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Monitoring austenite decomposition by ultrasonic velocity

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Abstract

The ultrasonic longitudinal velocity measured by the laser-ultrasonic technique is compared to dilatometry for the monitoring of austenite decomposition of low alloy steels. It is demonstrated that the technique could be an interesting alternative to dilatometry. The temperature dependence of the ultrasonic velocity and the various phases in steels is determined and used to calculate the decomposed austenite by a simple law of mixtures approximation. As a non-destructive and non-contact technique, laser-ultrasonics can be applied to monitor austenite transformation of real products in an industrial production line, which would be much more difficult with dilatometry.

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Keywords: Laser-ultrasonics; Steel phases; Austenite; Dilatometry

1. Introduction

The decomposition of high temperature austenite (fcc iron) into more stable carbon–iron compounds (ferrite, pearlite, bainite, etc.) during cooling, is of prime technological importance, given that this phase transformation determines to a considerable extent the microstructure, and consequently the properties, of steels. The experimental study of the transformation kinetics is complementary to modelling to determine optimised steel processing parameters. The development of advanced steels depends on quantitative understanding of austenite decomposition, calling for more accurate and robust experimental methods able to sense this phase transformation.

A technique commonly used to experimentally monitor steel phase transformation is dilatometry. This technique determines the remaining austenite fraction by assuming that the thermal expansion during decomposition is a volume-fraction-weighted average of the thermal expansion of the various decomposition products. This simple method, in spite of its inaccuracy for medium-carbon steels, is widely used due to its simplicity and low cost. A more accurate estimation of decomposed fraction can be obtained by using more complex dilatometric data analysis that accounts for volume changes of carbon enrichment of austenite and the distinct specific volume of pearlite

and ferrite [1]. Other methods for real-time phase transformation monitoring are calorimetry and X-ray or neutron diffraction [2,3].

Previous works have shown that ultrasound velocity and attenuation are parameters that can vary considerably during austenite decomposition [4,5]. Although the change in the velocity is stated to be directly correlated to the transformed fraction [4], the quantitative correlation has not yet been demonstrated. This paper reports recent work on the validation of ultrasonic velocity to quantitatively monitor the austenite decomposition. Laser-ultrasonics, due to its non-contact characteristics, is the technique of choice to perform ultrasonic measurements at high temperatures. Its demonstrated capability to perform accurate real-time measurements in laboratory conditions [6], and in-line measurements in industrial environments [7], has generated great interest on the technique for monitoring metallurgical transformations.

2. Steel phases and ultrasonic velocity

The ultrasonic velocity dependence on the phase fraction of a multiphase compound is a classical and complex problem of acoustics. The main input parameters to models are the elastic constants, density and the morphology (geometry) of the constituent phases. For constituent phases of similar elastic properties and densities, these models can be greatly simplified to a volume-fraction-weighted average of individual constituent

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Table 1
Properties at room temperature of cementite [8], ferrite [9], martensite [10] and austenite [9,4]

| Property | Ferrite (bcc) | Martensite | Fe ₃ C | Austenite (fcc) |
|------------------------------|---------------|------------|-------------------|--------------------|
| Density (g/cm ³) | 7.877 | 7.863 | 7.68 | 8.147 |
| T Curie (°C) | 770 | – | 215 | – |
| Young's modulus (GPa) | 211 | 208 | 190 | ? |
| Shear modulus (GPa) | 82 | 81 | 72 | ? |
| Longitudinal velocity (m/s) | 5900 | 5900 | 5945 | ≈5600 ^a |

^a Obtained by extrapolation from high temperatures.

velocities, and they become independent of phases morphology. The most common steel phases, ferrite (bcc), martensite (metastable bct), cementite (Fe₃C) and austenite (fcc), have similar elastic moduli and densities. The ultrasonic velocity of the compound (V) can then, with a good approximation, be expressed by a law of mixtures:

$$V = f_{\alpha} V_{\alpha} + f_M V_M + f_{\text{Fe}_3\text{C}} V_{\text{Fe}_3\text{C}} + f_{\gamma} V_{\gamma} \quad (1)$$

where f_i is the volume fraction and V_i is the velocity for the i -phase ($\sum f_i = 1$). The steel phases being considered are ferrite (α), martensite (M), cementite (Fe₃C) and the fraction of remaining (or retained) austenite (γ).

The ultrasonic properties for these steel phases found in literature are often contradictory. Table 1 shows ultrasonic velocities and other related properties for steel phases at room temperature taken from selected references. The most reliable values are those for ferrite. For the other phases, the properties are difficult to obtain due to the instability of the phase at room temperature (austenite) or problems in obtaining bulk samples of 100% phase (cementite). The elastic constants of low alloy austenite at room temperature are, to the best knowledge of the authors, unknown. Extrapolating the ultrasonic velocity measured at high temperatures by assuming a linear dependence on temperature, give a value of about 5600 m/s [4,11] that is a significantly lower value compared with other steel phases. The velocity of each phase has its own temperature dependence, which is often a linear function of temperature. One important exception for this habitually linear behavior is for ferromagnetic phases, where the magnetic component of the elastic modulus is not linear with temperature and the ultrasonic velocity shows significant perturbations near magnetic transitions.

For compounds of more than two components (often the case for steels), Eq. (1) does not permit the volume fraction determination for each phase because there are more unknown variables (the various fractions) than known variables (measured velocity). This also is the limitation for dilatometry, where the specific volume of each phase as a function of temperature is known, but only one parameter is measured (the sample length). However, in practice, the determination of more than two phases is often possible because they do not transform simultaneously. For example, a hypo-eutectoid steel, when cooled at low cooling rates, has the austenite-to-ferrite transformation completed before the austenite-to-pearlite transformation takes place. The measurement of other ultrasonic parameters, like another wave mode (e.g. shear wave), should provide an independent measurement that could permit simultaneous determination for three

phases, but this possibility will not be further investigated in this paper.

3. Experimental methods

The laser-ultrasonic technique, described elsewhere [5,6], is used to measure the ultrasonic velocity in steel samples heated and cooled in a Gleeble 3500 thermomechanical simulator. Most of the samples used are about 2 mm thick, 150 mm long and 30 mm wide. The laser-ultrasonic measurements are done in the center of the sample, close to the welded thermocouple. The heating and cooling rate was 1 °C/s unless otherwise specified. Early measurements were done with an excimer laser for generation and a long pulse Nd:YAG for detection but later measurements were done with a compact laser-ultrasonic system based in a Nd:YAG laser doubled frequency for generation and the pulsed Nd:YAG for detection. Because both are high-performance systems, a very good signal-to-noise ratio is obtained and results are independent of the system used. The velocity values are calculated by the ratio of the time delay between echoes and the thickness measured at room temperature. The velocities reported in this paper are of longitudinal waves. The correction for thickness variation due to thermal expansion is small and is not done to simplify data processing. Dilatometric measurements are simultaneous to the laser-ultrasonic measurements, with quartz grips of the dilatometer attached to the sample to measure the variations of the width of the sample. With this configuration, due to temperature gradient found in the sample, the dilatometer does not provide accurate measurements on the location where the temperature is measured, but an averaged value for a certain region of the sample. This is a problem for wide samples with sudden phase transformations. Although not optimal, the dilatometric measurements provide valuable qualitative information about the start and end of the austenite decomposition.

4. Ultrasonic velocity and austenite decomposition for plain carbon steels

The ultrasonic velocity is monitored during cooling for different carbon content plain carbon steels in order to investigate the dependence of ultrasonic velocity with temperature and phases. Fig. 1 shows the ultrasonic velocity measured during cooling for a low carbon steel (0.08% C). The austenite decomposition temperature range, as determined by dilatometry with a 10–90% criteria, also is indicated in the figure. Although the lower temperature phases are a combination of ferrite and pearlite, the

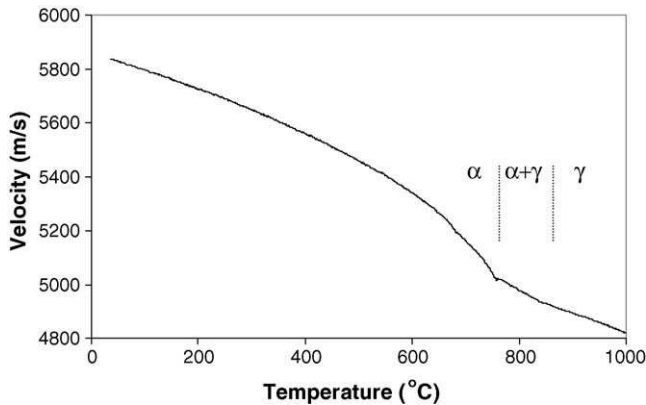


Fig. 1. Ultrasonic velocity measured during cooling for a 1008 steel sample.

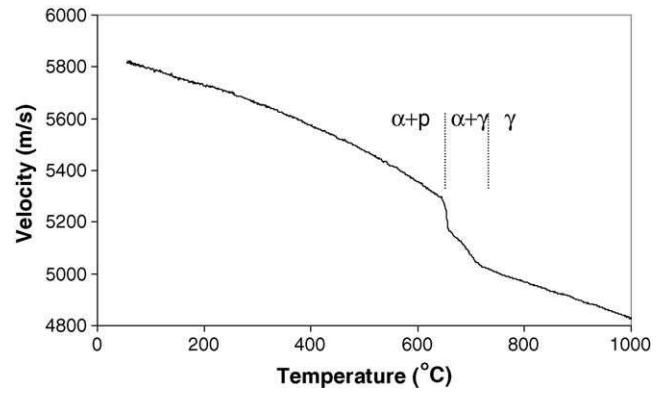


Fig. 3. Ultrasonic velocity measured during cooling for a 1035 steel sample.

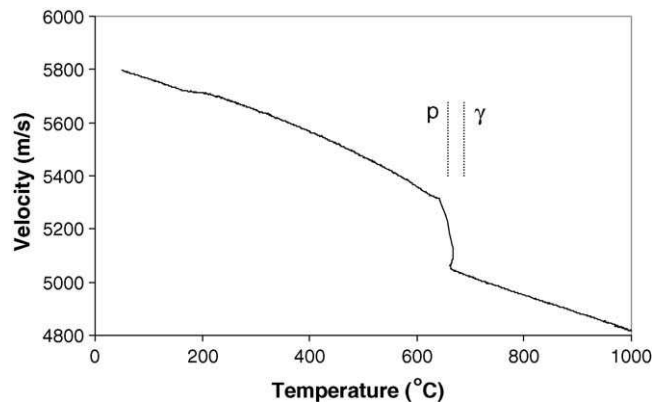


Fig. 4. Ultrasonic velocity measured during cooling for a 1074 steel sample.

pearlite fraction is small (about 10%) and this steel can be considered mostly a ferritic phase steel. The velocity variation in the austenite range (down to about 850 °C) is mostly linear. During the austenite decomposition (from 850 to 750 °C), the velocity dependence remains almost linear, but with a different slope than that of the austenite range. For the ferritic phase (below 750 °C), the velocity follows a non-linear behavior already reported for pure iron and low carbon steels [4,11–14] that shows a prominent inflection at the Curie temperature (about 770 °C). Fig. 2 shows the velocity curve for a 0.2% C steel. In the austenite phase (down to 770 °C), the velocity also presents a linear dependence to temperature. There is a clear inflection when the ferrite starts to form (770 °C) and another inflection when the remaining austenite decomposes into pearlite (670 °C). The velocity curve presented in Fig. 3 for the 0.35% C steel is similar to that of 0.2% C steel, but with a lower temperature for starting the decomposition (730 °C). Also, the pearlite formation is more pronounced at about 650 °C. For the 0.74 % C steel presented in Fig. 4, the velocity curve has a sharp inflection at 660 °C, where the austenite starts to decompose into pearlite. The transformation ends about 20 °C below. There is a small self-heating of the sample, due to the well-known exothermal character of this transformation. Fig. 5 shows the curves for the four plain carbon steels with a zoom in the temperature range of 600–1000 °C. The slope of the velocity dependence on temperature in the austenite phase seems to be mostly independent of carbon content.

The absolute accuracy of the ultrasonic velocity for relatively thin samples is limited by the accuracy of thickness determination and is estimated for the present measurements to be 0.5%. This error is larger than the difference of velocity observed for different carbon contents samples. Therefore, a possible effect of the carbon content on the velocity in the austenite phase is small and within the error bar of the present measurements. The ultrasonic velocity dependence on temperature for ferrite and cementite is more complex. The ferrite has an important perturbation near the Curie temperature, at about 750 °C, due

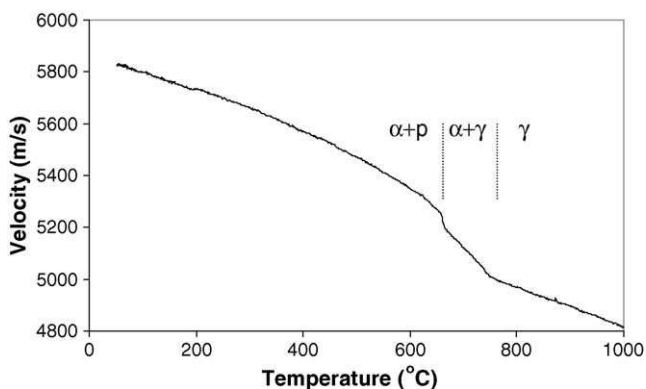


Fig. 2. Ultrasonic velocity measured during cooling for a 1020 steel sample.

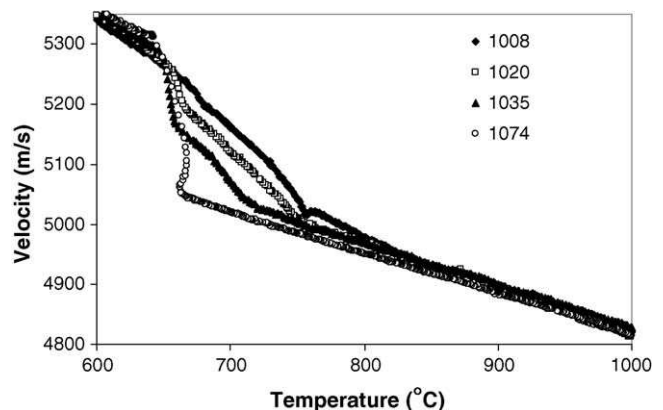


Fig. 5. Ultrasonic velocity for the different carbon content steels in the range of austenite decomposition.

the ferromagnetic to paramagnetic transformation. In the ferromagnetic domain, the slope of the velocity versus temperature curve for ferrite becomes increasingly negative when approaching the Curie temperature (see Fig. 1). For paramagnetic ferrite, the slope of the velocity curve cannot be determined for the steel grades tested, because there is always some phase transformation in this temperature range. Measurements on ultra-low carbon steels have shown a linear behavior with a slope slightly more negative than that of austenite. The cementite has a Curie temperature at about 200 °C that is easily observed by an inflection in the velocity curve when pearlite fraction is significant (see Fig. 4).

5. Ultrasonic velocity lever-rule method

The possibility of using the differences of ultrasonic velocity for each iron–carbon phases to quantitatively determine the phase contents and, more specifically, to fully characterize the austenite decomposition, is discussed below. As already mentioned, there is only one parameter being measured, the longitudinal ultrasonic velocity. Consequently, only two phases could be unambiguously evaluated. The results presented in the previous section show the velocity dependence on temperature for austenite, ferrite and pearlite. No martensite is expected for the present steel grades and cooling rate. Results from the literature suggest that there is not a significant difference for the ultrasonic velocity in ferrite and martensite at room temperature. In this paper, decomposition of austenite in martensite will not be further investigated. For practical purposes it will be considered to have the same ultrasonic velocity as ferrite.

5.1. Austenite decomposition into ferrite

The general form of the velocity curves for austenite and ferrite is illustrated in Fig. 6. The phase fraction for a given temperature and measured velocity can be obtained via a lever-rule method commonly used in dilatometry. With zero fraction of martensite and cementite in Eq. (1) and with the sum of ferrite and austenite fraction equal to one, Eq. (1) can be re-written as:

$$f_{\gamma} = \frac{V - V_{\alpha}}{V_{\gamma} - V_{\alpha}} \quad (2)$$

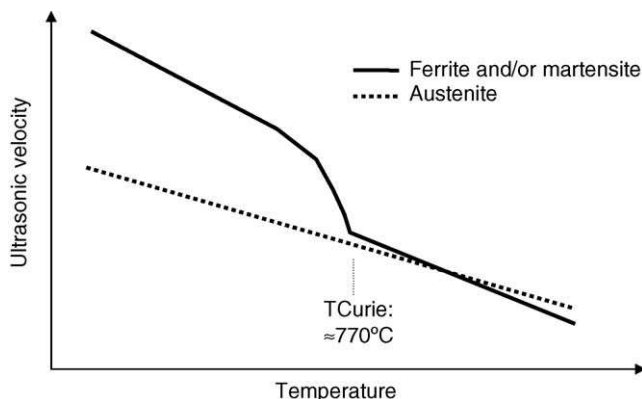


Fig. 6. Ultrasonic velocity dependence on temperature for various phases.

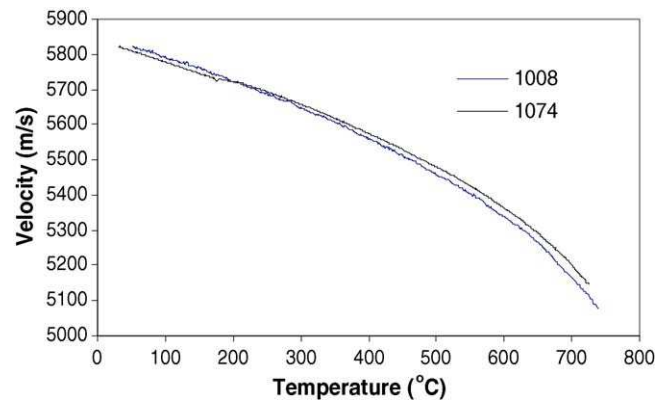


Fig. 7. Velocities for a mostly pearlitic (0.75% C) and a mostly ferritic (0.08% C) steel.

From Fig. 6, it is evident that the method will be much more precise below the Curie temperature, where the velocity difference between the phases is larger. At temperatures where the velocity for austenite and ferrite are similar, the method cannot be applied.

5.2. Austenite decomposition into pearlite

The procedure to determine the pearlite fraction should be the same as that of ferrite, except for the use of the velocity curve for pearlite. The velocity difference for a mostly pearlitic and a mostly ferritic steel is shown in Fig. 7. This difference is larger for higher temperatures, where the velocity for pearlite is higher, but this difference diminishes for lower temperatures. Below cementite Curie temperature (≈ 200 °C), the pearlite has the velocity lower than that of ferrite. The larger differences are still small and in the order of 0.8%, which is about one tenth of the difference between austenite and ferrite below Curie temperature. Therefore, if the ferrite velocity curve is used to calculate the fraction of austenite decomposed into pearlite, the error should be of less than 10%.

6. Ultrasonic and dilatometry lever-rule method comparison

The conventional dilatometric lever-rule method is compared to the ultrasonic lever-rule first approximation, where the velocity differences between ferrite and pearlite are ignored. Fig. 8 shows the velocities for ferrite and austenite determined experimentally and extrapolated for low temperatures for the austenite and for high temperatures for ferrite. The figure also shows the velocity measured during cooling for a low alloy steel. The fraction of austenite in function of temperature for this steel sample, calculated by Eq. (2), is shown in Fig. 9, where the fraction determined by dilatometry also is shown. Another example is shown in Fig. 10, where identical thermal cycles are imposed on a pearlitic steel sample in a dilatometric furnace and to a same material sample in a Gleeble thermomechanical simulator (for the laser-ultrasonic measurements). The example of Fig. 9 suggests that, although the techniques show the same pattern, they slightly differ quantitatively on the start and end of different

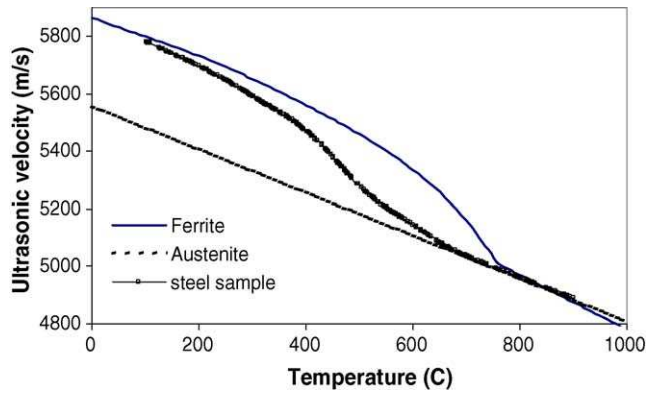


Fig. 8. Ultrasonic velocities for ferrite (solid line), austenite (dotted line) and measured for low alloy steel during cooling (line + symbol).

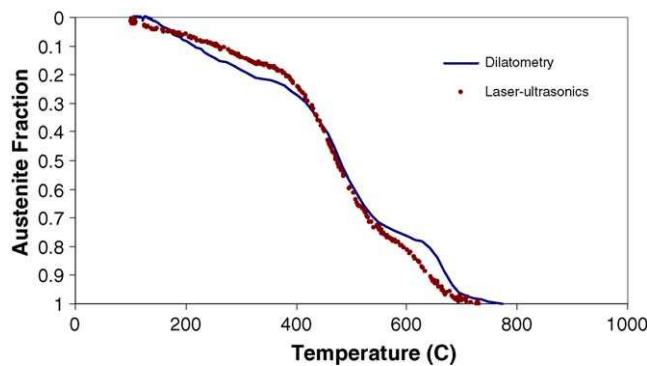


Fig. 9. Austenite fraction determined by the velocity curve shown in Fig. 8 (solid line) and determined by dilatometry (squares).

phases. For the example presented in Fig. 10, the transformation measured by ultrasonic velocity occurs in a shorter temperature range and does not show an overshoot at the end of the transformation. The differences found in the ultrasonic velocity and the dilatometric techniques can have many origins. First, the measurements by both techniques were not simultaneous. Different samples from the same material were used. Smaller differences in thermal cycles and measured temperatures are possible and the samples could have metallurgical differences. This is the most probable reason for differences between the techniques found

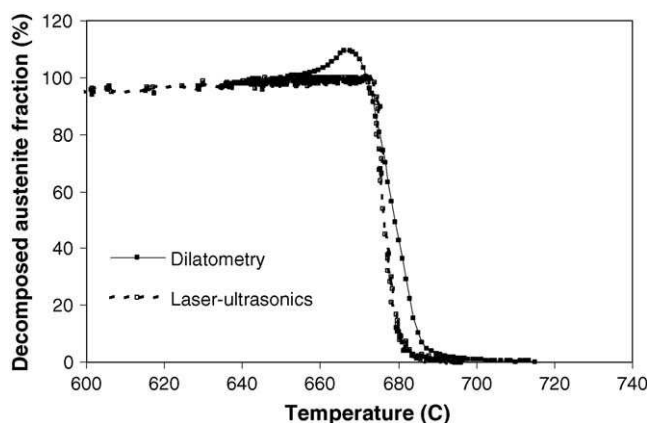


Fig. 10. Austenite fraction determined by dilatometry (line + symbols) and laser-ultrasonics (symbols).

in the example of Fig. 10. Second, for both techniques, a rough approximation was used. For the dilatometry, the enrichment of the austenite by carbon and the effect of different thermal expansion of cementite were not taken into account. For the ultrasonic velocity technique, the effect of the cementite was not taken into account. These are probable reasons for the differences found in the example of Fig. 9. These examples demonstrate the potential of ultrasonic velocity measurement as a new experimental tool to monitor austenite decomposition. The advantages of the laser-ultrasonic technique compared to dilatometry appear to be:

- As a remote all-optical, non-destructive technique, it could be applied in harsh industrial environments. It has the potential to verify model predictions in real products and to be used continuously as an in-line quality control tool.
- Absolute measurements of velocity permit the determination of the remaining austenite at any time. This should be especially useful after deformation, where a reference of sample length is lost.
- The dependence of the velocity on carbon in solid solution seems to be negligible, making accurate determination of the transformed fraction more robust. This is a serious limitation for the accuracy of dilatometry.
- Measures on materials with large gradients of temperature are possible, provided the sound path is in a region where the temperature is constant.
- Measurement of other ultrasonic parameters, like the velocity of the transversal waves, is possible. If such a parameter has a distinct value for the present material phases, the fraction three phases could be simultaneously determined.

The limitations of the technique are:

- Measurement system is more complex and more expensive.
- Precise measurements can be made only below the Curie temperature of iron ($\approx 770^\circ\text{C}$).
- For very coarse microstructures in thin samples, the statistics can be poor and the measured velocity can be dependent on only a few grains.
- If the start or final phases are strongly textured, the quantitative determination of each phase fraction will not be accurate.

7. Conclusion

Quantitative monitoring of austenite decomposition by the ultrasonic velocity in low alloy steels is demonstrated. The proposed method, based on the rule of mixtures of present phases, is very simple to apply. It also seems to be more robust than similar analysis applied to dilatometry, due to the insensitivity of the ultrasonic velocity to carbon in solid solution. As a non-contact technique already in use in harsh industrial environments, laser-ultrasonics can be used to verify, in real production conditions austenite decomposition models, which is much more difficult with dilatometry. Also, because accurate absolute measurement of velocity is possible, it is possible to determine remaining (or retained) austenite fraction in steels, making the technique especially attractive for quality control of steels with a fraction

of retained austenite, like TRIP steels or hardened high carbon steels.

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