Determination of Grain Size in Deep Drawing Steel Sheet by Laser Ultrasomics

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The laser ultrasonic technique has been used to determine grain sizes in Interstitial Free (IF) steels. With an analysis method that employs scattering theory, absolute values of the average grain size have been calculated directly from the ultrasonic traces. These results indicate that the laser ultrasonic technique may be incorporated on-line for direct measurements of grain size during steel production.


(Received August 21, 2013; Accepted March 13, 2014; Published May 30, 2014)

Keywords: laser ultrasonic, attenuation, on-line measurement, grain size, interstitial free (IF) steel

1. Introduction

The grain size and crystalline texture of metal sheets and plates strongly affects its formability. These characteristics play an important role in determining the subsequent formability of the metal into such finished parts as beverage cans and automotive components.¹,² It is of great importance that the material’s quality should be homogeneous over the entire length of a strip. Since grain size is one of the factors which influence the material properties of steel products, it is important to maintain the grain size within a permissible range.

It is well known that information from laser-ultrasonic (LUS) techniques about microstructural and metallurgical properties such as texture and grain size can be used to extract the measured ultrasonic wave velocity and attenuation in the materials.³⁻⁵ Results have also been published on austenite recrystallization and grain growth in steel alloys, studied in-situ by Dubois et al. and Moreau et al., among others.⁶⁻⁷

In rolled strip steels, the grain size is influenced by various kinds of thermo-mechanical processes applied by the rolling mill (hot and cold rolling and annealing). Characterization of steels, during production as well as development, is usually carried out using costly and time-consuming methods such as microscopy and mechanical tests. The combined results of the studies presented in the literature can be used as a continuous, on-line measuring method.

Ultrasonic waves can penetrate several centimeters in metals, although the wave amplitude becomes progressively attenuated by the microstructure as it travels over longer distances. The degree of attenuation experienced is dependent on grain size with large grains having a dominating influence. Depending on the wavelength/grain size ratio ($\lambda/D$), the attenuation $\alpha$ can be described as a Rayleigh region ($\alpha(\lambda, D) = C_D D^4/4$, $\lambda \ll D$), a stochastic region ($\alpha(\lambda, D) = C_D D/2$, $\lambda \approx D$), and a diffusive region ($\alpha(\lambda, D) = C_D D^2/2$, $\lambda \gg D$), where $C_D$, $C_s$, and $C_a$ are constants of the material and $D$ is the diameter for spherical grains.

For different kinds of steels, it is not expected that the measurement of grain size will be an easy task; in such cases, the authors believe that one kind of steel should have a specific equation and material constant $C_R$. In this paper, the constant, $C_R$, of Interstitial Free (IF) steel at room temperature in the Rayleigh region is calculated, and the average grain size and errors are predicted by the calculated constant of IF steel $C_R$.

2. Experimental

The samples inspected are hot rolled, IF steel. Four samples with dimensions 75 mm × 65 mm × 3.35 mm were cut from the coil for experimental study. Three of them were annealed in order to adjust the grain size, with a holding temperature of 800°C and holding time of 30⁻90 min. The chemical composition and annealing experiment of the four samples is shown in Table 1.

Figure 1 shows the LUS measurement model. A Q-switched Neodumium: Yttrium–Aluminum–Garnet (Nd:YAG) pulsed laser (wavelength = 532 nm, Continuous Laser Power = 100 mW, and pulse width = 4 ns) acted as the transmitter of the ultrasonic wave. The detection equipment is an IOS TWM-1550 laser ultrasonic receiver (bandwidth 120 MHz, wavelength 1550 nm).

3. Analysis and Results

3.1 Analysis

After acid pickling and electrolytic polishing, the grain sizes of the four samples were determined from EBSD analyses by measuring the average linear intercepts. The
microstructure and average grain size of the four samples were shown in Fig. 2.

In this section, a method to calculate the grain size from measurements of the ultrasonic attenuation in the detected IF steels will be presented. It is assumed that the main contribution to the attenuation is the scattering of the ultrasonic pulse at the grain boundaries, while other contributions such as absorption at dislocations and precipitates will be neglected.

The surface vibration caused by ultrasonic wave is detected by the IOS TWM-1550 laser ultrasonic receiver. Figure 3 shows examples of the detected ultrasonic waveforms and Fourier transforms of the first echo (1P) and the second echo (3P). The vertical axis of the detected ultrasonic waveforms (Fig. 3, left) represents the amplitude of ultrasonic wave detected by the laser ultrasonic receiver, and the horizontal axis shows the time following irradiation by the pulsed laser. It is found that the echoes become weaker as the grain size increases, to the point where IF3 and IF4 (samples with large grain size) only have two. From Fig. 3 (right), the variations of intensity as a function of frequency have been obtained by Fourier transform of the 1P and 3P echoes. It appears that the intensity of the echoes become weaker at higher frequencies, and in most cases, the intensity of the 1P echo is larger than the 3P echo. This is mainly due to scattering during the ultrasonic wave propagation through the steels.

3.2 Results

One way to obtain the attenuation from the ultrasonic measurement is from the relative difference in intensity between two successive echoes. In this way, the attenuation parameter can be calculated by the relationship in eq. (1), where, $U_1$ and $U_2$ are the intensities of the 1P and 3P echoes, respectively, and $L$ is the thickness of the sample.

$$\alpha = \frac{20}{2L} \log(U_1/U_2)$$ (1)

Based on the Fourier transforms of the 1P and 3P echoes, the attenuation parameters of each sample were calculated and are presented in Table 2. As the average grain size increases, it is found that attenuation parameters also.

The grain size can be determined using the Rayleigh’s scattering law (eq. (2)), where $D$ is the grain size (mm), $\alpha$ is the attenuation rate (dB/mm), $f$ is the frequency (Hz), and $C_r$ is the constant of the material.

$$\alpha(D) = C_r D^3 f^4$$ (2)

With a known average grain size by EBSD and the attenuation parameters of each sample by LUS, the material constant $C_r$ can be then calculated (Table 3).

Using the calculated material constant $C_r$ of IF1 presented, the average grain size of IF2 can be calculated. Comparing the calculated average grain size of IF2 (7.4 µm) to the detected average grain size, the size is underestimated with an error of 6.7%. In a similar fashion, the calculated average grain sizes for sample IF3 and IF4 are 38.6 and 69.7 µm, with respective errors of 15.8 and 33.8%.

From the results presented in Table 3, there exists a close relation between grain size obtained from EBSD image analysis and grain size calculated from ultrasonic wave analysis for IF1 and IF2. However, when the grain size is significantly larger between reference samples, i.e., IF3 and IF4 compared to IF1, the technique begins to fail as evidenced by larger errors. This implies that ultrasonic wave analysis can be used to determine grain size, within some limitations.

4. Industry Application in the Future

Industries involved in the production and use of iron require products with high performance and uniformity quality. The conventional testing for quality and mechanical properties mainly relies on static, offline methods that are destructive. Although these methods can obtain the performance parameters of the final products, they are less efficient,
prone to high variability, cannot detect the microstructure and mechanical properties of the steel, and are unable to control and monitor the quality of the products during the actual production process. It is therefore of strategic significance to investigate and develop on-line quality control and property determination technology for use as a real-time, non-destructive part integrated into the steel production process. In this way, the overall quality of steel products can

Table 2: Attenuation parameters of IF steel samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Average grain size</th>
<th>IF1</th>
<th>IF2</th>
<th>IF3</th>
<th>IF4</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>µm</td>
<td>MHz</td>
<td>dB/mm</td>
<td>MHz</td>
<td>dB/mm</td>
</tr>
<tr>
<td>IF1</td>
<td>5.2 µm</td>
<td>9.901 MHz</td>
<td>0.7816 dB/mm</td>
<td>0.8247 dB/mm</td>
<td>38.6 µm</td>
</tr>
<tr>
<td>IF2</td>
<td>7.4 µm</td>
<td>6.623 MHz</td>
<td>0.7311 dB/mm</td>
<td>0.8016 dB/mm</td>
<td>69.7 µm</td>
</tr>
<tr>
<td>IF3</td>
<td>38.6 µm</td>
<td>9.901 MHz</td>
<td>0.7816 dB/mm</td>
<td>0.8247 dB/mm</td>
<td>38.6 µm</td>
</tr>
<tr>
<td>IF4</td>
<td>69.7 µm</td>
<td>6.623 MHz</td>
<td>0.7311 dB/mm</td>
<td>0.8016 dB/mm</td>
<td>69.7 µm</td>
</tr>
</tbody>
</table>

Table 3: Calculated results and errors.

<table>
<thead>
<tr>
<th>Sample name</th>
<th>Detected grain size/µm</th>
<th>Material constant /dB/(mm·Hz)^-1</th>
<th>Calculated grain size/µm</th>
<th>Error/%</th>
</tr>
</thead>
<tbody>
<tr>
<td>IF1</td>
<td>5.2</td>
<td>5.96E-13</td>
<td>5.2</td>
<td>6.7</td>
</tr>
<tr>
<td>IF2</td>
<td>7.4</td>
<td></td>
<td>6.9</td>
<td>6.7</td>
</tr>
<tr>
<td>IF3</td>
<td>38.6</td>
<td></td>
<td>44.7</td>
<td>15.8</td>
</tr>
<tr>
<td>IF4</td>
<td>69.7</td>
<td></td>
<td>46.1</td>
<td>33.8</td>
</tr>
</tbody>
</table>

Fig. 3 Ultrasonic waveforms (left) and Fourier transforms (right) for the four steel samples (1P: the first echo; 3P: the second echo; 5P: the third echo).
be improved and products with a high degree of stability, uniformity, consistency of quality, and performance can be produced.

The ultimate purpose of an on-line measurement in the production line is feedback control of the production process itself. This is only possible if the quality of interest that is to be controlled for can be quantitatively measured. To accomplish this goal, laboratory-scale equipment is being assembled for on-line measurement by laser ultrasonic at the University of Science and Technology Beijing. Figure 4 shows the sketch of a proposed on-line laser ultrasonic measurement system. A Nd:YAG laser is employed to simultaneously generate these ultrasonic modes, and for the ultrasonic detection, a long pulse, another Nd:YAG laser is coupled to a photorefractive interferometer by optical fibers.

5. Conclusion

The average grain sizes and material constants, \( C_r \), of four IF steels were determined and analyzed by laser ultrasonic. Using the calculated \( C_r \), the average grain sizes of other IF steel samples were calculated and a close relationship between the grain size obtained from EBSD image analysis and grain size calculated from ultrasonic wave analysis was found. These results imply that grain size determination by laser ultrasonics may be useful for on-line measurement.

The sketch of an on-line laser ultrasonic measurement system was given and laboratory-scale equipment is being assembled at University of Science and Technology Beijing to test the feasibility of this type of grain size detection setup. The eventual goal of an on-line measurement system is to provide for feedback control of the production process itself.

Acknowledgement

This research is supported by the National Key Technology R&D Program of the 12th Five-year Plan of China (Grant No. 2012BAF04B02). The authors also wish to acknowledge Dr. Troy Munro for the English writing.

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