APPLICATIONS OF LASER ULTRASONICS (LUS) TO STUDIES OF MICROSTRUCTURAL AND MECHANICAL PROPERTIES OF METALS

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Abstract: During the last three years the Swedish Institute for Metals Research (SIMR) has done extensive work on applications of laser generated ultrasound (LUS). This paper reports results from several experiments mainly aimed at industrial on-line applications.

LUS has been used to determine grain sizes in commercial low-carbon (LC) and stainless steels. The analysis method employs a scattering theory that yields grain sizes in absolute values, without any free-parameter fits or calibration curves. The correlation is linear to grain sizes determined with optical microscopy.

Dynamic changes during annealing of cold rolled ferritic steels, such as phase transformation and recrystallisation, have been evaluated using the LUS technique. These phenomena are clearly revealed from changes in ultrasonic velocity due to changes in crystallographic texture.

LUS has also shown to be an alternative method to determine elastic properties of metals. By applying an isotropic approximation to the material symmetry, parameters like Young's modulus and Poisson's ratio may be calculated for rolled sheets. The results are compared with a method based on mechanical resonance and standard tensile testing.

Introduction: Ultrasound is a stress wave and the characteristic features of its propagation depend on several mechanical properties of the propagation medium. Characteristics of the ultrasonic source together with specimen geometry and position of the detection spot with respect to the generation spot determine which waves will be registered. Common ultrasonic wave types are bulk (P and S) waves, surface (Rayleigh) waves and plate (Lamb) waves.

Ultrasonic waves that travels through a metal sample interact with the microstructure and the registered wave spectra will carry information about the microstructural state of the penetrated volume. An ultrasonic spectrum is mainly characterised by the time of arrival for various echoes, i.e. the wave velocities, and the amplitude of the successive declining echoes (attenuation). For certain applications more detailed information can be extracted using a frequency analysis which reveals velocity or attenuation for specific frequency components within a specific wave.

Two principal types of microstructure information can be obtained. The wave velocity depends on the material type – its phase constitution and crystallographic texture. The wave attenuation depends mainly on grain size and on the dispersion of second phases if these are present. Table 1 lists various parameters that can be measured from ultrasonic velocity and attenuation.

Wave characteristics:	**Depends on:**	**Features that can be registered:**
VELOCITY (time of arrival)	Elasticity of single crystals Crystallographic texture	Recrystallisation Phase transformation Dimension Temperature
ATTENUATION	Scattering and absorption from various microstructure components	Grain size Second phases Precipitation Dislocations

Table 1: Parameters that can be measured from ultrasonic velocity and attenuation

Ultrasonic velocity is determined by the elasticity of the polycrystalline aggregate and is hence dependent on a combination of the elastic properties of the crystal structure together with as the crystallographic texture. The ultrasonic velocity will as a consequence differ in different directions of measurement for a textured sample. In general, metals always show more or less pronounced textures and, in principal, all processing steps such as deformation and annealing will alter the microstructure and also the texture, so affecting the ultrasonic velocity.

Ultrasonic waves can penetrate several centimetres in metals although the wave amplitude becomes progressively attenuated by the microstructure as it travels over longer distances. A propagating wave is scattered at grain boundaries and absorbed by dislocations, grain boundary scattering being the dominant effect. The wave will also be attenuated by dispersions of second phases or defects. The attenuation experienced is dependent on grain size such that large grains have a dominating influence; also higher frequencies are more strongly affected and will decline more rapid within the spectra. Depending on the wavelength/grain size ratio (λ/D), the attenuation α can be described as $\alpha \sim D^3 f^4$ for $\lambda >> D$ or $\alpha \sim Df^2$ for $\lambda \approx D$ [4].

An ultrasonic spectrum will hence contain various amounts of a specific frequency component depending on the grain size. The literature contains many examples where ultrasonic attenuation shows correlation with grain size. Theories relating grain size and ultrasonic attenuation can be found in the literature e.g. [4].

Microstructural changes that occur during annealing - recrystallisation, phase transformations and grain growth - are among the most important metallurgical processes occurring in metals from an industrial point of view and extensive efforts are spent around the world trying to describe mechanisms and kinetics for these processes. For physically based models it is extremely important to have relevant microstructure data in order to verify proposed models or hypotheses. Information about microstructure development during annealing is generally collected from interrupted annealing trials followed by sample sectioning, preparation and microscopy work. This is very time consuming and some structures like austenite are very difficult or even impossible to maintain at room temperature. Several non-destructive techniques do exist to monitor recrystallisation continuously but they all suffer from limitations that restrict their use in an industrial environment. A continuous measure of the microstructural state on-line during industrial annealing would be of great importance for maintaining high quality and uniformity of properties in the final products. Today, material that does not fulfil correct specifications has to be downgraded or even reprocessed causing considerable economic losses.

The laser ultrasonic device at SIMR was designed and built by Accentus, UK and was installed at SIMR in February 2001. The design of the system allows high flexibility together with a high safety standard so the system is fully enclosed and most measurement set-ups are done from a control unit and a PC. Figure 1 show an overview of the system and Table 2 gives the most important technical specifications. A lens system facilitates the IR beam to be positioned either on opposite or the same side of the sample as the detection laser. There is also a loop-controlled device that controls the power of the incident detection laser beam. This device helps the system to manage variations in amount of light reflected from the sample surface during for example oxidation due to heating or geometric changes during compression.

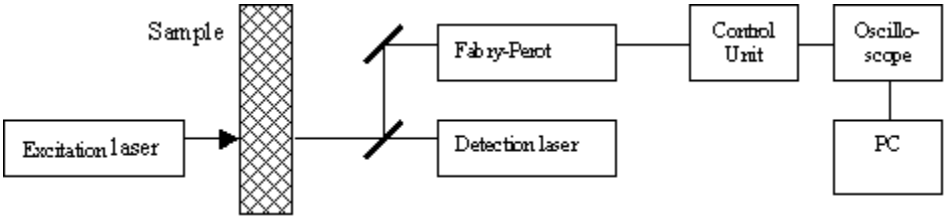

Figure 1: Overview of the LUS system at SIMR.

Excitation laser	Continuum "Surelite I", YAG. Wavelength: 1064nm. Pulse duration: 5ns. Pulse energy: 450mJ (ablative regime). Repetition rate: 20Hz.
Detection laser	Coherent "Verdi", Nd:YVO4. Wavelength: 532nm. Line width: <5MHz. Maximum power: 5W
Interferometer	Confocal Fabry-Pérot (working in transmission or reflection mode) Frequency range: 1-100MHz.
Data aquisition	1 GHz oscilloscope and PC

Table 1: Technical specifications of the LUS system at SIMR

Results: The elastic modulus is possible to calculate using LUS since the characteristic features of the propagation of ultrasonic waves depend on the elastic properties of the propagation medium, together with its density. In particular, propagation velocity is a function of the elastic tensor. A relation between phase velocities and tensor components can be found by solving Christoffel's equation [1]. Exact analytical solutions to Christoffel's equation exist only for simple cases of isotropic solids and some low index directions in high symmetry crystals. For all other cases approximations or numerical solutions must be used.

For the isotropic case, a simple expression relates the elastic modulus to two bulk wave (P, S) velocities. In turn the Rayleigh wave velocity may be simply expressed in terms of the same bulk wave velocities. This applies for Lamb waves as well, but only for the very first symmetric and asymmetric modes (S_0 and A_0) and in regimes where $\lambda >> d$ or $\lambda << d$, d being the sample thickness[1].

Hence, by using an isotropic approximation the elastic modulus can be calculated if one P and one S wave velocity is known. The P and S wave velocities may be found directly from the signal's time or frequency domain. In most cases, the P wave velocity is easy to extract direct but the S wave is often hard or impossible to

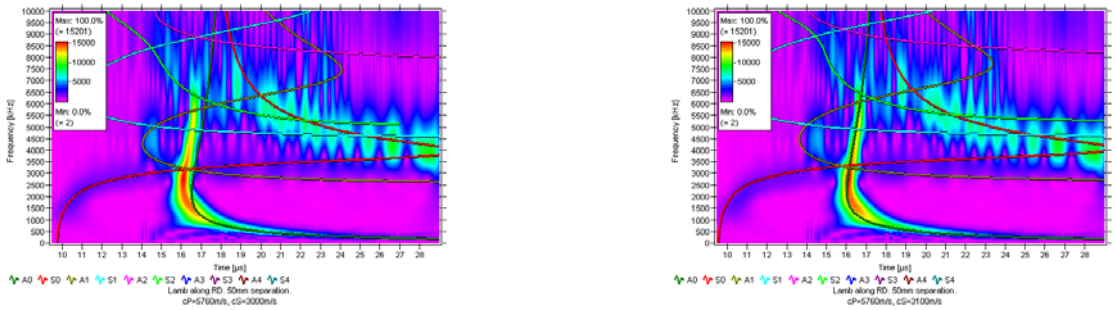

Figure 2 : Lamb waves propagating in the rolling direction in a 0.6mm steel sheet. Experimental data compared to theoretic dispersivity of isotropic Lamb waves (lines). P and S velocity: Left [5760m/s, 3000m/s], Right [5760, 3100m/s].

find and identify. If so, the S wave velocity may be derived from the arrival time of the Rayleigh wave and/or the Lamb wave (at $\lambda >> d$ or $\lambda << d$) by using the isotropic approximation. However, a better method is to compare Lamb waves from experiment to the theoretic dispersivity of Lamb waves. For an isotropic approximation this dispersivity may be calculated if P and S velocity are known. So, if the P velocity is known the S velocity may be derived by finding the S velocity that fits experimental Lamb waves to theoretic Lamb waves. This is shown in Figure 2 above. Figure 3 summarises the LUS measurements of the elastic modulus. There is also a comparison to results from other methods such as tensile testing and a method based on mechanical resonance [2,3]. All measurements consider hot or cold rolled sheets where P wave velocity was measured in the normal direction (ND, to rolling plane) and S wave velocity was measured in either rolling direction (RD) or perpendicular to RD.

Recrystallisation was studied in stainless steel plates that had been annealed industrially where different degrees of recrystallisation would have resulted in a large scatter in the mechanical properties. SEM-BSE micrographs were used to measure the volume fraction of recrystallisation and P-wave velocities were determined through the plate. In this case it is seen that both wave velocity and tensile strength vary almost linearly with the fraction of recrystallisation, Figure 4.

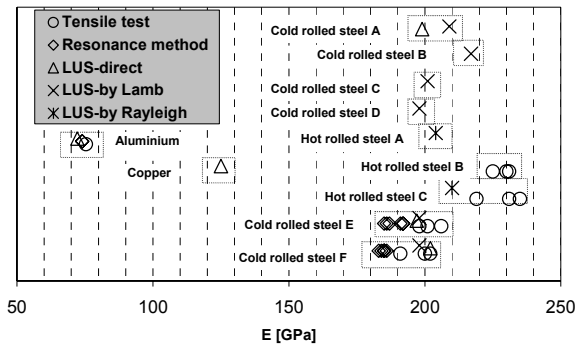

Figure 3: Summary of measurements of elastic modulus using LUS.

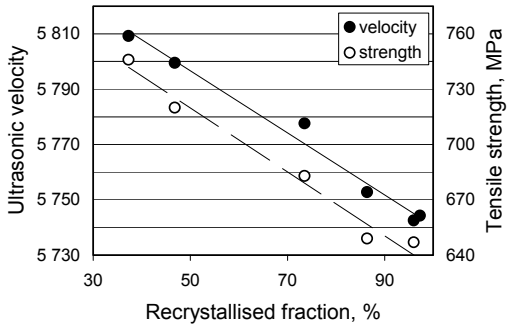

Figure 4: P wave velocity and tensile strength vs. fraction recrystallised for annealed stainless steel plates

Heating trials were performed with the aim of monitoring recrystallisation and phase transformation kinetics in real time, simulating continuous annealing of cold rolled steel sheets. Without using brazing atmosphere, the samples were heated to about 1100°C at a rate of 8°C/s. Epicentric positioned lasers gave spectra of reverberating P waves that enabled accurate time-of-flight measurements. Figure 5 show examples where the

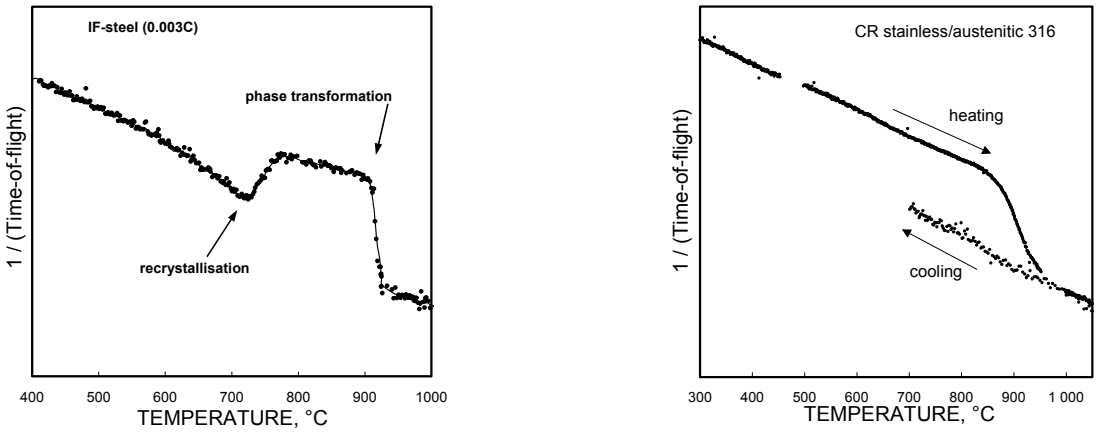

Figure 5: Time-of-flight (~velocity) for a P wave monitored during annealing of different steel types. Heating rates were 8°C/s.

ultrasonic velocity is plotted against temperature for annealing of two different steel types. A continuous decrease in velocity with temperature is registered in all figures due to the effect of temperature on elasticity. Other changes are, however, related to changes in the microstructure. The left graph shows annealing of a cold rolled interstitial-free (IF) steel. The distinct increase in velocity at about 750°C corresponds to recrystallisation and the marked drop above 900°C is from transformation from ferrite to austenite. The right graph concerns annealing of a cold rolled austenitic stainless steel. The texture development is different from the carbon steels and the velocity shows a decrease upon recrystallisation. The steel is austenitic at room temperature so no phase transformation takes place. Also a part of the cooling curve was traced; the velocity drop is permanent upon cooling since the texture is constant.

Grain size has been studied in samples from a hot rolled 4mm low carbon steel sheet collected from different band positions. The samples had various grain sizes – measured using optical microscopy and linear intercepts. An epicentric configuration with the two lasers opposite each other was used for the ultrasonic measurements. Ultrasonic spectra showed compression wave echoes – P1, P3, P5, P7… with successively weaker amplitude. Ultrasonic attenuation $\alpha(f)$ was evaluated for different frequencies using the FFT of two successive echoes:

$$\alpha(f) = \frac{1}{2L} \cdot \log \frac{FFT\left[y\left({}^{n}P\right)\right]}{FFT\left[y\left({}^{n+1}P\right)\right]} \quad , \qquad \begin{cases} y\left({}^{n}P\right) = \text{time domain signal of n}^{\text{th}}\ \text{Pwave} \\ L = \text{propagation distance between successive echoes} \end{cases}$$

Theoretical attenuation was then calculated for different frequencies and grain sizes using scattering theory [4] together with single crystal elasticity data. By comparing experimental values for attenuation with calculated

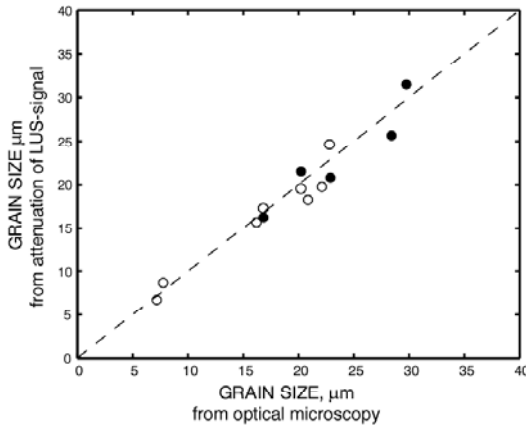

Figure 6: Grain sizes in hot rolled low carbon steels determined from attenuation of ultra-sonic waves versus linear intercept measurements from optical spectroscopy.

results it was possible to determine absolute values of grain size, without the use of any fitting parameters. The results give an excellent correlation as shown in Figure 6 where ultrasonic grain size is plotted versus grains size determined metallographically. Further details from this work were reported elsewhere [5].

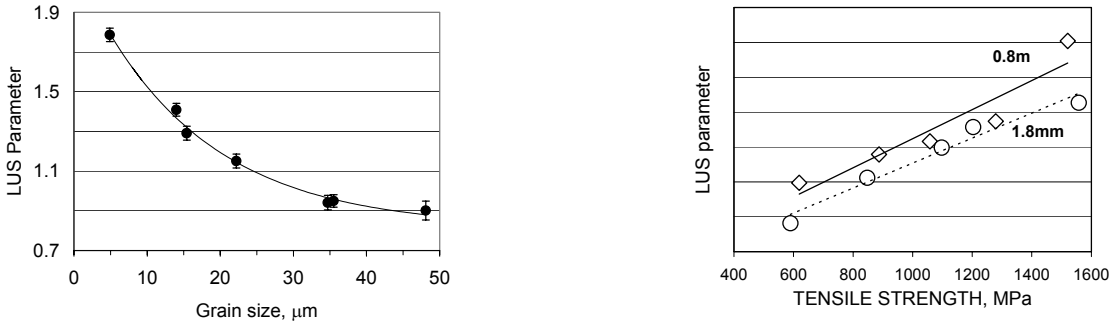

Figure 7: Ultrasonic attenuation versus grain size for 2mm thick stainless steel sheets.

For thin samples where the echoes overlap it is not possible to extract FFTs for individual peaks. An alternative approach has proved to give good correlation to grain size although it cannot provide absolute values of grain size. This approach is also more automatic and is well adopted for large amount of data sets. Instead of handling individual peaks, the FFT is applied to the whole spectrum. High frequencies are assumed to be most sensitive to grain size variations and an integral over an assumed frequency range is used. Results using this approach are shown in Figure 7. The left graph shows the LUS-parameter versus grain size for 2mm stainless samples. The right graph shows LUS-parameter versus strength for samples from steel where grain size is the dominating parameter for controlling strength.

Discussion: Figure 3 show values of the elastic modulus *E* measured using different techniques. The *E* values measured using LUS agrees well with *E* values found in literature and values achieved with other techniques. The accuracy of the LUS-technique used in this application depends on the precison of velocity measurements and whether the isotropic approximation is applicable or not. By using cross correlation methods, the determination of the P wave velocity can be done very accurately – to less than 0.1%. The accuracy of the measured velocity of the S wave depends mainly on the thickness of the sample. For thick (>2mm) sheets, the S velocity may be found by a) using an epicentric laser configuration and apply cross-correlation on successive S echoes, or b) by separating

generation and detection spots for determination of the Rayleigh wave velocity which in turn will be used to derive the S velocity. Neither method is very exact. The S wave echoes are often too weak to be used in a cross correlation. The Rayleigh wave method suffers from a similar problem since there is only one wavefront and nothing to cross correlate to. The accuracy of the S velocity using this method was estimated to 7-8%. For thin sheets, the S velocity may be found by a) using an epicentric laser configuration and use the signal's frequency domain to find the 'resonance' of the S wave and determine its velocity, or b) by separating generation and detection spots for detection of the Lamb waves which in turn will be used to derive the S velocity. The accuracy of the S velocity using the first method was estimated to <1.5%. The latter method has shown to be applicable only for Lamb wave propagation along RD. As shown in Figure 2, experimental data show a very good correlation to the theoretic dispersivity at 'correct' S velocity. However, the graph shows Lamb wave propagation in RD and for wave propagation perpendicular to RD it is not possible to get a fit for any value on S or P wave velocity. This indicates that the isotropic approximation is applicable only for propagation along RD. The accuracy of the S velocity using this method was estimated to <1%.

The results show that it is possible to monitor both recrystallisation and phase transformation in real-time using Laser-Ultrasonics. The velocity change is determined by the actual texture development taking place and it is evident that different steel types behave in different ways, as should be expected. Tracing changes in velocity are straightforward which makes data analysis quick and simple. No effort has been put on measuring the velocity in exact numbers and compensation for phenomen like thermal expansion has not been considered. At this level, the main object has simply been to classify the status of a specimen as recrystallised/phase transformed or not. All experiments were performed without the use of brazing atmosphere. During heating, some of the examined steel types suffered from oxidation and reduced reflectivity that caused difficulties for the wave detection device. The device for automatic control of power of detection light managed most of the cases but samples suffering from severe oxidation had to be Ni-coated to faciliate measurements.

This paper presents two approaches for determination of grain size in steel. The method that combines experimental data and scatter theory [4] results in absolute values of grain size that without use of any fitting parameters gives excellent correlation. However, since the method uses FFTs of individual peaks there is a lower limit for specimen thickness to allow for P wave peaks to be well separated. The approach for thinner samples is to study integral values of high frequency components of the signal since theory states that the frequency dependent attenuation is strongly affected by grain size. The choice of suitable frequency range varies for different steel types and has to be set individually. This method provides good correlation to grain size although it cannot provide absolute values of grain size. Comparing the two methods; Being able to extract grain size in absolute values is excellent, but for some in-situ applications relative values are sufficient. The data analysis for the latter method is far more simple, quick and also applicable to most specimen thicknesses.

Conclusions: The ability to measure mechanical and microstructural properties continuously and non-intrusively means that the LUS technique can save much time and effort compared to normal procedures which necessitate repeated heat treatments and/or specimen preparation. Laser-Ultrasonics is a technique that permits in-situ studies of microstructure changes during annealing using remote sensing. Ultrasonic velocity is governed by the elastic properties of the polycrystal sample, which allows texture changes during recrystallisation to be monitored. Both recrystallisation and phase transformations have been registered in real time during in-situ annealing for a variety of steel types showing different texture development. Attenuation measurements have been shown to give a good measure of grain size in metals, in agreement with theoretical calculations. This opens the possibility for making continuous measurements of grain growth during annealing treatments.

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