced Ultra-High Strength Steels" *Proceedings of the First International Symposium on Laser Ultrasonics: Science Technology and Applications*, Montreal QC, 16-18 July 2008. Published at <http://www.ndt.net/>, October 2008 issue.

S. E. Kruger and E. B. Damm. "Monitoring austenite decomposition by ultrasonic velocity". *Materials Science and Engineering A* 425 (2006) p. 238-243.

Marc Dubois, André Moreau, and Jean F. Bussière. "Ultrasonic Velocity Measurements During Phase Transformations in Steels Using Laser-Ultrasonics." *Journal of Applied Physics* **89**, 11 (1 June 2001) p. 6487-6495.

M. Dubois, A. Moreau, M. Militzer and J. F. Bussière. "Laser-Ultrasonic Monitoring of Phase Transformations in Steels." *Scripta Materialia* **39**, 6 (1998) p. 735-741.

M. Dubois, A. Moreau, M. Militzer and J. F. Bussière. "Monitoring of Attenuation During Phase Transformations in Steel Using Laser-Ultrasonics." *Nondestructive Characterization of Materials VIII*. Edited by Robert E. Green, Jr. (Plenum Press, New York, 1998). p. 323-328.

## 5.5 Internal friction

The LUMet is not the ideal setup to measure internal friction because the effect of internal friction on attenuation and velocity is often small compared to the effect of changes in grain size or texture. Nevertheless the effect of internal friction is sometimes observable or may contribute a small fraction (and even sometimes a large fraction) of the total effect that is observed. Internal friction is usually a contributing factor when monitoring phenomena involving magnetoelasticity (below the Curie temperature) or dislocations (such as during recovery). The references below can be used as a starting point to understanding the effect of internal friction on LUMet measurements.

### References

S. Bolognini and A. Moreau. "Ultrasonic absorption in ultra-low carbon steel." *Journal of Applied Physics* **94**, 6 (15 September 2003) p. 3771-3780.

S. E. Kruger, S. Bolognini, G. Lamouche and A. Moreau. Monitoring Microstructure Evolution of Nickel at High Temperature. *Review of Progress in Quantitative Nondestructive Evaluation* **21B**. Edited by D. O. Thompson and D. E. Chimenti (American Institute of Physics, New York, 2002). p. 1518-1525.

A. Moreau, M. Lord, D. Lévesque, M. Dubois, and J. F. Bussière. "Laser-Ultrasonic Absorption Measurements in Low Carbon Steels." *Journal of Alloys and Compounds* **310**, 1-2 (2000) p. 427-431, *Proceedings of the Twelfth International Conference on Internal Friction and Ultrasonic Attenuation in Solids*, Buenos Aires, Argentina, 18-23 July 1999.

## 5.6 Review articles and other references

Dynamic Systems Inc. "LUMet – Laser Ultrasonics Sensor for In-situ Metallurgical Studies"

A. Moreau, S. E. Kruger, M. Côté, and P. Bocher. "In-situ monitoring of physical simulations using laser-ultrasonics" *Proceedings of the First International Symposium on Laser Ultrasonics: Science Technology and Applications*, Montreal QC, 16-18 July 2008. Published at <http://www.ndt.net/>, October 2008 issue.

A. Moreau and S. E. Kruger. "In-situ monitoring of physical simulations using laser-ultrasonics" *Proceedings of the 5<sup>th</sup> International Conference on Physical and Numerical Simulation of Materials Processing*. Zhengzhou, China, 23-27 October 2007.

A. Moreau. "Laser-Ultrasonic Characterization of the Microstructure of Aluminum" *Proceedings of the 10<sup>th</sup> International Conference on Aluminum Alloys* 9-13 July 2006, Vancouver BC. Edited by W. J. Poole, M. A. Wells, D. J. Lloyd. Also published in *Materials Science Forum* **519-521** (2006) p. 1373-1378 (<http://www.scientific.net>) Trans Tech Publications, Switzerland.

S. E. Kruger, A. Moreau, C. Bescond and J.-P. Monchalin. "Real-time Sensing of metallurgical Transformations by Laser-Ultrasound" *Proceedings of the World Conference on Nondestructive Testing 2004*. Montreal QC, 30 August – 3 September 2004. CD-ROM.

André Moreau, Martin Lord, Daniel Lévesque, Marc Dubois, Jean Bussière, Jean-Pierre Monchalin, Christian Padioleau, Guy Lamouche, Teodor Veres, Martin Viens, Harold Hébert, Pierre Basséras, Cheng-Kuei Jen. "AISI/DOE Advanced Process Control Program. Vol. 4 of 6: On-line, Non-destructive Mechanical Property Measurement Using Laser-Ultrasound" Edited by Joe Vehce, American Iron and Steel Institute, 2001, 106 pages.  
[http://www.osti.gov/energycitations/product.biblio.jsp?osti\\_id=794988](http://www.osti.gov/energycitations/product.biblio.jsp?osti_id=794988)

M. Dubois, A. Moreau, A. Dawson, M. Militzer and J. F. Bussière. "Laser-Ultrasonic Measurement of Microstructure Evolution During Metals Processing." NATO RTO Workshop I - Intelligent Processing of High Performance Materials, Brussels, Belgium, 13-14 May 1998. Published in RTO MP-9 (November 1998), p. 11.1-11.9.