5th International Conference on Recrystallization and Grain Growth (ReX&GG V), May 5-10, 2013, Sydney, Australia

In-situ measurements of grain growth and recrystallization by laser ultrasonics

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Keywords : Laser ultrasonics, velocity, attenuation, recrystallization, austenite grain size

Abstract. Laser ultrasonics for metallurgy (LUMet) is an innovative sensor technology for in-situ measurement of microstructure evolution during thermomechanical processing. This unique sensor has been attached to a Gleeble 3500 thermomechanical simulator for dedicated laboratory studies during processing of steel, aluminum, magnesium and titanium samples. Advanced processing software has been developed for the measurement of grain size and texture evolution from laser ultrasonic signals. Results of austenite grain growth measurements in low carbon steels will be described to demonstrate the capabilities of the LUMet technique. Further, applications of the system to measure recrystallization of ferrite and austenite formation during intercritical annealing simulations of dual phase steels will be presented. The ability to rapidly acquire data both during a single test and for multiple conditions over a range of conditions from different samples has important implications on expediting process modelling and alloy design. Although certain limitations exist, the LUMet technique offers a very reliable characterization platform with a number of potential applications in metallurgical process engineering.

Introduction

The development of microstructure modelling tools and advanced materials processing paths may be aided by in-situ measurements of microstructure evolution including grain growth and recrystallization. Laser-ultrasonics for metallurgy (LUMet) is a new sensor specifically developed for in-situ monitoring of metallurgical phenomena during thermomechanical processing of metals and alloys. This non-destructive technique is fast and well adapted to remotely characterize the evolution of microstructure at high temperature [1]. During the development stage laser ultrasonics has been demonstrated to quantify elastic moduli and microstructure phenomena such as grain growth, recrystallization and phase transformations in steels and selected non-ferrous alloys [2],[3],[4]. The first commercial LUMet system has now become available as an attachment to a Gleeble thermomechanical simulator and constitutes an exciting innovative characterization platform for metallurgists to minimize labour intensive metallographic studies.

One of the key metallurgical parameter that can be accessed with laser ultrasonics is the austenite grain size. In steel, this parameter must be carefully controlled as it influences the subsequent phase transformation during cooling and thus affects the final microstructure and resulting mechanical properties. Another mechanism that can be captured with this method is the recrystallization process. In cold rolled multi-phase steel, the recrystallization may overlap with the austenite formation during intercritical annealing depending on the process parameters. This overlapping mechanism modifies the evolution of the austenite formation whereby austenite grains preferentially grow in the recrystallized area.

The present paper reports on technical specifications of the LUMet system and describes some of the first measurements carried out to determine austenite grain size evolution and ferrite recrystallization in low alloyed steels.

Experimental

LUMet system. Laser ultrasonic inspection is a recent technique in which ultrasound waves are generated and detected at the surface of a sample being processed. Ultrasound properties are characteristics of the materials bulk properties [5]. Ultrasound attenuation can be related to the average grain size and ultrasound velocity depends on the elasticity and density of the bulk material. The newly developed LUMet is designed as an attachment to the rear door of the Gleeble 3500 thermomechanical simulator. The laser ultrasonic measurements are conducted inside the Gleeble chamber in the centre of the sample in the pulse-echo configuration. A frequency-doubled Q-switched Nd:YAG laser with a wavelength of 532 nm is used for the generation of a wide band compressive ultrasound pulse. The duration of the laser pulse is approximately 6 ns and has a maximum energy of 72 mJ. The ultrasound pulse propagates back and forth through the thickness of the sample and looses part of its amplitude by interacting with the material and its microstructure. Successive arrival of the ultrasound pulse at the generation surface is detected with a frequency-stabilized Nd:YAG pulsed laser which illuminates the surface with an infrared radiation at a wave length of 1064 μm and a pulse duration of 90 μs. The infrared detection laser light reflected on the specimen surface is demodulated inside a photo-refractive crystal using an active interferometer approach, based on the two-wave beam mixing method [6].

Grain size measurements. Ultrasonic grain size measurements are based on monitoring attenuation. The ultrasonic attenuation resulting from scattering by grains is extracted from other dispersive contributions using a signal from a fine grained reference sample where contributions from grain scattering are negligible [3]. The part of the attenuation due to grain scattering is then expressed as a function of grain size and frequency by $\alpha_{Sc} = a + b f^n$ where a accounts for contributions such as internal friction, damping due to magnetic properties or dislocations and b is related to the average grain size, D, i.e. $b = K(T)D^2$ where $K(T)$ is a temperature dependent calibration parameter. For example, a suitable calibration was provided by Kruger et al. for austenite grain size in a range of plain C-Mn steels [7]. The equivalent area diameter of austenite grains were determined by metallography and related to the frequency dependent ultrasonic attenuation. From this calibration data, the grain size can be determined in steels with various compositions.

Recrystallization measurements. 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 compressive wave velocity is a function of the elastic modulus of the material in the propagation direction. During cold rolling, certain crystallographic orientations are stabilized resulting in a macroscopic anisotropy of velocity in the principal direction of the sample. During a recrystallization process, a new family of grains forms from the deformed structure resulting in a modification of the bulk texture in the material. These texture variations are directly associated with the variation of the ultrasonic wave velocity [8]. The change in the directional ultrasonic velocity accompanying texture variations during recrystallization is used to determine the evolution of recrystallized fraction by application of a lever rule principle on the ultrasonic velocity curve. X-ray diffraction measurements were conducted to explicitly quantify the texture changes during recrystallization.

Materials. The austenite grain size measurements are carried out in an X80 microalloyed low carbon linepipe steel with a composition (in wt %) of 0.06C-1.65Mn-0.11Si-0.034Nb-0.014Ti-0.24Mo-0.005N. In a previous study, similar ultrasonic measurements had been carried out using the laser ultrasonic instrument at the IMI Laboratory in Boucherville where the LUMet technique was developed to the stage of commercial readiness. Based on these previous studies a grain growth model was proposed that is coupled to a NbC dissolution model. The present investigation on the

same material and similar heat treating conditions provides a series of validation tests for the new LUMet system.

The ferrite recrystallization kinetics is measured for 50% cold rolled dual phase (DP) 600 steel with a composition (in wt %) of 0.105C-1.858Mn-0.157Si-0.012Ti-0.009Mo-0.006N. The initial microstructure consists of a ferrite-bainite matrix with elongated grains and 5% pearlite.

Results and Discussion

Austenite Grain Growth. Austenite grain growth tests on the linepipe steel were conducted by heating a sheet sample with a thickness of 1.5 mm from room temperature to an austenitizing temperature (in the range of 950 to 1250 °C) at a rate of 100 °C/s and subsequently holding for 20 minutes. The ultrasonic volumetric grain size evolution measured with LUMet is shown in Fig.1a and compared with metallography data and the predictions from the previously developed grain growth model for this steel. Within the experimental scatter, the grain size obtained with the newly installed LUMet system is in good agreement with the metallographic analysis and the grain growth model. At the austenitizing temperature of 1050˚C, the laser ultrasonic grain size is about 10 µm larger than the comparison data. This austenitizing temperature corresponds to the so-called grain coarsening temperature which is associated with the beginning of the dissolution of NbC precipitates. The onset of substantial austenite grain growth at about 1050˚C is further confirmed with continuous heating grain growth tests, as illustrated in Fig. 1b. Here, the samples were heated at 100˚C/s up to 900˚C, the temperature at which the austenite formation is completed. The evolution of the austenite grain size is then measured during continuous heating at 3˚C/s up to 1250˚C. Fig. 1b shows the results of two LUMet measurements and their comparison with the combined grain growth dissolution model previously developed. Comparing the two LUMet measurements indicates good reproducibility. Below 1050˚C, the grain size stays relatively small due to the strong grain boundary pinning associated with the presence of NbC precipitates. Above this temperature, NbC precipitates start to dissolve thereby reducing the pinning force such that a sudden increase of the grain growth rate is predicted and well captured by the laser ultrasonic measurements.

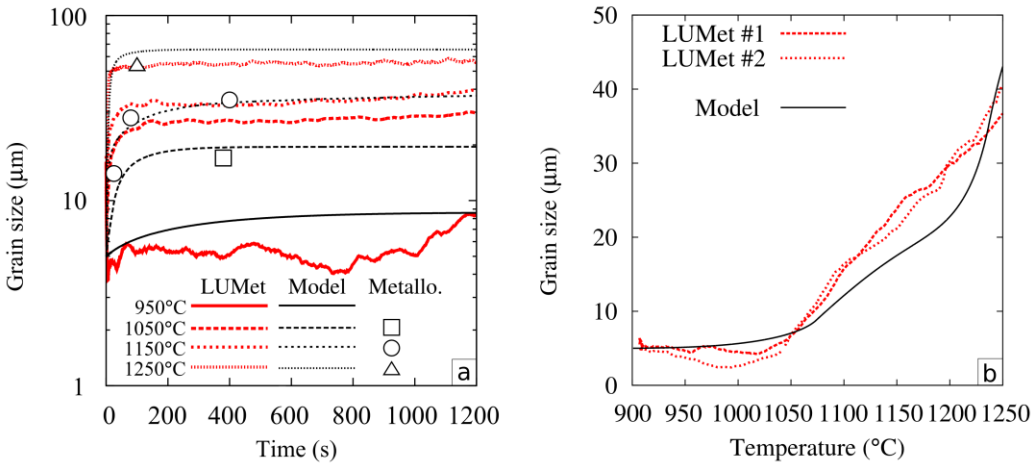

Fig. 1 – *a*: Isothermal austenite grain growth in the X80 linepipe steel at various temperatures. Time zero coincides with the sample reaching 900˚C. *b*: Comparison of measured and predicted austenite grain growth kinetics in the X80 linepipe steel during continuous heating at 3˚C/s to 1250˚C

Recrystallization. The recrystallization kinetics of the 50% cold-rolled DP 600 steel was measured during isothermal holding at various temperatures namely 600, 625 and 650 ºC. The holding temperatures were reached at a heating rate of 50 ºC/s. Fig. 2a shows the evolution of

ultrasonic velocity as obtained with LUMet. Time zero corresponds to the beginning of the isothermal holding. Initially the velocity is constant at each temperature and then increases by about 0.1 mm/µs. The evolution of the recrystallized fraction is determined by the application of the lever rule method on the velocity data. Linear parts of the velocity curve are fitted before and after recrystallization occurs. The recrystallized fraction obtained by laser ultrasonics is compared with values obtained by conventional metallographic analysis. There is excellent agreement between the laser ultrasonic fraction and the metallographically obtained kinetics of recrystallization, as illustrated in Fig. 2b.

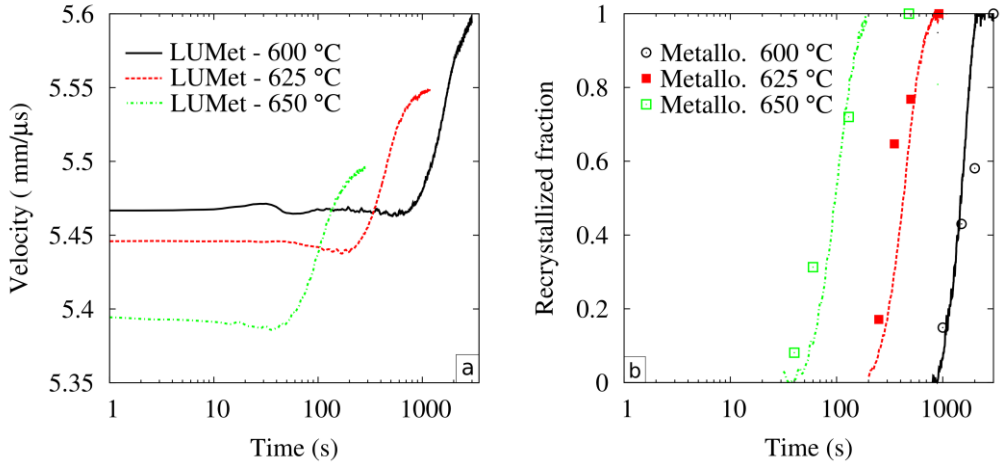

Fig. 2 – *a:* Evolution of the ultrasonic velocity during isothermal holding at various temperatures for the 50% cold rolled DP600 steel. *b:* Comparisons of recrystallized fraction obtained from LUMet (lines) with metallographic data.

The texture change during recrystallization was confirmed by X-ray diffractometry on as-cold rolled and recrystallized samples. The recrystallized sample was held for 300s at 650 ºC and naturally cooled down to room temperature. The orientation distribution functions (ODFs) for $\Phi_2 = 45°$ is shown in Fig. 3. The intensity of texture along the ODF is proportional to the intensity of grey level. In the as-cold rolled sample, higher intensities measured at $\Phi_1 = 0°$ and $\Phi < 45°$ indicate the presence of the alpha fiber. After recrystallization, the maximum intensity of the ODF is present mainly along the gamma fiber for $0° < \Phi_1 < 90°$ and $\Phi = 54.7°$.

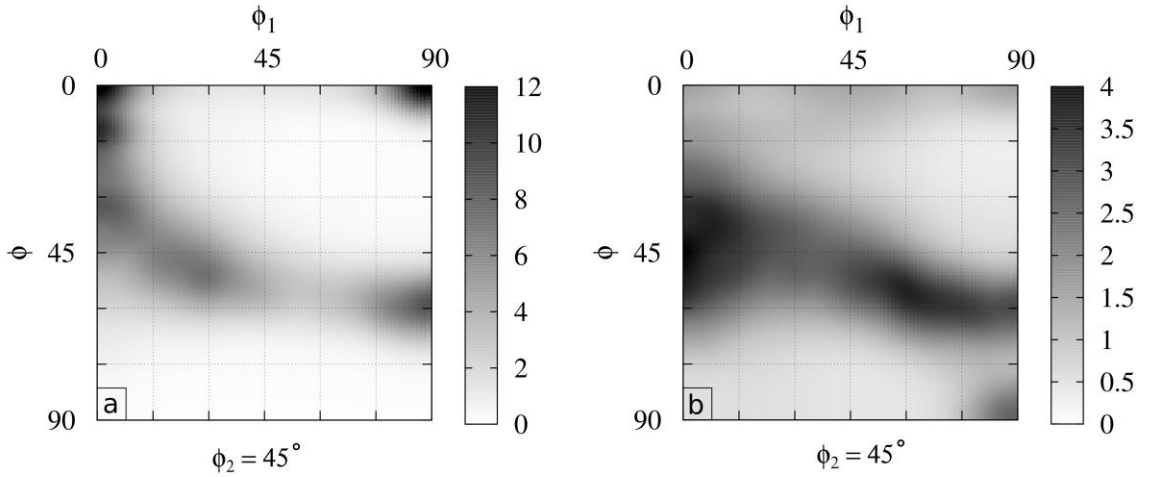

Fig. 3 - Orientation Distribution Function at $\Phi_2 = 45°$ of *a:* as-cold rolled and *b:* recrystallized DP 600 steel measured by X-Ray diffractometry.

A second set of experiments was carried out with the same steel where the recrystallization process was monitored during continuous heating for two different heating rates, i.e. 1 and 10 °C/s. Fig. 4 shows the temperature variation of the ultrasonic velocity for the two heating rates. Considering the velocity evolution at 1 °C/s the following is observed. The ultrasonic velocity decreases non-linearly with temperature up to 678°C and shows then a sudden raise of 0.04 mm/µs between 678 and 700°C. Heating the specimen further leads to velocity decreases with three distinct stages, i.e. (i) decrease of 0.1 mm/µs from 700 to 745°C, (ii) decrease of 0.15 mm/µs from 745 to 830°C and (iii) decrease at an even further reduced rate above 830°C. This velocity-temperature behaviour can be rationalized as follows. The decrease of velocity with increasing temperature in α-iron below the Curie temperature is related to the non linear temperature dependence of the magnetic component in the elastic constants for the ferrite phase. This velocity decrease is interrupted due to recrystallization which results in a velocity increase. The two final stages of velocity decrease above 745°C are related to the formation of austenite. The first region represents the two phase region austenite-ferrite and the final stage shows the linear velocity change with temperature in the completely austenitic state. The austenite formation region between 745 and 830 °C is consistent with dilatometric measurements of austenite formation by Kulakov et al. [10]. For the higher heating rate of 10 °C/s recrystallization, i.e. increases of velocity with temperature, is shifted, at least in part, into the range of austenite formation. In this case the relative increase in velocity is much lower than for 1°C/s and only a single step drop is monitored rather than the two step drop seen in the 1°C/s experiment before reaching the linear austenite regime above 830 °C. This single step behaviour at the higher heating rate indicates that ferrite recrystallization and austenite formation occur in the same temperature range whereas these two processes are well separated for the lower heating rate. This finding is consistent with dilatometer measurements of austenite formation and metallographic observations by Kulakov et al. [10].

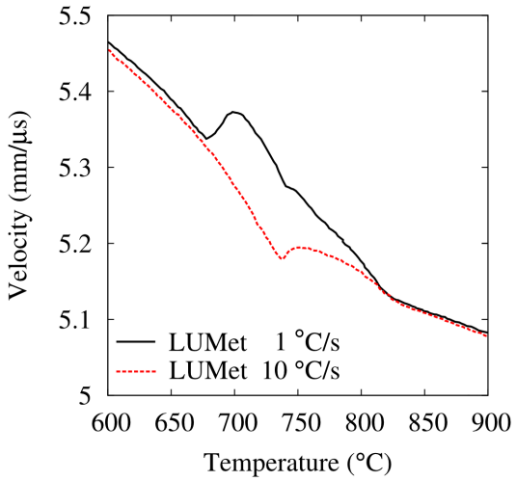

Fig. 4 –Evolution of the ultrasonic velocity during continuous heating of the 50% cold rolled DP600 steel.

Summary

The new 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.

Austenite grain size evolution was measured in low-carbon steel during isothermal holding and continuous heating experiments. Results were in good agreement with metallographic data and a phenomenological model combining grain growth and NbC dissolution as long as the grain

structure is well described by the average grain size. Sudden change in growth rate was well detected at the temperature where NbC precipitates begin to dissolve.

Cold-rolled dual phase steel was used for in-situ characterization of ferrite recrystallization at various temperatures. The variations of ultrasonic velocity captured during the recrystallization process were associated with texture variation. The laser ultrasonically measured evolution of the recrystallized fraction was validated with metallographic observations.

The in-situ laser ultrasonic measurements are expected to strongly accelerate the development and validation of microstructure based process models.

Acknowledgment

The financial support received from the Natural Sciences and Engineering Research Council (NSERC) of Canada, Canada Foundation for Innovation, Evraz Inc. NA and TransCanada Pipelines Ltd. is acknowledged with gratitude. Further, we would like to thank Dr. Andre Moreau for his helpful support of the laser-ultrasonic data analysis.

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