Segregation Neutralised Steels: Microstructural Banding Elimination from Dual-Phase Steel Through Alloy Design Accounting for Inherent Segregation

CARL SLATER, BHARATH BANDI, PEDRAM DASTUR, and CLAIRE DAVIS

Banding in commercial dual-phase steels, such as banded ferrite and pearlite or ferrite and martensite microstructures, is inherited from segregation during solidification in continuously cast material, predominantly from Mn segregation, and subsequent rolling. The banded microstructures lead to anisotropic mechanical properties which is generally undesirable. This paper presents an alloy design approach (termed “segregation neutralised” steels) to remove banding of the second phase by utilising co-segregation of both austenite and ferrite stabilisers to reduce local variability in second phase stability. The new composition proposed also considers achieving the same strength levels through maintaining the same second phase fraction, grain size and solid solution strengthening increments. Phase field modelling has been used to predict the segregation and phase transformation behaviours for a commercial composition dual-phase steel and the new composition segregation neutralised steel. A 5 kg laboratory alloy production route (casting, hot rolling and coiling simulation, cold rolling and annealing) has shown that the banded structure seen in commercial dual-phase steels is accurately reproduced and that banding has been reduced dramatically in both the hot rolled condition as well as after cold rolling and annealing in the new segregation neutralised steel. Chemical analysis has shown that in the segregation neutralised alloy the second phase distribution shows no correlation to the segregation bands, due to the achieved balance in austenite and ferrite stabilisers.

https://doi.org/10.1007/s11661-022-06674-6
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I. INTRODUCTION

SEGREGATION in steel can take many forms; from macro/centreline segregation resulting in loss in toughness,[1] local interdendritic micro-segregation which can locally deplete areas of expensive microalloying elements,[2,3] and/or grain boundary segregation of P and S which can significantly limit the strength/stress corrosion cracking susceptibility of a steel.[4] These phenomena are well known and understood, and generally heat treatments, that can be time consuming and expensive, are the most effective way to reduce their impact on processability and/or product properties.

One particular case where segregation has a pronounced impact is in ferrite + pearlite strip and plate steels and dual-phase (DP) automotive strip steels with a ferrite + martensite microstructure. Hot rolled ferrite + pearlite steels are used in structural applications and typically have a banded pearlite distribution[5] and anisotropic mechanical properties. Common automotive dual-phase steels (DP600, DP800 and DP1000) have helped transform the market since their implementation, contributing to the reduction of the mass of steel used in cars by up to 300 kg.[6,7] These DP steels rely on specific thermal cycle(s) to achieve, typically, a mixture of hard tempered martensite in a soft ferrite matrix ranging from around 15 to 80 pct martensite depending on the strength requirements.[8] This gives an excellent combination of strength and ductility. Most DP steels show a banded second phase distribution with martensite “islands” being elongated, and connected, in the rolling direction; the severity of this banding depends on the amount of rolling reduction, the volume fraction of second phase and the thermal processing. The spatial distribution of the martensite plays a critical role on the ductility and anisotropy in mechanical properties. For example, the anisotropy in strength has been reported to be up to 200 MPa for a 30 pct martensitic dual-phase steel when the martensite islands (with an aspect ratio of 6) were orientated along the tensile direction or at 45 to 60 deg.[9] DP steels are low alloy, being predominantly based on C-Mn compositions, potentially with microalloying additions. Mn, as the main alloying element present...
(typical range for DP steels: 1.5 to 3 wt pct)\(^{10}\) shows a strong segregation tendency, both micro and macroscopically. Moreover, the low diffusion coefficient for Mn (\(10^{-26}\) m\(^2\)/s) means that the solidification segregation is mostly retained throughout the downstream processes and exists in the final product.\(^{11-13}\) Typically values for interdendritic segregation give enriched Mn levels 30 to 50 pct above the mean composition after the reheating stage for conventional C-Mn steel grades (around 1 wt pct Mn) and high Mn specialist steels such as TWIP/TRIP grades (>20 wt pct Mn).\(^{14-16}\) Although some highly alloyed grades will specifically add Mn to obtain a homogenisation treatment to reduce these effects.\(^{17}\) Interdendritic segregation distributions are influenced by the secondary dendrite arm spacing (SDAS) during solidification and are dependent on the cooling rate, therefore casting route (e.g., thin slab, thick slab), composition (minor effect for the C-Mn steels) and location (slab surface, quarter thickness etc.). Typical SDAS values for C-Mn steels produced by thick slab continuous casting are 150 to 300 \(\mu\)m.\(^{18}\) After continuous casting the steel slab is hot rolled to plate or strip and then potentially cold rolled to final thickness, followed by annealing. The deformation from slab (80 to 250 mm thickness for thin/thick slab) to hot rolled strip (typically 2 to 10 mm thick) results in Mn rich bands in the rolling direction and therefore pearlite banding in the hot rolled product. Due to the strong austenite stabilising effect of Mn, these Mn rich bands favour austenite formation during intercritical annealing at much lower temperatures compared to the “solute poor” regions, resulting in a heavily banded martensitic microstructure in the DP steels. During the intercritical annealing stage used to generate the desired DP steel microstructure the peak temperature is chosen to attain the desired volume fraction of austenite that is then quenched to martensite. Austenite nucleates preferentially in the following areas:

- Regions of high austenite stabilisers (present due to solidification segregation).
- Regions of high strain (due to strain partitioning during cold rolling or incomplete recrystallisation)
- Deformed pearlite (typically synonymous with the high Mn bands)
- Recrystallised ferrite grain triple points

The balance of these nucleation points is highly dependent on composition and process history.\(^{19,20}\)

Various approaches have been reported on achieving a more desirable dispersed (non-banded) second phase distribution in two phase microstructures, particularly DP steels, using additional thermal cycles and/or changing the intercritical annealing parameters. Many authors have looked into the role of heating rates on the final microstructures of DP steels.\(^{19,21,22}\) By altering the heating rate and peak intercritical annealing temperature the overlap between ferrite recrystallisation and austenite formation can be changed; although the cold rolling reduction also needs to be considered. The heating rates employed in the studies ranged from 1 to 1000 °C/s, and in most cases increasing the heating rate results in a greater overlap between recrystallisation and austenite nucleation, which results in large blocky martensite islands as the nucleation sites are promoted in the retained high strain areas as well as Mn rich bands. However, it should be noted that banding is still prevalent in these steels, but the coarseness of the martensite islands is changed, which affects the mechanical properties. These microstructural changes are highly dependent on the alloying content and the percentage of cold reduction. However, according to the published literature, heating rates above 10 °C/s are generally required to see significant overlap between recrystallisation and transformation to austenite in steels cold rolled > 50 pct.\(^{13,20,23}\) It is well reported in the literature that the presence of these coarse and banded martensite morphologies increase the strain incompatibilities between the ferrite and martensite phases leading to premature failure and reduced ductility.\(^{19,22,24}\) Therefore, it is critical to develop microstructures with more dispersed martensite morphologies using industrially feasible chemical compositions and processing parameters. Etesami and Enayati\(^{25}\) researched complex heat treatment to achieve a more uniform second phase distribution where the processing required quenching after hot rolling to attain a fully martensite structure, which was intercritically annealed to obtain a ferrite-martensite structure (40/60 pct volume fraction), before cold rolling to 80 pct reduction and then the final intercritical annealing cycle common for DP steels. The result was a laboratory produced DP microstructure with refined ferrite grain size (< 2 \(\mu\)m compared to typical ferrite grain sizes in commercial DP steels of around 5 \(\mu\)m) due to the highly localised strain in the ferrite. The martensite showed much less banding than in conventional DP steels, however the additional intercritical annealing step after hot rolling is not commercially attractive and cold rolling a ferrite + martensite microstructure, which will be significantly harder than current ferrite + pearlite microstructures, may not be commercially viable in all cold mills. Similarly, Sajad et al., produced a refined laboratory microstructure with dispersed martensite islands using fully martensite hot rolled microstructures and repetitive intercritical annealing steps.\(^{26}\) The concept of multiple stage re-austenitisation and intercritical annealing has also been employed by Srivastava et al.\(^{27}\) to achieve a non-banded second phase distribution in a DP steel. In all of these cases using modified processing routes for DP steel the mechanical properties were improved (higher ductility for the same strength levels), however, commercially it is difficult to realise such complex and multistage heat treatments to coincide with currently annealing/galvanising lines. Other literature have also reported successful removal of some or all of the banding in laboratory produced DP steels using various additional heat treatments, such as 4-stage or cyclic heat treatments,\(^{16,27-29}\) but these are also not commercially desirable.

This study reports on an alternate approach to generate non-banded ferrite and second phase microstructures in steel that is cast and hot rolled, and also after cold rolling and annealing, mimicking
commercial production process parameters. The steel composition has been designed to balance austenite and ferrite stabilising element segregation such that there is no local preferential austenite stabilisation due to compositional segregation, as seen in current commercial C-Mn steels. In addition, the other characteristics of the benchmark steel chosen as a comparison (DP800 grade), such as second phase fraction, solid solution strengthening contribution and ferrite grain size, has been matched in the new alloy design.

II. MODELLING AND METHODOLOGY

A. Modelling Simulations

Phase field simulations to consider segregation behaviour and phase transformations were carried out with Miecrss 6.4\cite{30} using the Thermo-Calc databases TCFe10 and FEMOB4. For all simulations a $500 \times 2000 \mu m$ 2D field was used with a $2 \mu m$ cell size. A sensitivity study to cell size was performed showing that using a cell size $< 5 \mu m$ showed little/no difference to the predicted segregation levels. However, should aspects such as solid/liquid interface velocity, dendrite tip radii etc be needed from Miecrss simulations then a finer mesh size should be defined. In this case, the Miecrss model was used as guidance in the alloy design process rather than offering absolute predictions.

Symmetrical boundary conditions were placed on all four boundaries. The solidification simulation is seeded with two grains at the (0,0) and (500,0) positions with the [100] direction of the two grains orientated at 0 and 20 deg respectively to the axis corresponding to the maximum cooling orientation, i.e., the through thickness direction of the simulated as-cast slab section.

Literature has shown that cooling rates in a continuous caster for thick slab steel can range between 12 °C/s\cite{31,32} in the shell down to 0.5 to 3 °C/s in the core of the slab.\cite{31,32} With the high cooling rate shell only comprising around the first 20 mm,\cite{31} then the majority of a 230 mm continuous cast slab is solidified at the lower end of this cooling rate range. Therefore, initial simulations have been carried out under Newtonian cooling according to Eq.\cite{1} where the cooling rate during solidification is 2.5 °C/s with a thermal gradient of 0.07 °C/m. The steel is allowed to cool to 1000 °C at which point the simulation finishes.

\[ T(t) = 950 + (T_L - 950)e^{-0.05t} \]  

where \( T(t) \) is the time dependant temperature (°C) and \( T_L \) is the liquidus temperature of the material.

Recalculations of both the linearised thermodynamic data and diffusion coefficients were set to update every 2 °C during cooling. Additional parameters used in the model can be seen in Table I. Austenite nucleation in the delta ferrite is limited to form at temperature below 1503 °C (determined through ThermoCalc TCFe10 predictions). This steel has a peritectic composition and therefore austenite nucleation is set to form at the ferrite/liquid boundaries. To minimise metastable nucleation then once a nucleation event occurs a region of 20 µm diameter around the nucleation point has been defined to prevent further nucleation for 0.4 seconds. The growth of the nucleated phase is based on solute redistribution as the temperatures at which this occurs means that boundary mobility is unlikely to be the limiting factor.\cite{30} The higher solubility of most elements in austenite compared to delta ferrite results in very little sensitivity of the final segregation to the parameters for nucleation suppression (diameter and time frame of ‘no nucleation’); for example a difference of only $< 1$ pct in peak segregation values were seen when the parameters to prevent metastable nucleation were increased to 100 µm and 1 seconds.

B. Experimental Methods

The material chosen as the baseline steel for this study, which shows strong banding in the hot rolled and final intercritically annealed condition, is a commercial DP800 grade steel (more details can be found in Reference 34) with a composition of 0.13C–1.86Mn–0.25Si–0.55Cr–0.03Nb.

Laboratory scale production of the benchmark material was carried out using the Rapid Alloy Production (RAP) suite at WMG, University of Warwick, which has been compared to commercial production material to ensure that representative microstructures in the hot rolled, cold rolled and final intercritically annealed condition are achieved.\cite{34} 5 kg ingots were cast using a Consarc 30 kW vacuum induction melting furnace into a 200 × 80 × 30 mm mould. Once cooled, the ingots are reheated and hot rolled using a Hille 25 rolling mill at 1050 °C from 30 mm down to 3.5 mm in 7 passes with short (approximately 5 minutes) reheats in between each pass to maintain workpiece temperature.

After hot rolling the strip is directly placed into a fluidised bed at 600 °C and naturally cooled to simulate commercial coil cooling after hot rolling.

Samples were taken from the hot rolled strips for standard metallography procedures including mounting, grinding, and polishing before etching using 2 pct nital solution. Microstructural characterization of hot rolled samples has been carried out via optical microscopy (Nikon Eclipse LV150N).

Sections from the hot rolled strips (around 450 × 80 × 3.5 mm) were cold rolled to 75 pct reduction (0.875 mm) using the Hille 25 rolling mill with rolling reductions of 0.2 mm per pass. 100 × 20 × 0.875 mm sections were taken from the cold rolled strip for intercritically annealing.

A typical intercritical thermal cycle is given in Figure 1 where heating at two (decreasing) rates are applied to 670 °C, after which a rate of 1.2 °C/s is applied to the target intercritical temperature (750 °C for the benchmark DP800 grade). Intercritical annealing trials were carried out in a Carbolite 15/3 Muffle furnace using a Eurotherm Nanodac controller and a k-type thermocouple spot welded to the centre of the sample. After the intercritical hold the sample was water quenched to generate martensite from the austenite

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formed during the hold to allow microstructural studies of the distributions in the two-phase structure.

The distribution of martensite in the final processed microstructures was analysed by scanning electron microscopy (JEOL JSM-7800F) using an electron back scatter diffraction (EBSD) detector. A final polishing was performed using a vibratory polisher on the samples to prepare for EBSD analysis. The EBSD analysis was carried out using a step size of 200 nm and scan size of 300 µm × 400 µm. AZtec software (by Oxford Instruments) was used for post processing of EBSD data, where discrimination between the phases was achieved by a specific method used in the AZtec software, which utilized a combination of band contrast, band slope and pattern index quality maps.[35]

III. ALLOY DESIGN

Figure 2 shows the Micress simulation result for the benchmark DP800 steel. It has been reported previously by Ohno et al.[36] that 2D representation of micro-segregation over predicts that compared to 3D and experimental data. It was reported that the initial 80 pct of solidified material shows good agreement (within 1 to 2 pct) in Mn segregation between experimental and 2D simulations in an Fe–Mn alloy, the latter portion of the solidification was over predicted the Mn in 2D by around 10 pct. Therefore, whilst the 2D Micress simulations here may show some small discrepancy in absolute peak values in segregated composition, the overall trends and the ability to quickly simulate > 100 different compositions makes the 2D simulation approach a useful tool in alloy design.

The growth of the delta ferrite phase from liquid, in the form of dendrites, and the subsequent nucleation and growth of austenite is shown in Figure 2. The resultant SDAS has been determined to be 53 mm; this is in good agreement with literature data,[18,34] where Eq. [2] gives a predicted SDAS of 55 mm for a solidification rate of 2.5 °C/s.

\[ \text{SDAS} = 84CR^{-0.45} \]  

From the final Micress frame at 1000°C (fully austenitic structure, shown as 480 seconds in Figure 2) histograms for the local composition distribution were taken using the values for each 2 µm cell. The resultant composition histograms can be seen in Figure 3 where significant spread around the average composition is seen due to segregation into locally solute enriched and depleted regions. Despite back diffusion during cooling being included in the simulation there is a significant level of segregation with local regions (cells) having up to 30 pct higher solute levels than that of the average composition. These results are representative of typical final product composition profiles with reports in the literature showing experimentally measured Mn segregation values ranging from 30 to 50 pct more enriched in the interdendritic region compared to the average composition.[37,38] Si and Cr can both be seen to segregate as well, but to a much-reduced level compared to Mn (20 and 4 pct respectively maximum composition variation to average composition).

The consequence of this segregated solute distribution is to locally vary the Ae1 and Ae3 temperatures and therefore the local stability and volume fraction of austenite formed during intercritical annealing. The segregation has a strong spatial distribution linked to the SDAS, which is then deformed during hot rolling leading to the typical banded microstructure, where during intercritical annealing the Mn rich regions transform to austenite first and form martensite on cooling. By reducing the Mn level in the steel, the difference between the local Ae1 temperatures is much lower, as seen in Figure 4. However, it is only by reducing the Mn level down to 0.2 wt pct that a difference between the segregated solute rich and solute poor regions gives an Ae1 difference of less than 5 °C. Whilst this is a major improvement over the initial 59 °C difference in the benchmark commercial alloy, the required significant reduction in Mn will reduce the solid solution strengthening contribution in the material. There is, therefore, a need to compensate for this strength loss whilst, ideally, also reducing any impact of segregation on the local phase stabilisation and transformation behaviour.

In an attempt to neutralise the effect of segregation, the alloying strategy considered is to include a ferrite stabilising element that will segregate into the interdendritic regions, offsetting the effect of the austenite stabilising Mn segregation, whilst also providing some solid solution strengthening. Of the key alloying elements commonly used in steel, Al is a strong ferrite stabiliser that weakly negatively segregates (i.e., partitions into the solidifying steel rather than the liquid),[39] although care is needed to prevent formation of detrimental AlN particles. Cr has a low partitioning coefficient and therefore segregates very little, is a more effective delta ferrite stabiliser but provides little solid solution strengthening contribution (with some literature suggesting that Cr causes a slight reduction in the inherent strength of ferrite).[40] Mo segregates very strongly but also gives limited solid solution strengthening and is an expensive alloying addition. Si is a ferrite stabiliser, with a solid solution strengthening contribution greater than Mn per wt pct addition. Figure 3 shows that whilst Si does not segregate as strongly as Mn, the interdendritic regions are enriched in Si with around a 20 pct increase in Si compared to the bulk composition for the benchmark steel considered. A summary of the alloy selections can be seen in Table II.

To achieve a non-banded second phase DP microstructure there is a need to reduce the Mn content and increase the Si content sufficiently such that the segregation profiles result in the same austenite stabilisation (from Mn) as ferrite stabilisation (from Si) in both the interdendritic and dendritic regions i.e., no net preferential phase stabilisation composition distribution. The additional constraint is to achieve a similar total solid solution strengthening in the steel from the alloy additions. The bcc iron solid solution
| Parameter                                      | Value                      |
|-----------------------------------------------|----------------------------|
| Number of Cells                               | 250 x 1000                 |
| Time Step                                     | 0.25 s                     |
| Interfacial Energy Between Liquid and Solid Phases | 2.04 x 10^{-5} J/cm²     |
| Interfacial Energy Between BCC and FCC        | 4 x 10^{-5} J/cm²          |
| Anisotropy of Interfacial Mobility            | Cubic 0.3                  |
| Time Stepping                                 | 1 x 10^{-3} s              |

Fig. 1—Thermal profile for the standard intercritical annealing cycle of DP800.

Fig. 2—Micress simulation results showing the phase distribution during cooling and solidification of the benchmarked DP800 grade. Red—Liquid, Orange—BCC, White—FCC and Blue—Interfaces (Color figure online).
strengthening of Si (72 MPa/wt pct) is much more effective than that of Mn (35 MPa/wt pct) [41].

Figure 5(a) shows the combined contribution of Mn and Si towards solid solution strengthening in ferrite as a heat map, determined using the solid solution strengthening contribution factors in Table II. For the benchmark grade (1.86 wt pct Mn and 0.25 wt pct Si) the solid solution strengthening contribution equates to around 72 MPa in ferrite. Figure 5(b) is the heat map for the difference in the $A_{e1}$ temperature between the solute enriched and solute depleted segregated regions, which was created from 50 phase field simulations using different combinations of Si and Mn contents. Figure 5(b) shows the difference in the $A_{e1}$ temperature for the benchmark steel composition (1.86 wt pct Mn and 0.25 wt pct Si) is approximately 59 °C. The results from Figure 4 for Mn only effects are shown by the x-axis in Figure 5(b). As shown in Figure 3, Mn has a stronger tendency to segregate and therefore has a stronger effect, compared to Si, on the difference between $A_{e1}$ temperatures. Although Si has a similar partition coefficient to Mn, the higher diffusion coefficient of Si results in a greater amount of back diffusion and therefore a reduced impact on the $A_{e1}$ difference between segregation regions. Figures 5(a) and (b) can be combined to determine the preferred compositional window to achieve the alloy design aims. The alloy composition selected for experimental verification of the proposed segregation neutralisation design is given in Table III. The other alloying elements (C, Cr and Nb) have been kept constant to provide comparable behaviour in terms of the carbon content (and hence strength) of martensite, hardenability from Cr and grain size control from Nb. These alloying elements could be varied in future iterations of the alloy to achieve improved properties, but proof of concept that the alloy design concept to achieve a non-banded DP structure using current commercial processing approaches does not require this.

Micress simulations for the new composition produced the compositional histograms seen in Figure 6 for the segregation behaviour during solidification and cooling to 1000 °C, including back diffusion. The histograms show similar trends to those of the benchmark grade, shown in Figure 3, with Mn and Si showing peak values around 40 and 20 pct greater than the bulk composition, respectively, i.e., microsegregation has occurred but in this case there is balanced segregation of both austenite and ferrite stabilising elements into the interdendritic solute enriched areas.
A digital line scan from the simulations taken between coordinates (250, 500) and (250, 1500) for both the benchmark DP800 and modified alloy can be seen in Figure 7. The position of the peaks and troughs correlate to the spatial distribution of the segregation, which in this case highlights the secondary dendrite arm spacing. Whilst Mn and Si both have very similar partition coefficients, the increased diffusion rate of Si results in a softening of its segregation compositional peaks compared to those of Mn. From each Micress cell composition, the local Ae1 temperature was calculated and can be seen for both alloys in Figure 7. The benchmark DP800 steel shows significant variation in Ae1 temperature of up to 20 °C which correlates well with the location of the Mn peaks. For the modified alloy however, the Ae1 temperature variation is below 3 °C. The segregation and consequent large difference in the Ae1 temperature between the solute enriched and solute depleted regions results in the banded structures seen for conventional hot rolled ferrite and pearlite, and DP, steels i.e., pearlite and martensite form in the Mn enriched bands, respectively. However, the narrow Ae1 temperature range (almost constant value) in the modified composition is expected to result in a more uniform distribution of ferrite nucleation sites on cooling after hot rolling, and more uniform austenite nucleation sites during the intercritical annealing, resulting in a non-banded more uniformly distributed second phase.

IV. EXPERIMENTAL VERIFICATION

Figure 8 shows the microstructures of the benchmark composition and new modified composition after VIM casting, hot rolling and slow cooling (representing the thermal profile expected for commercial strip coiling). These microstructures show the ferrite + pearlite phases typical of slow cooling after hot rolling, with both steels showing similar pearlite volume fractions (20.1 and 22.8 pct ± 3 pct pearlite, respectively) due to the carbon content being the same in the two grades. It is, however, evident that the benchmark DP800 microstructure shows strong banding of the second phase, which is typical of commercially produced material. During the slow cooling after hot rolling (> 900 °C finish rolling temperature) ferrite forms in the solute poor regions, meaning that pearlite forms in the final austenite to transform in the highly segregated, austenite stabilising Mn rich regions. In the modified steel no such pearlite bands occur because there is no segregated austenite stabilised regions, and therefore ferrite nucleation occurs on prior austenite grain boundaries and triple points, giving more random nucleation sites. The pearlite forms in the last austenite to transform, typically in the central area of the prior austenite grains as the ferrite grains consume the grain boundary regions, and thus independent of any segregation profiles within the microstructure. The grain size for the benchmark and modified alloy in the hot rolled condition is 5.2 and 7.1 μm, respectively suggesting that

| Table II. Summary of Alloying Elements Attributes to Segregation, Phase Stability and Solid Solution Strengthening |
|---------------------------------------------------------------|
| **Alloying Element** | Mn | Al | Cr | Si |
|---------------------|----|----|----|----|
| Partition Coefficient Between Liquid and Delta Ferrite | 0.76 | 0.97 | 0.95 | 0.77 |
| Change in Ae1 Temperature (°C/wt pct) | −78 | +140 | −17 | +71 |
| Solid Solution Strengthening Contribution (MPa/wt pct) | 35 | 1 | 3 | 72 |

All data are based on a binary system with pure iron using ThermoCalc TCFe10.

| Table III. Proposed New Composition (All wt pct) to Achieve a Non-banded Dual-Phase Steel Using Current Commercial Processing Schedules |
|----------------------------------------------------------------------------------------------------------------------------------|
| **Fe** | **C** | **Mn** | **Si** | **Cr** | **Nb** |
|---------|------|-------|-------|-------|-------|
| Bal.    | 0.135| 0.20  | 0.75  | 0.55  | 0.03  |

A digital line scan from the simulations taken between coordinates (250, 500) and (250, 1500) for both the benchmark DP800 and modified alloy can be seen in Figure 7. The position of the peaks and troughs correlate to the spatial distribution of the segregation, which in this case highlights the secondary dendrite arm spacing. Whilst Mn and Si both have very similar partition coefficients, the increased diffusion rate of Si results in a softening of its segregation compositional peaks compared to those of Mn. From each Micress cell composition, the local Ae1 temperature was calculated and can be seen for both alloys in Figure 7. The benchmark DP800 steel shows significant variation in Ae1 temperature of up to 20 °C which correlates well with the location of the Mn peaks. For the modified alloy however, the Ae1 temperature variation is below 3 °C. The segregation and consequent large difference in the Ae1 temperature between the solute enriched and solute depleted regions results in the banded structures seen for conventional hot rolled ferrite and pearlite, and DP, steels i.e., pearlite and martensite form in the Mn enriched bands, respectively. However, the narrow Ae1 temperature range (almost constant value) in the modified composition is expected to result in a more uniform distribution of ferrite nucleation sites on cooling after hot rolling, and more uniform austenite nucleation sites during the intercritical annealing, resulting in a non-banded more uniformly distributed second phase.

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the bands may be restricting growth in the normal direction in the benchmark steel.

After hot rolling, the strips were cold rolled to 75 pct reduction before intercritical annealing. To achieve the desired approximately 35 pct martensite, the benchmark DP800 steel was intercritical annealed at 750 °C. This is higher than the temperature ThermoCalc TCFe10 predicts (equilibrium 730 °C) for the formation of 35 pct of austenite during intercritical annealing. This offset was needed to compensate the kinetic effect of the short hold time at the intercritical temperature and the lower stability of austenite which forms in the solute poor regions. Figures 9(a) and (c) show the EBSD maps before and after data processing to reveal the martensite distribution for the benchmark DP800 after intercritical annealing. The banded structure has persisted with the austenite forming preferentially in the pearlite (solute rich) regions and growing along the bands before being quenched to martensite. The volume fraction of second phase (martensite) in the annealed benchmark sample is 38 pct compared to 21 pct pearlite seen in the hot rolled product. The additional 17 pct second phase comes from the coarsening of the austenite formed from the banded pearlite with potentially a few additional austenite islands being nucleated at ferrite triple points/isolated cementite particles.

For the modified alloy, a higher annealing temperature was used due to the reduction in Mn and increase in Si content changing the Ae1 to Ae3 temperatures. According to ThermoCalc TCFe10, the equilibrium temperature required to achieve the desired 35 pct austenite is around 800 °C. Due to the more homogeneous austenite formation in the modified alloy, the same predicted temperature of 800 °C was selected for the intercritical annealing temperature of the modified alloy. Figures 9(b) and (d) show the EBSD maps before and after data processing for the modified composition steel after intercritical annealing at 800 °C. For the modified alloy, with 35 pct martensite, it is difficult to
distinguish between the austenite present from transformation of pearlite and any additional austenite islands that may have newly nucleated from ferrite triple points and/or isolated cementite particles, suggesting the driving force for these mechanisms are very similar.

As both of these steels were cold rolled to 75 pct and the heating rate used during the intercritical annealing cycle is relatively slow (< 4 °C/s) it is assumed that recrystallisation completes before austenite nucleation or the presence of blocky martensite would be seen. This agrees well with Bandi et al. [23] who stated that for a heating rates up to 10 °C/s, full recrystallisation would occur by 601 °C for a similar alloy, well below the A1 temperatures of 630 and 745 °C of the benchmark and modified alloy respectively (even taking into account segregation).

Figures 10 and 11 present histograms showing the size and morphology (aspect ratio) of the ferrite grains and martensite islands for the benchmark and modified alloy, respectively. From Figure 10(a) it can be seen that the equivalent circle diameter (ECD) ferrite grain size is around 2 μm larger in the modified alloy compared to the benchmark steel. In addition to the difference between the hot rolled grain size, this is consistent with the higher annealing temperature, allowing more ferrite grain growth before the austenite forms and restricts further growth. The benchmark grade shows strong martensite banding, which aids in minimising ferrite grain growth in the normal direction. This can be seen when comparing the ferrite grain aspect ratios in Figure 10(b), where the largest grains in the benchmark sample show a higher aspect ratio. This suggests that the austenite is very effective at pinning the growing ferrite grains in the normal direction, with growth in the rolling direction remaining comparatively unimpeded.

Figure 11 shows the martensite island distribution in the two alloys. There is a clear difference in both the area fraction histogram and the aspect ratio of the martensitic islands between the steels. The martensitic morphology in the benchmark grade is dominated by the large area of connected martensite that form along
the segregation bands (and therefore along the rolling direction), whereas the modified alloy shows a mode ECD of 30 μm for the martensite islands. The mean aspect ratios of the benchmark and modified alloy martensite islands are 6.1 and 2.2, respectively. The dispersed lower aspect ratio martensitic islands are more favourable for reducing mechanical property anisotropy as well as distributing stress throughout both the ferritic and martensitic regions during mechanical testing more effectively.

For the modified alloy with a second phase aspect ratio close to unity in the hot rolled condition, after 75 pct rolling reduction, an aspect ratio of 16 would be expected. Figure 11(b) shows an upper aspect ratio value of 9, but with the majority of the second phase showing a much lower aspect ratio. This could be the result of strain partitioning during cold rolling, with the softer ferrite deforming more favourably compared to the pearlite. Research carried out by Basantia et al.\cite{42} and Teixeira et al.\cite{43} states that for pearlite fractions of 30

![Fig. 10—Histograms showing (a) ferrite equivalent circle diameter and (b) ferrite aspect ratio of both the benchmark and modified alloys.](image1)

![Fig. 11—Histograms showing (a) martensite equivalent circle diameter and (b) martensite aspect ratio of both the benchmark and modified alloys.](image2)
and 16 pct, that approximately only 75 and 67 pct respectively of the applied strain is accommodated by the pearlite. This suggests that an aspect ratio of 3.5 to 5 after cold rolling would be expected, much more in line with that seen in Figure 11(b). Therefore, the neutralisation of the segregational effects on the hot band pearlite distribution appears to dominate the final distribution. Further reduction in the aspect ratio of the martensite could be due to the intercritical temperatures involved being sufficient to dissolve the pearlite and redistribute the carbon, so that austenite nucleates and grows much more evenly and/or the austenite islands are spheroidised due to the higher intercritical temperatures.

Table IV shows the hardness of the two alloys after hot rolling and after intercritically annealing. After hot rolling the benchmark grade has a slightly higher hardness compared to the modified alloy. The smaller hot rolled ferrite grain size (5.2 compared to 7.1 μm) is predicted to account for around 15 HV, similar to the measured difference between the two steels. HV of the difference assuming a Hall-Petch coefficient of 600 MPa μm 1/2. After cold rolling and annealing however very little difference can be observed between the two steels. The difference in cold rolled ferrite grain size between these two conditions predicts the benchmark grade ferrite to be 9 HV harder, which with the lower fraction of ferrite in the annealed microstructures would account for the measured difference in hardness. With both the hot rolled and cold rolled/annealed hardness variations being accountable through grain size differences, this suggests that the balance of solid solution strengthening was achieved through alloy modification.

To determine the levels of segregation and relationship between the solute rich regions and second phase distributions EDX lines scans across the second phases were carried out. Figures 12 and 13 shows representative EDX line scans (minimum of 10 scans carried out per sample) for the benchmark and modified alloy in both the hot rolled and final annealed conditions. From Figure 12(a) a conventional Mn profile can be seen through the pearlite (i.e., high Mn, and Si values, corresponding to the pearlite region), whereas for the modified alloy the two peaks in composition (high Mn and Si levels) that can be seen in Figure 12(b) do not coincide with the pearlitic region, suggesting that the balance of Mn and Si segregation in the modified alloy is sufficiently balanced to reduce any local composition driven stability of austenite and hence pearlite. This is consistent with the earlier discussion where ferrite nucleation occurs on austenite triple points and grain boundaries on slow cooling after hot rolling with pearlite forming in the remaining austenite (grain centres) rather than transformation being linked to local compositional variations.

Similar profiles can be seen in Figure 13 for the steels after intercritical annealing. For the benchmark alloy all the Mn peaks are positioned within a martensitic island, but not all martensite islands coincide with a Mn peak. There is a higher volume fraction of martensite in the annealed microstructure than pearlite in the hot rolled microstructure (and interdendritic solute rich regions after solidification), therefore once the pearlite/high Mn regions have transformed to austenite, then these austenite regions will grow and other preferential nucleation sites (ferrite triple points/isolated cementite particles) will result in austenite (and hence martensite) regions to achieve the 38 pct final second phase volume fraction. Therefore, there will be martensite regions (e.g., growth away from segregated bands or formed from ferrite triple points/isolated cementite particles nucleation) that will not show a relationship with Mn. For the modified alloy however, the critical difference is that none of the Mn peaks are positioned within a martensitic island, suggesting that this alloy has successfully removed the local variation in Ae1 temperatures to a level where triple points/pearlite and isolated cementite particles are all favourable austenite nucleation sites.

V. CONCLUSIONS

The work presented describes an approach to use alloy composition design to avoid the detrimental effects of elemental segregation during solidification on microstructural development and hence mechanical properties—‘segregation neutralised’ steels. In the case study discussed the effect of Mn segregation usually causing second phase banding in dual-phase (DP) steels has been considered. The extent of segregation and microstructural development during processing (hot rolling, cold rolling and annealing) in standard DP steel has been used as a benchmark to compare to a proposed modified composition (a segregation neutralised steel) to eliminate the effect of segregation on second phase banding. The following conclusions can be made:

- Micrress phase field modelling has been used to predict the segregation profiles of the main alloying (Mn, Si, Cr) elements in the benchmark steel during solidification. The local variation in composition between the solute rich (interdendritic) and solute poor (dendritic) regions results in differences in the Ae1 temperature for the benchmark DP steel composition of up to 70 °C; this is primarily due to Mn segregation. The solute rich interdendritic areas are

| Table IV. Microhardness (HV1) Measurements of the Benchmark and Modified Alloy |
|-----------------|-----------------|
| Hot Rolled (HV0.5) | Cold Rolled Annealed (HV0.5) |
| Benchmark        | 166 ± 3         | 286 ± 4         |
| Modified Alloy   | 147 ± 3         | 281 ± 3         |

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deformed during hot rolling and are the last areas to transform on cooling resulting in the banded pearlite distribution.

- To reduce the effect of local compositional stabilisation from Mn on austenite resulting in the banded dual-phase microstructure, Mn levels have been lowered in the modified steel composition. Si has been identified as an appropriate alloying addition to compensate for the lower levels of Mn to achieve similar solid solution strengthening. The relative levels of Si and Mn required in the modified steel have been determined by balancing the ferrite stabilisation from Si and austenite stabilisation from Mn.

- The new composition to achieve a non-banded DP steels has lower Mn and higher Si contents, whilst maintaining the C content (to give the same strengthening from pearlite fraction (hot rolled material) and in the martensite (final annealed material), Cr content (for hardenability) and Nb content (for grain size control).

- Experimental casts were made of both the benchmark composition and the new DP composition and processed (cast, hot rolled and coil simulation cooled, cold rolled and intercritically annealed) to strip. The benchmark grade showed significant second phase banding after hot rolling/coil cooling and in the final intercritically annealed microstructure. The benchmark microstructures are representative of those produced commercially. The modified (segregation neutralised) composition had a randomly distributed second phase distribution in the hot rolled and annealed conditions with no banding.

- Minor differences in the ferrite grain size were observed between the benchmark and modified alloy in the hot rolled condition which is suggested to be

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Fig. 12—EDX line scans showing the Mn and Si profiles across a pearlitic region in the hot rolled condition of (a) the benchmark alloy and (b) the modified alloy.
related to the austenite bands pinning ferrite grain growth normal to the bands in the benchmark steel.

- Microhardness measurements of the hot rolled and annealed samples suggest similar solid solution strengthening contributions in the benchmark grade and modified alloy.
- EDS line scans show a clear correlation between the Mn segregation pattern and the second phase distribution in the benchmark alloy. There is no clear correlation between microsegregation (solute rich regions) and the second phase in the modified alloy which supports the alloy design principle of using balanced ferrite and austenite stabilising element segregation. This removes the local composition variation (solute rich) giving preferential stabilisation of austenite and reduction in local A1 temperatures. Therefore, after hot rolling during cooling ferrite forms on austenite triple points/grain boundaries and the pearlite forms in the last austenite (grain centre) to transform. During intercritical annealing after cold rolling austenite nucleation will occur in the deformed pearlite regions and also on ferrite triple points/isolated cementite particles resulting in a more random martensite distribution than in the benchmark DP steel.

ACKNOWLEDGMENTS

The authors would like to thank the University of Warwick VC scholarship for funding of a doctoral student. In addition, the authors would also like to thank EPSRC (grant number EP/S005218/1) and RAEng (Research Chair funding) for support of the research group and relevant input data. Special thanks to Tata Steel for the open discussions about the concepts behind this work.
CONFLICT OF INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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