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IN-SITU EVALUATION OF METALLURGICAL PHENOMENA USING LASER GENERATED ULTRASONIC WAVES

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Abstract

Over the past decade, laser ultrasonics has gained tremendous maturity to in-situ characterize microstructure evolution during thermo-mechanical processing of metals and alloys. In this technique, pulse lasers are used for the generation and detection of ultrasound in materials. The ultrasound wave properties are correlated with microstructure parameters such as grain size and phase fractions transformed or fraction recrystallized. Meanwhile the Laser Ultrasonics for Metallurgy (LUMet) system has been developed such that this technology can be used as a user-friendly tool in the laboratory. Attached to a Gleeble thermomechanical simulator, it allows for the systematic in-situ investigation of a large number of metallurgical phenomena observed in metals and alloys with complex microstructures including advanced steels, superalloys and titanium alloys. This contribution aims at demonstrating that this technology provides fast and reliable measurements of microstructure evolution during complex thermomechanical treatments including rapid heating and cooling as well as hot deformation conditions.

Introduction

The control of phase transformations and grain structure in metals is a crucial aspect in thermomechanical processing of advanced metallic materials used in transportation, infrastructure and energy applications. Real time measurements of microstructure including phase transformation, grain growth and recrystallization can therefore accelerate the development of microstructure modelling tools and advanced materials processing paths. Laser Ultrasonics for Metallurgy (LUMet) is a technology specifically developed for in-situ monitoring of metallurgical phenomena during thermomechanical processing of metals and alloys [1]. This non-destructive technique is fast and perfectly adapted to remotely characterize the evolution of the microstructure at high temperature. For more than two decades, the technology has been in constant development and is now validated to monitor various microstructure phenomena such as grain growth, phase transformation and recrystallization in steels and selected non-ferrous alloys [2-5]. In 2012, the first commercial LUMet system became available as an attachment to a Gleeble thermomechanical simulator and constitutes an innovative characterization platform for metallurgists to minimize the labour intensive metallographic studies [6].

During hot-rolling of steels, the austenite grain structure and subsequent austenite decomposition must be carefully controlled as it influences the final microstructure and resulting mechanical properties. In addition, recrystallization occurring during or between hot rolling passes is also of major importance as it influences the austenite structure, the subsequent phase transformation and the overall mechanical strength of the final product. All these microstructure changes can be assessed in real time with the LUMet technology.

The present paper reports on technical specifications of the LUMet system and describes some of the measurements carried out with LUMet to determine the evolution of the austenite grain structure during and after hot-deformation tests and the kinetics of austenite decomposition in low alloyed steels.

Experimental

The LUMet system

The laser ultrasonic inspection is conducted during thermomechanical processing with the LUMet attached to a Gleeble 3500. The LUMet system consists of a probe design to be safely attached to the rear door of the Gleeble chamber and of two enclosed cabinets containing optics and electronics required for light demodulation, digital conversion and data acquisition. A frequency-doubled, short pulse, Nd:YAG laser at a wavelength of 532 nm is used for the generation of a wide band ultrasound pulse. Thanks to the very short duration of the generation laser pulse (approximately 6 ns) the power-density of the light is high enough to generate a wide-band compressive wave by ablating a thin surface layer of the sample with a typical spot size of 2 mm. The ultrasound pulse propagates back and forth through the thickness of the samples and interacts with the microstructure. Simultaneously, a frequency-stabilized Nd:YAG pulsed laser illuminates the surface with an infrared radiation at a wave length of 1064 nm and a pulse duration of 90 µs. The successive arrival of the ultrasound wave at the specimen surface causes a microscopic displacement which modulates the detection laser light reflected on the sample surface. After collection in the optical system, the reflected light is demodulated using an active interferometer approach, based on the two-wave beam mixing method [7]. The specific characteristics of the crystal allow for the demodulation of the signal light beam and provide an output signal with an amplitude that is proportional to the surface displacement. Figure 1 depicts a typical ultrasound waveform that can be obtained up to 50 times per second on a sheet sample.

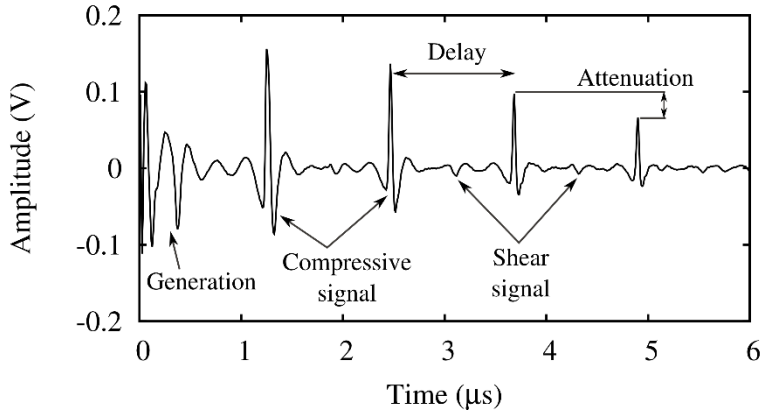

Figure 1: Ultrasound waveform measured with LUMet on a low-carbon steel sheet sample with a thickness of 3 mm at 1000 °C

The initial period of the signal is disturbed by the ultrasound generation and is seen as large oscillations present during the first 0.5 to 1µs of the signal. The compressive signal are identified as large amplitude echoes arriving periodically at the generation surface. The shear signal can be observed as smaller oscillations measured between two compressive signals. The properties of this ultrasound signal are correlated to the microstructure parameters. In elastically anisotropic materials like steel, attenuation is primary due to the elastic scattering

by grains and can be correlated to the average grain size. Further, the time between two successive echoes or delay is used for the evaluation of the ultrasound velocity. This second parameter is related to the bulk elastic properties that can vary during recrystallization and phase transformation as a response to the change in the density and crystallographic arrangement of grains in the material.

Grain size measurement

The measurement of grain size is based on the evaluation of the ultrasonic attenuation. The part of attenuation associated to grain scattering is calculated by comparing, in the frequency domain, the amplitude of an echo measured in the test sample relative to the amplitude of an echo measured in a sample where contributions from grain scattering are negligible [8]. The attenuation is then fitted to a power law as a function of frequency such as:

$$\alpha(f) = a + b f^{n} \quad (1)$$

where n is an exponent depending on the wave scattering regime; a and b are the scattering parameters. Here, b is a function of the grain size D, i.e. $b = \beta D^{3}$ where β is a temperature dependent calibration parameter. It is commonly accepted that the exponent n can be approximated with a value of 3 for the employed frequency range (5-30 MHz). A suitable calibration was generated by Kruger et al. for austenite grain size in a range of plain C-Mn steels by relating laser ultrasonic attenuation to the equivalent area diameter of austenite grains as determined by metallography [9]. This calibration is suitable for austenite grain size measurements in most low alloyed steels for which the elastic anisotropy is very similar to that of the C-Mn steels used for the construction of the calibration.

Phase fraction measurement

The evaluation of phase fraction is based on the evaluation of the velocity change during the phase transformation [3]. The ultrasonic longitudinal velocity is experimentally obtained from the ultrasound signal by the ratio of the propagation distance to the delay between two successive echoes. The sensitivity of ultrasonic velocity to phase fraction is due to the different elastic constants and densities of the different phase components. The velocity in a two phases domain is in good approximation the sum of the velocities of the parent and product phases weighted by their volume fractions. The fraction transformed can therefore be obtained by a classical rule of mixture between the velocity measured for the parent phase and the velocity measured for the product phase. When the material used has no or weak magnetic properties, the ultrasonic velocity of the parent and product phase varies linearly with temperature and can be extrapolated for the application of a classical lever rule. In steel, the velocity of ferrite varies non-linearly with temperature below the Curie temperature of 770 °C, but varies linearly above this temperature and the velocity in austenite varies linearly for all temperatures where it is present. Therefore, the classical lever rule cannot be directly applied to the measured velocity for the evaluation of the austenite fraction transformed upon cooling. Instead, the lever rule is applied between the velocity in ferrite measured upon heating and the velocity of austenite extrapolated to low temperature from its domain of stability [10].

Materials

An example for the austenite decomposition measurements is presented for a low-carbon linepipe steel (steel I) with a composition (in wt %) of 0.06C-1.49Mn-0.2Si-0.047Nb-0.038Al-0.0094N. Ultrasonic measurements of the fraction transformed during cooling are validated with dilation measurements conducted simultaneously. The initial microstructure

consists of a ferrite-pearlite mixture with 9 % pearlite. The laser ultrasonic measurements are conducted in the thickness direction of sheet samples of dimensions 60 mm x 10 mm x 2.1 mm.
The evaluation of austenite grain size during austenite recrystallization following hot-deformation is conducted with a laboratory steel (steel II) in the form of forged bars with a composition (in wt %) of 0.19C-1.5Mn-1.6Si-0.2Mo. This steel is also used for the evaluation of the ultrasound attenuation and velocity during uni-axial hot-compression experiment in austenite. Uniaxial compression tests are carried out with cylindrical samples of 10 mm in diameter and 15 mm in length.

Results and Discussion

Real time evaluation of austenite decomposition

Three sheet samples labeled A, B and C of the low-carbon line-pipe grade steel are used for this investigation. Sample A and B are heated at a rate of 100 °C/s up to 950 and 1150 °C respectively. The samples are held for 5 min prior to a cooling at 3 °C/s. Sample C is heated up to 1100 °C at a rate of 100 °C/s followed by a holding for 5 min prior to a rapid controlled cooling at a rate of 60 °C/s. Figure 2a shows the evolution of the ultrasonic velocity during heating for sample A and cooling for sample A, B and C.

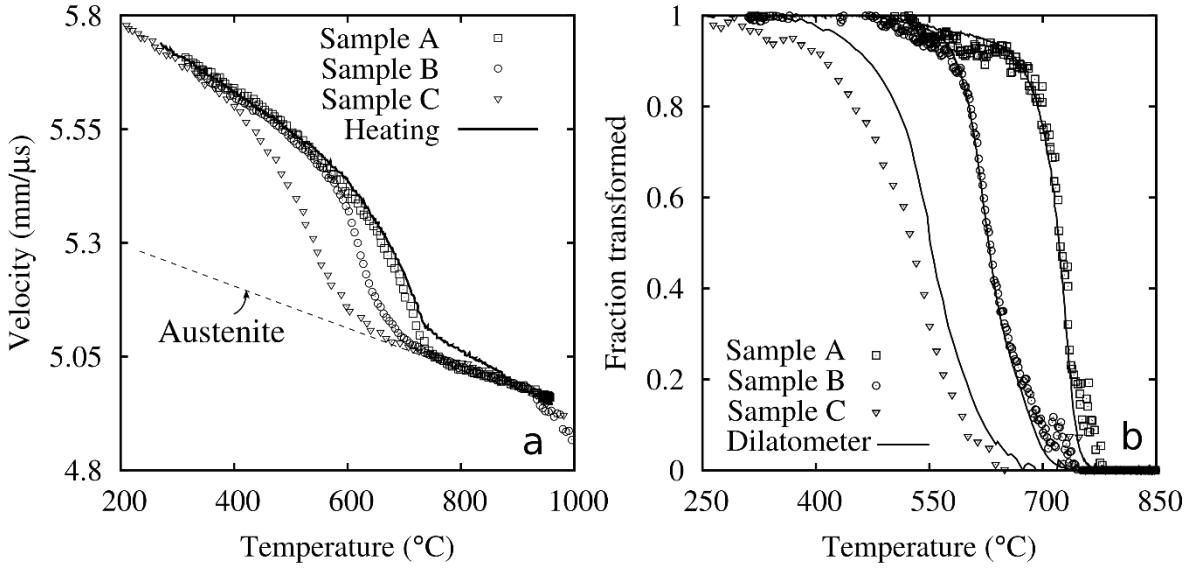

Figure 2: a) Evolution of the ultrasonic velocity during heating (Lines) and cooling (Symbols) measured in samples A, B and C of steel I. b) Calculated fraction transformed during the austenite decomposition upon cooling from dilatometer (Lines) and velocity (Symbols).

Upon heating the non-linear variation of velocity in ferrite is indicated with a continuous line. At 770 °C, the abrupt change in slope corresponds to the magnetic transition. This transition is followed by a linear domain prior to the austenite formation. Note that the transformation from ferrite to austenite around 900 °C upon heating is not associated with a large velocity change. For a steel composition initially weakly textured, the velocity of ferrite and austenite are very close in this temperature domain and the austenite formation cannot be monitored accurately with this technique. Upon cooling, the velocity variation is shown with open

symbol for samples A (square), B (circle) and C (triangle). Starting from the high temperature, the velocity of all three samples varies initially linearly with temperature. At the transformation start temperature, the velocity leaves the linear temperature dependence of the austenite phase (dashed line) and gradually approaches the velocity value measured initially upon heating. This transition from the velocity of austenite to the velocity in ferrite occurs at different temperatures for samples A, B and C. The lever rule is applied to the velocity measurements for each sample and is shown in Figure 2b. Quantitative analysis of dilation measurements conducted simultaneously with the LUMet measurements provides a direct validation of the measurements obtained from laser ultrasonics. The accuracy of the LUMet measurements is confirmed by excellent agreement between the conventional dilatometer data and the lever rule applied to the velocity signal.
The ultrasonic attenuation measured in real time during these three tests provides additional information on the evolution of the microstructure. This parameter primarily related to the scale of the grain structure is already validated for the measurement of the austenite grain size. The austenite grain size measured with LUMet at the end of the soaking is 16 µm for sample A, 70 µm for sample B and 60 µm for sample C. Moreover, the attenuation measurement can be used to discriminate between various transformation products [2]. Figure 3 shows the resulting microstructure obtained using conventional metallography including Nital etching. Sample A is composed of a mixture of polygonal ferrite grains and pearlite colonies. The pearlite fraction is estimated to be 9 % from quantitative image analysis. Sample B shows a more irregular ferrite grain structure. Sample C a much finer structure mainly composed of bainite laths. The ultrasonic attenuation measured at room temperature in these three structures is 1.7, 1.5 and 1.1 dB/mm for sample A, B and C, respectively. This example illustrates the potential of LUMet to differentiate between different transformation products which cannot be accomplished with conventional dilatometry.

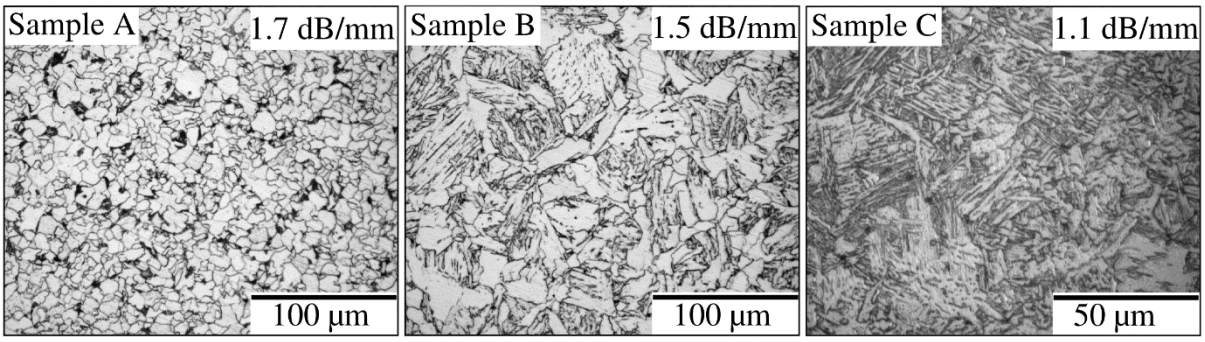

Figure 3: Optical metallography of samples A, B and C etched with Nital 2%.

Austenite grain size evolution during recrystallization

Austenite grain size evolution is monitored during the recrystallization process occurring after hot-deformation. The methodology adopted for these tests is that used by Sarkar et al. [11]. For the uniaxial compression tests, the samples are heated up to 1000 °C at a rate of 5 °C/s and then held for 2 min at this temperature prior to a cooling at 10 °C/s to the deformation temperature at 900 °C. Following deformation, the samples are held for 4 min at the deformation temperature where the attenuation is measured continuously in order to monitor the evolution of the austenite grain size. Immediately after the end of the holding time, the samples are rapidly cooled down to room temperature with high pressure Helium gas at a rate of approximately 60 °C/s. This rapid cooling is applied in order to generate a fine ferrite grain structure. The quenched samples are suitable for the acquisition of a reference

signal required in the LUMet grain size measurement method. The initial grain size prior to deformation is approximately 40 μm as reported by Liu et al. [12] for the steel investigated here. Figure 4 shows the evolution of the austenite grain size during soaking at 900 °C after deformation with a strain of 0.2 (continuous line) and 0.4 (dashed line), respectively. Symbols in the graph represent the time to achieve 5 and 95 pct recrystallization for these tests conditions, as measured from double hit tests experiments reported by Liu et al. [12]. The LUMet grain size measured during the soaking time remains approximately constant up to the onset of recrystallization. During recrystallization, the grain size drops continuously and remains constant after completion of recrystallization. The recrystallized grain size measured with LUMet at the end of the recrystallization is 21 μm and 12 μm, respectively, for a strain of 0.2 and 0.4. These measurements are in good agreement with the recrystallized grain size model established for this alloy by Liu et al. which predicts grain sizes of 19 μm and 11 μm, respectively, for the above deformation conditions [12]. These comparisons constitute a quantitative validation of onset and finish of recrystallization as well as the total change of grain size measured by LUMet in these conditions.

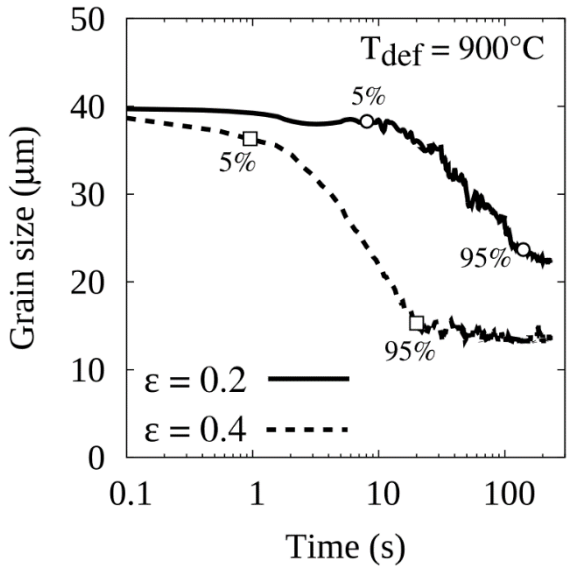

Figure 4: Evolution of the LUMet austenite grain size measured after deformation in steel II. Symbols in the graph represent the time to achieve 5 and 95 pct recrystallization as measured from double hit experiments as reported in [12].

Laser ultrasonics measurement during hot-deformation

For the first time, laser ultrasonics is applied to the measurement of microstructure evolution during hot-deformation in austenite. The laser probe is positioned on a translation stage connected with the Gleeble console. In this manner, while the piston side jaw (or stroke) moves during the deformation, the laser probe follows the center of the sample during the deformation. The maximum stroke rate that can be achieved with this system is 1 mm/s and the length to diameter ratio of the sample during deformation must be larger than 0.6 in order to limit the parasite reflection of the ultrasound pulse on the parallel surface of the sample. In the present configuration, the maximum strain rate is $0.1s^{-1}$ and the maximum total strain is 0.5 for a sample of 15 mm length and 10 mm in diameter. In order to evaluate the influence of strain rate on the occurrence of dynamic processes, the samples of steel II are austenitized at 1050 °C for 20 s and then deformed at strain rates of 0.001, 0.01 and 0.1 s^{-1}, respectively.

The attenuation and velocity are extracted from the analysis of the first and second echo measured on each waveform. The signal measured from the dilatometer placed in the same section of the LUMet measurement provides accurate evaluation of the changes in the propagation distance. The effect of strain rate on the flow stress curves measured at 1050°C is shown in Figure 5a. In the three selected deformation conditions, the flow stress curves show a single broad peak indicative of dynamic recrystallization. The peak stress is shifted toward higher values of true strain as the strain rate is increased. Figure 5b and 5c summarize the measurements of ultrasound attenuation and velocity, respectively, for the three strain rate conditions.

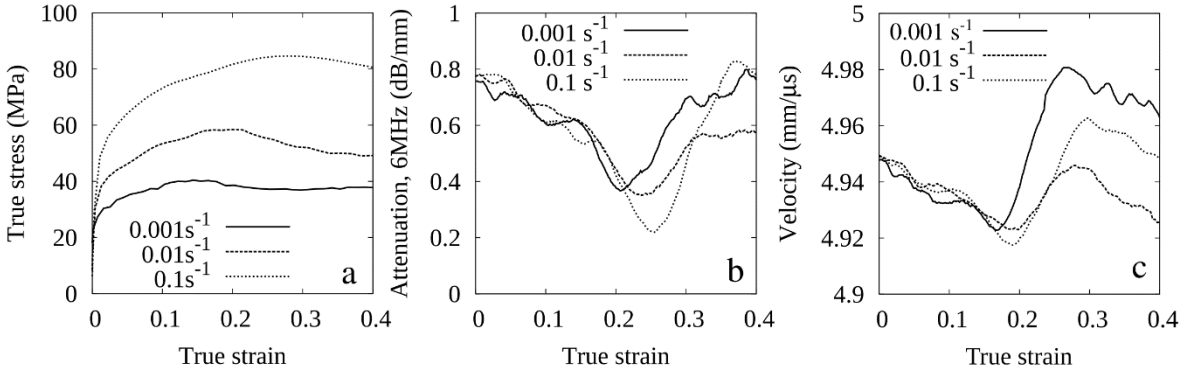

Fig 5 a) Flow stress curve for steel II at 1050 °C and strain rates of 0.001, 0.01 and 0.1 s^{-1}. b) Evolution of the ultrasound attenuation at 6 MHz and c) ultrasound velocity with respect to the true strain for the investigated conditions.

Both attenuation and velocity show characteristic minima that approximately coincide with the peak strain value. The decrease in attenuation is accelerated near the minimum and may be correlated to the change in effective grain size associated to the occurrence of dynamic recrystallization where a necklace of fine grains forms reducing the effective size of the initial austenite grain. In order to further exploit the attenuation signal, one will need to observe the frequency dependence of the attenuation which is less affected by the variation of other contributions like diffraction that may change as the specimen geometry changes continuously. The velocity signal shows a significant increase in the region of dynamic recrystallization and this may be attributable to a variation in the bulk texture in austenite. The slight velocity decrease measured before the onset of recrystallization and as steady-state is approached at later times needs to be further investigated. These preliminary data need to be validated with measurements using a wider frequency range but strongly indicate that correlations can be constructed between variations of the ultrasound parameters and the occurrence of dynamic recrystallization in austenite.

Conclusion

The laser ultrasonics sensor LUMet attached to the Gleeble 3500 is an exciting characterization tool allowing measurements to be carried out in-situ during various material process simulations. The capabilities of LUMet have been illustrated here for austenite grain growth, recrystallization and decomposition in low-carbon steels.
In particular, the present study shows for the first time that ultrasound attenuation and velocity measurement can be conducted remotely during hot deformation of a cylindrical specimens under conditions where dynamic recrystallization occurs. Similarly studies are presently conducted for non-ferrous materials [13, 14]

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