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Phase Transformation Monitoring by Dilatometry and by Laser-Ultrasonic Velocity techniques on grades DP780 and QP

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Short Summary: Phase transformation during cooling is evaluated on two industrial steel grades, a DP780 and a QP, using two different techniques: the laser-ultrasonic velocity technique and the dilatometry technique. Results analysis for the DP780 grade show that the lever rule method used with the dilatometry technique needs to be corrected to consider two metallurgical phenomena to agree with laser-ultrasonic velocity results: the austenite enrichment in Carbon during austenite to ferrite transformation and the difference of volume between ferrite and pearlite. Results analysis for the QP grade show that the laser-ultrasonic velocity technique, in contrast to the lever rule method for dilatometry, detects some austenite transformation in ferrite at high temperature, prior to martensite transformation, which is confirmed by metallographic observations. Then the massive martensite transformation of QP at lower temperatures is detected correctly by both techniques, with no need of correction for the lever rule method for dilatometry. This is because martensite transformation does not involve any Carbon enrichment in austenite during transformation.

EXPERIMENTAL PROCEDURE

Steel grades:

Phase transformation during cooling has been monitored on two different steel grades using dilatometry and laser-ultrasonic velocity techniques. Chemical compositions are in Table 1.

Table 1: Steel grades chemical composition in major elements (weight %)

Grade	C	Mn	Si	Cr	Al	Mo	B
DP780 grade	0.136	2.09	0.208	0	0.027	0	0
QP grade	0.37	1.95	1 .95	0.35	0	0.12	0

DP780 produces mainly ferrite, pearlite and martensite phases, while the QP grade is expected to produce mainly martensite. As a result, the two techniques of transformation monitoring can be evaluated against this wide range of transformations with these two grades.

Dilatometric tests:

A Bahr dilatometer DIL805L was used for dilatometric tests with four different cooling rates: 0.1, 1, 5 and 10ºC./sec. after an austenitization of 950ºC during 300 sec. After the trials, microstructures of samples were analyzed by metallography. To evaluate the transformation kinetics with the dilatometry technique, the classic lever rule method is used**:** on the dilatometric curve, the experimental slopes for the austenite and austenite transformed phases are used for the lever rule formula.

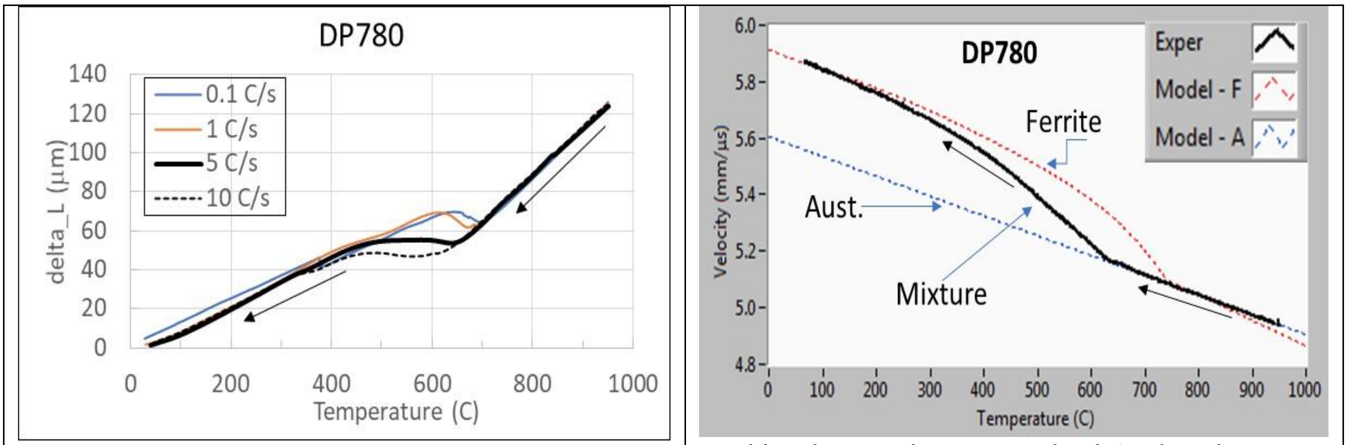
Laser-ultrasonic velocity tests:

Ultrasonic velocity measurements were performed during cooling tests conducted on a Gleeble-3500 machine on samples of same grade as dilatometric tests. The ultrasonic velocity method detailed in **[1]** was used to deduce the transformed austenite.

EXPERIMENTAL RESULTS and DISCUSSION

<u>Grade DP780:</u>

Figure 1 shows the measured sample length variations (dilatometry) and the measured ultrasonic velocity for DP780 grade: a large amount of ferrite, pearlite and possibly martensite is formed at all cooling rates.



a) dilatometric test results

b) ultra-sonic test results (5ºC./sec.)

Fig.1: DP780 grade during cooling

Using these measurements, the austenite phase fraction evolution has been estimated during cooling respectively by the lever rule method and by the ultrasonic velocity method **[1]**. Results on Figure 2 show a discrepancy between phase transformation kinetics obtained by dilatometry and by laser-ultrasonic velocity methods for the 4 cooling rates: the dilatometry tends to <u>over-estimates</u> the transformation kinetics compared to the laser-ultrasonics.

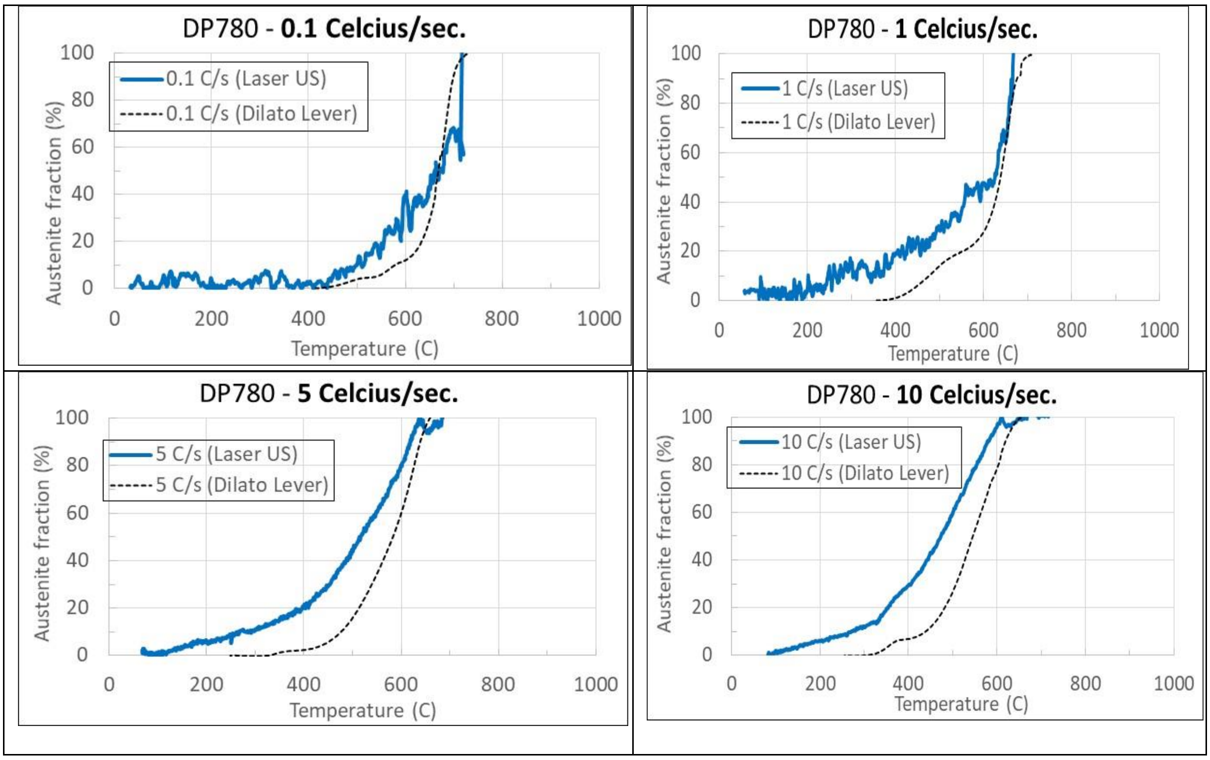


Fig.2: Transformation kinetics of DP780 obtained by classic dilatometry analysis **(lever rule method)** and by laser ultra-sonic analysis for different cooling rates

These discrepancies are believed to be due to the lever rule method used for dilatometry that does not consider Carbon enrichment in austenite during ferrite formation and that does not consider the significant difference of volume between ferrite and pearlite. Therefore, these two mechanisms have been introduced in the lever rule analysis using the following equations **[2]** to correct the dilatometric curves (lever rule corrected).

Lattice parameters equations for ferrite, austenite and pearlite used to correct lever rule **[2]**

TABLE I Lattice parameters of ferrite (α) and austenite (γ) and of the orthorombic phase cementite (θ) as a function of temperature T and the atomic fraction carbon (ξ) **[2]**

Phase	Lattice parameters (Å)
α	$a_\alpha = 2.8863$ Å $(1 + 17.5 \times 10^{-6}\,\mathrm{K}^{-1}\,[T - 800\,\mathrm{K}])$ $800\,\mathrm{K} < T < 1200\,\mathrm{K}$
γ	$a_\gamma = (3.6306 + 0.78\xi)$ Å $(1 + (24.9 - 50\xi)10^{-6}\,\mathrm{K}^{-1}\,[T - 1000\,\mathrm{K}])$ $1000\,\mathrm{K} < T < 1250\,\mathrm{K}; 0.0005 < \xi < 0.0365$
θ	$a_\theta = 4.5234$ Å $(1 + \{5.311 \times 10^{-6} - 1.942 \times 10^{-9}\,\mathrm{K}^{-1}\,T + 9.655 \times 10^{-12}\,\mathrm{K}^{-2}\,T^2\}\mathrm{K}^{-1}\,[T - 293\,\mathrm{K}])$ $b_\theta = 5.0883$ Å $(1 + \{5.311 \times 10^{-6} - 1.942 \times 10^{-9}\,\mathrm{K}^{-1}\,T + 9.655 \times 10^{-12}\,\mathrm{K}^{-2}\,T^2\}\mathrm{K}^{-1}\,[T - 293\,\mathrm{K}])$ $c_\theta = 6.7426$ Å $(1 + \{5.311 \times 10^{-6} - 1.942 \times 10^{-9}\,\mathrm{K}^{-1}\,T + 9.655 \times 10^{-12}\,\mathrm{K}^{-2}\,T^2\}\mathrm{K}^{-1}\,[T - 293\,\mathrm{K}])$ $300\,\mathrm{K} < T < 1000\,\mathrm{K}$

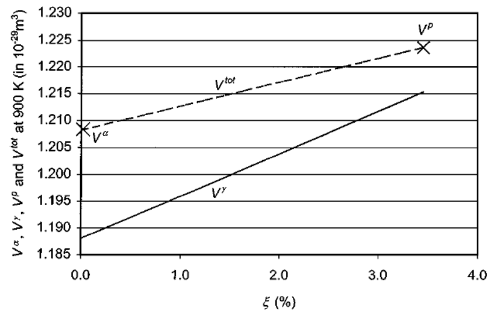


Figure 1 The atomic volumes of austenite, V^γ, and of a system composed of equilibrium fractions ferrite and pearlite, V^{tot}, depending on the carbon concentration at a temperature of 900 K. The atomic volumes of ferrite, V^α, and pearlite, V^p, are depicted as well.

Results on Figure 3 show that after correction, dilatometry and laser ultrasound are in very good agreement, except for the cooling rate 0.1 ^{o}C./sec. The reason for this remains unknown.

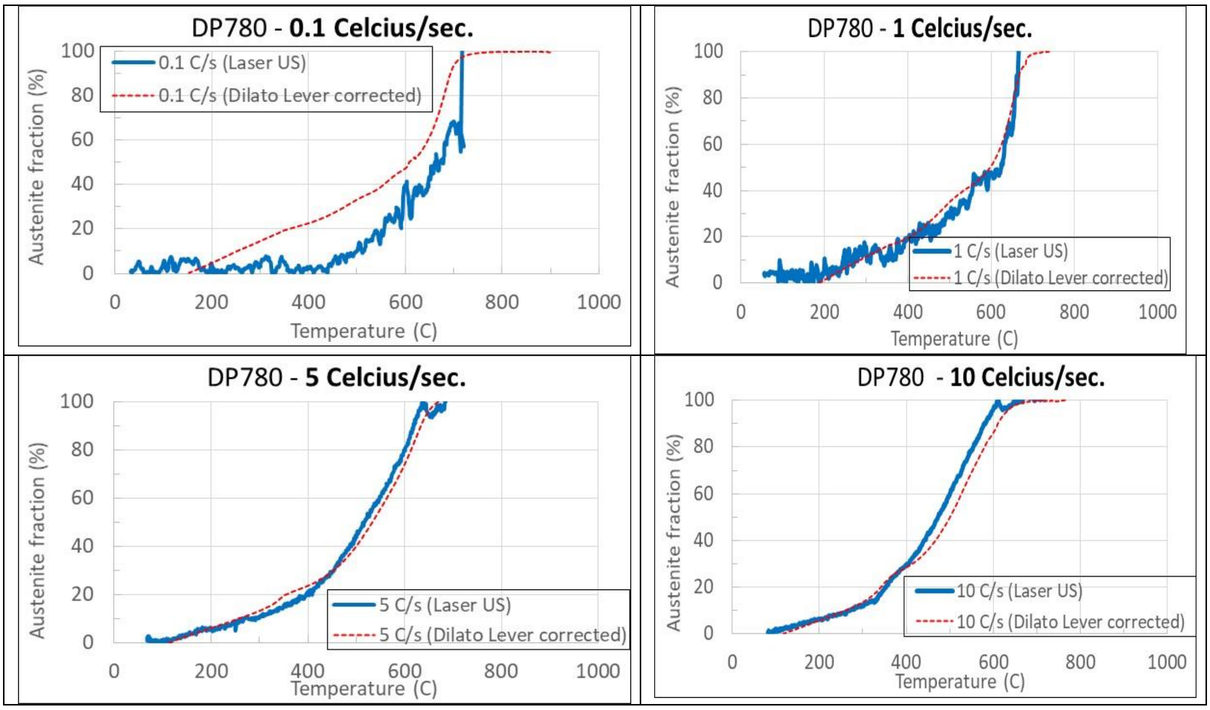


Fig.3: Transformation kinetics of DP780 obtained by modified dilatometry analysis **(lever rule method corrected [2]**) and by laser-ultrasonic analysis for different cooling rates

<u>Grade QP:</u>

Figure 4 shows the measured sample length variation (dilatometry) for the QP grade: at low cooling rate (0.1^{o}C./sec.), a large amount of ferrite (upper temperatures) and of bainite (lower temperatures) is formed. At higher cooling rates (1 and 5^{o}C./sec.), only martensite seems to form at ~300^{o}C.

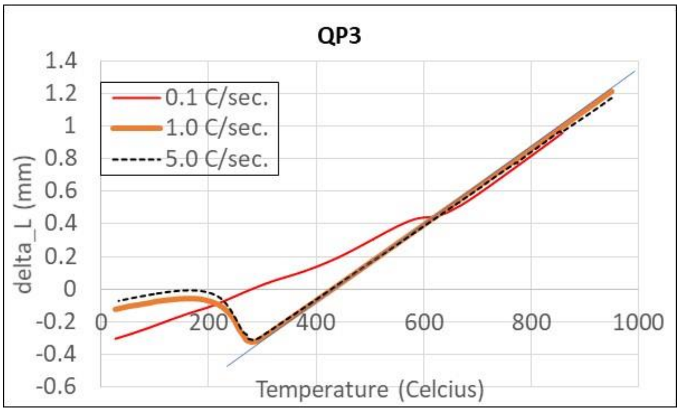


Fig.4: Dilatometric tests on QP grade during cooling at different cooling rates.

As for DP780 grade, the same comparison of transformation kinetics obtained by dilatometry and by laser-ultrasonic techniques has been made for the QP grade, as shown on Figure 5.

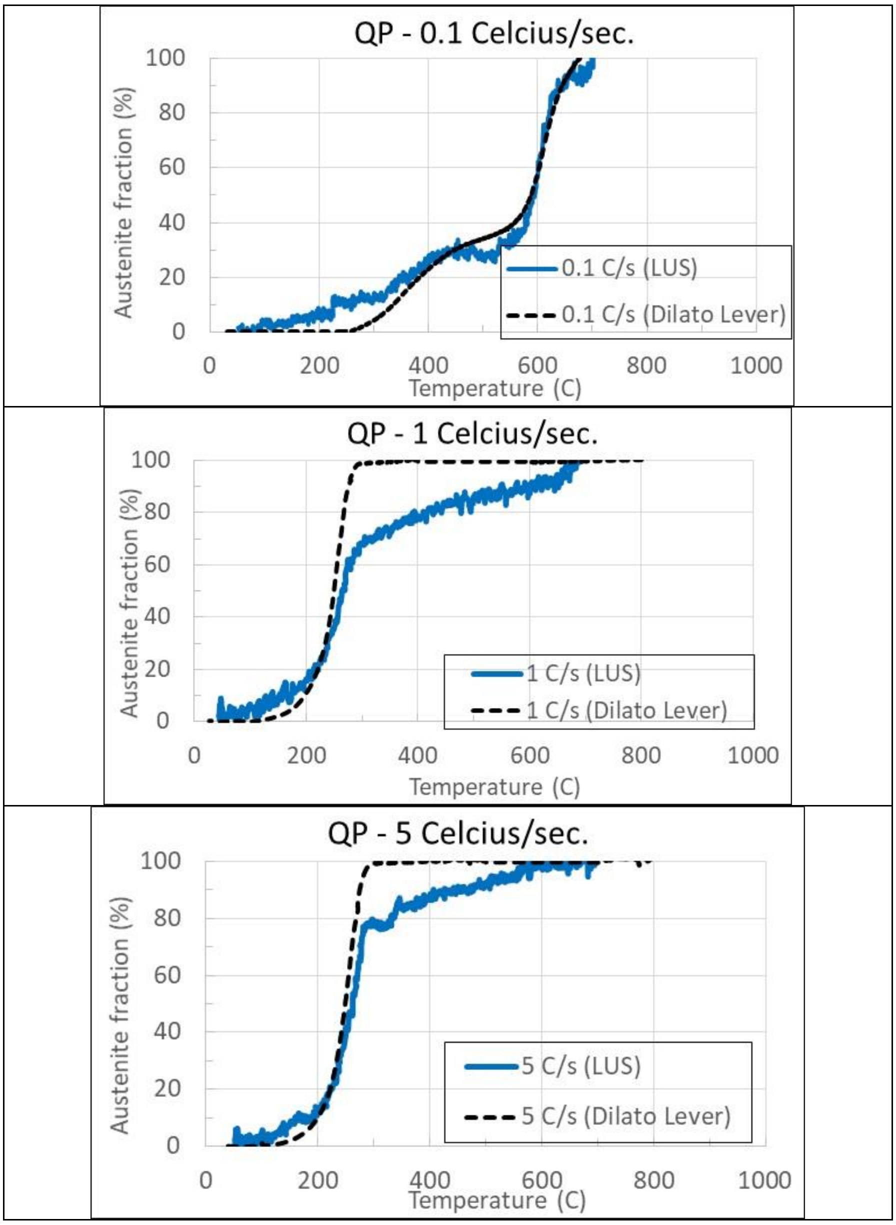


Fig.5: Transformation kinetics of QP grade obtained by classic dilatometry analysis **(lever rule)** and by laser ultra-sonic methods for different cooling rates

Here the massive transformation of martensite at ~300ºC. for cooling rates 1 and 5ºC./sec. is correctly monitored by the two techniques: the lever rule method seems to work relatively well here without any correction. This is because during austenite to martensite transformation, in contrast to DP780 (Figure 2), there is no Carbon enrichment in austenite so no need for correction. However, for 0.1ºC./sec., a correction of carbon enrichment should be needed because of the important amount of ferrite formed at ~ 600ºC. The reason for this remains unclear.

Also, Figure 5 shows an important difference between laser-ultrasonic and dilatometry analysis for high cooling rates (1 and 5ºC./sec.): the lever rule method indicates no other phase formation except martensite, while the ultrasonic velocity technique clearly indicates a certain amount of austenite has been transformed (probably to ferrite), prior to martensite transformation.

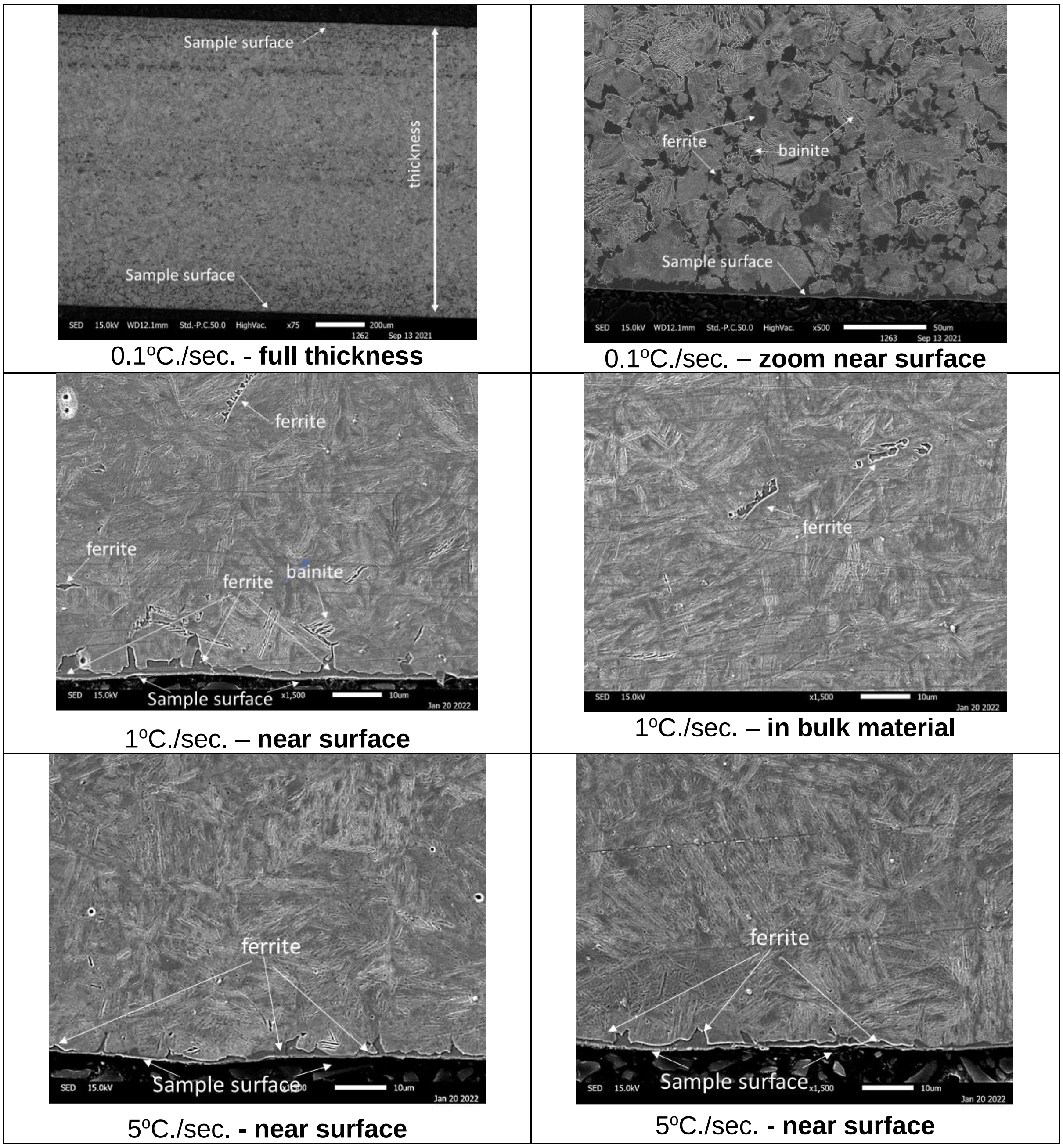


Fig.6: Microstructure obtained by metallography on QP grade after 3 different cooling rates (0.1, 1, 5ºC./sec.)

To verify this, metallography was conducted on the dilatometric samples: metallographic analysis is shown in figure 6.
At 0.1ºC./sec. (figure 6 top): a large amount of ferrite, bainite is observed, which confirms the indication of dilatometric curves of no martensite formation (Figure 4).
At 1ºC./sec. (Figure 6 middle): the presence of some islands of ferrite and bainite microstructures among a large amount of martensite microstructure is observed. These ferrite islands are also clearly seen near sample surfaces: this is probably due to a decarbonization of layer that promotes ferrite formation near the surfaces during cooling.
At 5ºC./sec. (Figure 6 bottom): ferrite seems present only near sample surfaces (decarbonization layer), while the bulk material seems exclusively martensite.

These results perfectly confirm the results of laser ultrasonic technique (Figure 5) that indicate a certain amount of ferrite and possibly bainite formation prior to martensite formation. Note that the laser-ultrasonic technique indicates that this amount of ferrite (prior to martensite) is higher at 1ºC./sec. than at 5ºC./sec. This is confirmed by metallography (Figure 6) which indicates a larger amount of ferrite/bainite islands in the bulk material at 1 ºC./sec. while at 5ºC./sec. ferrite/bainite islands are only at surfaces.

Therefore, the ferrite formation in the QP grade clearly detected by the laser-ultrasonic technique shows the superiority of this technique in the present conditions over dilatometry. The reason why dilatometry does not detect ferrite transformation at 1 and 5ºC./sec. for the QP grade is due to the remarkable linear behavior of the curve down to 300 ºC. (Figure 4) that is attributed to pure austenite before the martensite transformation around 300ºC.

CONCLUSION

Two different techniques have been evaluated and compared to monitor phase transformation on two different steel grades: the laser ultrasonic and the dilatometric techniques. The analysis reveals that the classic lever rule method used for dilatometry needs to be corrected to consider the enrichment in Carbon during phase transformation in DP780 from austenite to ferrite and to consider the difference of volume between ferrite and pearlite. The analysis of QP grade reveals that the laser ultrasonic technique, in contrast to dilatometry, detects the formation of ferrite at high temperature prior to martensite formation, which is in agreement with metallography. The massive phase transformation in martensite at lower temperatures is detected similarly by both techniques without any need of correction of dilatometry for this QP grade. This is because of the absence of Carbon enrichment in austenite during the transformation in martensite. It is concluded that the correction of lever rule method proposed by **[2]** for austenite to ferrite-pearlite seems useful for usual cooling rates (1 to 10 ºC./sec.). Also the method proposed by **[2]** based on volume difference between the phases could certainly be applied to the lever rule method of dilatometry to detect ferrite formation on QP grade (Figure 4). However, for very low cooling rates (0.1ºC./sec.), the need of this correction could not be evidenced by our experiments and further investigations are needed to understand why.

REFERENCES

[1] Monitoring austenite decomposition by ultrasonic velocity, S. Kruger, E. Damm, Materials Science and Engineering A 425 (2006) 238-243.
[2] Dilatometric Analysis of phase transformations in hypo-eutectoid steels, T.A. Kop, J. Sietsma, S. Van Der Zwaag, Journal of Materials Science 36 (2001) 519-526.