Characterization of Microstructure Variations by Laser-Ultrasound during and after the Heat Treatment of Metals

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Abstract. We demonstrate the potential of laser ultrasound for non-destructive testing applications in steel production and processing in two use cases: (1) Imaging of the grain structure transition between the thermally hardened surface and the unaffected core material with a comparison to sectional views by an optical microscope. We found very good correlation between the laser ultrasonic image and the microscopic view at the cross section. (2) In situ monitoring of Poisson's ratio of steel sheets during thermal processes by evaluation of resonant frequencies of Lamb waves. In our ongoing research the results will be correlated to metallurgical properties of interest.

1 Introduction

Extended knowledge about steel production and processing is the basis for meeting the increasing requirements in ever shorter development cycles and increasing energy efficiency, where in particular the steel industry has a high potential for savings. Laser-induced ultrasound is a non-contact technology that can provide relevant, supplementary insights into the complex microscopic processes of metals [1][2]. In our presented applications laser pulses of 5-10 ns and up to 100 mJ per pulse are fired at the surface from several centimetres distance and thus generate broadband ultrasonic waves by thermoelastic expansion and slight ablation. The surface displacement caused by the generated ultrasound waves is detected by a laser vibrometer. In the presented cases we used a robust, industrial interferometer developed to measure on rough surfaces [3][4]. Laser ultrasound (LUS) typically provides higher bandwidth signals than contact ultrasound and also allows for remote measurements on hot samples [5][6]. These properties are of great importance for the presented applications.

We show two use cases, where we use LUS to analyse the microstructure and the metallurgical phases in steel. These are (1) imaging of hardness penetration depth, (2) determination of the Poisson’s ratio of sheet steel during heat treatment. The state-of-the-art technique to obtain information on the microstructure of metals are etched microsections which can be prepared to show grains, texture, phase, precipitations and more [7]. To receive this information the sample needs to be cut and specifically prepared for microscopic analysis [8]. During heat treatment this is not directly possible. Instead, samples are quenched at certain points of the thermal process before being analysed ex situ. This requires a model-based reconstruction of the high-temperature condition, which introduces uncertainties and provides results only at the selected points [9].

LUS has the potential to provide in situ information with a sampling rate up to hundreds of Hz. The challenge and focus of current investigations are the correlation of observable characteristics of the
ultrasonic wave propagation with metallurgical properties of interest. We want to show that LUS can complement existing methods for the determination of the hardness penetration depth \[10\][11][12][13] and provide cost saving alternatives for in situ methods having their individual advantages and disadvantages.

2 Use case 1: Imaging of hardness penetration depth

Thermal hardening is commonly used to increase the surface resistance of steel components in heavily stressed areas such as bearings. Carbon-containing steel is heated and then cooled abruptly, so that a martensitic structure, strained by the carbon, is formed from the austenitic phase. We have developed a LUS setup, Figure 1, and applied a method to measure and image the grain structure transition between the finer grained, hardened zone near the surface and the unaffected core. For excitation we used a pulsed Nd:YAG laser, the Quantel Q-smart, with 532 nm, 10 ns pulses and a maximum energy per pulse of 430 mJ. The LUS detection was the Tecnar Lus system with an 80 μs long-pulse laser at 1064 nm.

The short generation laser pulse produces a broadband acoustic wave package inside the sample. According to scattering theory the waves are scattered at the grain boundaries due to changing elastic properties. Scattering strongly depends on the average grain diameter \(D\) and the acoustic wavelength \(\lambda\), thus, for the evaluated bandwidth of 10 MHz to 40 MHz, it mainly occurs in the non-hardened core regions with coarser grain structure. For the grain diameter in the hardened layer holds \(D \ll \lambda\) and therefore the acoustic waves are scattered very weakly compared to the core region. This behavior can be seen in Figure 2 (right). It shows a B-Scan with the lateral position on the sample plot on the x-axis, the depth down into the sample on the y-axis and the signal amplitude in grey scale. The backscattered signal starts occurring at approximately 3 mm depth which is in good agreement with the microscopic view shown in Figure 2 (left).

In some cases, the hardened zone is already optically visible on the cross section as it is the case with this induction hardened steel sample Figure 2 (left). A scan line of 15 mm length is performed in the vicinity of this sectional cut consisting of 150 point measurements with a total acquisition time of 15 s. There is excellent agreement between the depth and shape of the transition zone of the LUS image compared to the microscopic view on the cut surface. It can be clearly seen that the coarser grain of the unaffected core causes stronger back-scattering than the hardened layer closer to the surface, whereby

![Figure 1. Laboratory setup for LUS measurements on a thermally hardened part of a crankshaft. The laser paths of excitation (green) and detection (red) are indicated and intersect each other at the current measurement position.](image)
the first millimetre of the ultrasonic image can hardly be evaluated due to the intensive ultrasonic generation effect and it appears as horizontal white lines in the image.

![Ultrasonic image](image)

**Figure 2.** Left: Microscopic view on cross section of an induction hardened steel sample with visible fine grained (hardened) and coarser grained (unaffected) core. Right: Reconstruction of LUS data.

![Deformation and quenching dilatometer](image)

**Figure 3.** Deformation and quenching dilatometer based on Linseis L78 adapted for LUS application.

### 3 Use case 2: Determination of the Poisson’s ratio of sheet steel during heat treatment

Sheets or plates are a common product of the metal industry. This geometry sustains guided waves e.g. Lamb waves, which can be used in many NDT applications. In our presented approach we measure resonant frequencies of Lamb waves [14] [15][16][17] instead of the time-of-flight of longitudinal pulses. Using the central frequency of two resonances, the Poisson's ratio of a sample can be determined with high accuracy [15][16]. For this analysis knowing the thickness of the sample (which is subject to
thermal expansion during heat processes) is not required. The resonances occur in the range <10 MHz for typical thickness in the mm range. This allows for a strong limitation of the detection bandwidth and thus reduces electronic measurement noise compared to time-of-flight measurements.

A LINSEIS dilatometer type L78 has been adapted for in situ LUS [18][19] measurements, see Figure 3, adapted for inductive heating and cooling of sheets by a flat coil and both lasers coming confocally from the top window (2-3mm spot size each), see Figure 4.

The right side of Figure 5 shows a typical measured response spectrum with peaks caused by resonant zero group velocity (ZGV) resonances (~3 MHz) and by longitudinal thickness resonances (~6 MHz and ~9 MHz). Their frequency positions correlate to the local minimum (ZGV) and cut-off frequencies of the propagating modes (thickness resonances) in the dispersion curves of the sheet (Figure 5, left). The peaks could be resolved with satisfying SNR, without coming applying 50 mJ for the excitation pulses.

**Figure 4.** Left: LUS setup on dilatometer accessing the sheet sample through the top window. Excitation- (green) and detection laser (red) are indicated and aim at the same spot. Right: zoom of the steel sheet above flat, inductive coil with holes for gas cooling.

**Figure 5.** Left: dispersion curves for a 1 mm thick steel sheet with $c_L=6147.5$ m/s, $c_T=3329.3$ m/s and $\rho=7870$ kg/m$^3$. Right: response spectrum of sample at room temperature.
5

Figure 6. Left: Response spectra of a plate during a thermal cycle. Right: Temperature and calculated Poisson’s ratio.

Figure 6 shows the response spectra of a plate during a thermal cycle. From the maxima positions \( f_1 \) and \( f_2 \) of the peaks in Figure 5 we obtained the Poisson’s ratio \( \nu \) of the sample during the process, which is shown in the right subfigure. The data also reveals additional resonant modes appearing in certain ranges values of \( \nu \), which is in agreement with literature [14] and increases confidence in our results. The correlation of the Poisson’s ratio with metallurgical reference measurements is currently in progress.

4 Conclusion

LUS measurements were carried out on three different use cases to demonstrate the potential of this technology for the characterisation of steel properties. The results of use case 1 show the ability of LUS to image the hardness penetration depth on thermally hardened steel samples. The imaged depth is in very good correlation with the microscopic view on the cross section. By use case 2 we presented an approach for plate geometries to determine the Poisson’s ratio in situ which is independent of the plate thickness. This ongoing research aims to develop a robust method to correlate metallurgical parameters in sheet steel production.

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