Alloy Design for Enhanced Austenite Stabilization via Quenching and Partitioning

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ABSTRACT

Quenching & Partitioning (Q&P) has recently been proposed as a fundamentally new way of producing high strength steels containing martensite and carbon stabilized austenite. The increased carbon content of the austenite is obtained through carbon depletion of the martensite and transport to the austenite during a controlled heat treatment process. Alloying with carbide formation retarding elements is necessary.

The Q&P response of a variety of steel compositions was investigated via dilatometry. The alloys studied included Si, Al and/or Mo modifications. The (partial) replacement of Si by Al resulted in lower volume fractions of stabilized austenite which may have resulted from decomposition of the austenite at the quench temperature in the partitioning section. The addition of Mo to a CMnSi steel resulted in nearly constant austenite fractions as a function of partitioning time for all partitioning times examined. No significant volume expansion was measured in this case suggesting an increased austenite stability in this alloy.

INTRODUCTION

Quenching and Partitioning (Q&P)[1] is receiving increased attention as a fundamentally new way of producing advanced high strength steels (AHSS). The process consists of a two-step heat treatment: after soaking in the intercritical region or above the Ac3 temperature, the steel is quenched to a pre-determined quenching temperature (QT) within the Ms-Mf range resulting in a partially martensitic, partially austenitic microstructure. Both constituents inherit the carbon content of the austenite present at the soaking temperature. The second step of the Q&P process consists of either holding at the QT or reheating to a higher temperature and holding followed by a final quench to room temperature. The second step is referred to as partitioning and aims at carbon enrichment and stabilization of the austenite by carbon depletion of the martensite and transfer to the austenite. In this way, a martensitic microstructure is obtained along with significant retained austenite at room temperature after final quenching . Notably interesting mechanical properties have been reported via this heat treatment.[2,3,4,5,6] The TRIP effect has been shown to operate in these steels, effectively contributing to increased strain hardening and elongation.[4] Encouraging strength/ductility combinations have been obtained within a “gap” in the available property combinations for recently developed AHSS grades.[6,7]

Significant retained austenite volume fractions have been measured after Q&P[3,4,5] indicating the effectiveness of the heat treatment, although some fundamental aspects are not yet well understood. These aspects involve carbon "trapping" mechanisms in the martensite such as dislocation trapping and (transition) carbide formation which have been the focus of previous work,[8] as well as austenite decomposition via mechanisms which may involve interface migration[9] and/or bainite formation.[10] All of these mechanisms reduce the levels of retained austenite that might be achieved in comparison to the case of "ideal" partitioning under constrained carbon equilibrium (CCE).[a][1] CCE is a hypothetical concept involving "equilibrium" carbon partitioning from martensite into austenite without interface movement and in the absence of carbide precipitation. A better understanding of some of the mechanisms involved during Q&P may eventually yield enhanced austenite stabilization through improved alloy design.

In the present contribution, alloying modifications were examined to assess the effects of Al as a cementite retarding element which is also a ferrite stabilizer and known to increase bainite formation kinetics, and of the effects of Mo which is known to retard austenite decomposing transformations including bainite formation.[11] The effect of (a combination of) these alloying elements on austenite stabilization via Q&P is assessed and the Q&P process is studied *in situ* via dilatometry. A CMnSi grade is included as a reference.

EXPERIMENTAL PROCEDURE

The chemical compositions of the steel grades studied are given in Table I. Alloying modifications include carbon, silicon, aluminum and molybdenum. The steels were prepared as 100kg ingots in a Pfeiffer VSG100 vacuum melting and casting unit, operated under an Ar protective atmosphere. The ingots were cut into blocks of 250mm width, 120mm length and 25mm thickness. The blocks were reheated for 1 hour at a temperature of 1250°C and hot rolled in 6 passes to a final thickness of 3.5 mm. A coiling simulation was performed at 600°C. After pickling, the steels were cold rolled to a final thickness of 1mm. Square samples 15×15mm were cut from the sheets for heat treating using salt baths. After full austenitization, the steels were quenched to the appropriate quench temperature and held for 3s, followed by a partitioning treatment before final water quenching to room temperature. The method proposed by Speer *et al*.[1,12] was used to estimate the preferred quench temperatures and associated austenite fractions at the quench temperature. Partitioning was done at 350, 400 and 450°C for 10-300s. Austenite volume fractions were determined experimentally after processing using magnetic saturation, according to the method proposed by Wirthl *et al*.[13]

Table I: Chemical composition (in wt%), calculated optimum quench temperature QT (in °C) and austenite volume fraction (in vol%) predicted to be present at the QT.

	C	Mn	Si	Al	Mo	QT	$f\gamma$ at QT
CMnSi	0.20	1.63	1.63	-	-	240	19
C̲MnSi	0.24	1.61	1.45	0.30	-	250	23
C̲MnAlSi	0.25	1.70	0.55	0.69	-	260	24
MoC̲MnAl	0.24	1.60	0.12	1.41	0.17	270	20
MoCMnSi	0.21	1.96	1.49	-	0.25	240	21

Dilatometry was carried out using a DIL805A/D Bähr type dilatometer in quenching mode operating under vacuum (10^{-4} mbar). Specimens 4×10×1mm were prepared from the cold rolled sheet by spark erosion. Quenching was done with He gas allowing a high quenching rate of 100°C/s in order to avoid ferrite/pearlite and bainite formation during intitial cooling to the QT.

RESULTS AND DISCUSSION

The effect of carbon, silicon, aluminum and molybdenum additions on austenite stabilization after Q&P was assessed by investigation of the steels listed in Table I. The resulting fractions of retained austenite are given in Figure 1 for the indicated steels and partitioning conditions. The effect of carbon content is reflected in Figure 1a although it should be noted that the Si and Al contents differ slightly in the two grades shown. The austenite fractions generally decrease with increased holding time (Pt) at the partitioning temperature (PT). Partitioning at 450°C for longer times (Pt > 120s) results in the lowest austenite fractions in the C̲MnSi grade. In general, fairly similar volume fractions are stabilized in both grades suggesting that a carbon content difference of ~0.05 wt% does not alter the obtained austenite fractions significantly within the range of steels employed. Note that the measured austenite volume fractions were lower than the calculated values of Table I. Figure 1b shows the retained austenite fractions for the C̲MnSi and C̲MnAlSi grades, having similar carbon contents. Lower austenite fractions are obtained for the C̲MnAlSi grade for the same partitioning conditions, indicating that the partial replacement of Si by Al results in less stabilization during Q&P. Both grades show decreasing austenite fractions with partitioning time. The highest fractions in the Al modified grade are obtained after partitioning at 350°C. The effect of

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[a] There are some special instances where interface migration can increase the potential levels of retained austenite.

Mo addition to a CMnSi grade is given in Figure 1c; both grades again have similar carbon concentrations but the MoCMnSi has a higher Mn content. In general, the MoCMnSi steel contains greater retained austenite fractions. Different trends with partitioning time are also observed. At a PT of 350°C the values show a small decrease with partitioning time, while partitioning at 400°C leads initially to decreasing values, followed by increased austenite fractions above 120s. At 450°C, the austenite fraction increases with partitioning time until a maximum of ~16 vol% γ_{ret} is reached at 120s. The effect of Mo alloying in an Al grade is given in Figure 1d. As shown in Table I, this Mo containing grade contains 1.41wt% Al and very little Si (0.12wt%). The other grade shown in Figure 1d is a CMnAlSi steel containing less Al (0.69wt%) and more Si (0.55wt%), and a slightly higher carbon level. Continuously decreasing austenite fractions with partitioning time are observed for both grades. The austenite fractions drop significantly and very small amounts are retained at partitioning times longer than 120s for both grades. Partitioning at 350°C results in similar values for both compositions. At higher partitioning temperatures slightly lower values are obtained for the MoCMnAl grade. Based on the results in Figure 1b, it appears that the higher Al and lower Si content of the MoCMnAl grade may explain the lower observed austenite fractions for this grade. The replacement of Si by Al does not seem to be compensated by the beneficial effect of Mo alloying used here.

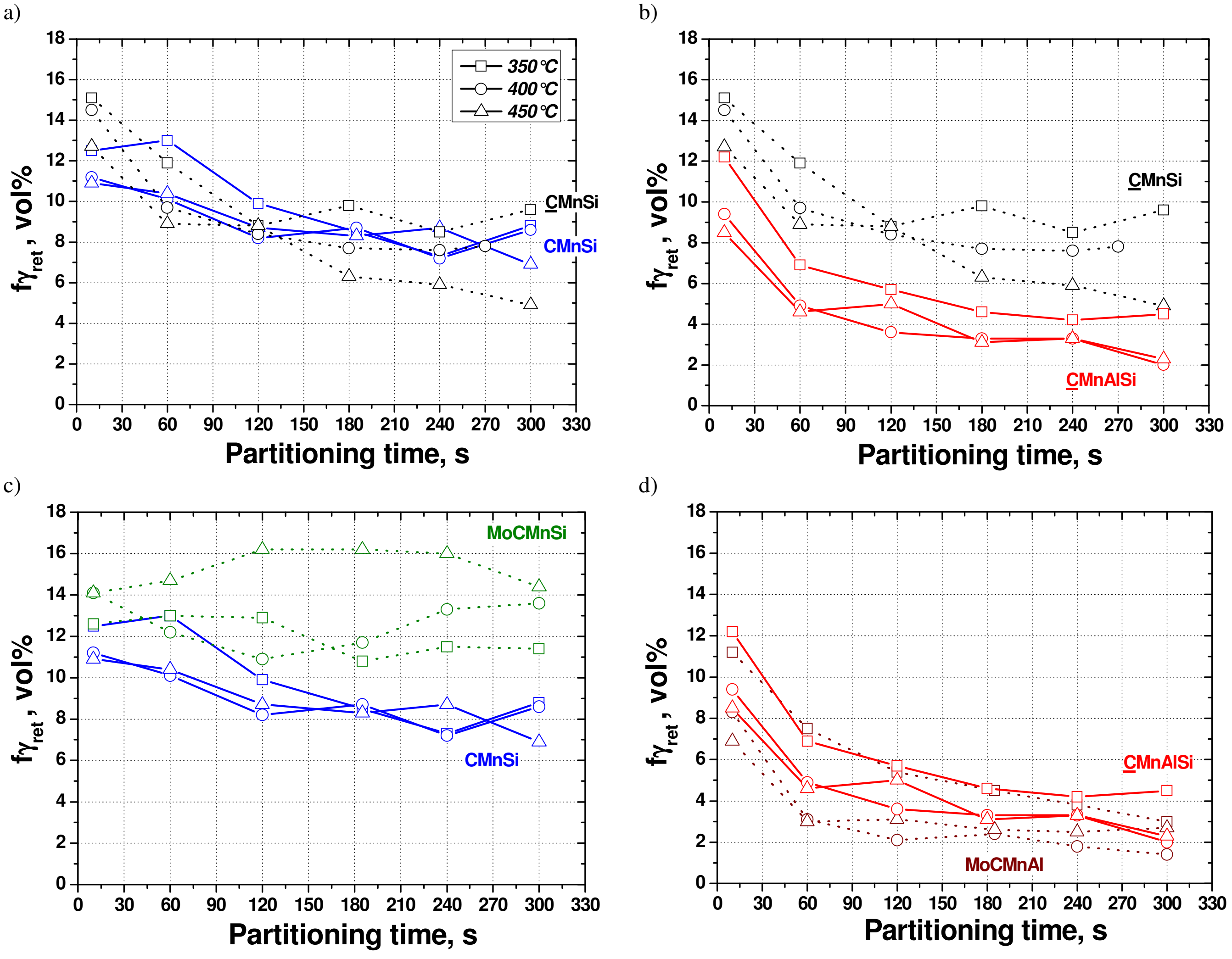


Figure 1: Effect of alloying on the retained austenite volume fractions stabilized via Q&P: effects of a) carbon content b) partial replacement of Si by Al c) Mo additions to a CMnSi grade, or d) Mo additions to an Al grade.

Of all the steel compositions examined here, the MoCMnSi grade gives the greatest amount of austenite stabilization and is less sensitive to holding at the PT for longer times. (Partial) replacement of Si by Al results in lower retained austenite fractions. The lowest γ_{ret} fractions are observed for the grade with the highest Al content. Note that the γ_{ret} obtained at the plateau exhibited by the MoCMnSi grade for partitioning at 450°C approximates the calculated value more closely than is the case for all other grades examined, although higher austenite fractions are predicted by the CCE calculations than are experimentally observed in any of the steels. Hence, “ideal partitioning” is not obtained and competing reactions may occur involving carbon trapping in the martensite (such as by carbide formation) or decomposition of the austenite present at the QT (e.g. by interface migration). These mechanisms are expected to be influenced by alloying. Dilatometry has proven to be a good experimental technique for *in situ* observation of Q&P[14] and was chosen here to study these alloying effects. The CMnSi, CMnAlSi and MoCMnSi were selected for this study, to focus on the effects of aluminum and molybdenum.

A representative dilation curve is shown in Figure 2 for the CMnSi steel, along with the thermal profile used for the one-step Q&P heat treating (i.e. with PT=QT) simulation that was applied. After heating to 950°C and holding for 2 minutes, the steel was quenched to 250°C and held at this temperature for 15min followed by quenching to room temperature. The heating and cooling rates are indicated in Figure 2b. It is clear that at the soaking temperature applied, the steel is fully austenitized and that ferrite, pearlite and bainite formation is avoided during cooling to the QT. A volume expansion is observed during partitioning, as reported recently by Kim *et al.*[14] Similarly, two-step Q&P cycles were simulated for a range of partitioning temperatures varying by 50°C increments up to 500°C. A holding time of 3s was applied at the QT. A rate of 20°C/s was used for reheating from the QT (fixed at 250°C) to the PT. Partitioning for 15min was again applied. The dilatometric data are shown in Figure 3 for the two-step simulations. The dilatation during partitioning shows a pronounced dependence on partitioning temperature applied. At low partitioning temperatures (PT<400°C) an expansion is observed with increased partitioning time whereas partitioning at higher temperatures (PT>400°C) leads to volume contraction with Pt. At 400°C an initial expansion is followed by contraction as shown in Figure 3b and the inset of Figure 3a.

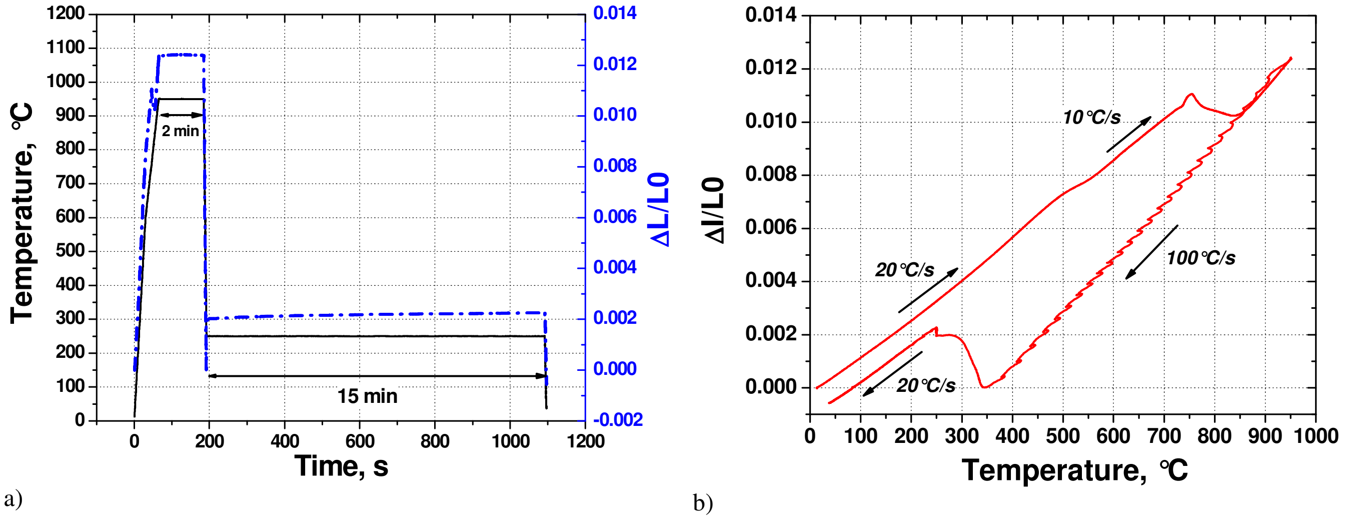
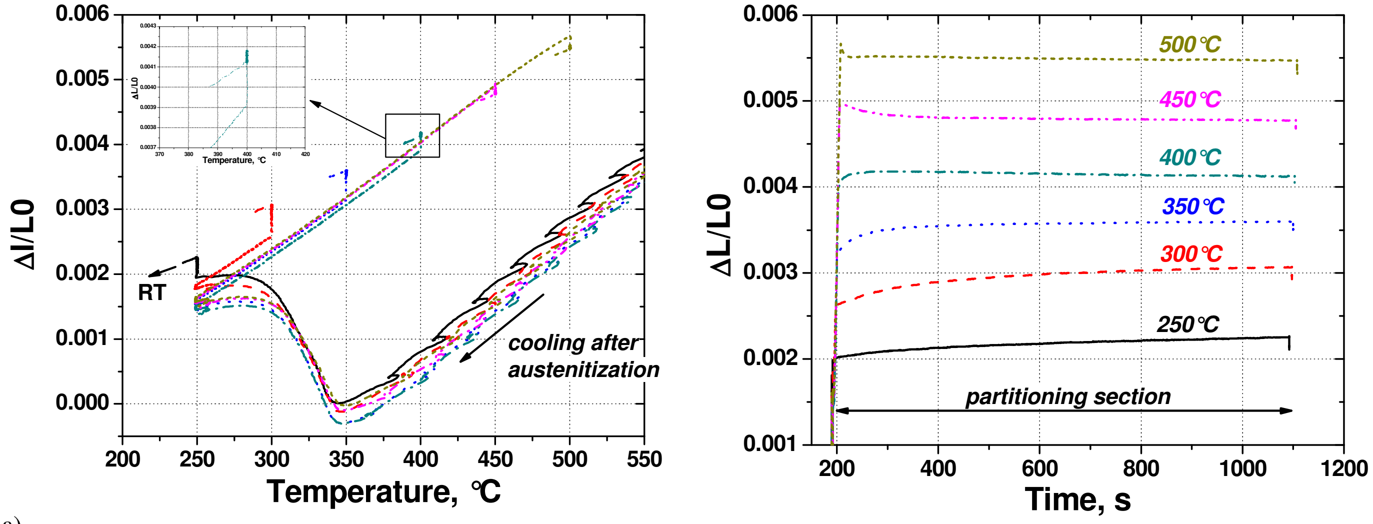


Figure 2: Dilatometry curves resulting from a one-step Q&P simulation using the CMnSi steel with reheating at 950°C for 120s, quenching to 250°C and isothermal holding for 15min at this temperature followed by quenching to room temperature. a) thermal profile (full line) and relative dilatation as function of time (dashed line) b) relative dilatation as function of temperature. Note the volume expansion during isothermal holding at the QT.

The dilatometric signal measured during partitioning in the Q&P heat treating simulations has a complex nature due to the multiple reactions that may occur during partitioning. Such mechanisms include carbon partitioning, (transition) carbide formation, austenite decomposition, interface migration, etc. The predominant mechanism involved is determined by the relative kinetics of each mechanism, and is likely influenced by alloying and processing (including partitioning temperature). An overview of plausible mechanisms is given in Table II. Carbon partitioning results in a contraction of the martensite and expansion of the austenite likely leading to an overall contraction of the sample during partitioning. Austenite decomposition results in an expansion, whether by conventional processes such as bainite formation, or by other processes such as “isothermal martensite” formation which has been proposed recently[14]. The austenite may also be consumed by interfacial movement of the martensite/austenite interface in the direction of the austenite.[9] This movement is made possible via the driving force available for iron atom movements.[9] (Thermodynamic conditions may also exist where driving force is available for iron atoms to move in the opposite direction, leading to interfacial movement whereby austenite consumes martensite, leading to a volume contraction.) Martensite tempering via carbide formation may also occur during partitioning. TEM evidence for ε/η transition carbide precipitation in Q&P microstructures has been reported [3,15,16,17] as well as cementite precipitation for longer partitioning times.[3] Cementite precipitation in martensite is associated with volume contraction whereas transition carbide formation results in volume expansion.[18,8] Expansion observed at lower partitioning temperatures may hence involve transition carbide formation. A higher number density of carbides has been observed at lower partitioning temperatures.[8] It is therefore useful to examine dilatometric effects during conventional martensite tempering of these steels to quantify the potential contribution of carbide precipitation to the dilatation response.



a)

b)

Figure 3: Dilatometric results from two-step Q&P simulations with the CMnSi steel using different partitioning temperatures. a) dilatation as function of temperature, inset: Q&P cycle for partitioning at 400°C b) dilatation as a function of time during partitioning. Partitioning at low temperatures is associated with volume expansion, while contraction is observed at higher PT.

Dilatometric results during conventional martensite tempering for the same CMnSi steel are shown in Figure 4. Austenitizing occurred similarly as in the Q&P cycles, and quenching was carried out at the same cooling rate but in this case was continued all the way to room temperature. The samples were then heated to the desired tempering temperature at a rate of 20°C/s. Tempering temperatures ranging from 250°C to 500°C were applied. The samples were held at these temperatures for 15min. The results in Figure 4 show that tempering at 250°C and 350°C leads to a slight volume increase. An earlier calorimetric study of martensite tempering of this steel showed that ε/η carbide precipitation occurs in this temperature range[8]. At 400°C contraction occurs, which is believed to result from cementite formation and associated decarburization of the martensite[8]. Tempering at 450°C and 500°C leads to a more significant length decrease due to these mechanisms.

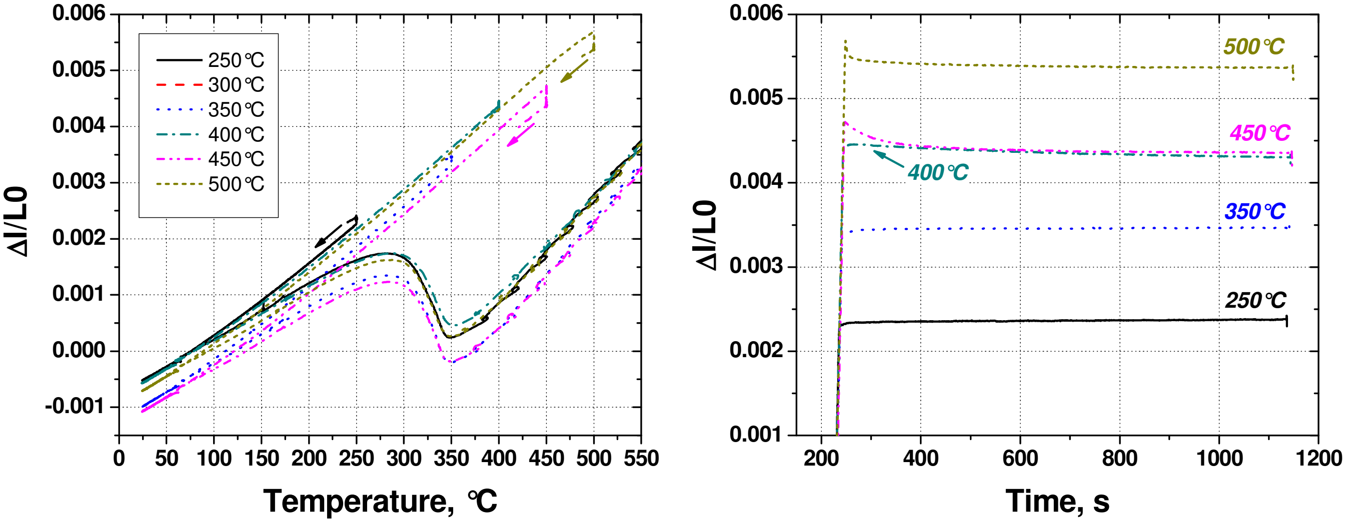
Table II: Plausible mechanisms acting during partitioning, and associated dilatometric response

Mechanism	Dilatation
C partitioning	contraction of martensite expansion of austenite
austenite decomposition	expansion
interface movement	contraction or expansion[9]
martensite tempering via - transition carbide formation - cementite formation	 expansion [8] contraction[8,18]

Comparing the dilatometric data obtained during tempering with the dilatometric data obtained during Q&P processing, the following observations are made:

- the expansion observed at low temperatures (250°C and 350°C) is greater for partitioning than for martensite tempering.
- the contraction observed at higher temperatures (450°C and 500°C) is less pronounced for Q&P than for martensite tempering.
- at 400°C, the initial expansion is greater for partitioning whereas the subsequent contraction is greater for tempering.

The volume expansion observed during partitioning at lower temperatures can hence not be explained solely by transition carbide formation. Austenite decomposition is thus believed to be the main contributor. The observed volume contraction at higher temperatures during partitioning and tempering is believed to be a result of decarburization of the martensite and associated shrinkage of the crystal structure. The contraction is irrespective of whether this carbon decrease is primarily the result of carbon partitioning into austenite or cementite formation. The smaller contraction observed during partitioning may be due to the lower martensite fraction present in the Q&P microstructure leading to a lower overall contraction and to decomposition of austenite resulting in an expansion. In addition, a reduced likelihood of cementite formation during Q&P may exist due to martensite decarburization. The dilatation observed at 400°C is consistent with observations made at lower and higher temperatures. The lower initial expansion observed during tempering may be related to decomposition of the austenite in the heating section from room temperature to 400°C. The austenite consumed during heating will no longer contribute to the isothermal decomposition signal.



a) b)

Figure 4: Dilatometric data of martensite tempering of the CMnSi steel at different tempering temperatures. The samples were fully austenitized at 950°C and quenched to room temperature prior to tempering for 15min.

It is clear that decomposition of austenite is usually not desired and should be avoided by, for example, careful selection of partitioning conditions. However, continuous strip processing facilities such as annealing and particularly galvanizing lines may not allow substantial variations of these conditions. It is therefore useful to understand alloying effects on austenite decomposition during partitioning. This may result in a better understanding of the measured retained austenite fractions given in Figure 1 as well as the mechanisms at play during partitioning, and strategies to better control microstructure during industrial processing.

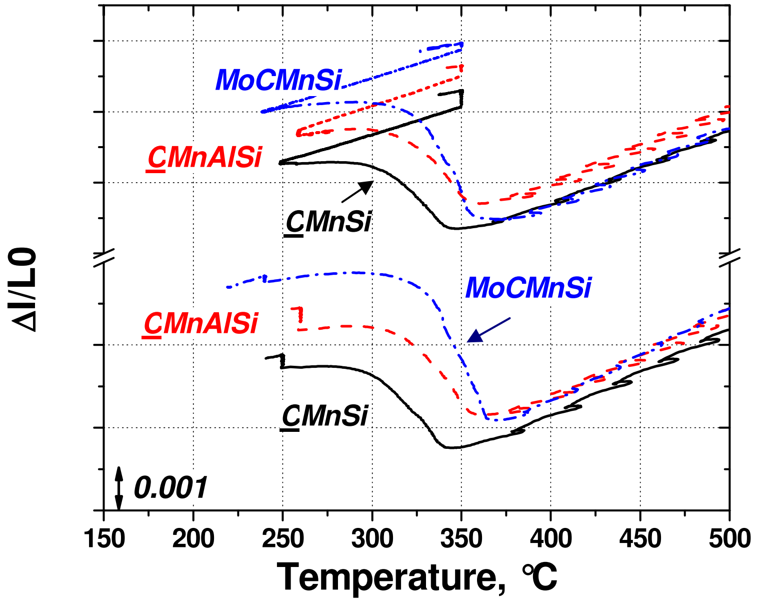


Figure 5: Dilatometric data for the CMnSi, CMnAlSi and MoCMnSi steels for Q&P cycles involving partitioning for 15min at the QT (lower graphs) or at 350°C (upper graphs).

Figure 5 shows dilatometric data obtained during two types of Q&P cycles applied to the CMnSi, CMnAlSi and MoCMnSi compositions. The same soaking and cooling/heating rates were applied as for the heat treatment shown in Figure 2. Partitioning was done at the respective quench temperatures (lower graphs) and at 350°C (upper graphs) for 15min. The curves were shifted vertically to improve the clarity of the figure. A holding time of 3s at the QT was applied for the 2-step heat treatment. A volume expansion is observed for all three steels during partitioning (at the QT) in the one-step Q&P cycles. The CMnAlSi shows the greatest expansion, the MoCMnSi the smallest. This indicates that more austenite decomposition is obtained when Si is (partially) replaced by Al, and that decomposition is slowed when Mo is added. During the 3s hold at the quench temperature in the two-step Q&P heat treatments, a similar composition-dependent expansion is also observed. The CMnAlSi grade again shows more expansion than the CMnSi steel, and no significant volume change is observed for the MoCMnSi steel. For the two-step Q&P cycles, partitioning at 350°C results in the greatest expansion for the CMnSi grade, and the MoCMnSi again exhibits the smallest expansion. Austenite decomposition is possible during heating to the partitioning temperature, and Figure 6 compares the dilation curves during heating from the quench temperature to 500°C at a constant rate of 20°C/s. Note that the curves were again shifted vertically for clarity. The dilatation curve for a CMnSi sample quenched to room temperature before heating (i.e quenched and tempered) was also added for comparison. A significant volume expansion is observed for the Q&P sample compared to the Q&T sample, presumably due to the greater austenite fraction present at the QT compared to room temperature. The expansion observed during partitioning is significantly influenced by steel composition. Very limited expansion is observed for the MoCMnSi grade in the heating section as well as during the 3s hold at the QT, suggesting highly stable austenite. In contrast, the CMnAlSi grade shows some austenite decomposition at the QT, comparable to the expansion observed in the CMnSi grade.

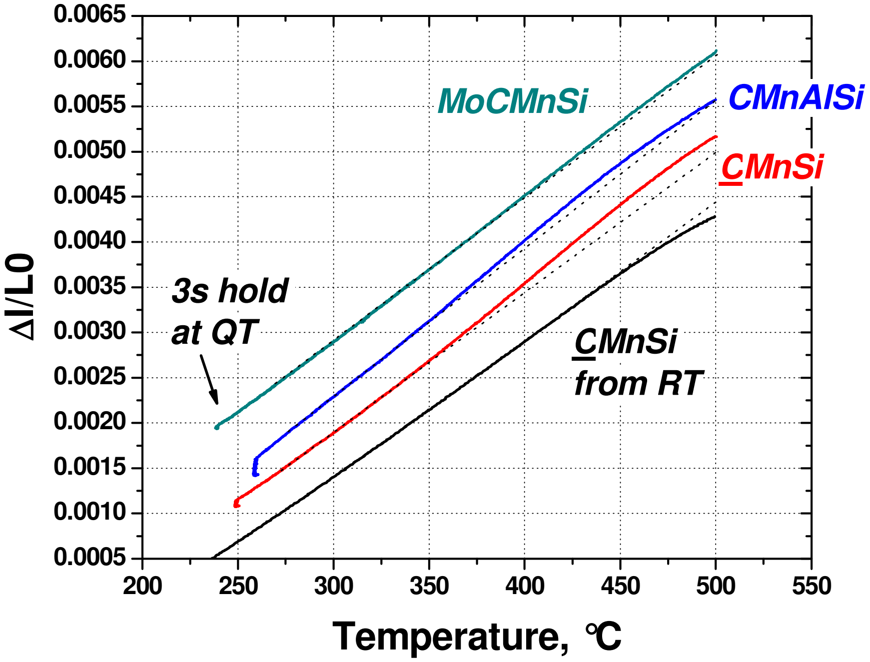


Figure 6: Dilatometric data of the heating section from the respective quench temperatures to 500°C at 20°C/s for the CMnSi, CMnSiAl and MoCMnSi steels. Data for heating from room temperature is also shown for the CMnSi grade.

The dilatometric results help explain the measured retained austenite fractions given in Figure 1. A Mo addition to a CMnSi grade resulted in higher retained austenite fractions, and this behavior is consistent with the lower expansion observed from the dilatometry results during isothermal holding at the QT, during heating, and during partitioning.[b] The greater resistance to austenite decomposition during heating is perhaps consistent with the increased austenite volume fractions obtained at higher partitioning temperature. Higher partitioning temperatures result in increased carbon mobility and thus in greater potential for austenite stabilization after final room temperature quenching via partitioning. However, austenite decomposition must be avoided in the heating and partitioning section. Austenite decomposition may occur via (carbide-free) bainite formation or α_M/γ interface movement. Mo is known to slow down the kinetics of austenite decomposition into ferrite/pearlite and, to a lesser extent into bainite. The

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[b] Molybdenum is also expected to influence the propensity for austenite to transform during cooling to the quench temperature. This effect may be especially important at cooling rates applicable to commercial processing, but was not an important factor at the relatively high cooling rates employed during the dilatometry simulations conducted here.

retardation of bainite formation by Mo has been ascribed to a solute drag like effect (SDLE) by adsorbed Mo at the ferrite/austenite boundaries,[19, 20] along with strong affinity of carbon atoms for molybdenum, which may retard carbon transport and austenite decomposition. While the SDLE mechanism was proposed for austenite decomposition via bainite formation upon cooling, the theory should also be applicable to the mixed martensite/austenite microstructure present at the QT, and in particular for austenite decomposition resulting from interfacial movement.

The partial replacement of Si by Al results in lower retained austenite fractions after quenching and partitioning. Previous work on martensite tempering indicates that Al may be less effective in retarding carbide formation,[8] and as a consequence, Al additions may reduce that amount of carbon available to stabilize austenite. In addition, more austenite decomposition is observed at the QT in the Al-containing steel, even at short holding times of 3 seconds. Al is a ferrite stabilizer and has been reported to increase bainite formation kinetics.[21,22] The second stage of tempering i.e. austenite decomposition has also been reported to proceed with increased kinetics in Al modified grades.[8] The exact mechanisms involved during Q&P of different alloys are not yet clear and would benefit from further examination and *in-situ* studies e.g. hot-stage TEM. It appears clear however, that alloying with the strong ferrite stabilizing element Al results in lower retained austenite fractions after Q&P heat treating whereas Mo additions lead to significantly higher stabilized austenite fractions. In alloys with sluggish bainite and interface migration kinetics, carbon partitioning should be the primary contributor to austenite stabilization, and appears to be an effective mechanism in the Si-Mo containing steel.

SUMMARY

Retained austenite stabilization via quenching and partitioning was examined in steels with Si, Al and Mo modifications. Partial replacement of Si by Al results in lower retained austenite fractions. Mo additions to a CMnSi steel results in higher austenite fractions, increasing with increasing partitioning temperature, although a small Mo addition to the CMnAl grade did not overcome the loss of retained austenite that resulted from replacement of Si by Al. Composition dependent volume expansion associated with austenite decomposition was measured via dilatometry at the quench temperature, during heating section from the QT to the PT and during partitioning. Austenite decomposition is strongly retarded by Mo alloying in a MoCMnSi grade, presumably resulting in the higher austenite fractions retained after Q&P. Some austenite decomposition was observed during holding at the quench temperature in a CMnAlSi grade.

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