Effect of Carbon Content on Variant Pairing in Bainitic Low Alloy Steel

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Variant pairing in bainite was evaluated in four different commercial low alloy steels with medium to high carbon content. The steels investigated were austempered in the temperature range 275 °C to 450 °C to obtain a bainitic microstructure. It was found that the V1–V6 is the most frequent variant pairing at lower temperature, while it gradually decreases towards intermediate temperatures, and at the highest austempering temperatures the variant pairing with low misorientation boundaries such as V1–V4 and V1–V8 is the most frequent. The preferred variant pairing is the least pronounced after austempering of steels with higher carbon contents at intermediate temperature. Nonetheless, a continuous increase of variant pairing within the same Bain group was observed with increasing austempering temperature for all steels. Furthermore, it was observed that the deviation from the theoretical Kurdjumov–Sachs orientation relationship increases with increasing austempering temperature for all steels.

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1. INTRODUCTION

The use of low alloy steels is extensive in a wide range of applications due to the range of properties that can be obtained at a low cost by only alternating the carbon content and heat treatment parameters. Changes in the heat treatment process and the carbon content of the steel result in differences in the austenite transformation and the resulting microstructure, which can be tuned to obtain the desired properties for an application in mind. When high strength and hardness are sought for in the application, bainite or tempered martensite are mostly the desired microstructures. Bainite is slightly more difficult to obtain considering the austempering where a salt bath or metal bath is required instead of oil, polymer or water quenching used to obtain a martensitic microstructure. A major benefit with the bainitic microstructure is that it has good toughness without any post-processing treatment as in the case of martensite where tempering is always needed to obtain appropriate toughness.

The bainitic microstructure is known to change with austempering temperature where the structure formed at high temperature is coarser with the cementite in between the plates of bainitic ferrite, while bainite formed at the lower austempering temperatures becomes finer with the cementite within the plates. It should be noted that there are two hypothesis regarding the role of carbon diffusion during the growth of bainite, the diffusional and the diffusionless hypothesis. It should though be emphasized that both hypothesis agree that bainite is formed by a displacive transformation mechanism. Bainite has historically often been defined as lower or upper bainite using the terminology originally proposed by Mehl, even if other definitions also have been proposed. However, studies have shown that the transition of the bainitic structure from lower to upper bainite with increasing temperature of formation may be better described as a gradual shift from cooperative to degenerate eutectoid transformation. In both hypotheses for the bainitic transformation some diffusion of carbon is involved, either only in the nucleation of the bainitic ferrite (diffusionless) or in both the nucleation and growth (diffusional); this is distinct from the martensitic transformation which is completely diffusionless.

The similarities of the bainitic and martensitic microstructures can be related to their displacive phase transformation mechanisms, which lead to similarities in the local orientation relationships (OR) between the parent austenite and bainitic ferrite plates or martensitic units. By studying the changes in the local crystallography with changing bainitic austempering temperature

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and carbon content it is possible to learn more about the bainitic transformation. Different ORs such as Kurdjumov–Sachs (K–S), Greninger–Troiano (G–T) or Nishiya-Wassermann (N–W) have been proposed,[17–22] and in this work the K–S OR is used as reference. The K–S OR has 24 possible orientations for the product ferrite to form in relation to the parent austenite and each distinct orientation of a ferritic plate can be referred to as a variant.[23] The 24 different variants may be divided into four groups where the six variants sharing the same parallel planes are referred to as CP-groups. Variants are most commonly related to variant V1[24–26] and the relation between neighboring variants is referred to as variant pairing, which is known to be affected by alloying and austempering temperature.[22,23,25–29] In addition to CP-groups, variants may also be grouped into three different Bain groups based on the Bain correspondence, where the variants within the same Bain group have a relatively low misorientation with respect to each other.[26] It is known that the experimentally determined OR rarely agree exactly with the theoretical OR and it is, therefore, common to present the difference between theoretical OR and experimental OR by a deviation angle for CPP (Close Packed Planes) and CPD (Close Packed Directions) between austenite and ferrite/martensite.[26,30–32]

Studies on the effect of austempering temperature on the variant paring in bainite is still limited, and most studies have either been conducted at high austempering temperature, above 400 °C, and/or on samples with a carbon content lower than 0.5 mass pct.[26–29] Furthermore, these studies have been conducted at specific austempering temperatures for specific alloys, which limits the possibility to compare and draw general conclusions. In an earlier work by the present authors the unexpected two distinct shifts in the variant paring in a 64Ni4 steel with increasing austempering temperature[24] was revealed and the microstructure was characterized in-depth.[24,33] For that specific steel it is clear that the bainitic microstructure is more complex than inferred by the standard division into upper and lower bainite. However, further investigations on additional steels after austempering at various temperatures are needed in order to improve the understanding of the effect of austempering temperature and carbon content on variant paring in bainite. Therefore, in the present work a study of the variant pairing over a range of carbon contents from 0.61 to 0.99 wt pct and austempering temperatures in the range from 275 °C to 450 °C is performed. Variant pairing analyses of martensite for the same steels were also included for comparison.

II. EXPERIMENTAL

The composition of the four investigated steels are given in Table I. Samples for dilatometry were cut to the dimension 10 × 4 × 1 mm³ by wire electrical discharge machining and a Quenching/Deformation Dilatometer Linseis L78 RITA was used for the first heat treatments. A heating rate of 5 K/s up to 880 °C was applied under vacuum, the samples were thereafter soaked for 10 or 20 minutes (64Ni4) prior to quenching with helium gas to the austempering temperature with a cooling rate of about 200 °C/s to 300 °C/s to avoid any undesired diffusional transformations. The samples were finally held at different austempering temperatures of 275 °C to 450 °C for 1 hour before quenching to room temperature, Figure 1. From dilatometry tests it was concluded that all steels studied herein are fully bainitic after one hour of austempering treatment for the investigated temperature range. The time to reach a fully bainitic microstructure increases as the austempering temperature is decreased, see Table II. Samples were also quenched directly to room temperature to determine the Mₜ temperature, which also was the lowest austempering temperature, since fully bainitic microstructures were desired.

Further heat treatments were conducted for microstructural analysis in a Bi–Sn metal bath at different temperatures between 275 °C and 450 °C for 1 hour to achieve a fully bainitic microstructure before quenching in brine. The samples were cut into pieces of 6 × 6 × 1 mm³ and austenitized at 880 °C for 10 or 20 minutes (64Ni4) in a tube-furnace with argon atmosphere prior to the isothermal heat treatment. Fully martensitic samples were obtained by direct quenching in brine after austenitization and subsequent cryogenic cooling in liquid nitrogen.

Samples for secondary electron imaging in the Scanning Electron Microscope (SEM) were mechanically polished down to 1 μm diamond suspension and thereafter etched with 2 pct picric acid, whereas for Electron Back Scatter Diffraction (EBSD) final polishing with 0.02 μm colloidal silica was conducted. The SEM analyzes were performed in a field emission gun (FEG) SEM, JEOL JSM-7800F, operated at 12 kV with a working distance of 7 mm for imaging, whilst for EBSD it was operated at 12 kV with a working distance of 20 mm using a step size of 50 nm. The EBSD data were analyzed using MTEX version 5.2.[34,35] The boundaries between the variants were calculated using the fast multiscale clustering method and then imported into the graphical user interface developed by Nyyssönen et al.[36–39] to determine the variant pairing. The definitions of variant pairing are based on the work of Morito et al.[23,40,41] and Miyamoto et al.[23,40,41]

III. RESULTS

IPF colored EBSD images of the four investigated steels austempered at 300 °C, 350 °C and 450 °C are presented in Figure 2. It can be seen that the structures are plate-like for all four steels at the lower austempering temperature, 300 °C, and since each plate forms in one single parent austenite grain (PAG) the PAGs become more distinguishable at these austempering temperatures compared to the higher austempering temperatures.[24] At the intermediate austempering temperature, 350 °C, a difference between the steels is clear. For the steel with the lowest carbon content, 64Ni4, the structure still appears plate-like but with increasing carbon content the plate-like structure diminishes and
for the steel with the highest carbon content the structure consists of rather small and blocky areas with less apparent relation to its surroundings. When the austempering temperature is increased even further, to 450 °C, no plates are observed and all structures are bulky and more irregularly shaped and is again rather similar in all four steels.

In Figure 3, the variant pairing of bainite for all four steels austempered between 275 °C and 450 °C is presented together with the one for martensite. For all four steels, it is clear that V1–V6 is the most frequent variant pairing at the lowest temperatures studied, whereas V1–V4 together with V1–V8 are the most frequent variant pairing at the highest temperatures. For 64Ni4, V1–V2 is the dominant variant pairing at intermediate temperatures as previously reported.[24] In the other steels with higher carbon contents, an increase in V1–V2 can also be observed at the intermediate temperatures, but it never becomes dominant and it can be observed that no other variant pair is dominant neither at those intermediate austempering temperatures. These changes are more clearly visualized in Figure 4 where the fraction of the variant pairings is plotted vs the austempering temperature, clearly showing that V1–V6 decreases while V1–V4 increases with increasing austempering temperature for all four steels. The fractions presented varies depending on the amount of background. However, for the intermediate temperature it is hard to identify any clear trends for the steels with higher carbon content. Martensite was also included in Figure 3 for comparison, and it can be seen that the V1–V16 variant pairing increases with increasing carbon content but V1–V2 still remains the most frequent variant pairing in all four steels with martensitic microstructure.

The variant pairing presented in Figure 3 is also analyzed with respect to the Bain groups[26] presented in Table III, and the result is presented in Figure 5. It is shown that the fraction of variant pairs belonging to the same Bain group increases continuously with increasing austempering temperature. Variants belonging to the same Bain group are separated with low angle misorientation boundaries, and thus it is shown that the fraction of variant pairs with low angle misorientation boundaries is increasing rather linearly with increasing austempering temperature. This trend is very similar for all four steels regardless of the carbon content. However, a small deviation can be seen for 64Ni4 at 350 °C.

In Figure 6, SEM micrographs showing the carbide morphology in the bainitic microstructure after austempering at 300 °C, 350 °C and 450 °C are presented for 64Ni4 and C100 and as expected a larger fraction of cementite is observed in C100 due to the higher carbon content. At 300 °C it is observed that the cementite in adjacent variants are growing with an angle of 120 deg to each other in both steels as previously described for 64Ni4.[24] This was observed for all four steels and here exemplified by 64Ni4 and C100. At the intermediate temperature the cementite in the V1–V2 variant pairs is growing parallel to each other in 64Ni4, as can be seen Figure 6(c) and previously described.[24] The same distinct relation could not be found in the three other steels, and as seen in Figure 6(d), the carbides are more randomly formed and no distinct relations could be identified. For higher temperature it is observed that all different steels have a more degenerate growth rather than cooperative growth which is indicated by cementite located between the ferrite plates, in agreement with the observations by Yin et al.[42]

The deviation of the crystallographic OR between the parent phase, austenite, and the product phase, ferrite, were calculated for the close packed planes (CPP) and close packed directions (CPD) compared to the theoretical K–S OR.[17,30–32] The results are presented in Figure 7, showing the effect of carbon content and austempering temperature on the deviation. For all four steels it is seen that the deviation from the ideal CPP is increasing rather linearly with increasing austempering
temperature. The CPD is roughly the same within each of the four steels, but with a slight increase at intermediate temperatures. This change is, however, small and is hard to differentiate from the variations between different austempering temperatures. It can also be seen that steels with higher carbon content has a higher deviation from the ideal CPD. For martensite the deviation from K–S from the ideal CPP is rather constant, but a slight decrease can be observed for the steels with higher carbon content. For the CPD it can be seen that an increase in carbon results in a smaller deviation from the K–S OR. Additional experimental data from literature are included for comparison in Figure 7.[26,30–32]

**IV. DISCUSSION**

Previous studies on variant pairing in bainite have usually only considered a limited temperature range or a single material. An exception is a study by the present authors for a medium carbon steel[24] including low, intermediate and high austempering temperatures. The reason that variant pairing for bainite formed at lower austempering temperatures rarely is reported is presumably because low carbon steels[27–29] has been more frequently investigated. This limits the possibility of studying bainite at lower austempering temperatures due to the relatively high MS temperature of these steels. Hence, the temperatures investigated are usually in the
intermediate or high temperature range and will here be discussed in relation to the present results.

Kaneshita et al.\textsuperscript{[27]} investigated steels with varying carbon content 0.2 to 0.75 wt pct, down to 400 °C. They observed V1–V2, for the alloys with lower carbon content, and variant pairs with low misorientation boundaries such as V1–V4 and V1–V8 at their higher temperature, 500 °C. The same trend was also observed by Furuhara et al.\textsuperscript{[28]} and Takayama et al.\textsuperscript{[26]} but they only used steels with low carbon content. These observations with increasing fractions of variant pairs with low misorientation boundaries at high austempering temperatures are in good agreement with the observations in this work, Figures 3(f), (g), 4 and 5 even if the exact temperatures have changed due to the fairly large differences in the carbon content between the investigated alloys. It should also be emphasized that this increase with temperature of variant pairs separated

Fig. 3—Variant pairing from V1–V24 relative to V1 for (a) martensite and (b) through (g) for bainite formed at increasing austempering temperature, 275 °C to 450 °C. The four steels are indicated as 64Ni4 (1), C75 (2), C85 (3) and C100 (4).
with a low misorientation boundary is continuous for all four steels, Figure 5. Further, the observations by Kaneshita et al. and Furuhara et al. also correspond well with the V1–V2 found in Figure 3(e1) for the medium carbon steel. The reason that the same dominant V1–V2 relation could not be found for the steels with higher carbon content in Figure 3 may be due to a weaker variant selection at the intermediate temperature. This weaker variant selection with increased carbon have previously been described by Kaneshita et al.\[27\] They concluded that the variant pairing tends to follow a specific variant selection to a lower degree when the amount of carbon is increased which is confirmed in this study. They suggested that this effect was attributed to the increased strength of the austenite as a result of the increased amount of carbon, where slip deformation becomes more difficult resulting in multiplication of the variants.\[27\]

Morito et al. observed V1–V6 variant pairing for bainite formed at lower temperatures\[43\] in accordance with the present work. However, in their study only two temperatures were investigated, and thus the two types of dominant variant pairing which were found were coupled with lower bainite for the V1–V6, and upper for the V1–V2. Thus, they did not include the variants with low-misorientation boundaries which typically are found at higher temperatures. It can, however, be concluded from the results presented herein that even if the carbon content is increased from 0.61 to 0.99 the V1–V6 variant pair will remain dominant at the lower austempering temperatures. The conclusion by Kaneshita et al.\[27\] that increased austenite strength leads to multiplication of the variants is not supporting the observation of the variant pairing, V1–V6, observed herein at the lower austempering temperatures and it thus requires additional investigation. It is noted that V1–V6 observed herein is below the lowest temperature of 400 °C that was studied by Kaneshita et al.\[27\] Thus, it could be assumed that if Kaneshita et al.\[27\] would have included lower temperatures in their study they would have found a strong variant selection of V1–V6 for their high carbon sample which is similar to the C75 steel herein.

In this study it is further observed that the overall changes in the microstructure to a large extent is similar for medium to high carbon steel, except for the intermediate temperature range where it is more arbitrary for the steels with higher carbon content, Figures 3 and 6. In all four steels, the bainite formed at lower temperature is more cooperative where the ferrite and cementite have grown together forming a lamellar structure, while the bainite formed at higher temperature have more characteristics of degenerate eutectoid growth with cementite in-between the ferrite plates.\[13–16\] However, the microstructural difference observed at intermediate austempering temperatures correspond well to the observed difference in variant pairing with no clear dominant variant pair even if a slight increase in

Fig. 4—Fraction of variant pairs in bainite after austempering for 1 h as function of austempering temperature from the data in Fig. 3, for (a) 64Ni4, (b) C75, (c) C85 and (d) C100.
V1–V2 can be observed for the three steels with higher carbon content. Even if the complexity at the intermediate temperatures not is fully understood nor the mechanisms behind it, it is here clearly shown that all the changes in the bainitic microstructure appears gradually with austempering temperature which is consistent with Yin et al.\textsuperscript{[32,42,44,45]} Thus, definitions such as upper and lower bainite is not sufficient to describe the bainite structure over the full austempering temperature range since there is a region in-between that also needs to be addressed.

Further, a comparison between earlier published data and the data presented within this work was made by comparing the deviation angle for CPP and CPD between austenite and ferrite, Figure 7(a). For bainite a close correlation to the relationship suggested by Greninger–Troiano and an almost linear increase in the deviation from K–S for CPP with increasing austempering temperature is shown in Figure 7(b). The increasing deviations is continuous with temperature for all four steels investigated herein. It is also possible to extend the temperature trend further with data from Miyamoto et al.\textsuperscript{[52]} and Takayama et al.\textsuperscript{[26]} towards higher austempering temperatures, 450 °C to 580 °C. With their data it is shown that there is a continuous increase in the deviation from CPP with increasing austempering temperature for bainite from 275 °C to 580 °C, which also is consistent with a gradual change in the formation of bainite with temperature. An overlap
in temperature between the current dataset and the dataset by Takayama et al.\textsuperscript{26} shows a similar deviation from K–S for CPP at 450 °C (colored in yellow according to the temperature scale). It was further noted that the increasing amount of carbon slightly increases the deviation for CPD comparing the four steels investigated herein. Comparing the result with Miyamoto and Takayama, their CPD is in the lower range of our data but they also had a lower carbon content. Similar effects are noted in the plot for martensite are the IF-Steel has a lower deviation in CPD than the alloys containing 0.15 to 0.45 wt pct carbon. Thus, some differences in the deviation between low and higher carbon samples is not entirely unexpected for the CPD, however, the lack of data in the range 0.15 to 0.5 wt pct carbon for bainite, differences in austenitization temperature affecting the PAG size, and difference in carbon content makes it hard to draw any further conclusions. Comparing the deviations for martensite herein the CPP is fairly stable, with a small decrease. The data also provide a good correlation with other available data,\textsuperscript{30,31} it can be mentioned that in References 30 and 31 the CPP and CPD axes were switched. For the martensitic samples there is a clear trend that increased carbon content results in a relation that moves closer to K–S. It could be speculated that the temperature effect shown for the bainitic samples could be similar for the martensitic samples. For the martensitic samples it is seen that the CPP increases with decreasing carbon content, and since the $M_s$
temperature increases with decreasing carbon content, CPP increases with increasing formation temperature in a similar way as bainite. However, it is still a significant difference in the deviation from K–S between bainite formed at the lower austempering temperatures and martensite for all four steels, here investigated, Figure 7(c). This is consistent with the earlier observations in Figures 3(a) and (b), thus further indicating that the transformation mechanisms are different between bainite and martensite.

It can be noted that there are differences in substitutional alloying elements between the steels herein, Table I, which could have an impact. However, when comparing the differences in-between the four steels with other steels presented in the literature the differences are small. Steels with as much as 9 wt pct nickel,[28,29] or 1 to 2 wt pct of manganese,[26,27,32] or very limited alloying additions,[25,43] all show a good agreement in variant pairing regarding austempering temperature and carbon content. Therefore, it is concluded that the effect of substitutional alloying elements should only have a limited effect compared to the effect of carbon content. There are also some differences in the PAG size, Figure 2, both between and within the samples. The differences which are observed between the PAG sizes are acceptable, and influence on the variant pairing selection is assumed to be limited. It will, however, have some influence on the background as a smaller PAG size would result in more PAG boundaries being included in the variant-pairing analyzes. It should be mentioned that in other studies of variant pairing the PAG size is much larger than in the current work. It should also be emphasized that the method used to determine the variants differs. Finally, it should be mentioned, that the method used herein analyzes the whole IPF image, while at least in some other works the analyzes have been done on a number of specific PAG meaning that a smaller number of grains are analyzed and that variants pairs over PAG boundaries are avoided.[30,32]

V. CONCLUSION

In all four steels and thus regardless of medium or high-carbon content it is observed that:

1. The variant pairing in bainite formed in the lower temperature region is of type V1–V6 and when increasing the austempering temperature variants separated with low misorientation such as V1–V4 and V1–V8 are favored.
2. The fraction of variant pairing in bainite from the same Bain group show a continuous increase with increasing austempering temperature.
3. Changes in the bainite microstructure with temperature of formation appear gradual.
4. The CPP deviation from K–S increases almost linearly with increasing austempering temperature for bainite.

A difference observed between the four steels studied is that the variant pairing of V1–V2 does not become dominant in the high carbon steels as it does in the medium carbon steel.
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CONFLICT OF INTEREST

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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