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AUSTENITE GRAIN SIZE MEASUREMENTS BY LASER-ULTRASONICS ON A MODERN LOW CARBON STEEL

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ABSTRACT

Laser-ultrasonics is a non-contacting technique where the attenuation of ultrasonic signals can be measured and related to the grain size of the investigated material. In the present study, this novel technique was used to monitor the austenite grain size evolution of a selected microalloyed low carbon (0.05C in wt%) complex-phase steel. Using a calibration previously developed for higher carbon grades, the grain sizes obtained from the ultrasonic measurement were found to be in good agreement with the average grain sizes measured by conventional metallographic techniques provided the grain size distribution is log-normal. The challenges in quantifying grain sizes during abnormal growth will be discussed and the advantages of laser-ultrasonics to monitor grain size evolution in modern low and ultra-low carbon steels will be delineated.

INTRODUCTION

Austenite grain size plays an important role in determining the recrystallization of austenite and the subsequent phase transformation during processing of steels using a hot rolling mill. A fine austenite structure is desired to produce finer transformed products. In modern generation microalloyed steels, various microalloying elements such as niobium, vanadium and/or titanium are used in combination with a suitable thermo-mechanical processing to obtain the austenite grain size distribution at elevated temperature that will lead to a desired microstructure after phase transformation [1-2]. Hence, it is desirable to have reliable knowledge of the austenite grain size evolution during hot rolling of steel. At present, metallographic studies, such as conventional metallography, thermal grooving, oxidation, carburization, glass etching and ferrite/cementite delineation of prior austenite grain boundaries are the primary tools employed to investigate austenite grain growth behaviour [3]. Unfortunately each method has its own limitations to quantify the grain size to an appreciable extent. Conventional metallography becomes difficult for low and ultra low carbon steel when direct quenching from high temperature to low temperature does not produce a completely martensite structure. Other techniques, such as thermal grooving, glass etching, oxidation or carburization of austenite grain boundaries are limited to surface grains where the grain growth behaviour may demonstrate different characteristics than that of bulk grains [2].

Alternatively, laser-ultrasonics can be used to obtain information on grain growth. Ultrasonic attenuation is a measure of decay of an ultrasonic wave as it propagates through the material. The attenuation process is known to be sensitive to material parameters such as grain size [4], porosity [5] etc. The laser-ultrasonic technique relies on lasers for both the generation and detection of ultrasound waves and is non-contacting; this makes laser-ultrasonics suitable for high temperature measurements [6,7]. At high temperature, the rate of attenuation of the ultrasonic signal can be correlated to the austenite grain size [8,9,10] and, to a lesser extent, to various internal friction mechanisms [11].

In previous studies [9,10], laser-ultrasonics has been used to monitor the austenite grain growth for various C-Mn steels with carbon values mostly in the range of 0.08wt% to 0.7wt%. At present, this new technique is applied for a selected grade of complex-phase (CP) 0.05wt% C steel, which also contains Nb and Mo as microalloying elements. A calibration previously developed for various medium and high carbon grade steels [6] is used to quantify the grain size of the present steel grade for temperatures ranging from 900°C to 1200°C. The grain size values measured by laser-ultrasonics are then compared to measurements made by conventional metallography for selected austenitizing conditions to investigate the effectiveness of the laser-ultrasonic method to measure austenite grain growth in a quantitative manner.

EXPERIMENTAL PROCEDURE

The chemistry of the CP is shown in Table 1. The steel was lab cast and received in the form of forged bars. Samples for ultrasonic measurements having a dimension of 200mm × 20mm × 3mm were machined from these forged bars. The surfaces were machined to a smooth finish to keep the effect of surface roughness on ultrasound attenuation to a minimum.

Table 1 – Chemical composition (wt%) of the CP steel

C	Mn	Si	S	P	Nb	Mo	Al	N
0.05	1.88	0.04	0.007	0.005	0.048	0.49	0.05	0.004

The laser-ultrasonic experiments were carried out using a Gleeble 3500 thermo-mechanical simulator equipped with a laser-ultrasonic system. Resistance heating is employed to heat up the samples and the temperature was monitored with a thermocouple (K type thermocouple for 900°C to 1000°C; R type thermocouple for 1050°C to 1250°C) spot-welded approximately 5mm away from the sample edge but on the same vertical line of the laser detection spot. A second thermocouple was placed approximately 1cm away from the first thermocouple to verify the temperature distribution over the sample. The variability of temperature was within 5°C over an area of 1x1cm^2 during the entire thermal process. Steel samples were heated at 5°C/s to various holding temperatures in the range of 900°C to 1250°C and held for approximately 15min to monitor austenite grain growth.

The pulse-echo configuration (reflection mode) was used for attenuation measurement. During the whole thermal cycle, measurements were made as follows: A short (5 ns) and energetic (150mJ) green (532nm) light pulse from a frequency-doubled Nd:YAG laser (Neodymium:Yttrium Aluminium Garnet laser) is focused on the sample to a circular area of 2mm diameter. This light pulse generates a wideband acoustic pulse by ablating a very thin surface layer of negligible thickness. A second laser (detection laser) with longer pulse (Nd:YAG infrared laser operating at 1064 nm, with a pulse duration of 50μs and pulse energy of approximately (70mJ) was used for detection of ultrasonic displacement. The detection laser beam was focused on the sample into a uniform disk approximately 2mm in diameter that was superimposed onto the generation laser spot. A phase shift in the reflected infrared wave at the detection location is essentially proportional to the surface displacement and is picked up by the optical interferometric system attached to the signal processing system. The experimental set-up for the current ultrasonic measurement is presented in Figure 1. The measurements were made approximately once every 3 seconds for each thermal cycle.

To verify the validity of ultrasonic measurements, some samples were quenched to room temperature either employing helium gas or water quenching after completion of

the grain growth experiments. In addition, selected samples were subjected to grain growth treatments that replicated the laser-ultrasonic experiments but for a shorter soaking time and hence were interrupted by water quenching to room temperature. Saturated picric acid with a small amount of copper chloride and wetting agent [12] solution was used to reveal prior austenite grain size for all samples. The individual equivalent area diameter (EQAD) of each grain was first measured using an image analyser and the average EQAD was calculated for approximately 400-500 grains for each sample. Prior austenite grain size could not be revealed for grain sizes below 10μm, i.e. for temperatures of 950°C and lower.

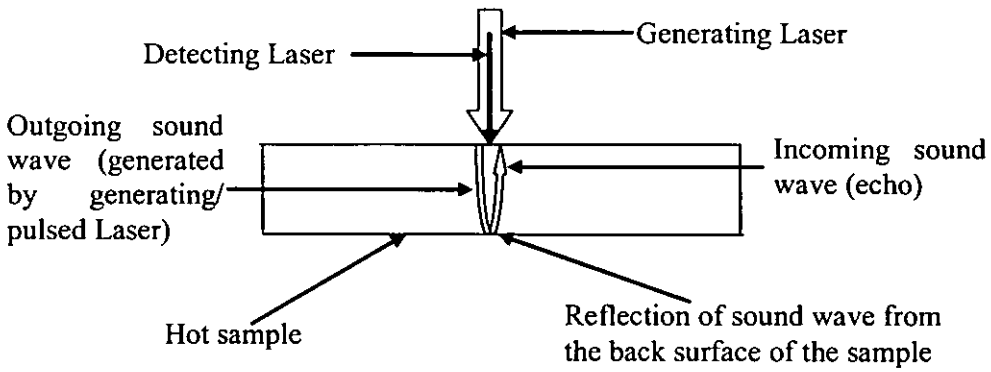


Figure 1 – Schematic diagram of the laser-ultrasonic setup

BACKGROUND THEORY AND SIGNAL PROCESSING

Ultrasonic attenuation is a measure of the decay in ultrasonic wave amplitude and is caused by various microstructural parameters. In polycrystalline metals, grain scattering is usually considered to be the dominant mechanism. When a metal is hit by an intense laser pulse, two physical phenomena occur to generate an ultrasound pulse on the metal surface namely 1) thermal expansion and 2) ablation, including the generation of plasma at the sample surface. Either of these effects induces an ultrasonic wave in the test sample which propagates within the sample and is attenuated due to grain scattering. This scattering mechanism is frequency dependent and hence exhibits different attenuation rates at different frequencies. Generally, the attenuation measurements can be done in a narrowband system (measurement is carried out at a particular frequency) or can be carried out using a broadband system involving a large frequency domain.

The acoustic pulse bounces back and forth between the two parallel faces of the sample and is detected as a surface vibration by laser interferometry. Figure 2 shows a typical ultrasonic signal obtained from the steel sample at 1050°C. The signal obtained in the first few hundreds of ns is caused by plasma generation at the sample surface and is

not acoustical. Later, two acoustic echoes corresponding to the travel through twice and four times the sample thickness are observed. The ultrasonic attenuation can be measured between two corresponding echo signals at each frequency by comparing their amplitude after Fourier transforming each echo from the time domain to the frequency domain. Only the longitudinal part of the wave-front is considered for calculation as compared to the shear part (the small negative amplitude signal near 2 μs).

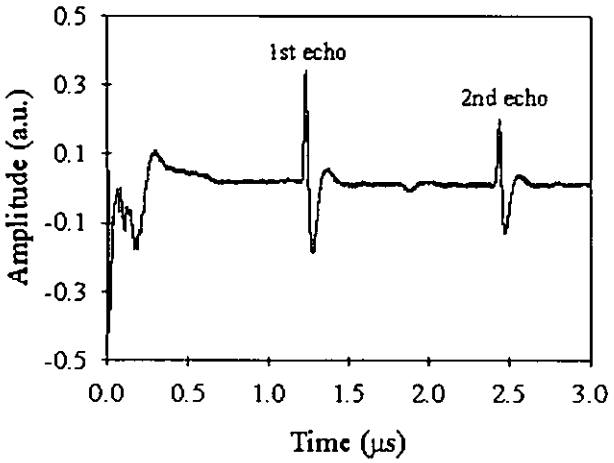


Figure 2 – A typical ultrasonic signal acquired during grain size measurement at 1050°C for CP steel

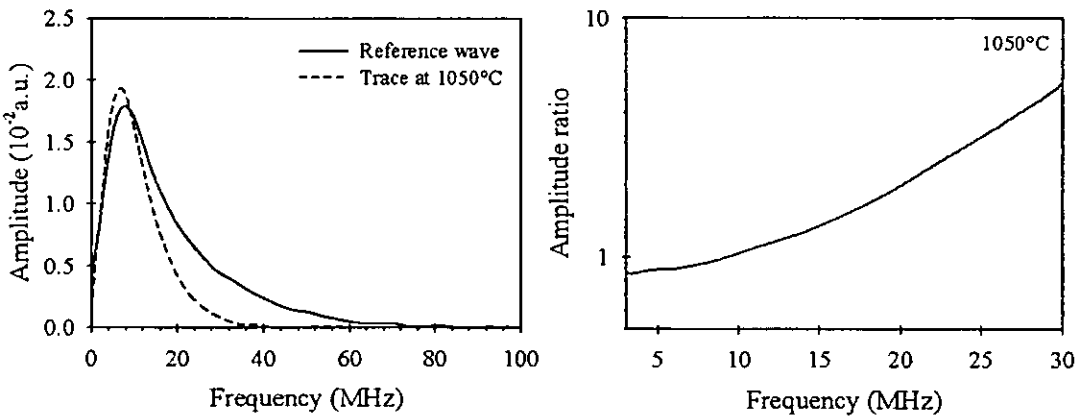
Finally, the attenuation is calculated by taking the ratio of the amplitude spectrum of the first echo with that of a reference echo and divided by twice the sample thickness to obtain an attenuation spectrum. This attenuation spectrum is then fitted to a power law for the corresponding selected suitable frequency domain. The power law is in the form of

$$\alpha = a + bf^n \tag{1}$$

where n, a and b are the three fitted parameters, α is attenuation and f is frequency in the selected frequency domain. The parameter a is an arbitrary offset and has no effect on determining the material grain size. The parameter b is proportional to $D^{(n-1)}$, where D is a measure of grain size, i.e. here the average EQAD.

Unfortunately, when the samples are thick (several to tens of mm) or when the grain size is large (hundreds of microns), the calculated attenuation based on the two-echo method is sometimes affected by noise and the accuracy of the measurement is reduced; especially the signal-to-noise ratio (SNR) can be very poor for the second echo. The other difficulty involving the conventional two-echo method is the requirement for correction of diffraction effects of the ultrasonic pulse, or the requirement that the

measurement be done either in the near or far acoustic field, which present strong limitations on the experimental geometry. The present study employed a more recent technique for the calculation of the amplitude spectrum. In this technique, the amplitude spectrum of the test material is compared with that of a reference system for a single echo signal (generally the first echo is considered for calculation). The reference broadband ultrasonic pulse was obtained from the same sample at room temperature because room temperature attenuation is very low, which is a prime requirement for selecting the reference echo. Taking the ratio between amplitude spectrum at test temperature to that at room temperature for the same sample takes care to a great extent of the diffraction effect with respect to the broadband ultrasonic pulse because both measurements are made with the same experimental geometry, and in particular, for the same propagation distance. Finally, the room temperature signal shows excellent SNR value. Figure 3 compares the amplitude spectrum of the test material at 1050°C with that obtained at room temperature (reference system). Figure 4 exhibits a good fit of power law (Equation 1) with the attenuation spectrum (generated from Figure 3(b) for a frequency domain between 3MHz to 30MHz.



(a) (b)

Figure 3 – (a) Example of typical amplitude spectrum for the test object at 1050°C and at room temperature (reference spectrum); (b) Ratio of two amplitude spectra presented in (a) or amplitude ratio spectrum for the particular trace measured at temperature 1050°C

Generally three separate regimes are considered for the conventional analysis [4]:

Rayleigh regime ($\lambda >> D$)	$b = K_R D^3 f^4$	(2a)
Stochastic regime ($\lambda \approx D$)	$b = K_S D f^2$	(2b)
Diffusion regime ($\lambda << D$)	$b = K_D / D$	(2c)

where λ is the acoustic wavelength and K_R, K_S and K_D are constants of the material. For the present case, the relationship between the ratio of the ultrasonic wavelength to the

average grain size due to grain scattering lies between Rayleigh and Stochastic regimes. This assumption generates a single equation to explain the relationship between attenuation with the average grain size of the material,

$$b = K(T)D^{n-1}f^n \tag{3}$$

where $K(T)$ is a material property that depends only on temperature and n is the power law exponent that varies between 2 to 4 and for the present situation was determined to be approximately 3. Eq. (3) may be viewed as an empirical generalization of Eqs. (2) but it can also be derived from modern scattering theories, in which case it can be shown that n is a slowly varying function of the product of grain size times frequency, and n can be assumed constant over the frequency range considered here. As a result, Eq.(1) can still be used to fit the data. The fitted parameter b is proportional to the square of grain size, and a, the frequency-independent term, accounts for sensitivity changes of the system as a function of temperature (signal amplitude may increase or decrease as a function of temperature for various reasons, including change in surface reflectivity, acoustic generation efficiency, etc.). The function $K(T)$ was obtained by calibration [8].

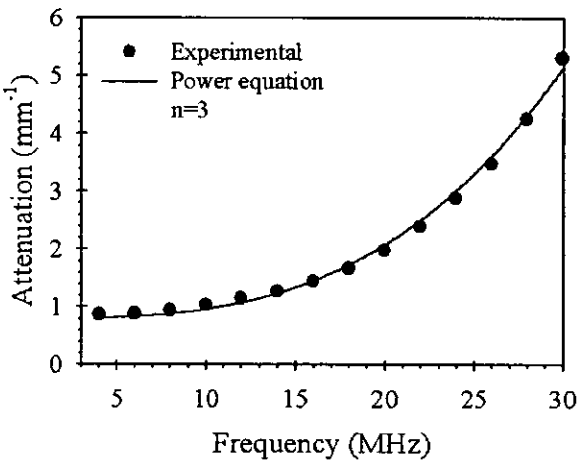


Figure 4 – Experimentally measured attenuation spectrum for a particular trace at 1050°C and its description by power law (Equation 1) for the selected frequency domain

RESULTS AND DISCUSSION

Figure 5 shows the austenite grain growth behaviour of the present CP steel grade as measured by laser-ultrasonics (based on the previously described calibration procedure) at selected temperatures (1050°C and 1150°C) that fall into different grain growth regimes. For each temperature two complete cycles of measured grain size data is presented in Figure 5. Although ultrasonic measurements show some scatter for any test temperature, the grain size evolution as measured by laser-ultrasonics shows

reproducibility that is at least as good as that obtained from traditional metallographic techniques. As can be seen from Figure 5, at 1050°C continuous austenite grain growth was observed for the present CP steel, whereas in the case of holding at 1150 °C, grain sizes increase rapidly in the first two to three minutes followed by a much slower grain growth rate at longer holding time. Figure 5 also presents the metallography results at these temperatures. At 1050°C, laser-ultrasonic results show reasonable agreement with grain size data obtained from metallography. The situation is more complex for the grain size evolution at the higher test temperature of 1150°C. For short holding times (e.g. 2min at 1150°C) the apparent austenite grain size obtained by laser-ultrasonics is significantly larger than the grain size measured from conventional metallography, whereas at longer time (after 15min) both predictions are consistent with each other. Conventional metallography revealed abnormal grain growth for 2min holding at 1150°C, as shown in Figure 6. Here, a bimodal grain size distribution is observed for the austenite microstructure. In comparison, Figure 7 shows the micrograph for holding at 1150°C for 15 min where a log-normal distribution of grain sizes is observed.

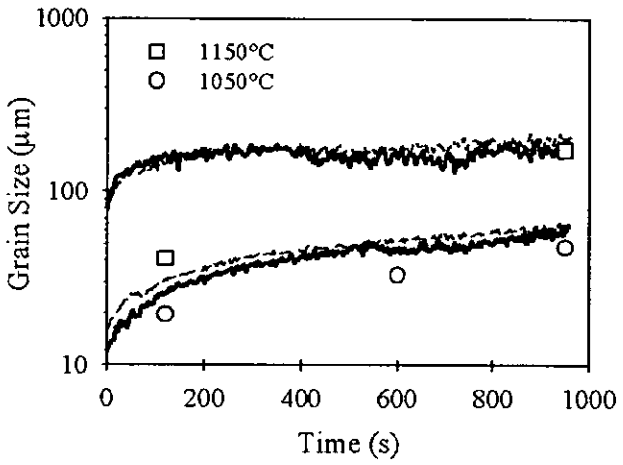


Figure 5 – Comparison of measured average grain sizes by laser ultrasonics (lines) and conventional metallography (symbols). For each temperature, two laser-ultrasonic measurements are presented (thin vs. bold data points).

Figure 8 summarizes the mean austenite grain size obtained for the present CP steel grade by laser-ultrasonic technique at seven austenitizing temperatures between 900°C to 1200°C for a soaking period of fifteen minutes. Measurements were also made at 1250 °C, but were rejected because of poor signal-to-noise ratio caused by high attenuation. The laser-ultrasonics grain size data were smoothed by using best fit polynomial functions at each test temperature. As can be seen from Figure 8, the laser-ultrasonic measurements are consistent with what one would expect, i.e. the austenite grain size increases with temperature and soaking time.

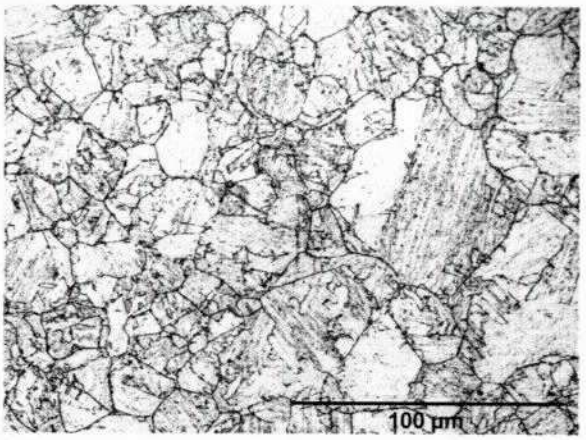


Figure 6 – Abnormal grain growth observed during grain growth treatment at 1150°C for 2min soaking

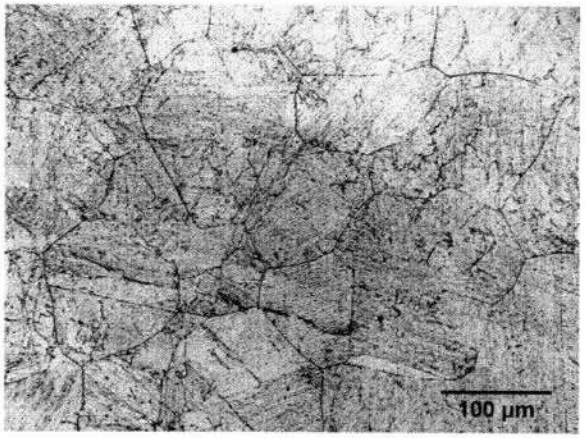


Figure 7 - Normal grain growth observed during treatment at 1150°C for approximately 15min soaking

As presented in Figure 8, the entire austenite grain growth phenomenon can be divided into three distinct grain growth regimes. At low temperatures (900°C to 1000°C), there is negligible grain growth probably due to the strong pinning effect on austenite grain boundary motion by precipitates of the microalloying elements. Previous study on microalloyed steels [13] suggested that the dissolution of the Nb-rich precipitates starts at temperatures of 1100°C and above. The larger grain growth rates observed at 1050°C and 1100°C, probably reflect a growth stage when the pinning effect of precipitates on grain boundary motion is gradually becoming weaker. Complete dissolution of precipitates may not be possible at these temperatures. The situation becomes more complex at higher temperatures like at 1150°C. It appears that at 1150°C, there is rapid (more or less complete) dissolution of precipitates promoting abnormal grain growth for shorter soaking time, whereas longer holding time at 1150°C stabilizes normal grain growth of

the coarse structure that emerges from the abnormal growth stage. These situations are verified by conventional metallography as presented in Figures 6 and 7. Grain growth at 1200°C is similar, i.e. rapid initial growth is followed by much slower growth rates for longer holding times.

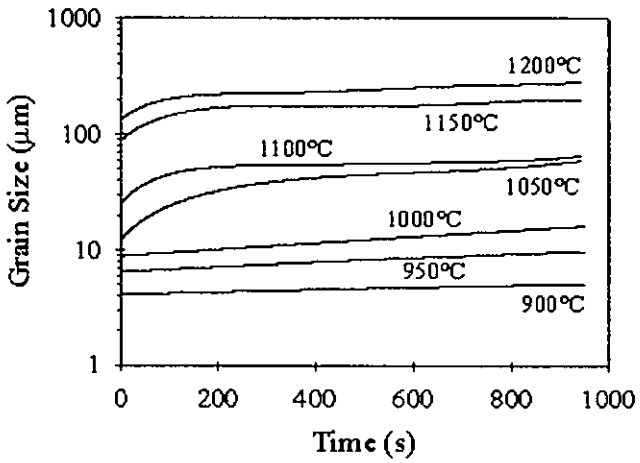


Figure 8 – Austenite grain growth behaviour at various temperatures as obtained by laser-ultrasonics

During the abnormal grain growth regime, large grains dominate the ultrasonic response. A simple explanation can be given if the material can be modeled as a mixture of two components, one with a distribution of large grain sizes, and one with a distribution of small grain sizes. Each volume fraction will contribute to the total attenuation according to Eq. (3). Because the attenuation increases as the square of grain size, the volume fraction of large grain sizes will dominate the attenuation behaviour. In contrast, a lower average grain size is obtained using conventional metallography and analysis. However, the bimodal grain size distributions that result from abnormal grain growth cannot be simply characterized by an "average" grain size; proper care has to be taken to give equal importance on the distribution of small and large grains. At much longer holding times and/or higher temperatures (e.g. 1200°C and 1250°C), the precipitates are readily dissolved and hence grain growth behaviour reverts back to a normal grain growth regime. However, at higher temperatures, such as at 1200°C and 1250°C, the number of grains in the analyzed volume is comparatively small so that the statistics are poor. This factor may then decrease the accuracy of the measurements both by laser-ultrasonics and metallography.

CONCLUSION

Laser-ultrasonics using first-echo processing is used to measure the austenite grain size for a low-carbon complex-phase steel containing 0.05wt%. A previously developed calibration procedure for plain carbon steels with carbon levels ranging from

0.08 to 0.7wt% can also be employed to measure the grain size for this present low-C steel, as demonstrated by comparing the laser ultrasonic measurements with austenite grain sizes measured using conventional metallography. To a first approximation, grain size values obtained from the two different techniques match well with each other. However, during abnormal grain growth regime (at temperature range above 1100°C for shorter holding time), the laser-ultrasonic technique probably measures the grain size of the larger grains as opposed to the mean of the small and large grains. Similarly at much higher temperatures (1200°C and 1250°C), the attenuation is large and difficult to measure and the number of measured grains becomes relatively small. These two factors can limit the capability of the technique for measuring austenite grain size at higher temperatures.

However, the laser-ultrasonic technique has important distinct advantages as compared to other techniques. First of all, laser-ultrasonics is less time-consuming as compared to the conventional metallography technique. In addition, from an application point of view in industry, it has immense potential, especially in the high temperature metal industry. Being a non-contacting technique, there is potential to use this approach for on-line high temperature grain size measurement in an industrial setting, e.g. as required during hot rolling of steel, and such on-line measurements are already being made [8]. The real-time determination of the austenite grain size can provide an impressive tool to control microstructure in a closed loop controlled method during hot rolling of steel or other materials.

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